



# FEDERAL REGISTER

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Vol. 80

Thursday,

No. 14

January 22, 2015

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Part III

## Environmental Protection Agency

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40 CFR Parts 110 and 300

National Oil and Hazardous Substances Pollution Contingency Plan;  
Proposed Rule

## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Parts 110 and 300

[EPA-HQ-OPA-2006-0090; FRL-9689-9-OSWER]

RIN 2050-AE87

### National Oil and Hazardous Substances Pollution Contingency Plan

**AGENCY:** U.S. Environmental Protection Agency (EPA).

**ACTION:** Proposed rule.

**SUMMARY:** The Environmental Protection Agency (EPA or the Agency) proposes to amend the requirements in Subpart J of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) that govern the use of dispersants, other chemical and biological agents, and other spill mitigating substances when responding to oil discharges into waters of the United States (U.S.). The proposal addresses the efficacy, toxicity, environmental monitoring of dispersants, and other chemical and biological agents, as well as public, state, local, and federal officials' concerns regarding their use. Specifically, the proposal amends the Subpart J regulatory requirements for the NCP Product Schedule (Schedule) by adding new listing criteria, revising the efficacy and toxicity testing protocols, and clarifying the evaluation criteria for removing products from the Schedule. The Agency also proposes amended requirements for the authorities, notifications, monitoring, and data reporting when using chemical or biological agents in response to oil discharges to the navigable waters of the United States and adjoining shorelines, the waters of the contiguous zone, and the high seas beyond the contiguous zone in connection with activities under the Outer Continental Shelf Lands Act, activities under the Deepwater Port Act of 1974, or activities that may affect natural resources belonging to, appertaining to, or under the exclusive management authority of the United States, including resources under the Magnuson Fishery Conservation and Management Act of 1976. These requirements are anticipated to encourage the development of safer and more effective spill mitigating products, and would better target the use of these products to reduce the risks to human health and the environment. Further, the amendments are intended to ensure that On-Scene Coordinators (OSCs), Regional Response Teams (RRTs), and Area Committees have sufficient

information to support agent preauthorization or authorization of use decisions.

**DATES:** Comments must be received on or before April 22, 2015.

**ADDRESSES:** Submit your comments, identified by Docket ID No. EPA-HQ-OPA-2006-0090, by one of the following methods:

- *Federal Rulemaking Portal:* <http://www.regulations.gov>. Follow the on-line instructions for submitting comments.

- *Mail:* The mailing address of the docket for this rulemaking is EPA Docket Center (EPA/DC), Docket ID No. EPA-HQ-OPA-2006-0090, 1200 Pennsylvania Avenue NW., Washington, DC 20460.

- *Hand Delivery:* Such deliveries are only accepted during the Docket's normal hours of operation, and special arrangements should be made for deliveries of boxed information.

*Instructions:* Direct your comments to Docket ID No. EPA-HQ-OPA-2006-0090. EPA's policy is that all comments received will be included in the public docket without change and may be made available online at <http://www.regulations.gov>, including any personal information provided, unless the comment includes information claimed to be Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Do not submit information that you consider to be CBI or otherwise protected through

<http://www.regulations.gov>. The <http://www.regulations.gov> Web site is an "anonymous access" system, which means EPA will not know your identity or contact information unless you provide it in the body of your comment. If you submit an electronic comment, EPA recommends that you include your name and other contact information in the body of the comment and with any disk or CD-ROM you submit. If EPA cannot read your comment due to technical difficulties and cannot contact you for clarification, EPA may not be able to consider your comment. Electronic files should avoid the use of special characters, any form of encryption, and be free of any defects or viruses. Comments and suggestions regarding the scope of any future rulemaking should be clearly differentiated from comments specific to this proposal (e.g., label Suggestions for Future Rulemaking and Comments on Current Proposal).

*Docket:* All documents in the docket are listed in the <http://www.regulations.gov> index. Although listed in the index, some information is not publicly available, e.g., CBI or other

information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will be publicly available only in hard copy. Publicly available docket materials are available either electronically in <http://www.regulations.gov> or in hard copy at the EPA Docket, EPA/DC, EPA West, Room 3334, 1301 Constitution Avenue NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is 202-566-1744 to make an appointment to view the docket.

**FOR FURTHER INFORMATION CONTACT:** For general information, contact the Superfund, TRI, EPCRA, RMP, and Oil Information Center at 800-424-9346 or TDD at 800-553-7672 (hearing impaired). In the Washington, DC metropolitan area, contact the Superfund, TRI, EPCRA, RMP, and Oil Information Center at 703-412-9810 or TDD 703-412-3323. For more detailed information on this proposed rule contact Gregory Wilson at 202-564-7989 ([wilson.gregory@epa.gov](mailto:wilson.gregory@epa.gov)) or Vanessa Principe at 202-564-7913 ([principe.vanessa@epa.gov](mailto:principe.vanessa@epa.gov)). The contacts address is: U.S. Environmental Protection Agency, Office of Emergency Management, Regulations Implementation Division, 1200 Pennsylvania Avenue NW., Washington, DC 20460-0002, Mail Code 5104A, or visit the Office of Emergency Management Web site at <http://www.epa.gov/oem/>.

**SUPPLEMENTARY INFORMATION:** The contents of this preamble are:

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### I. General Information

In April 2010, the Deepwater Horizon underwater oil well blowout discharged significant quantities of oil into the Gulf of Mexico. The blowout discharged oil from one mile below the sea surface. Approximately one million gallons of dispersants over a three-month period were deployed on surface slicks over thousands of square miles of the Gulf, and approximately three quarters of a million gallons of dispersants were, for the first time, injected directly into the oil gushing from the well riser. This use of dispersants raised many questions about efficacy, toxicity, environmental trade-offs, and monitoring challenges that EPA seeks to address through the proposed revisions to Subpart J.

The proposed revisions to Subpart J address the use of dispersants and other chemical and biological agents to respond to oil discharges into waters of the U.S. Over the past several years, EPA's Office of Research and Development (ORD) has conducted research on improved laboratory protocols for dispersant and bioremediation efficacy, and revisions to Subpart J to address these new protocols were under consideration. As a result of this research and the Deepwater Horizon event, the new protocols in the proposed revisions, in addition to increasing the overall scientific soundness of the data, take into consideration not only the efficacy but also the toxicity, long-term environmental impacts, endangered species protection, and human health concerns raised during responses to oil discharges, including the Deepwater Horizon blowout. Additionally, area planning requirements for dispersant use authorization, toxicity thresholds and advanced monitoring techniques are also proposed. The proposed amendments are a major component of

EPA's effort to inform the use of dispersants and other chemical or biological agents when responding to oil discharges. They incorporate lessons learned from the federal government's experiences in the Gulf, and address recommendations specific to agent testing and use in response to oil discharges from the National Commission on the BP Deepwater Horizon Oil Spill and Offshore Drilling Report to the President.

The proposed amendments would help to ensure that only products that perform effectively in laboratory testing would be listed on the NCP Product Schedule (Schedule) for use in mitigating the effects of oil discharges in the environment. Manufacturers would be required to provide more detailed product application materials, ecological toxicity data, and human health and safety information, including more detailed instructions for product application in the field. Prohibitions on using products in certain areas under certain conditions determined by On-Scene Coordinators (OSCs), Regional Response Teams (RRTs), and EPA would help ensure that first responders are better able to mitigate environmental effects when spills occur. The required submission of additional product toxicity information would aid OSCs and RRTs when evaluating specific product information and when deciding whether and which products to use to mitigate hazards to the environment and human health caused by discharges or threatened discharges of oil.

Specifically in this action, the Agency proposes, for the following areas:

- **Definitions.** Amend definitions for Bioremediation agents, Burning agents, Chemical agents, Dispersants, Sinking agents, Sorbents, and Surface washing agents; add new definitions for Bioaccumulation, Bioconcentration, Biodegradation, Biological agents, Bioremediation, Herding agents, Products, and Solidifiers; and remove definitions for Miscellaneous Oil Spill Control Agents (MOSCA) and Surface collecting agents.

- **General Requirements.** Revise to reflect new and amended regulatory definitions.

- **Authorization of Use.** Revise to clarify planning and preauthorization responsibilities, establish limitations and prohibitions on the use of certain agents, establish requirements for storage and use of agents, clarify authorities for requiring supplemental testing, monitoring and information on agents, establish requirements for agent recovery from the environment, and establish reporting requirements for agent use.

- **Monitoring the Use of Dispersant.** Establish monitoring requirements for dispersant use in response to major discharges and/or certain dispersant use situations.

- **General Product Information for Schedule Listing.** Revise and establish requirements, including designation of and testing for all product categories under which the listing is requested, Safety Data Sheets, sample product labels, shelf life, collection and recovery, persistence in the environment, storage and use conditions, physical and chemical properties, component identities, concentration limits on National Water Quality Criteria and Standards contaminants, laboratory accreditations, submission of all testing data and calculations, production capabilities, and any other data or certification informing the product's performance capabilities or environmental benefits.

- **Dispersant Testing and Listing Requirements.** Revise the efficacy testing methodology using a baffled flask test, establish new developmental and sub-chronic toxicity testing requirements, revise the acute toxicity testing methodologies, revise the listing criteria, and establish use limitations to saltwater environments.

- **Surface Washing Agent Testing and Listing Requirements.** Revise the acute toxicity testing methodology and listing requirements, establish efficacy testing requirements and listing criteria, and establish use limitations based on product testing for salt and/or freshwater environments.

- **Bioremediation Agent Testing and Listing Requirements.** Revise the efficacy and acute toxicity testing methodologies and listing criteria, establish exceptions for specified non-proprietary products, and establish use limitations based on product testing for salt and/or freshwater environments.

- **Solidifier and Herding Agent Testing and Listing Requirements.** Revise the acute toxicity testing methodology and listing criteria, and establish use limitations based on product testing for salt and/or freshwater environments.

- **Sorbent Requirements.** Establish a list of known, non-proprietary sorbents to be made publicly available in lieu of listing sorbents on the Schedule, and requirements for data and information for sorbent products with components other than those specifically identified in the rule.

- **Submissions of Confidential Business Information.** Revise the allowable confidential business information claims and reporting procedures.

- *Addition of a Product to the Schedule.* Revise the submission requirements including the package contents, EPA’s review of submission package, request for review of decision, changes to a product listing, and transition of listed products from the current Schedule to the new Schedule.

- *Mandatory Product Disclaimer.* Revise the product disclaimer requirements.

- *Removal of a Product from the Schedule.* Revise the basis for removal and appeals process.

- *Appendix C to part 300.* Revise the requirements for product testing protocols and summary test data including new dispersant baffled flask efficacy and toxicity tests; new standard acute toxicity tests for bioremediation agents, surface washing agents, herding agents, and solidifiers; and revised bioremediation agent efficacy test.

- *Appendix E to Part 300—Oil Spill Response.* Remove this appendix from the NCP.

EPA estimates industry may incur total incremental costs of approximately \$668,000 to \$694,000 annually. The benefits of the Subpart J amendments are assessed qualitatively. Such benefits include, for example, greater clarity of regulatory requirements, as well as less toxic products. This action does not pose significant impacts on a substantial number of small entities. The Regulatory Impact analysis, which can be found in the docket, provides more detail on the cost methodology and benefits of this action.

**COST OF THE PROPOSED RULE**

	Annualized costs, 20 years	
	Annualized at 3%	Annualized at 7%
Costs .....	\$667,610	\$694,343

**II. Entities Potentially Affected by This Proposed Rule**

Industrial category	NAICS code
Chemical Manufacturing .....	325
Merchant Wholesalers, Non-durable Goods .....	424
Professional, Scientific, and Technical Services .....	541
Waste Management and Remediation Services .....	562
Oil and Gas Extraction .....	211

The list of potentially affected entities in the above table includes manufacturers and users of chemical and biological agents, and other oil spill mitigating devices and substances used as countermeasures against oil discharges. The Agency’s goal is to

provide a guide for readers to consider regarding entities that potentially could be affected by this action. However, this action may affect other entities not listed in this table. If you have questions regarding the applicability of this action to a particular entity, consult the person(s) listed in the preceding section entitled **FOR FURTHER INFORMATION CONTACT**.

**III. Statutory Authority and Delegation of Authority**

Under sections 311(d) and 311(j) of the Clean Water Act (CWA), as amended by section 4201 of the Oil Pollution Act of 1990 (OPA), Public Law 101–380, the President is directed to prepare and publish the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) for removal of oil and hazardous substances. Specifically, section 311(d)(2)(G) requires the President to include a schedule identifying “dispersants, other chemicals, and other spill mitigating devices and substances, if any, that may be used in carrying out” the NCP. The authority of the President to implement this portion of the CWA is currently delegated to EPA in Executive Order 12777 (56 FR 54757, October 22, 1991). Subpart J of the NCP governs the use of dispersants, and any other chemical or biological agent to respond to oil discharges (40 CFR part 300 series 900).

**IV. Background**

*A. Historical Background*

The Council on Environmental Quality (CEQ) first published the National Oil and Hazardous Materials Pollution Contingency Plan in 1970 (35 FR 8508). Among its elements was Annex X—Schedule of Dispersants and other Chemicals to Treat Oil Spills. Annex X provided a basic regulatory framework that included authorization of use, restrictions, and information requirements to be submitted to the Federal Water Quality Administration (FWQA). This initial schedule advocated mechanical and other control methods, and the removal and proper disposal of oil from the environment. It also specified that dispersants might be used in accordance with the schedule if other control methods are judged to be inadequate or infeasible, and if certain information requirements and usage requirements were met. As a listing requirement, manufacturers had to provide the FWQA with methods for analyzing the chemical components in fresh and salt water, or reasons why such analytical methods could not be provided. Except to prevent or substantially reduce the hazard to

human life or limb, or substantial hazard of fire to property, dispersants were restricted from use on or in any: Distillate fuel oil; spill of oil less than 200 barrels in quantity; shoreline; waters less than 100 feet deep; waters containing major populations or breeding or passage for species of fish or marine life which may be damaged or rendered commercially less marketable by exposure to the dispersant or dispersed oil; waters where the winds and/or currents are of such velocity and direction that the dispersed oil mixtures would likely, in the judgment of the FWQA, be carried to shore areas within 24 hours; and waters where such use may affect surface water supplies. The CEQ revised the National Oil and Hazardous Materials Pollution Contingency Plan, renaming it the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) in 1971 (36 FR 16215). The NCP was amended again in 1971 (36 FR 18411) and 1972 (37 FR 28208), with no substantive changes to Annex X.

As a result of the Federal Water Pollution Control Act (Pub. L. 92–500) of 1972, CEQ again revised the NCP, including revisions to Annex X (38 FR 21887, August 13, 1973). The title of Annex X changed to the “*Schedule of Chemicals to Remove Oil & Hazardous Substances Discharges*.” While the Schedule still advocated mechanical and other control methods, and the removal and proper disposal of oil from the environment, it broadened its applicability to chemical agents, including dispersants. It also recognized separate authorizations of use for chemical agents on minor, medium and major discharges. In addition, the revised schedule required an official report from a recognized laboratory with a description of the analytical methods employed and results obtained in determining the chemical and biological characteristics of the chemical agent, but no longer required the submission of those analytical methods. Biological and burning agents were not part of the Schedule, which expressed caution on their use.

In 1975 CEQ again revised the NCP, including Annex X (40 FR 6282). Annex X was now the “*Schedule of Chemicals and Other Additives to Remove Oil & Hazardous Substances Discharges*,” and it was “. . . revised and expanded to provide more precise and definitive information, concerning substances which may be employed to remove discharges.” Additionally, Executive Order 11735 (38 FR 21243, August 3, 1973) made EPA responsible for Annex X. Chemical agents or any other substance not specifically defined in the

Schedule were to be considered for use on a case-by-case basis. The Schedule advocated the development and utilization of mechanical control methods to remove or mitigate oil, and to remove, mitigate, or neutralize hazardous substances discharges from the environment, with subsequent proper disposal. The Schedule intended that no harmful quantities of any substance were to be applied to waters to remove or mitigate the effects of oil or hazardous substances discharges. The Schedule also provided procedures for authorization of use for different agent categories, and separate regulatory authorization of use for dispersants or other chemical agents was established for minor, medium, and major discharges. Product shelf life, toxicity and effectiveness, and analytical methods needed to obtain such data were among the technical data requirements. Similar provisions were tailored to surface collecting agents and biological additives.

In 1982, EPA amended the NCP; the revision included rewriting of Annex X as Subpart H of 40 CFR part 300 of the revised Plan (47 FR 31180). The Agency allowed OSCs to authorize the use of dispersants or other chemicals to treat discharges of oil, provided they were listed in the previous Annex X, with the following limitations:

- Authorization applies only to discharges of oil, not to releases of hazardous substances.
- OSCs may only authorize the use of dispersant or other chemicals on EPA's Acceptance list, which included the twenty-eight products tested and found acceptable for their intended purpose in the previous Annex X.
- State consultation provisions regarding the use of any dispersant or other chemicals in its waters required the OSC to obtain concurrence from the EPA representative to the RRT.

The new Subpart H of 40 CFR part 300 also provided for a case-by-case authorization by the EPA Administrator ("Administrator") or designee of the use of dispersants or other chemicals not on EPA's Acceptance list in treating oil discharges or hazardous substances releases. However, EPA did not include testing procedures or a process for designation of dispersants or other chemicals as acceptable for use.

In 1984, EPA published amendments to Subpart H, including adding Appendix C (49 FR 29192). The amendments specified testing and data requirements for listing of dispersants, surface collecting agents, or biological additives on the Schedule to ensure sufficient data was available for the OSC. Standardized testing procedures

generated comparable data regarding the products' effectiveness and toxicity. Appendix C detailed the methods and types of apparatus to be used in carrying out the revised standard dispersant effectiveness and toxicity tests, as well as the format required for summary presentation of product test data. Listing of a product on the Schedule was neither a recommendation nor authorization for use on an oil discharge, but rather a confirmation that data submission requirements had been satisfied. EPA's listing on the Schedule did not confirm its safety or effectiveness or constitute an endorsement; in fact, a new requirement was established that either a written disclaimer to this effect or EPA's written disclaimer be included in all product technical literature or advertisements. Products previously listed under Annex X were included on the new Schedule as the previous data requirements were sufficient to permit OSCs to make informed decisions about product use.

The amendments also provided for OSC authorization for use of burning agents on a case-by-case basis with the concurrence of the EPA RRT representative and the States, and prohibited the use of sinking agents in waters of the United States. They encouraged advance planning by allowing the OSC to act without the concurrence of the RRT and affected States if these parties have approved a plan identifying the products that may be used in specific contexts. The amendments also allowed the OSC to authorize the use of any product (including products not on the Schedule) without obtaining the concurrence of the EPA representative to the RRT or the affected States if the use of a dispersant, surface collecting agent, or biological additive is necessary to prevent or substantially reduce hazard to human life, and there is not sufficient time to obtain concurrence. However, the OSC was to inform the EPA RRT representative and the affected States of the use of a product as soon as possible, and obtain their concurrences for the product's continued use in the situation once the threat to human life had subsided. These provisions were designed to eliminate delays in life threatening situations, such as spills of highly flammable petroleum products in harbors or near inhabited areas. Subpart H was re-designated as Subpart J with minor changes in 1990 (55 FR 8666), with those definitions present in Subpart H moved to Subpart A, and a new definition and data requirements for miscellaneous spill control agents added.

In 1994, EPA revised the NCP in response to the passage of the Oil Pollution Act in 1990 (59 FR 47384). The final rule significantly revised Subpart J to its current regulatory requirements with respect to authorization of use, data requirements, dispersant effectiveness and toxicity testing protocols, surface washing agent toxicity testing protocol, bioremediation agent effectiveness testing protocol, and requirements for adding products to the Schedule. As a result, several dispersants were re-classified as surface washing agents because they did not pass the dispersant efficacy test threshold, but were effective in removing oil from solid surfaces.

#### *B. Current Statute and Regulation*

Section 300.910 of Subpart J addresses the authorization of the use of products on the Schedule and specifies the conditions under which OSCs may authorize the use of dispersants, other chemicals, and other spill mitigating substances. Sections 300.915 and 300.920 describe the data requirements and the process for adding products to the Schedule. To list a product on the Schedule, Subpart J currently requires the submission of technical data on the product to EPA. Data on dispersants, surface collecting agents, surface washing agents, and miscellaneous oil spill control agents must include the results of the toxicity test set for these products in Appendix C of the NCP. Data on dispersants must also include the results of the dispersant effectiveness test, while bioremediation agents must include results of the bioremediation effectiveness test, also set forth in Appendix C. These tests are conducted at the expense of the product manufacturers and must be performed by laboratories experienced with Appendix C protocols.

The raw data and a summary of the results from these tests are then submitted to EPA, where they are reviewed to confirm that the data are complete and that the specified procedures were followed. The data requirements for placement of a product on the Schedule are designed to provide sufficient data for the OSCs to judge whether and in what quantities a product may be used to control a particular discharge. Inclusion of a product on the Schedule means only that the data submission requirements have been satisfied. The listing of a product on the Schedule does not mean that the product is recommended or authorized for use on any specific oil discharge nor does it imply that EPA has in any other way endorsed the product for the use listed or for other

uses. The standardized testing procedures set forth in Appendix C provide OSCs comparable data regarding the toxicity, effectiveness, and other characteristics of different products.

### C. Advanced Response Planning

Under the current 300.910(a) regulation, RRTs and the Area Committees (ACs) are required to address, as part of their planning activities, the desirability of using appropriate dispersants, and other chemical or biological agents. The RRTs and ACs generally develop "preauthorization plans" which address the specific context in which products can be used under OSC direction. Preauthorization plans are approved with concurrences from EPA, Department of Interior (DOI) and Department of Commerce (DOC) natural resource trustees, and the state(s) with jurisdiction over the water to the area, which they apply. When a preauthorization plan approves in advance the use of certain products under specified circumstances, the OSC may authorize the use of the products without obtaining the specific concurrences described elsewhere in that section of the regulation. The use of chemical or biological agents is only one approach of many available, such as mechanical collection or *in-situ* burning, and decisions about their use should be weighed to achieve greater overall environmental protection.

To facilitate the best possible response, it is important that the regional-level and area-level contingency planning efforts of the RRTs and ACs, respectively, are coordinated closely with each other and are consistent. Section 300.910(a) authorizes the RRTs to review and either approve, disapprove, or approve with modification the preauthorization plans developed by Area Committees that addresses dispersants or other spill mitigating substances. This advanced planning has allowed the OSC to authorize the use of products without obtaining the specific concurrences, if the RRT representatives from EPA and the states with jurisdiction over waters to which a preauthorization plan applies, and DOC and DOI natural resource trustees approve their use in advance. The OSC primarily uses the Schedule to confirm if a product is listed, analyze toxicity and efficacy data, note worker health and safety precautions, understand proper product application, and compare one product to another in order to make the most informed decision on how to mitigate an oil discharge.

AC responsibilities include enhancing contingency planning; ensuring preplanning of joint federal, state, and local response efforts; and expediting decisions on the use of dispersants and other spill mitigating devices and substances. The Area Contingency Plan (ACP) must list the equipment, dispersants or other spill mitigating substances, and personnel available to ensure effective and immediate removal of a discharge. ACPs must also ensure the mitigation or prevention of a substantial threat of a discharge; provide a description of the procedures to be followed for obtaining an expedited decision regarding the use of dispersants (which may be addressed in applicable preauthorization plans); and identify the means to monitor use of chemical countermeasures. Many coastal ACPs include some type of preauthorization zones for dispersants, while most Regional Contingency Plans (RCPs) address other product categories, such as bioremediation and surface washing agents. RRTs, in cooperation with the states and federal agencies, have addressed the requirements for the conduct of *in-situ* burns (ISB) of oil discharges in their RCPs.

This planning has allowed consideration of chemical agent use on oil discharges as a viable response tool in combination with other mitigation measures. These agents have increasingly been considered and used in the field, as evidenced by research and case studies presented at national and international oil spill conferences, research and development funding from private and government stakeholders, RRT efforts to plan for their use, requests for EPA and other federal experts to advise field personnel on the use of such products, and by the response to the Deepwater Horizon oil spill. While many research efforts have helped to clarify issues and added information on the toxicity, efficacy, proper use, and human health impacts of these response technologies, uncertainties still arise. The Agency has an interest in resolving the issues that arise from the use of chemical and biological agents in spill responses, as its representatives are asked to concur with chemical and biological agent use for marine and freshwater spills.

The use of non-mechanical oil spill mitigating technologies, such as dispersants, surface washing agents, sorbents, solidifiers, bioremediation agents, and others are among the available oil response options that responders may consider in the United States and the world. The National Academies' National Research Council report titled "Oil Spill Dispersants:

Efficacy and Effects" (2005), recommends that steps be taken to better support policymakers and spill responders faced with making choices regarding the use of dispersants as part of spill contingency planning efforts or during actual spills. The United States Coast Guard has promulgated new requirements for certain vessels to have only Schedule listed dispersant response capabilities while operating in designated dispersant preauthorization zones (74 FR 45004, August 31, 2009).

The proposed amendments are aimed at ensuring that chemical and biological agents have met efficacy and toxicity requirements, that product manufacturers provide important use and safety information, and that the planning and response community is equipped with the proper information to authorize and use the products in a judicious and effective manner.

### V. This Action

This proposal addresses the efficacy, toxicity, environmental monitoring of dispersants, other chemical and biological agents, and other spill mitigating substances, as well as public, state, local, and federal officials concerns on their authorization and use. The sections below explain the proposed requirements and revisions, and EPA is requesting comments by section. Alternatives offered should include rationale and supporting information in order for the Agency to include the alternative in any final rule.

#### A. Discharge of Oil

The Agency is proposing revisions to harmonize 40 CFR part 110.4 with the definitions for chemical and biological agents proposed for Subpart J. The current language in § 110.4 is specific to dispersants and emulsifiers. The proposal replaces the terms "dispersants and emulsifiers" with the broader terms of "chemical agent" and "biological agent" as proposed to be amended in § 300.5. The proposed definition for chemical agents includes elements, compounds, or mixtures designed to facilitate the removal of oil from a contaminated environment and mitigate any deleterious effects. The proposed definition for biological agents includes microorganisms (typically bacteria, fungi, or algae) or biological catalysts, such as enzymes, able to enhance the biodegradation of a contaminated environment. Chemical and biological agents would include both the dispersants and emulsifiers cited in § 110.4. By revising 40 CFR part 110, the Agency is clarifying that any chemical or biological agent added to a discharge of oil with the intent to circumvent any

provision of 40 CFR part 110 is prohibited. To further reflect the proposed revisions, the Agency is proposing to also amend the section title to “Chemical or biological agents.” EPA believes the proposed amendment is consistent with U.S. Coast Guard (USCG) regulations, which prohibit the discharge of chemicals or other substances into the sea that circumvent discharge conditions specified in their regulation (33 CFR 151.10(g)). EPA requests comment on these revisions.

#### B. Subpart A—Introduction

##### 1. Definitions

The Agency is proposing revisions to § 300.5 that amend the definitions for Bioremediation agents, Burning agents, Chemical agents, Dispersants, Sinking agents, and Sorbents. The Agency proposes to revise the term Surface washing agent and amend its definition. Additionally, the proposal includes new definitions for Bioaccumulation, Bioconcentration, Biodegradation, Biological agents, Bioremediation, Herding agents, Products, and Solidifiers. Finally, the Agency is removing the definitions for Miscellaneous Oil Spill Control Agent (MOSCA) and Surface collecting agents.

##### (a) Revised Definitions

**Bioremediation agent**—The Agency is proposing to revise the definition of bioremediation agents to identify as such biological agents and/or nutrient additives. These agents would be deliberately introduced into a contaminated environment to increase the rate of biodegradation, which in turn would assist in mitigating deleterious effects caused by contaminants. The proposed definition identifies as bioremediation agents microorganisms and enzymes. It also identifies nutrient additives such as fertilizers containing bio-available forms of nitrogen, phosphorus, and potassium. The proposed definition clarifies the current definition and adds specific examples of bioremediation agents. This clarification will help manufacturers of products in identifying the type of product, and hence, what testing requirements they will need to comply with to have a product listed on the Schedule.

**Burning agents**—The Agency proposes to revise the definition of burning agents to identify as such those additives that improve the combustibility of the materials to which they are applied. This could be achieved through either physical or chemical means. Burning agents include inorganic compounds in the form of gelling agents, such as aluminum soap.

For example, the fuel used in helitorch systems is a mixture of powdered gelling agents with either gasoline, jet fuel, or a diesel/gas mixture, which are organic compounds.<sup>1</sup> The Agency believes both the inorganic gelling agent and the organic fuel (*e.g.*, gasoline) meet the burning agent definition by improving the combustibility of the materials to which they are applied through physical or chemical means. The Agency considered including ignition devices in the definition of burning agent because improving the combustibility of oil and igniting that oil could be considered one and the same. The intent would be to clarify the potential that not only substances, but also the devices holding those substances, be considered in the case-by-case authorization of use of such agents. The Agency rejected this approach since many devices either deliver the agent to the oil to be burned and do not enter the water, or are consumed in the burn along with the agent. The Agency requests comment on whether it should add ignition devices to the definition of a burning agent.

**Chemical agents**—The Agency proposes to revise the definition of chemical agents to identify as such those elements, compounds, or mixtures that are designed to facilitate the removal of oil. These agents may be used to mitigate deleterious effects of the oil on a contaminated environment. The proposed definition would include under the chemical agent category burning agents, dispersants, herding agents, sinking agents, solidifiers, surface washing agents, and those bioremediation agents that consist of nutrient additives. The proposed language reflects the distinction the Agency is now making between chemical and biological agents, allowing product manufacturers to better target the testing requirements and OSCs to better inform their authorization of agent use in specific situations. The proposal also removes from the definition agent categories that are either being eliminated, prohibited or amended to conform to the changes, as discussed below.

**Dispersants**—The Agency is proposing to revise the definition of dispersants to identify them as those agents that promote the formation of small droplets of oil in the water column by reducing the oil-water interfacial tension. Dispersants are proposed to be defined as typically mixtures of solvents, surfactants

(including biosurfactants), and additives. The proposed definition specifically addresses the process through which these agents assist in mitigating the consequences of a discharge, clarifying for manufacturers which testing requirements they will be subject to when seeking to list a product on the Schedule.

**Sinking agents**—The Agency is proposing to revise the definition of sinking agents to identify them as those substances deliberately introduced into an oil discharge to submerge the oil to the bottom of a water body. The current definition is ambiguous in regards to how much sinking below the water surface would qualify as a sinking agent, as some submersion below the surface but generally not to the bottom of the water body can also be associated with other agents, such as dispersants. The proposed definition specifies these agents purposely sink the oil to the bottom of a water body. The Agency is prohibiting the use of sinking agents in the remediation of oil discharges in water because of their potential for causing adverse effects on benthic organisms vital to the food chain of the aquatic environment.

**Sorbents**—The Agency is proposing to revise the definition of sorbents to identify them as inert, insoluble substances that readily absorb and/or adsorb oil or hazardous substances. The proposed definition specifies that sorbents are not combined with or act as any other chemical or biological agent. The proposed definition also specifies that sorbents are generally collected and recovered from the environment and that they may be used in their natural bulk form, or as manufactured products in particulate form, sheets, rolls, pillows, or booms. The proposed definition identifies sorbents as substances consisting of: (1) Natural organic substances (*e.g.*, feathers, cork, peat moss, and cellulose fibers such as bagasse, corncobs, and straw); (2) inorganic/mineral compounds (*e.g.*, volcanic ash, perlite, vermiculite, zeolite, clay); and (3) synthetic compounds (*e.g.*, polypropylene, polyethylene, polyurethane, polyester). The proposed changes simplify the definition by removing the definitions of absorption and adsorption that are embedded in the current definition of sorbents. The Agency believes this is appropriate given these are generally recognized scientific terms and the proposal does not distinguish sorbents or in any way restrict their use based on whether they absorb or adsorb the oil. The definition also adds the “natural” qualifier to organic substances, indicating that organic substances that

<sup>1</sup> Fingas, Mervin, (Ed) (2011), Oil Spill Science and Technology, Gulf Professional Publishing, pp. 836, ISBN: 978-1-85617-943-0.

have been treated with other substances would not necessarily fall under this category of agents and should not immediately be considered a sorbent. It also expands on and simplifies the examples by removing the references to the type of birds that feathers could come from, by adding bagasse to the examples for natural organic substances, and by adding clay to the examples for inorganic/mineral compounds. The Agency requests comments on whether the definition is appropriate or if there are other materials that should also be included.

Additionally, the Agency requests comments on particulate materials (*e.g.*, clay) as sorbents. There is concern that particulate materials with densities greater than the seawater (or freshwater depending on where they may be used) have the potential to become sinking agents, settling to the seabed and posing potential risks to benthic organisms. This question is also relevant when considering emerging response technologies such as the use of particulate materials to form oil-mineral aggregates (OMAs) to promote dispersion. OMAs are stable microscopic entities formed when particulate materials interact with the oil droplets resulting in distinct oil and mineral phases. These fine mineral particles could be intentionally introduced by themselves to promote physical dispersion by preventing the oil to re-coalesce, or can also be used in conjunction with dispersants to enhance chemical dispersion. However, any particulate material that by itself, or when combined with oil (*e.g.*, OMA), results in overall densities less than the waters where it may be used, would not be prohibited as a sinking agent. The Agency requests comment on whether the use of sorbent materials in particulate form should be specified for use only in booms or other contained manufactured products, and whether there should be limitations on the authorization of use on water for sorbents in loose particulate form. Alternatively, the Agency also requests comment and supporting rationale on other approaches, including whether particulate materials in loose form, or specifically OMA technologies, should be excluded from the regulatory definition of sorbents.

Finally, EPA also requests comments on the qualifier phrase “that are generally collected and recovered from the environment.” For example, a natural organic and biodegradable sorbent (*e.g.*, bagasse) may not necessarily need to be removed after it has absorbed/adsorbed the oil when used in a wetland or salt marsh. Such

removal may cause more harm than the oil itself due to trampling in the wetland or salt marsh. Once the oil is brought to the surface by the sorbent, biodegradation of both the oil and the sorbent can take place, especially if nutrients are added to enhance biodegradation. Another example would be the use of OMA technology to promote dispersion, which might not lend itself to collect or remove the aggregates from the environment. Thus, the Agency is requesting comment on whether testing and/or authorization of use requirements should be considered for particulate materials in loose form or OMA technologies that may be used in discharge situations where they would not be collected and recovered from the environment.

*Surface washing agents*—The Agency is proposing to revise the term surface washing agent and the definition for surface washing agents. The proposed definition would identify surface washing agents as those substances that separate oil from solid surfaces (*e.g.*, beaches, rocks, metals, or concrete) through a detergency mechanism. This detergency mechanism would lift and float the oil for collection and recovery from the environment. The use of these agents results in minimal dissolution, dispersion, or transfer of oil into the water column. The proposed changes revise the term from singular to plural to be consistent with the other agent definitions and clarifies that these agents are to be recovered from the environment along with the oil being treated.

#### (b) New Definitions

The Agency is proposing to add several new definitions that serve as the foundation for the new proposed biological agent classification: Bioaccumulation, Bioconcentration, Biodegradation, Biological agents, and Bioremediation. The proposed definitions include basic terminology and are consistent with definitions of these terms generally understood by the scientific community.

The Agency is also proposing new definitions for the terms herding agents and solidifiers. The proposed definitions address types of agents originally captured under the surface collecting agent or the miscellaneous oil spill control agent categories respectively, and are terms that are more commonly used in industry. The definitions more specifically describe the specific process through which the product affects the oil for the categories and are consistent with definitions of these terms generally understood by the scientific community.

Finally, the Agency is proposing a new definition for the term product to clarify the difference between a specific product and an agent type or category. All of the proposed new definitions can be found in the § 300.5 of this action.

#### (c) Removed Definitions

The Agency is proposing to remove the definitions for surface collecting agent and for miscellaneous oil spill control agent (MOSCA). The surface collecting agent definition is being removed and replaced with a new herding agent definition to better reflect the common terminology used in industry for these agents. The MOSCA definition is being removed and replaced with a number of new and/or revised definitions for types of agents. The original MOSCA category was used as a catchall for all types of products that did not meet other agent definitions. As the Agency adds new, more stringent testing requirements for listing products on the Schedule, there is a need for more specific category definitions to assist manufacturers in determining which of those testing requirements apply to their products. The Agency believes it has identified categories that capture all products to be listed on the Schedule; we request comment on whether the MOSCA category should be retained, and whether the proposed categories are appropriate, including sufficient information and rationale to support the addition of any new categories.

#### C. Subpart J—Use of Dispersants, and Other Chemical and Biological Agents

##### 1. General

EPA is proposing to amend § 300.900 by revising paragraphs (a) and (c), and by adding paragraph (d) to reserve for “Releases of Hazardous Substances”. The proposed revisions clarify that Subpart J addresses not only chemical agents, but also those agents that now fall under the newly proposed biological agent category. The revisions reaffirm the notion that Subpart J is not only comprised of a Schedule of chemical and biological agents, but also includes testing requirements and authorization of use procedures. Consistent with current Subpart J regulatory requirements the Agency is proposing to reserve a section for “Releases of Hazardous Substances” to take place of the current placeholder in § 300.905, which is proposed to be removed. Based on all relevant circumstances, testing data and information, and in accordance with the authorization of use procedures (including the appropriate concurrences and consultations), the waters and



quantities in which a dispersant, or other chemical or biological agents may be safely used are to be determined in each case by the OSC. When taken together, these testing requirements, listing of agents and authorization of use procedures address the types of waters and the quantities of listed agents that may be used in response to discharges of oil or hazardous substances. The wide variability in waters, weather conditions, organisms living in the waters, and types of oil that might be discharged requires this approach.

## 2. Authorization of Use

Section 300.910 sets forth the provisions for the authorization of use of products on the Schedule in response to oil discharges. Subpart J does not state or imply that chemical or biological agents are preferred over other response options such as mechanical recovery devices. EPA believes that the circumstances surrounding oil discharges and the factors influencing the choice of response methods are many. During the DWH response, a priority countermeasures scheme was established to first use mechanical recovery via skimming/booming or in-situ burning followed by subsea dispersant and lastly surface dispersant use. Following DWH, EPA and the NRT issued Interim Actions regarding the use of dispersants on oil spills to NRT members and RRT co-chairs<sup>2</sup> and to EPA Regional Administrators<sup>3</sup> for consideration during response planning. While response actions are incident specific, the availability of response methods that address the specific discharge situation depends largely on contingency planning activities and on how these requirements are implemented through the RCPs, ACPs and vessel and facility response plans. In order for a response to be effective, the NCP requires coordination between the regulatory and planning entities responsible for all these response plans. The Agency believes that preauthorization or expedited decision making plans are critical elements of contingency planning activities. Regularly reviewing or revising preauthorization or expedited decision making plans provides those agencies in

charge of preparedness planning the opportunity to identify and resolve concerns in advance, leading to quick and effective operations during removal actions. The Agency believes these proposed revisions to the authorization of use provisions will assist OSCs, RRTs, and ACs in their advanced planning activities as they consider response methods that result in the greatest environmental protection. The goal is to ensure that preauthorization or expedited decision making plans are developed and maintained to effectively support decisions by OSCs during removal actions. In carrying out advanced planning activities, the Agency believes the NRT can assist RRTs by providing guidance on national level issues that may arise during planning activities.

EPA is amending § 300.910 by revising paragraphs (a) through (f); and by adding paragraphs (g), (h) and (i). EPA is not proposing major changes to the current authorities granted to OSCs, RRT representatives to the RRTs, States, DOC, DOI, or other National Response Team decision makers with regards to the authorization and application of chemical or biological agents. However, EPA recognizes that the planning for and prolonged use and monitoring of chemical agents, especially dispersants, may require additional planning activities and monitoring requirements. Thus, the Agency is proposing to reorganize this section; to add titles to the paragraphs for ease of use; to add several requirements addressing the storage and use of agents, notification of agent use and recovery from the environment; and to revise language to clarify established EPA policy. The proposed revisions and clarifications are highlighted for each paragraph under § 300.910.

EPA is also confirming, consistent with the intent of the NCP, that use of chemical or biological agents in response to oil discharges to waters of the U.S. or its adjoining shorelines must be authorized by an OSC in accordance with Subpart J. The unauthorized use can result in violations of section 301 and 311 of the CWA. Section 301(a) makes unlawful “the discharge of any pollutant by any person,” except in compliance with certain provisions of the CWA. In addition, section 311(b) establishes penalties for persons who fail or refuse to comply with any regulation issued under section 311(j) of the CWA.

(a) Use of Agents Identified on the Schedule on Oil Discharges Addressed by a Preauthorization Plan

The Agency is proposing revisions to § 300.910(a) of Subpart J to address the preauthorized use of chemical and biological agents identified on the Schedule. The proposed revisions clarify the process for preauthorization, the responsibilities of all involved parties, and the factors to consider during the preauthorization process, including the authorization for the use of agents by the OSC at the time of a discharge. EPA is also proposing to reorganize paragraph (a) to provide greater clarity by making the regulatory text easier to read and follow. The Agency believes that the proposed revisions do not change its fundamental policies regarding roles of Federal, state and local representatives involved in planning for and responding to an oil discharge, but rather clarify the current requirements and further explain the responsibilities for each party. The Agency is also proposing added procedures and review requirements intended to ensure preauthorization plans are up-to-date so they are most effective when implemented in case of a discharge.

EPA believes RRTs and ACs must work together in order to effectively and successfully manage contingency planning. Thus, the proposed revisions continue to require that, as part of their planning activities, RRTs and ACs address in the preauthorization plan whether the use of chemical and biological agents listed on the Schedule on certain oil discharges is appropriate. The Agency believes that the proposed revision clarifies the meaning of the desirability of using appropriate chemical or biological agents. The Agency is removing “. . . the desirability of using appropriate burning agents.” from paragraph (a) and addressing the use of burning agents under paragraph (c) to provide greater flexibility to OSCs for authorization of use.

Under the current paragraph (a), ACs are responsible for developing preauthorization plans. ACs are also responsible for developing ACPs, providing a forum to evaluate the environments within a jurisdiction and establishing protection priorities. The information gathered during the ACP development process should inform the development of preauthorization plans. Identified representatives from the RRTs are responsible for approving or requesting modifications of preauthorization plans developed by the ACs. ACs can advocate for local

<sup>2</sup> Tulis, Dana S., EPA Chair and Caplis, Captain John, USCG Vice-Chair, National Response Team, “Use of Dispersants on Oil Spills—Interim Actions,” Memorandum to NRT Members and RRT Co-Chairs; December 16, 2010.

<sup>3</sup> Stanislaus, Mathy; Assistant Administrator, OSWER EPA; “Revision of Area Contingency Plans/Regional Contingency Plans Regarding Use of Dispersants on Oil Spills—Interim Actions,” Memorandum to EPA Regional Administrators, November 2, 2010.

concerns to be reflected in the preauthorization plans they develop, and the RRTs will decide if the plans are adequate and address region-wide and cross-regional issues. Since the RRTs should be in a position to provide guidance to ACs on common attributes within a region, EPA continues to believe RRTs and ACs should work together to develop preauthorization plans, particularly when identifying discharge situations where chemical or biological agents may be used. There may be circumstances where it is appropriate to allow either the RRTs and/or ACs to develop preauthorization plans. Preauthorization plans developed by an RRT would allow for these plans to better reflect overarching regional circumstances. Therefore, the Agency is proposing to include RRTs as another planning entity with responsibility for developing preauthorization plans, and is requesting comment on this change, and on the advantages or disadvantages of keeping the development of these plans at the AC level.

ACPs generally describe discharge situations for the geographical area for which they apply. Discharge situations developed as part of area contingency planning activities may consider likely scenarios from vessels, and onshore or offshore facilities. While RRTs and ACs should consider the discharge scenarios described in ACPs when identifying discharge situations in developing preauthorization plans, the Agency believes they should also have the flexibility to consider other discharge scenarios. The proposed language states preauthorization plans must specify limits for the quantities and duration of use, and use parameters for water depth, distance to shoreline, and proximity to populated areas for discharge situations identified in which agents may be used. The Agency believes that clearly stating the use parameters in a preauthorization plan will make it easier for planners to address concerns of preauthorizing agent use and in turn for responders to authorize their use.

In meeting these provisions, the preauthorization plans should document how both regional and logistical factors were addressed when establishing dispersant use limits and parameters. Regional factors include the likely sources and types of oil that might be discharged, various discharge scenarios, and the existence and location of environmentally sensitive resources or restricted areas that might be impacted by discharged oil. Logistical factors include inventory, storage locations and manufacturing capability of available agents, availability of equipment needed for

agent use, availability of adequately trained operators, and the availability of appropriate means to monitor agent use. While the proposed revisions simplify the language and clarify the requirements, the Agency believes it is necessary to keep in place the fundamental elements that should be considered. The proposed factors generally parallel those under the current 300.910(a) regulation. Several revisions include identifying some factors as “regional” and others “logistical.” The Agency is identifying “logistical” factors to ensure the availability of chemical and biological agents to address discharge situations identified in the preauthorization plan. The Agency added “various discharge scenarios” as a regional factor to be considered because preauthorization plans may cover more than one ACP with multiple discharge scenarios that RRTs and/or ACs may want to consider. The Agency also added the existence and location of “restricted areas” along with “environmentally sensitive resources” as a factor to consider. Environmentally sensitive resources would include fish, wildlife and their habitats, and other special areas of ecological sensitivity that may be adversely affected by a discharge. While “restricted areas” may include “environmentally sensitive resources” some areas may be restricted from certain activities because of biologically sensitive topographic features or critical habitats, such as submerged rock formations colonized by species (*e.g.*, coral) and the organisms they interact with and support. NOAA’s Environmental Sensitivity Index maps, the Fish and Wildlife and Sensitive (FWS) Environments Plan in ACPs, or environmental impact statements, may contain relevant information on “environmentally sensitive resources” for the RRTs and ACs to consider.

The proposal revises the phrase “potential sources and types of oil that might be spilled” to “likely sources and types of oil that might be discharged.” EPA believes the phrase “likely sources and types of oil” better focuses on the sources and types of oil specific to the preauthorization plan for which agents may be used. In addition, the proposal revises “spill” to “discharge” to be consistent with terminology in the NCP. The proposal also revises the phrase “the available means to monitor product application and effectiveness” to “means to monitor agent use in the environment” as the Agency believes it provides for additional flexibility for the RRTs and/or the ACs to consider the scope of the monitoring, and to include

other endpoints beyond product application and effectiveness. The Agency is proposing to eliminate the “available” qualifier, as it believes it is unnecessary. Likewise, the Agency proposes to eliminate the “available” qualifier before “product and storage locations” and revise the phrase “product and storage location” to the broader “inventory, storage locations and manufacturing capability of available agents” to address lessons learned from the Deepwater Horizon incident, including the challenges posed by the potential sustained use of dispersants.

When developing preauthorization plans, RRTs and ACs should use the best available scientific information to assess environmental trade-offs, including those identified by conducting an ecological risk assessment. Environmental trade-offs should be considered in determining response options that provide the greatest environmental protection. The RRTs and ACs should identify the affected biological resources and their habitats likely to be negatively impacted, as well as those that are expected to benefit. The natural resource trustees are critical partners that can assist in conducting these analyses.

As previously stated, all members of the RRT are afforded an opportunity to review and provide input on a draft preauthorization plan. However, only the RRT representatives from EPA and the state(s) with jurisdiction over the waters and adjoining shorelines within the preauthorization plan area and the DOC and DOI natural resource trustees may approve, disapprove, or approve with modification the draft preauthorization plan. The Agency believes this remains the correct approach. Given preauthorization plans are developed during the contingency planning phase, the Agency believes that DOC and DOI natural resource trustee concurrence is preferred over just consultation because it provides for sufficient time to identify and resolve natural resource concerns. As noted in the 1994 NCP final rule, the requirement for concurrence during the advanced planning phase ensures trustee involvement in decision-making (59 FR 47398). Addressing in advance concerns that might otherwise slow the action ensures that operations during a removal action can be carried out quickly and effectively. EPA believes natural resource trustee concurrence with preauthorization plans satisfies the consultation obligation since the preauthorization plans specify the use parameters for chemical or biological

agents. Thus, the Agency is retaining this concurrence requirement for preauthorization plans.

The Agency is proposing to revise the “Approved preauthorization plans shall be included in the appropriate RCPs and ACPs” in the current § 300.910(a) regulation to “RRTs and ACs shall, as appropriate, include applicable approved preauthorization plans in RCPs and ACPs.” The Agency is proposing to modify the qualifier “approved” with “applicable” to clarify that RRTs and ACs need to include the preauthorization plans only in those RCPs and ACPs to which they apply, and remains consistent with current requirements.

The Agency considered adding specifically the EPA Administrator and the senior EPA representative to the NRT (*e.g.* NRT Chair) to § 300.910(a) and other paragraphs to make clear the EPA Administrator’s and senior EPA NRT representative’s existing authority under section 311(d)(2)(G) of FWPCA and Executive Order 12777, along with the OSC to authorize any chemical or biological agent use. The Agency is clarifying that if the preauthorization plan is approved in advance for chemical or biological agent use under specified discharge circumstances, then the OSC may authorize the use of the agents on the Schedule for their intended purpose without the incident specific concurrences and consultations described in paragraphs (b) of this section unless otherwise directed by the Administrator in accordance with current concurrence authority. The Agency believes this clarification would not impede rapid decision-making on the part of the OSC, and that for the majority of discharge situations, the OSC will remain as the sole authorizing entity for discharge situations covered by preauthorization plans. Note that in situations like a spill of national significance (SONS) or an event of extended duration, the Administrator already has the authority for, and is likely to have a more direct role in chemical or biological agent use decisions. The authority, jurisdiction, and implementation provisions in the NCP flow from section 311 of the Clean Water Act and are reflected in Executive Order 12777. All authorities under CWA 311 are delegated either directly to the Administrator by Congress, or by Executive Order 12777 from the President to the Administrator. While the Administrator’s authority may be further delegated through senior management on down to the RRT representative, the Administrator (and other delegates) retain the authority to act. The mere delegation of authority

does not prohibit the delegator from exercising said authority. However, given these situations are rare and that this is an existing authority, the Agency is not proposing a regulatory amendment to clarify the Administrator’s authority at this time. We request comment on this issue.

The Agency is clarifying that chemical or biological agents may only be used for their intended use, given the different listing requirements proposed for the various categories of chemical or biological agents. For example, a chemical agent that is listed on the Schedule solely as a surface washing agent cannot be authorized for use as a dispersant, nor can a chemical agent that is listed on the Schedule solely as a dispersant for use under saltwater conditions be used in freshwater.

The Agency is proposing specific procedures for concurrence withdrawals, allowing agencies to do so if they believe the preauthorization plan no longer addresses or reflects existing situations if it were to be implemented. While an agency with concurrence authority may now decide to withdraw concurrence from an approved preauthorization plan, there are currently no set procedures to promptly address those situations. The proposal would require the RRT and the ACs to address the withdrawal of approval of the preauthorization plan within 30 days of the withdrawal, allowing an opportunity to address the concerns. The proposal also calls for the RRT to notify the NRT of the final status of the preauthorization plan within 30 days from the withdrawal. The Agency requests comments on whether this 30 day notification requirement should also include notification to the public. In the event of an Agency withdrawing its concurrence from an approved preauthorization plan, EPA believes the advanced planning process should continue with consideration for all the elements specified in paragraph (a) of this section. While the absence of a preauthorization plan requires that authorizations for agent use be conducted according to paragraph (b) of this section, the Agency continues to believe that preauthorization plans serve as a valuable advanced planning tool that provides a strong foundation to support decision-making and strongly encourages the resolution of any withdrawal.

Finally, EPA proposes a new requirement for RRTs and/or ACs to review, and revise as needed, preauthorization plans: (a) At least every 5 years to address revisions of the Schedule; (b) after a major discharge or a spill of national significance (SONS);

(c) to reflect new listings of threatened and/or endangered species or; (d) after any other change such as a new or revised worst case discharge estimate that may impact the conditions under which the use of chemical and biological agents is preauthorized. A 5-year review cycle is consistent with facility response planning requirements; as those plans are revised and updated, it seems reasonable that preauthorization plans should be reviewed and revised accordingly. The Agency recognizes that development of preauthorization plans can be resource intensive; however, once developed, a periodic review and revision as needed should require much less effort. EPA welcomes comment on this timeframe and suggestions with supporting information for alternatives.

This review requirement is intended to ensure that preauthorization plans are actively maintained and updated to reflect revisions to the Schedule. Preauthorization plans, as well as the facility and vessel response plans reflected in them, may include information on products listed on the Schedule. A review at least every 5 years is expected to provide greater consistency not only between any Schedule revisions, but also between any ACPs, facility, and vessel response plans. For example, an ACP revision that results in a change in the worst-case discharge scenario could trigger a preauthorization plan review. Additionally, the requirement specifically includes plan review and revision requirements as appropriate to reflect new listings of threatened and/or endangered species that may occur. The EPA RRT representative, the DOC and DOI natural resource trustees, and, as appropriate, the RRT representative from the state(s) with jurisdiction over the waters of the area to which a preauthorization plan applies must review and either approve, approve with modification, or disapprove any revisions to the preauthorization plans. This review and approval is intended to focus on any revisions, and is not intended as a requirement for review and approval for those portions that do not require modifications.

#### (b) Use of Agents Identified on the Schedule on Oil Discharges Not Addressed by a Preauthorization Plan

The Agency is proposing revisions to § 300.910(b) of Subpart J to address use of chemical or biological agents identified on the Schedule for discharge situations that have not been addressed in preauthorization plans. The proposed revisions clarify the authorities and responsibilities of all involved parties,

and the factors to consider when authorizing the use of listed chemical or biological agents in these situations. The Agency believes the proposed revisions do not change its fundamental policies regarding the roles of Federal, state and local representatives involved in an oil discharge response.

The proposed revisions maintain, with the appropriate concurrences and consultations, the OSC's authority to authorize the use of chemical or biological agents on the oil discharge, provided that the agents are listed on the NCP Product Schedule. The concurrence of the EPA representative to the RRT and, as appropriate, the concurrence of the RRT representatives from the states with jurisdiction over the waters and adjoining shorelines threatened by the release or discharge is maintained. The requirement for consultation with the DOC and DOI natural resource trustees is also maintained. However, the language is amended by removing "when practicable" with respect to consultation with the DOC and DOI natural resource trustees. The Agency believes that the case-by-case decision making should include consultations with natural resource trustees since these discharge situations may present unique challenges when selecting a response option that involves chemical or biological agents. While the Agency recognizes the time-critical nature of decision making during a response, advances in communication technology (e.g., smart phones, email) provide OSCs with increased capabilities to communicate quickly. Therefore, the Agency believes it is reasonable to expect an OSC to be able to notify and explain the circumstances requiring use of the certain agents to natural resource trustees in a timely manner. Of note, while consultation with the trustees on removal actions as required by OPA does not equate to a concurrence requirement, the Agency believes that such concurrence is highly desirable. The Agency is also proposing to revise the term "navigable waters threatened" to "waters and adjoining shorelines threatened" to be consistent with the provisions in paragraph (a) of this section.

The preauthorization plan requirements in paragraph (a) proposes to remove the term "specific context" currently used and instead clearly establish what the term refers to. The proposal specifically identifies the parameters that must be considered by the OSC for authorizing agent use. Similar requirements are proposed under paragraph (b). Thus, in meeting the provisions of § 300.910(b), the OSC

must consider and document the parameters for the use of agents including, but not limited to, quantities to be used, the duration of use, the depth of water, the distance to shoreline and proximity to populated areas, and should address factors such as environmentally sensitive resources or restricted areas that might be impacted, agent inventory and storage locations, agent manufacturing capability, availability of equipment needed for agent use, availability of adequately trained operators and appropriate means to monitor agent use in the environment. These considerations are parallel to those proposed under paragraph (a) for preauthorization and planning purposes. While the Agency is not including other factors that may inform preauthorization planning development, such as various discharge scenarios, this does not mean these factors cannot or should not be considered if RRTs and/or ACs choose to develop expedited decision making plans. The Agency believes these are fundamental elements that would inform an assessment of the overall ecological risks for the OSC to consider when authorizing the use of chemical or biological agents.

Finally, while not required, EPA strongly recommends advanced planning for expedited decision making for cases where the discharge situation is not addressed in the preauthorization plans. Some RRTs have developed expedited approval guidelines that are not part of the preauthorization plans, but that offer an opportunity for advanced contingency planning by gathering information on the key parameters discussed above. Because discharge situations not covered by preauthorization plans need incident specific (i.e., case-by-case) authorization concurrence,<sup>4</sup> expedited approval guidelines can be used to support expedited incident specific authorizations. For chemical or biological agents listed on the Schedule that are not authorized for use under a preauthorization plan, the ACs and RRTs should work together to outline the process for expedited authorization decisions regarding their use. It is important to note that while the NCP requires that the ACPs include procedures for expedited decisions, these procedures can include disapproving the use of agents, or approving the use of agents with certain operational conditions. For example, areas may be designated in which the use of certain agents or other discharge

mitigating devices is prohibited, situations where limits are placed on the quantities of agents used, or situations that require certain monitoring requirements be in place.

#### (c) Burning Agents

The Agency is proposing to replace the current authorization of use for burning agents in § 300.910(c) with a provision that provides greater flexibility to OSCs for authorizing the use of burning agents. Specifically, the Agency proposes that OSCs may authorize the use of burning agents for authorized in-situ burns. The proposed amendments recognize that relatively small quantities of burning agents are ignited prior to or immediately after they are introduced to an oil discharge. Furthermore, they are composed of substances that are expected to rapidly burn off during use, which serves to remove them from the water. The Agency also recognizes that ISB has become an important response option that is used more frequently and the proposed revisions would allow OSCs to authorize the use of burning agents for authorized burns. For example, a significant number of ISBs were conducted during the Deepwater Horizon oil spill,<sup>5</sup> and ISBs appear to be gaining a more prominent role as a response option in federal waters in remote locations, such as the Arctic. Therefore the Agency believes the proposed revisions better address OSC authorities for these situations, without compromising environmental concerns. Further, because of the nature of burning agents and the proposed revisions to the authorization of use for these products, the Agency continues to believe it is not necessary to require product submissions for burning agents. Thus, the proposal removes the provisions for burning agents under the current data requirements. The Agency requests comments on this approach.

#### (d) Exceptions

The proposed rule maintains the provision allowing OSCs to authorize the use of any agent, including products not on the Schedule, when it is determined that the use of the agent is necessary to prevent or substantially reduce a threat to human life. The proposed revisions do not change previous policy, but rather clarify the intent of the exception. The Agency believes that the protection of human life is the primary consideration in responding to an oil discharge. Life-

<sup>4</sup> [http://www.epaosc.org/sites/5083/files/rrt6\\_nearshore\\_dispersant\\_eap\\_031605.pdf](http://www.epaosc.org/sites/5083/files/rrt6_nearshore_dispersant_eap_031605.pdf).

<sup>5</sup> <http://www.restorethegulf.gov/release/2011/08/19/operations-and-ongoing-response-august-17-2011>.

threatening oil discharges (e.g., spills of highly flammable petroleum products in harbors or near inhabited areas) may occur at locations where chemical agents on the Schedule are not immediately available. The Agency believes that in such cases, an OSC must have the ability to use agents that, in his professional judgment, would effectively and expeditiously mitigate the threat to human life. Allowing this authorization to occur without the required concurrences for preauthorization or authorization of use for products on the Schedule under paragraphs (a) and (b) respectively, eliminates delays in responding to potentially life-threatening situations. The Agency is proposing to include “without obtaining the immediate concurrence” to clarify the scope of the exception. The proposed revisions are consistent with the intent of the current regulation which recognizes that once the threat to human life has subsided, the continued use of a product shall be in accordance with authorization of use paragraphs (a) and (b) of the section. In addition, this exception is intended for those extraordinary situations in which time is of the essence to mitigate the threat to human life; revising the language to replace “hazard” with “threat” clearly establishes this. The Agency emphasizes this authority is not intended to circumvent the authorization of use provisions in paragraphs (a) and (b) of this section, which serve to address all other situations.

The proposed revisions also specify that the OSC immediately notify, and document the reasons for the use of an agent to the EPA RRT representative and the affected states as soon as possible, and must obtain their concurrences where continued use of chemical or biological agents extends beyond 48 hours. The Agency believes that advances in communication technologies (e.g., smart phones, email) provide OSCs the increased capabilities to communicate quickly. Therefore, it is reasonable to expect that the OSC notify and explain the circumstances requiring use of the agent to the designated EPA RRT representative and, as appropriate, the RRT representatives from the affected states and the DOC/DOI natural resources trustees within 48 hours. The Agency is requesting comments on these revisions, and specifically on the 48 hour timeframe within which the OSC shall be operating in accordance with authorization of use paragraphs (a) and (b) of the section.

#### (e) Prohibited Agents

The Agency is maintaining the current prohibition for the authorization of use of sinking agents, and is clarifying that this prohibition also applies to any other chemical agent, biological agent, or any substance that acts as a sinking agent when mixed with oil. While certain chemical and biological agents may submerge oil below the water surface (e.g. dispersants are designed to break up oil into small, near neutrally buoyant particles that are entrained in the water column between the surface and the bottom), they would not be considered “sinking agents” for purposes of the proposed definition and this prohibition, given that they do not completely submerge oil to the bottom of the water body when applied to an oil discharge. Sinking agents, when applied to oil discharges, function by sinking floating oil to the bottom of any body of water where used, potentially causing adverse effects on benthic organisms vital to the food chain of the aquatic environment. Additionally, the oil and these agents are very difficult to remove. The Agency has similar concerns regarding substances that could directly cause the oil to submerge to the bottom of the water body when used in an oil spill response, and thus it is specifically proposing to exclude their use.

The Agency is also proposing to add a prohibition from listing on the Schedule and from authorizing use of any chemical or biological agents that contain nonylphenol (NP) or nonylphenol ethoxylates (NPEs) as components. This prohibition reflects the Agency’s concern for these substances, as presented in the *EPA Nonylphenol and Nonylphenol Ethoxylates Action Plan*, released August 18, 2010. The Action Plan specifically addresses nonylphenol (NP) and nonylphenol ethoxylates (NPEs). NP and NPEs are produced in large volumes, with uses that currently lead to widespread release to the aquatic environment. NP is persistent in the aquatic environment, moderately bioaccumulative, and extremely toxic to aquatic organisms. NP’s main use is in the manufacture of NPEs. NPEs are nonionic surfactants that are used in a wide variety of industrial applications and consumer products. Many of these, such as laundry detergents, are “down-the-drain” applications. Some others, such as dust-control agents and deicers, lead to direct release to the environment. NPEs, though less toxic and persistent than NP, are also highly toxic to aquatic organisms, and, in the environment, degrade into NP. Both NP and NPEs have been found in

environmental samples taken from freshwater, saltwater, groundwater, sediment, soil and aquatic biota. NP has also been detected in human breast milk, blood, and urine and is associated with reproductive and developmental effects in rodents. EPA has encouraged the ongoing voluntary phase-out of NPEs in industrial laundry detergents, and intends to evaluate how releases and exposures are mitigated through the phase-out action prior to taking any final regulatory action under the Toxic Substances Control Act.<sup>6</sup> The Agency believes this prohibition would not adversely affect product manufacturers given there are viable alternatives to the use of NP and NPEs in product formulations. However, we are requesting comment on the potential impacts of modifying existing products to meet this new requirement, including cost.

Alternatively, EPA considered a broader prohibition from listing and from authorizing the use of chemical or biological agents formulated with any endocrine disrupting compounds (EDC). EDCs impact exposed organisms by altering the hormonal and homeostatic systems that allow them to interact with and respond to their environment. The group of molecules identified as potential endocrine disruptors is highly varied and may be present in chemicals used as industrial solvents or surfactants that can be found in dispersants and surface washing agents. Because of the common properties of these compounds and the similarities of the receptors and enzymes involved in the synthesis, release, and degradation of hormones, no endocrine system is immune to endocrine disrupting compounds.<sup>7</sup>

The 1996 Food Quality Protection Act directed EPA to develop a screening program, using appropriate validated test systems and other scientifically relevant information, to determine whether certain substances may have hormonal effects in humans; the 1996 amendments to the Safe Drinking Water Act authorized EPA to screen substances that may be found in sources of drinking water for endocrine disruption potential. In response to these mandates, the Agency’s Endocrine Disruptor Screening Program is developing requirements for the screening and testing of pesticides, commercial chemicals, and

<sup>6</sup> For more information, refer to <http://www.epa.gov/oppt/existingchemicals/pubs/actionplans/np-npe.html>.

<sup>7</sup> For more background information on endocrine disrupting compounds and their human health and environmental effects, please see <http://www.epa.gov/endo/pubs/edspsoverview/index.htm>.

environmental contaminants for their potential to disrupt the endocrine system.<sup>8</sup> The science related to measuring and demonstrating endocrine disruption is relatively new and validated testing methods are still being developed. When complete, EPA will use these validated methods or assays to identify and characterize the endocrine activity of pesticides, commercial chemicals, and environmental contaminants, specifically in relation to estrogen, androgen, and thyroid hormones. EPA plans to use the assays in a two-tiered screening and testing process: Tier 1 will serve to identify chemicals that have the potential to interact with the endocrine system; and Tier 2 will determine the endocrine-related effects caused by each chemical and obtain information about effects at various doses. With this two-tiered approach, the Agency will gather information needed to identify endocrine-active substances and take appropriate action, as mandated by Congress. To date, EPA has developed and validated 11 assays that comprise the Tier 1 Screening Battery. EPA's validation effort continues with the current focus on Tier 2 tests and potential replacement assays for Tier 1.<sup>9</sup> When all tests are finalized and chemical information begins to be collected, EPA may further consider how to incorporate that information into the Subpart J NCP requirements, including additional testing and listing requirements. Because validated testing methods are still being developed, the agency is not proposing any test requirements with respect to endocrine disruption for products to be listed on the Schedule.

However, because NP and the NPE are extremely or highly toxic to aquatic organisms, the Agency is proposing a prohibition specific to Subpart J products containing NP and NPE as components. The Agency is not proposing to include a broad prohibition on Subpart J products that contain substances in which at least part of the toxic mode of action may include disruption of the organism's endocrine system, but is requesting comments on prohibiting similar substances that may be found in products/agents that could be listed on the Schedule, or to what the criteria should be in order to make that determination. As an alternative, the Agency could require that all product components be tested for a toxic mode of action that includes disruption of the

organism's endocrine system and is requesting comment on this approach as well.

The Agency believes the proposed prohibitions are appropriate in all cases, notwithstanding the proposed provisions for case-by-case use authorization of burning agents under § 300.910(c), or for authorization of use of any chemical or biological agent when it is necessary to prevent or substantially reduce an immediate threat to human life under § 300.910(d). There are chemical and biological agent alternatives to sinking agents and to agents containing NP or NPE, as well as mechanical methods for responding to oil discharges, including those situations that pose extreme threats or are time critical.

#### (f) Storage and Use of Agents

Section 300.915 currently requires that information be provided on recommended conditions of storage and use for each product at the time an application for listing a product under Subpart J is submitted to the Agency. This information is summarized in EPA's *NCP Product Schedule Technical Notebook*. Specifically, this information includes: Special handling and worker precautions for storage and field application; maximum and minimum storage temperatures (optimum ranges and temperatures that will cause phase separations, chemical changes, or other alterations to product efficacy); shelf life of the product; recommended application rates and procedures, concentrations and conditions (considering water temperatures, salinity, types and ages of the oil); and any other application restrictions.

The Agency proposes to add a new paragraph (f) to this section that complements the existing information requirements for the person or entity submitting a product for listing ("submitter"). The proposed requirements focus on the use of this information by the responder and the OSC. Specifically, the revised provisions require the OSC to only authorize for use those products that are stored under the conditions specified by the submitter of the product for listing, including the maximum, minimum and optimum temperatures, humidity and any other relevant conditions. Additionally, the Agency proposes to require that the OSC only authorize for use those products whose date of use does not exceed the expiration date listed on the container's label at the time of an incident, unless the

responsible party<sup>10</sup> provides the OSC documentation, developed in consultation with the submitter of the product for listing, prior to product use, affirming the product has maintained its integrity, including no changes in the composition, storage conditions, efficacy, and toxicity of any product. In such cases where the product expiration date has passed but the owner or operator of the facility/vessel storing the product still wants to be able to use the product inventory, EPA proposes that samples of the expired product lot representative of all storage conditions at any end user location be tested following the applicable testing protocols in Appendix C. The testing documentation is to include laboratory information (*i.e.*, contacts, accreditations) and all test data and calculations (*i.e.*, raw data and replicates, notes and observations, calculated means and standard deviations, stock solution preparations, source and preparations of test organisms, test conditions, chain of custody forms, and summary reports). Only if the owner or operator can demonstrate that the product has maintained its integrity is the OSC allowed to authorize the use of that product inventory. The owner or operator of the facility/vessel, or their representative, must re-label the tested product lots and maintain documentation of the test results until those lots are used or discarded, and must retest the expired product lot representative of the product at least once every 5 years to ensure efficacy and to allow an OSC to authorize the product for use. The intent of these proposed revisions is to ensure that products being authorized and used have maintained their efficacy, even though storage beyond the products' original shelf life may have occurred. The Agency believes it is the owner or operator's responsibility to ensure that any product it stockpiles for future use has maintained its efficacy and has not changed from its listed composition, including the possibility of degrading into more toxic byproducts, once the manufacturer's expiration date has passed. This retesting provision is supported by the proposed requirements that the submitter of the product for listing provide not only the recommended storage conditions, but the anticipated shelf life of a product at those conditions, and that the product label include both the manufacture and expiration dates, and conditions for storage. The flexibility in this proposed

<sup>8</sup> <http://www.epa.gov/endo/pubs/edspoverview/background.htm>.

<sup>9</sup> <http://www.epa.gov/endo/pubs/assayvalidation/index.htm>.

<sup>10</sup> Responsible party is defined in the NCP under 40 CFR 300.5.

revision allows for the use of available product inventory that may still be viable. To alleviate concerns that applications are submitted that establish an extended or indefinite shelf life for a product, the Agency is requesting comments on whether any additional data or information requirements should be included for product listing determinations specific to a product's shelf life, or whether alternative approaches, such as limiting the shelf life for product categories to a given timeframe, should be considered.

(g) Supplemental Testing, Monitoring, and Information

This proposal maintains RRT authority in that they may require supplementary toxicity and efficacy testing, or available data or information that addresses site, area, or ecosystem specific concerns relative to the use of a product for both planning and authorization of use. While parallel to the current provisions, the proposed revisions provide added flexibility. The current provisions allow RRTs to require supplementary toxicity and efficacy testing of products, in addition to those specified as technical requirements following the test methods described in Appendix C to part 300, due to existing site-specific or area-specific concerns when developing preauthorization plans. EPA proposes to remove the qualifier "When developing preauthorization plans" to provide greater flexibility for RRTs to require supplementary toxicity and efficacy testing, or available data or information that addresses site, area, or ecosystem specific concerns relative to the use of a product for situations even when preauthorization plans are not being developed. For example, RRTs may need additional testing or information for situations that fall under paragraph (b) of this section, including when developing an expedited decision making plan. The proposed revision also allows for RRTs to require supplementary product toxicity and efficacy testing, or available data or information for both planning and authorization of use situations, and based on that information may consider establishing limitations for the use of products in certain areas. The Agency is including "available data and information" to compliment the supplementary toxicity and efficacy testing provision recognizing that existing data or information that addresses site, area, or ecosystem specific concerns relative to the use of a product may be available. The Agency is also proposing to include "ecosystem" with area and site specific

concerns, as RRTs may want to gather additional information on the use of certain products when assessing the use of a product relative to the biological communities specific to their area. The proposal removes "in addition to the test methods specified in § 300.915 and described in appendix C to part 300." While RRTs may want to use the efficacy and toxicity testing protocols specified in Appendix C of the NCP for comparative purposes, the proposed revisions clearly establish the RRTs authority to require tests using parameters beyond those specified in Appendix C. Furthermore, there may be supplementary toxicity and efficacy testing information based on recognized standard testing methods already available that RRTs may want to consider when addressing site, area, or ecosystem specific concerns.

This proposal also provides the RRT authority to request that the OSC require a responsible party to conduct additional monitoring associated with the use of a product during a discharge incident. The proposed revision compliments the proposed monitoring requirements for dispersant use, but also including other chemical or biological agents, or other testing endpoints. The Agency believes the RRT must be afforded the ability to request that the OSC direct the responsible party to conduct additional monitoring under Subpart J for the use of a product in the environment. The RRT may request that the OSC consider additional monitoring during an oil discharge response to support operational decisions on dispersant use. For example, the RRT may want to monitor the exposure of marine mammals to oil constituents, including dispersed oil, or to monitor toxicity in the water column using biological assays. The Agency requests comments on these proposed revisions.

(h) Recovery of Agents From the Environment

The proposal identifies certain agent categories and substances intended to be removed from the environment following their use: Solidifiers, sorbents and surface washing agents. For those categories, the Agency expects the agents to be recovered from the environment to minimize any potential adverse impact. The proposal adds a new requirement that charges the responsible party, under OSC oversight, to recover these products from the environment. Recovery activities after the use of these agents would include containment of the agents in the water, collection of the agents mixed with oil or any residual agent, storage of the oil-agent waste prior to disposal, and

disposal of that oil-agent waste. The Agency also recognizes there may be situations where the safety of response personnel is threatened, or where additional harm to the environment could occur during recovery operations. Consequently, we are proposing these factors be considered when initiating recovery actions. While it is appropriate to have the OSC ensure these activities take place, it is ultimately the responsibility of the responsible party to conduct these activities and ensure that the agents are recovered from the environment. The Agency requests comments on this proposed requirement.

(i) Reporting of Agent Use

The proposal includes a new requirement for the OSC to provide to the RRT certain information after the use of a chemical or biological agent within 30 days of completion of agent operations. The information required in this report includes the information on any chemical or biological agent used, including product name, the quantity and concentration of the agent used during the response, the duration of use, the locations where the agent was used, and any data collected and analysis of efficacy or environmental effects. The proposal allows this information to be provided in the OSC report to the RRT or RRT as required under section 300.165 of the NCP, if such a report has been requested. While other existing notification requirements serve to activate an immediate response to an event, the proposed requirement gathers information that will be useful in specifically evaluating the use of chemical or biological agents in the response. It will also inform the review of preauthorization plans and provide a basis for any necessary changes to improve environmental protection. Given that response and removal actions can greatly vary depending on the discharge situation, the Agency requests comments on whether it would be appropriate to allow the timeframe for submitting the report to be agreed upon by the RRT, rather than establishing a set timeframe. The Agency is requesting comments on this proposed new requirement, including on the proposed timeframe.

3. Monitoring the Use of Dispersants

The goal of establishing a Schedule under the NCP is to protect the environment from possible damage related to spill mitigating products used in response to oil discharges. The proposal establishes a regulatory approach under Subpart J that includes test data and information requirements

for certain chemical and biological agents, procedures for authorizing the use of those agents, and monitoring requirements for certain discharge situations.

Each oil discharge represents a unique situation with distinct conditions, which may require various response methods. When dispersants are applied to an oil discharge, field monitoring can be used to inform operational decisions by gathering site-specific information on the overall effectiveness, including the transport and environmental effects of the dispersant and the dispersed oil. The revisions to product test data and information requirements are intended to provide OSCs, RRTs, and ACs with the best information available when selecting products for use on an oil discharge. While laboratory test protocols allow for comparison between different products under standardized laboratory conditions and may be useful during the monitoring and assessment of a discharge event and/or for selection of the agents used in the response, they do not necessarily reflect field conditions. Monitoring of agents in the field informs the OSC and other agencies on the overall effectiveness of dispersant use, including the environmental effects and transport of dispersed oil.

The Agency believes that comprehensive monitoring in certain discharge situations is necessary to determine the overall effectiveness of dispersants and should transcend from the initial dispersant application to include the transport and environmental effects of the dispersant and dispersed oil in the water column. Monitoring the overall effectiveness of dispersant use in the field provides those Agencies with responsibility for authorizing the use of dispersant product information for decision-making during subsurface or prolonged surface dispersant applications. Adverse effects on ecological receptors from exposures to dispersed oil depend on the length of time and concentration of the exposure, which in turn is dependent on the transport of dispersed oil. Because these exposures may vary depending on the discharge situation, the Agency believes comprehensive monitoring is important for certain discharge situations. This monitoring data will enhance the information needed for an effective response without delaying the use of agents under these conditions.

Equipment is being contemplated to inject dispersants subsurface, directly into the oil near the source of the

discharge.<sup>11</sup> This type of application is intended to minimize dispersant dilution in the water before the dispersant has had an opportunity to interact with the oil.<sup>12</sup> This application approach that is closer to the source is expected to reduce potential adverse environmental consequences from the use of excessive quantities of dispersants.<sup>13</sup> However, applying dispersant to an oil discharge does not result in the physical recovery of oil from the environment. Instead, dispersing oil increases the potential exposure of aquatic organisms to the dispersant-oil mixture, at least transiently, and subsurface application has the potential to more immediately and effectively increase these exposures near the discharge. The Agency believes this new subsurface application approach requires new environmental monitoring capabilities to support operational decision-making. These new monitoring capabilities must be able to meet the operational conditions (e.g., water depths, temperatures) and be supported by knowledgeable personnel familiar with them.

The proposed rule adds § 300.913 establishing requirements for the responsible party to monitor any subsurface use of dispersant in response to an oil discharge, surface use of dispersants in response to oil discharges of more than 100,000 U.S. gallons occurring within 24 hours, and surface use of dispersants for more than 96 hours in response to an oil discharge, as directed by the OSC. The purpose of monitoring subsurface application is to characterize the dispersed oil, follow the plume integrity and transport with the underwater current, and identify and assess the potential adverse effects from the dispersed oil. The proposal requires the responsible party to implement monitoring for any subsurface dispersant use in response to an oil discharge upon initiation and for the duration of subsurface dispersant use. The Agency believes monitoring subsurface use of dispersants is critical to inform response actions to minimize potential environmental effects.

While surface application of dispersants is not a new approach and understood for small, short duration discharges, the Agency believes it is appropriate to require comprehensive monitoring for situations where dispersants are used for an extended period of time or in cases of major oil

discharges. The Agency chose 100,000 U.S. gallons as a threshold criterion for a major oil discharge because the NCP classifies a discharge of more than this quantity to coastal waters as major. In addition, EPA is proposing to this quantity monitoring of dispersant use in response to major oil discharges occurring within 24 hours since a larger quantity of dispersant may be required in a short time frame for an incident of this scale. The Agency also believes comprehensive monitoring should be required when surface dispersant is used for more than 96 hours because of potentially longer exposures of biological aquatic resources to dispersant and dispersed oil. Further, many acute toxicity studies use 96-hour exposure durations, including the acute toxicity tests using *Menidia beryllina* described in this proposal. While other toxicity tests have shorter exposure durations (e.g., 24, 48, or 72 hours), the Agency believes 96 hours is a reasonable threshold given its commonality of use in the toxicology field. Therefore, the proposal requires a responsible party to implement monitoring for surface dispersant use in response to an oil discharge under these discharge conditions and for the duration of dispersant use. The proposal also requires the submission of a Quality Assurance Project Plan for approval to the OSC covering the collection of all environmental data to ensure and maximize its quality, objectivity, utility, and integrity. The Agency welcomes comments on the proposed monitoring requirements, including the thresholds. Specifically, the Agency requests comments on whether it should also consider a threshold for surface use of dispersants that is based on the quantity of dispersant used within a given timeframe. Alternative thresholds must include a rationale in order for the EPA to consider them for final action.

#### (a) Dispersant Application

The proposal requires the responsible party to document the characteristics of the source oil; best estimate of the oil discharge flow rate, periodically reevaluated as conditions dictate, including a description of the method, associated uncertainties, and materials; dispersant(s) product used, rationale for dispersant choice(s), including the results of any efficacy and toxicity tests, recommended dispersant-to-oil ratio; and the application method and procedures, including a description of the equipment to be used, hourly application rates, capacities, and total amount of dispersant needed. For subsurface discharges, the proposal also

<sup>11</sup> [http://marinewellcontainment.com/expanded\\_system.php](http://marinewellcontainment.com/expanded_system.php).

<sup>12</sup> NRC. (2005). Oil Spill Dispersants: Efficacy and Effects, The National Academies Press, pp. 138, [http://www.nap.edu/catalog.php?record\\_id=11283](http://www.nap.edu/catalog.php?record_id=11283).

<sup>13</sup> Ibid.



requires the responsible party to document the best estimate of the discharge flow rate of any associated volatile petroleum hydrocarbons, periodically reevaluated as conditions dictate, including as description of the method, associated uncertainties, and materials. This would provide the OSC with the necessary information for operational decision-making and coordination of the dispersant application monitoring.

Because of the many factors that influence the selection of a dispersant product (e.g., its availability, the type of oil it will be used on, the prevalent weather conditions, and the particular discharge situations) the Agency proposes that the responsible party document its product choice and selection rationale. Documenting the characteristics of the source oil provides specific chemical data to identify the oil associated with discharge and to inform decisions on dispersant use. Documenting application methods and procedures ensures that dispersant use is consistent with the dispersant manufacturer's recommended concentrations, and conditions provided as part of the data requirements for listing the product on the Schedule. Documenting results of any additional efficacy and toxicity testing, or available data or information specific to the area or site conditions will assist the OSC in establishing the appropriateness of the dispersant choice. Documentation and clear understanding of the estimated daily oil discharge flow rate and the recommended dispersant-to-oil ratio (DOR) allows a dispersant rate to be established that would aid the OSC in determining the adequate dispersant usage. The OSC can compare the recommended DOR to the manufacturer's recommended application concentration to ensure it is within the recommended range or to provide an opportunity for the responsible party to explain any deviations that may arise due to operational considerations.

The biodegradation of petroleum constituents may result in a reduction in the dissolved oxygen concentration in the water column that could lead to hypoxia, which could be detrimental to marine organisms. The best estimate of the oil discharge flow rate, periodically reevaluated as conditions dictate, is an important consideration for monitoring dispersant use since the estimated amount of oil discharged may be used to provide insight into the potential oxygen demand exerted on the water column and to characterize the potential oil distribution. The proposal would also require the responsible party to

document the best estimate of the discharge flow rate of any associated volatile petroleum hydrocarbons for subsurface applications. Volatile petroleum hydrocarbons (e.g., methane, and low molecular weight alkanes such as ethane, propane, and butane) associated with the discharge could contribute to a reduction in dissolved oxygen as a result of microbial degradation. The Agency is not proposing this requirement for surface applications given these compounds are expected to readily evaporate, making it unlikely they would be incorporated into the water column in quantities that would result in hypoxia.

The proposal requires the documentation of the dispersant application method and procedures to include a description of the equipment to be used, including hourly application rates, capacities, and total amount of dispersant needed. Dispersant and equipment should be available to maintain the hourly dispersant application rate without significant deviation from the set rate. The Agency believes this information will assist in ensuring a consistent dispersant application rate. Dispersant application rates outside of established parameters may result in their over or under use, altering their effectiveness. Significant fluctuations in dispersant application rates could also indicate equipment malfunctions, requiring a reassessment of the response technique. EPA welcomes comment on the proposed requirements for dispersant application. Alternatives must include a rationale in order for the Agency to consider them for final action.

#### (b) Water Column Sampling

The proposal requires the responsible party to collect representative background samples from the water column at the closest safe distance determined by the OSC from the discharge, and in any direction of likely transport. The sampling should consider surface and subsurface currents and oil properties. Establishing background information prior to oil contamination from the discharge source provides reference data to compare against the results from water samples taken during the response. In establishing the background information, the responsible party should consider other potential sources of petroleum hydrocarbons (e.g., natural seeps) in the water column. The background samples would be collected in areas not affected by the discharge of oil throughout the water column upon initiation of dispersant use, at the closest safe distance as determined by the OSC from the oil discharge.

Sampling would be conducted in any direction of likely transport considering surface and subsurface currents and the oil properties in those areas. The responsible party should consider using trajectory models that incorporate relevant factors such as oil type to determine the likely direction of the dispersed oil to inform where the water samples should be collected.

The Agency is requiring that sample collection follow established standard operating and quality assurance procedures that are reliable and defensible. An accurate assessment of environmental data depends on the reliability, timeliness, and integrity of the data collected. Standard operating procedures should describe the appropriateness of the sampling method; the equipment needed for sample collection; a description of potential interferences, problems that may be encountered and corrective actions that would be taken; the sample collection procedure, including the preparation steps, representative sampling considerations, and sampling steps for each sampling device used; sample preservation, containers, handling, and storage; decontamination of sampling equipment; and record keeping and quality control procedures. These elements are generally described in various guidance documents on standard operating and quality assurance procedures for environmental sampling.<sup>14 15</sup>

In addition to the background water sample collection, the proposal requires the responsible party to collect water column samples daily in the dispersed oil plume at such depths and locations where dispersed oil is likely to be present. EPA believes the dispersed oil plume captures the presence of oil from either the subsurface or the surface application of dispersant, including where oil could be rising to the surface from a subsurface discharge or the dispersed oil plume at the water surface as a result of surface dispersant application. The responsible party also would need to analyze for:

- *In-situ* oil droplet size distribution analysis, including the mass or volume mean diameters between droplet sizes ranging from 2.5 to 2000  $\mu\text{m}$ , with the majority of data collected between the 2.5 and 100  $\mu\text{m}$  sizes. Droplet size distribution, which is an important component to understanding the chemical and hydrodynamic effectiveness of dispersants, can be measured with devices such as the Laser

<sup>14</sup> [http://www.epa.gov/quality/qa\\_docs.html](http://www.epa.gov/quality/qa_docs.html).

<sup>15</sup> <http://www2.epa.gov/region8/environmental-sampling>.

In-Situ Scattering and Transmissometry (LISST) instrument or other similar instruments. Small oil droplets (*e.g.*, diameters in the tens of  $\mu\text{m}$  range) have such low rise velocities that they tend to remain suspended in the water column (neutrally buoyant), where they can become widely dispersed by advective forces, turbulent motion, and to a lesser extent diffusion. These small droplets are more readily amenable to biodegradation due to their higher surface area to volume ratio. In contrast, large oil droplets (*e.g.*, diameters  $>100\ \mu\text{m}$ ) will tend to re-coalesce and rise faster to the surface.<sup>16</sup> Effective application of dispersants is expected to generate a larger number of small droplets, reducing the average droplet size.<sup>17</sup> This will change the trajectory of rising oil that is subjected to stratified horizontal subsurface currents. Near the water surface, hydrodynamic and other environmental factors also influence the oil droplet size distribution. Mixing energy, especially from breaking waves, dictates the breakup of large oil droplets into smaller droplets and the depth of submergence of the droplets. Plunging breaking waves under experimental conditions produced the smallest oil droplets.<sup>13, 14</sup> Additionally, studies indicate that the type of chemical dispersant used is an important factor in controlling the dispersed oil droplet size distributions. Further, wave tank studies have shown that the chemically dispersed oil may result in a droplet size distribution that has not been observed without the use of dispersants (*i.e.*, physically dispersed oil), both in terms of a smaller volumetric mean diameter and a bimodal distribution for droplet size diameters below  $100\ \mu\text{m}$ .<sup>18</sup> EPA recognizes that the aforementioned studies were conducted under conditions characteristic of surface waters. However, the effect of the high velocities of oil that may occur from a discharge from a subsurface oil well on the oil droplet size distribution requires further scientific investigation to understand the contribution of physically dispersed oil relative to chemically dispersed oil. In either event, it is clear droplet size distribution

<sup>16</sup> Li, M. and Garrett, C., (1998) The Relationship Between Oil Droplet Size and Upper Ocean Turbulence, *Mar. Poll. Bull.*, Vol. 36, pp. 961–970.

<sup>17</sup> Li, Z., Lee, K., King, T., Boufadel, M.C., Venosa, A.D., (2008) Assessment of Chemical Dispersant Effectiveness in a Wave Tank under Regular Non-Breaking and Breaking Wave Conditions, *Mar Pollut Bull*, Vol. 56, pp. 903–912.

<sup>18</sup> Li, Z., Lee, K., King, T., Boufadel, M.C., Venosa, A.D., (2009) Evaluating Chemical Dispersant Efficacy in an Experimental Wave Tank: 2—Significant Factors Determining In Situ Oil Droplet Size Distribution, *Env Eng Scien*, Vol. 26, pp 1407–1418.

analysis provides important data useful in optimizing operational guidelines and decision making, modeling transport and fate, and potentially evaluating biological effects of chemically dispersed oil. The proposal also includes larger oil droplet sizes of up to  $2000\ \mu\text{m}$ , intended to provide information that can inform the oil distribution using trajectory modeling analysis.

- *In-situ* fluorometry. When exposed to ultraviolet (UV) light, certain compounds absorb energy that can elevate electrons into an excited higher energy level. The wavelength that excites electrons into a higher energy state is typically referred to as the excitation wavelength. Electrons unstable at the higher energy state return to a lower energy state, emitting energy at longer emission wavelengths resulting in fluorescence.<sup>19</sup> Many organic compounds fluoresce at specific excitation and emission wavelengths that allow for identifying many of the components of dissolved organic matter and other compounds (*e.g.*, crude oil) in seawater.<sup>20</sup> For crude petroleum oils, the aromatic fraction is responsible for the fluorescence property of petroleum, and these compounds are used to determine the specific excitation and emission wavelengths for monitoring. When subject to excitation at certain wavelengths in the near ultraviolet spectrum, polycyclic aromatic hydrocarbons (PAH) fluoresce over a range of higher wavelengths, depending on the number of aromatic rings in the structure.<sup>21</sup> Fluorometers can be targeted to the type of oil discharged, and the excitation and emission wavelengths chosen should match the aromatic properties of the oil discharged. Although this measure does not include all oil constituents, fluorescence is a valuable screening tool deployed during a response,<sup>22</sup> providing a rapid indication of potential dispersed

<sup>19</sup> Henry, C.B., Roberts, P.O., Overton, E.B., A Primer on In Situ Fluorometry to Monitor Dispersed Oil, *IOSC 1999:246*.

<sup>20</sup> Coble, P.G., (2007) Marine Optical Biogeochemistry: The Chemistry of Ocean Color, *Chem. Rev.*, Vol. 107, pp. 402–418.

<sup>21</sup> Bugden, J.B.C., Yeung, C.W., Kepkay, P.E., Lee, K. (2008) Application of Ultraviolet Fluorometry and Excitation-Emission Matrix Spectroscopy (EEMS) to Fingerprint Oil and Chemically Dispersed Oil in Seawater, *Mar Pollut Bull*, Vol. 56, pp. 677–685.

<sup>22</sup> Conmy, R.N., Coble, P.G., Farr, J., Wood, A.M., Lee, K., Pegau, W.S., Walsh, I.D., Koch, C.R., Abercrombie, M.I., Miles, M.S., Lewis, M.R., Ryan, S.A., Robinson, B.J., King, T.L., Kelble, C.R., and Lacoste, J. (2014) Submersible Optical Sensors Exposed to Chemically Dispersed Crude Oil: Wave Tank Simulations for Improved Oil Spill Monitoring, *Environ Sci Technol*, Vol. 48, pp. 1803–1810.

oil in the water column, as well as an indicator of dispersion effectiveness as discussed in the following section. The Agency request comments on alternative spectroscopy techniques such as absorption measurements that may be used to rapidly estimate oil concentrations in the water during a discharge event.

- Fluorescence signatures. In addition to *in-situ* fluorometry, the proposal requires the responsible party to conduct a fluorescence intensity analyses on water samples collected to determine fluorescence signatures of the dispersed oil. EPA proposes to use fluorescence signatures as a relatively simple and rapid means to assess dispersion effectiveness. This includes but is not limited to, identifying the peak wavelength position, magnitude and ratios of fluorescence intensity of the dispersed oil. For example, measuring the reduction in an intensity ratio after applying dispersant to the oil in seawater may provide an estimate of the effect of chemical dispersion.<sup>23, 24</sup> Ratios of fluorescence intensity are derived from the three dimensional excitation/emission matrix spectra (EEMS) acquired by combining multiple emission spectra obtained from oil exposed to individual excitation wavelengths typically using a scanning spectrofluorometer. If the optimal excitation and emission bands are known, then these ratios may be derived from measuring the fluorescence intensity at two different fixed emission wavelengths at a single fixed excitation wavelength. The ratios are calculated by dividing the fluorescence intensity of the shorter emission band by the fluorescence intensity of the longer band. Studies have shown that adding dispersants to crude petroleum oil increases the fluorescence intensity of the longer emission band, driving down the value of the ratio. Although individual petroleum oils may have common spectral characteristics, the ideal three-dimensional EEMS for the discharged oil should be obtained from scanning the source oil. Therefore, the monitoring should include procedures to obtain the EEMS using the source oil to determine the optimal excitation-emission wavelengths. The Agency

<sup>23</sup> Bugden, J.B.C., Yeung, C.W., Kepkay, P.E., Lee, K. (2008) Application of Ultraviolet Fluorometry and Excitation-Emission Matrix Spectroscopy (EEMS) to Fingerprint Oil and Chemically Dispersed Oil in Seawater, *Mar Pollut Bull*, Vol. 56, pp. 677–685.

<sup>24</sup> Kepkay, P.E., Yeung, C.W., Bugden, J.C.B., Li, Z., and Lee, K. (2008) Ultraviolet Fluorescence Spectroscopy (UVFS): A New Means of Determining the Effect of Chemical Dispersants on Oil Spills, *IOSC*, pp. 639–644. <http://ioscproceedings.org/doi/abs/10.7901/2169-3358-2008-1-639>.

request comments on alternative techniques or measurements that may be used to rapidly assess dispersion effectiveness during a discharge event.

- Dissolved oxygen (DO). Dissolved oxygen is an important variable to monitor in the application of dispersants, particularly in subsurface waters that may inform operational decisions. For surface dispersant application, dissolved oxygen is expected to be higher in the mixed layer in the surface water. Dissolved and/or dispersed oil represents a readily available carbon source for microbial oxidation, and this metabolic activity can lead to a decrease in the DO content in the water column. The oil degrading community in the water is an important DO sink for the mass transfer of hydrocarbons in the subsurface, and advantage can be taken of this fact during a response. The decline in DO due to hydrocarbon biodegradation could approach or exceed levels of concern (*i.e.*, hypoxia) and thus be detrimental to surrounding organisms (*e.g.*, invertebrates and vertebrates). Dissolved oxygen, which relies on the diffusion of molecular oxygen through a membrane in the sensor, is commonly measured during oceanographic survey cruises using an *in-situ* DO instrument that provides real-time results. However, a DO sensor instrument that passes through a dissolved and/or dispersed oil layer is subject to contamination by the oil and could lead to questionable measurements if such fouling of the electrodes occurs, particularly at depths where the dissolved and/or dispersed plume depth are expected to coincide with depressions in dissolved oxygen. Therefore, the Agency is concerned that relying solely on measurements from *in-situ* oxygen instruments may lead to an erroneous interpretation of oxygen data. Thus, the Agency believes that *ex-situ* confirmatory DO measurements should also be conducted using Winkler titrations to confirm *in-situ* dissolved oxygen measurements.

- Total petroleum hydrocarbons, individual resolvable constituents, including volatile organic compounds, aliphatic hydrocarbons, monocyclic, polycyclic, and other aromatic hydrocarbons, including alkylated homologs, and hopane and sterane biomarker compounds. The Agency is proposing that the responsible party analyze each water sample collected for total petroleum hydrocarbons (TPHs), individual resolvable constituents, including volatile petroleum hydrocarbons, and branched and normal aliphatic hydrocarbons. The analysis would also include monocyclic,

polycyclic and other aromatic hydrocarbons (*e.g.*, heterocyclic aromatic hydrocarbons), including their alkylated homologs, and hopanes and steranes biomarker compounds. TPHs are best measured using gas chromatograph (GC) equipped with a flame ionization detector (FID). TPH analysis measures total alkanes and aromatics, can be obtained relatively quickly, and provides a good measurement for initial screening. Given that PAHs and biomarkers are typically measured using GC/MS, the proposal includes GC/MS analysis to provide the alkanes, PAHs, and biomarker (*e.g.*, hopane) specificity important in identifying certain oil constituents that may be present in the oil discharge. Identifying the concentrations of PAHs and biomarkers assists responders in making informed operational decisions regarding the dispersant application because these compounds can be compared against the composition of the source oil, which serves as a reference to determine the degree of weathering for oil.

- Carbon Dioxide (subsurface only). The aerobic biodegradation of oil constituents not only consumes dissolved oxygen, but would also produce carbon dioxide. Increases in the concentration of carbon dioxide that coincide with decreases in the concentration of dissolved oxygen would provide credible evidence that biodegradation of oil is occurring. Thus, the Agency believes that measuring the *in-situ* carbon dioxide for subsurface dispersant applications would be a good indicator of microbial oxidation and inform the OSC on the potential fate.

- Methane if present (subsurface only). The microbial degradation of methane may contribute to a reduction in dissolved oxygen. Thus, the Agency believes that, when present, subsurface *in-situ* methane measurements are an important factor to consider in evaluating dissolved oxygen levels.

- Heavy metals analysis, including nickel and vanadium. Crude petroleum oil may contain certain heavy metals, including nickel and vanadium.<sup>25 26 27</sup> Dispersing oil may increase the bioavailability of certain associated heavy metals to marine organisms.

<sup>25</sup> Barwise, A. J. G. and Whitehead, E. V. (1980). Separation and Structure of Petroporphyrins. In: *Advances in Organic Geochemistry 1979*. (A. G. Douglas and J. R. Maxwell, eds.), Pergamon, New York, pp. 181–92.

<sup>26</sup> Barwise, A. J. G. (1990). Role of Nickel and Vanadium in Petroleum Classification. *Energy & Fuels*, 4, 647–52.

<sup>27</sup> All, M.F., Bukharl, A., and Saleem, M., (1983) Trace Metals in Crude Oils from Saudi Arabia, Ind. Eng. Chem. Prod. Res. Dev. 1983, Vol 22, pp. 691–694.

Therefore, the Agency is proposing to include heavy metal analysis as part of the sampling analyses to be conducted.

- Turbidity. Turbidity is a general measure of water clarity. Turbidity is determined by measuring how much material suspended in water decreases the passage of light through the water.<sup>28</sup> Suspended materials may include soil particles (clay, silt, and sand), algae, plankton, microbes, and other substances. This measure provides a quick assessment of suspended materials in receiving waters from other water bodies and is useful in determining the presence of materials that could interfere with oil particle size determinations. Alternatively, the Agency is requesting comments on whether there are other physical measurements in the water that would provide similar timely information, or that can serve as validation for turbidity values collected *in situ*.

- Water temperature. Water temperature typically is measured using an electronic thermometer on the conductivity-temperature-depth (CTD) instrument. When combined with salinity and pressure, temperature measurements are used to calculate water density, which may change along the vertical profile. Along with the horizontal subsurface currents and the rise velocity of the dispersed oil, the ambient density gradient is an important factor in determining the behavior of dispersed oil in the water column. Water temperature is also an important variable that may influence the effectiveness of dispersant applications. For example, cold temperatures may, among other environmental factors, impact the effectiveness of dispersants as it affects certain oil properties (*e.g.*, viscosity). Colder temperatures also may affect the degree of oil weathering (*e.g.*, evaporation), and the amount of dispersant/oil mixing energy (wave action) needed to effectively disperse oil relative to warmer temperatures.

- pH. The pH is a simple standard measurement used to analyze water quality that can affect chemical or biological processes in water. pH also determines the solubility and biological availability of chemical constituents such as heavy metals.

- Conductivity. Conductivity is measured by passing a current through the water in a CTD instrument, which is then compared against known salinities to obtain the water salinity. When combined with temperature, and pressure, salinity measurements are

<sup>28</sup> <http://water.epa.gov/type/rsll/monitoring/vms55.cfm>.

used to calculate water density, which may change significantly along the vertical profile. Along with the horizontal subsurface currents and the rise velocity of the dispersed oil, the ambient density gradient is an important factor in determining the behavior of dispersed oil in the water column.

The Agency requests comment on the proposed approach to water column sampling. Alternatives must include a rationale in order for the Agency to consider them for final action.

#### (c) Oil Distribution Analyses

The proposal requires that the responsible party, in consultation with the OSC and using best available technologies, characterize the dispersant effectiveness and oil distribution, considering the condition of the oil, dispersant, and dispersed oil components from the discharge location. EPA believes this is necessary to inform sampling locations by assessing the horizontal and vertical boundaries of the dispersed oil plume and the direction of likely transport. The majority of the sampling can then be focused on known or suspected locations and depths of the dispersed oil. Based on the initial water sampling results to characterize the boundaries of the dispersed oil, the sampling design can be tailored to optimize sampling. The Agency proposes that the responsible party characterize the dispersant effectiveness to determine the changes in the condition of the oil due to weathering, which can include changes in oil viscosity due to water uptake (*e.g.*, mousse). Weathered oil that mixes with water may result in significant emulsification (*e.g.*, water-in-oil emulsions), which in turn may decrease the dispersant's chemical effectiveness and diminish biodegradability. Therefore, the characterization of dispersant effectiveness should provide a reasonable estimate to the extent (*e.g.*, distance from the discharge source) in which the dispersant can be applied to the oil and still be effective. The Agency believes this will result in better dispersant applications and minimize excessive dispersant use. The Agency requests comment on this approach. Alternatives posed must include a rationale for the Agency to consider them for final action.

#### (d) Ecological Characterization

The Agency proposes that the responsible party characterize the ecological receptors (*e.g.* aquatic species, wildlife, and/or other biological resources), their habitats, and exposure

pathways that may be present in the discharge area, in consultation with the OSC. Exposure pathways, such as ingestion, refer to the way in which ecological receptors may come into contact with discharged oil and result in exposure that would be detrimental to ecological receptors. The Agency believes that the ecological characterization should include those species that may be in sensitive life stages, transient or migratory species, breeding or breeding-related activities (*e.g.*, embryo and larvae development), and threatened and/or endangered species that may be exposed to oil, dispersed oil, and dispersant, which in turn will assist the OSC in managing response actions, including those actions that were determined not to be needed. In developing the characterization of ecological receptors, the responsible party may refer to relevant sources of information such as applicable expedited decision-making plans, environmental assessments or statements, Federal and state environmental databases (*e.g.*, ACP—Fish and Wildlife and Sensitive Environments Annex; NOAA—Environmental Sensitivity Indices; NOAA—NMFS Southeast Area Monitoring and Assessment Program; EPA—National Coastal Assessment<sup>29</sup>) or through studies conducted by universities or other research-oriented institutions.

The proposal requires the responsible party to consult with the OSC to determine an acute toxicity level of concern for the dispersed oil using available dose/response information relevant to potentially exposed species. The envisioned approach would be to monitor acute toxicity in the water column concurrently with dispersed oil sampling for fluorometry, particle size, and water quality (*e.g.*, dissolved oxygen). The TPH concentrations in water samples taken throughout the water column could then be compared to TPH-based ecotoxicity benchmarks (EBs). Water samples collected for comparison of aqueous TPH concentrations to EBs would be analyzed and reported within the timeframe necessary to make operational decisions (*e.g.*, within 24 hours of collection). Sampling could also be performed in areas without dispersant application to distinguish toxicity associated with physically dispersed oil from that of chemically dispersed oil.

While EBs may have already been established during the development of contingency plans, they can also be

derived at the time of an incident. One approach is to use a species sensitivity distribution (SSD),<sup>30</sup> which allows for species relevant to the location of the discharge to be considered. An SSD is a probability distribution of the sensitivity of a group of species to a toxicant.<sup>31</sup> SSDs could be developed for representative oils (*e.g.*, crude oils) using existing acute toxicity values for mortality or immobility (*e.g.*, 48 and 96 hr LC<sub>50</sub>) where sufficient species diversity are available (*e.g.*, toxicity data for 10 or more species). For example, the EBs could be computed from the fifth percentile of the SSD as the hazard concentration 5 percent (HC5), as they are considered protective of 95% of species, have been used by EPA for developing ambient water quality criteria, and are generally accepted by the international community.<sup>32</sup> Chronic toxicity benchmarks may be derived applying safety factors to the acute toxicity EBs. The Agency requests comments on the proposed approach to ecotoxicity assessment, and whether it should consider making SSDs a requirement for deriving EBs. If alternative approaches are suggested, the commenter must include an appropriate rationale in order for the Agency to consider them for final action.

#### (e) Immediate Reporting

The proposal requires the responsible party to immediately report to the OSC any deviation of more than 10 percent of the mean hourly subsurface dispersant use rate for the total dispersant volume authorized for that 24 hour use, and the reason for the deviation (*e.g.*, equipment malfunction). The Agency believes that inconsistent dispersant application rates confound data sampling interpretation due to variations in the DOR. The proposal, however, provides a margin for variation within 10 percent of the mean hourly subsurface dispersant application rate to account for equipment performance. The Agency believes this margin to adequately account for variations in dispersant

<sup>30</sup> Barron, M.G., Hemmer, M.J., and Jackson, C.R., (2013) Development of Aquatic Toxicity Benchmarks for Oil Products Using Species Sensitivity Distributions, *Integr Environ Assess Manag*, Vol. 9, pp. 610–615.

<sup>31</sup> Aldenberg, T. and Jaworska, J.S., (2002) Uncertainty of the Hazardous Concentration and Fraction Affected for Normal Species Sensitivity Distributions, *Ecotox and Environ Safety*, Vol. 46, pp. 1–18.

<sup>32</sup> Barron, M.G., and Wharton, S., (2005) Survey of Methodologies for Developing Media Screening Values for Ecological Risk Assessment, *Integrated Environmental Assessment and Management*, Vol. 1, pp. 320–332.

<sup>29</sup> <http://www.epa.gov/emap/nca/>.

injection equipment without being overly restrictive. EPA requests comments on whether it should consider a similar requirement for dispersant surface application.

The Agency also is proposing to require the immediate reporting of ecological receptors, including any threatened or endangered species that may be exposed to dispersed oil based on trajectory modeling and the estimated acute toxicity level of concern. Results from daily sampling (e.g., droplet size distribution, TPH) would provide input data to refine predictions of the likely dispersed oil direction using trajectory modeling. Further, results may also inform decisions to alter dispersant application in order to minimize effects on biological resources.

#### (f) Daily Reporting

The proposal requires daily reporting of sampling and data analyses collected within the timeframe necessary to make operational decisions (e.g., within 24 hours of collection), including documented observations, photographs, video, and any other information related to dispersant use, unless an alternate timeframe is authorized by the OSC. Daily reporting would also include the total amount of dispersant used for the previous reporting period. Additionally, the proposal would require a schedule for any data analyses that require time beyond 24 hours due to analytical methods; this schedule is not to exceed 120 hours (i.e., 5 days) unless authorized by the OSC. Timely sample analyses afford the OSC and other responders with multiple relevant data that can be analyzed together to inform situational awareness of dispersant operations and adjust dispersant application if necessary. The Agency believes that a 120-hour window for analyses requiring additional time provides an adequate opportunity to conduct all requested analyses in a timely manner without being overly restrictive. Finally, the proposal requires the responsible party to report the estimated daily transport of dispersed and non-dispersed oil, and associated volatile petroleum hydrocarbons if applicable, and dispersants, using the best available trajectory modeling. This information is intended to assist response planners to coordinate response activities and to schedule monitoring activities based on the expected transport of oil and dispersed oil.

#### 4. Data and Information Requirements for Product Schedule Listing

The Agency is proposing to revise the data and information requirements for listing products on the Schedule in § 300.915 of Subpart J. The proposed amendments revise the efficacy and toxicity testing protocols and listing criteria for all chemical and biological agents on the Schedule. Additionally, the Agency proposes revisions to the requirements for general product information, Confidential Business Information (CBI) claims, submission package contents, EPA review and listing procedures, requests for decision review, changes to products, transitioning products from the current Schedule to the new Schedule, mandatory product disclaimer, and removal of products from the Schedule.

##### (a) General Product Information

The Agency is proposing to consolidate the general submission requirements applicable to all types of agents that may be listed on the Schedule in paragraph (a) of § 300.915. Subsequent regulatory paragraphs in this section are proposed to include requirements specific to each product category. Additionally, the Agency is proposing to require some additional information, as applicable, regarding products that are submitted for Schedule listing consideration. The proposed general information requirements for all products are proposed to be revised as follow:

- **Submitter:** Name, physical address, email, telephone number, identity of submitter (i.e., manufacturer, vendor, importer, distributor, designated agent for the manufacturer), and documentation of such identity.
- **General Product:** All name(s), brand(s), and/or trademark(s) under which the product is to be sold; Safety Data Sheet (SDS); sample product labels for all name(s), brand(s), and/or trademark(s); chemical or biological agent categories under which the product is submitted for listing, including information on the specific process(es) through which the product affects the oil, and the specific environment (waters and/or adjoining shorelines) on which it is intended to be used.
- **Supplier:** Names, physical addresses, emails and telephone numbers of the primary distributors, vendors, importers, and/or designated agent acting on behalf of the manufacturer.
- **Product Storage:** Maximum, minimum and optimum temperature, humidity and other relevant conditions

for product storage; impact on product performance if the product is not stored within recommended limits; and anticipated shelf life at the recommended storage conditions.

- **Product Use:** Recommended procedures, including product concentrations, application ratios, types of application equipment, conditions for use, and any application restrictions. The procedures must address variables such as weather, water salinity, water temperature, types and weathering states of oils or other pollutants, and product and oil containment, collection, recovery and disposal, and include supporting documentation and standard methods used to determine them.

- **Environmental Fate:** Known measured data and supporting documentation on the persistence, bioconcentration factor, bioaccumulation factor, and biodegradability of the product and all of its components.

- **Physical/Chemical Properties and Formulation:** Physical state and appearance; vapor pressure; flash point; pour point; viscosity; specific gravity; particle size for solid components; pH; citation of standard methods used to determine the physical/chemical properties; identity of all components in the product, including each specific component name; corresponding Chemical Abstract Service (CAS) Registry Number; the maximum, minimum, and average weight percent of each product component; and the intended function of each component (e.g., solvent, surfactant).

- **For products that contain microorganisms, enzymes and/or nutrients:** All microorganisms and weight percent by current genus and species, including any reclassifications; all enzymes and their International Union of Biochemistry (I.U.B.) number(s); Enzyme Classification (EC) code numbers; the source of each enzyme, units, and specific oil-degrading activity; name(s) and average weight percent of the nutrients contained in the product; citation or description of the methodology used to determine product components; certification, including data, methodology, and supporting documentation, indicating that the product does not contain levels that exceed the National Ambient Water Quality Criteria lowest density value for the following: Bacterial, fungal, or viral pathogens or opportunistic pathogens, including, but not limited to, enteric bacteria such as Salmonella, fecal coliforms, Shigella, Coagulase positive

Staphylococci, and Beta Hemolytic Streptococci or enterococci.

- National Water Quality Standard Contaminants: Certification, including data, methodology, and supporting documentation, indicating that the product does not contain levels that exceed the National Water Quality Standards lowest aquatic life acute value for the following contaminants:

Metals reasonably expected to be in the product including arsenic, cadmium, chromium, copper, lead, mercury, nickel, vanadium, zinc; cyanide; chlorinated hydrocarbons; pesticides; polychlorinated biphenyls (PCBs); and polynuclear aromatic hydrocarbons (PAHs).

- Certification, including data, methodology, and supporting documentation, indicating that the product does not contain prohibited agents (sinking agents, nonylphenol, or nonylphenol ethoxylates).

- Testing Laboratory Information: Name, address, contact name, email, phone number; national or international accreditations.

- Laboratory Testing Data and Reports: All applicable information, data and analysis specified in the testing protocols, including raw test data and replicates, notes and observations, calculated mean values and standard deviations, summary of stock solution preparation, source and preparation of test organisms, test conditions, and chain of custody forms.

- Production capacity: Estimated annual production volume; average and maximum volume per day; time needed to reach that maximum production rate (days).

- Performance Capabilities/Benefits: Recognition received from, if applicable; national and/or international product testing or use data, recognitions (e.g., EPA's Design for the Environment), and/or certifications, informing the performance capabilities or environmental benefits of the product.

The proposed revisions group together and simplify the general submission requirements applicable to all product types. EPA believes reorganizing the general requirements in a central location will clarify which requirements are applicable to all submissions, and which are specific to each product type by including them in separate sections. While most of the information listed above is currently required to be submitted under Subpart J, the Agency is proposing revisions to several of the existing general requirements and several new data and information requirements. The Agency believes the proposed revisions and added new requirements will better

characterize the nature of the product and will assist EPA in product listing decisions. The information will also assist the RRTs in their area planning activities, and the OSCs in authorizing the appropriate use of chemical and biological agents. Details on the proposed additions and revisions are as follow:

(1) *Identification and documentation by the submitter of its status in relationship to the product as the manufacturer, vendor, importer, distributor, or other designated agent of the manufacturer.* This proposed requirement is intended to clearly establish the point of contact responsible for the submission, and to avoid any conflicts or claims from unauthorized entities on products listed or submitted for consideration.

(2) *Chemical or biological agent categories under which the product is submitted for listing, including information on the specific process(es) through which the product affects the oil, and the specific environment(s) (water and/or adjoining shorelines) on which it is intended to be used.* Currently, when a product meets the definitions of two or more product categories, a submitter must provide technical product data for each of those product categories. After review of the submitted technical product data, EPA makes a determination on whether and under which category the "mixed product" should be listed on the Schedule. It has been Agency policy to list products only under one category to avoid confusion in the field during a response, and because the process(es) through which the products affect the oil for different product categories do not generally overlap or allow for multiple uses of one product. However, EPA has received comments from responders, vendors, and manufacturers who believe that it is appropriate for certain products to be listed under more than one category. The Agency is proposing to revise the current limitations for mixed products. The proposal allows for products to be considered under multiple categories provided they meet all of the listing requirements for each. To this end, the Agency is also requiring that information be provided identifying which environments the product is intended to be used for, in the water and/or on the shoreline. EPA is soliciting comments on the issue of multiple category product listing.

(3) *Copy of the Safety Data Sheet (SDS) required by the Occupational Safety and Health Administration (OSHA) under their Hazard Communication Standard (HAZCOM)*

*for the product.* EPA recognizes that chemical and biological agents may contain substances that could cause harm to oil spill responders who, if unaware of the product's formula, may not wear the proper personal protective equipment. SDSs describe the hazards that may be involved with the product and recommend safety measures that would minimize or avoid adverse consequences that may result from exposures. The Agency believes SDS information will be useful to both OSCs and responders when authorizing and using the product respectively, and that adding this new requirement for a product SDS is appropriate. While the Agency believes that the relevant SDS information should be that of the product, we request comment on the value for responders of requiring an SDS for each individual component. The Agency requests comments on this new requirement, as well as whether the submitter should provide any additional information on potential adverse human health effects based on the product's formula and application methods not captured in the SDS, and how to best communicate this information to product end users.

(4) *Sample product label for all names, brands, or trademarked versions of the product that include the manufacture and expiration dates of the product, as well as the conditions for storage.* The Agency would be allowing flexibility in complying with this requirement by specifying that the submitter does not need to affix new labels to comply with this section if existing labels already convey the required information. This proposed requirement is not intended in any way to supersede any other federal labeling requirement in place (e.g., OSHA's HAZCOM). The proposed requirement is intended to assist the OSC in ensuring that the product used to respond to an incident is still viable and effective, and the oil spill response organizations (OSROs) or any other responder that is storing the product to ensure that their stockpile is viable and available to be authorized for use.

(5) *Recommended product use procedures.* The Agency is proposing to revise the requirement for providing information on the recommended application procedures. While the proposal is maintaining the specific elements included in the current requirement, the supporting documentation and information on the standard methods the product manufacturer used to establish the procedures is requested. EPA believes that providing detailed information on the recommended product use

procedures is necessary to inform the OSC when authorizing these products. This supporting documentation and specific information on the methods and standards used to establish them will inform OSCs and other response personnel in selecting products that can be effectively used under the operating conditions encountered for any given incident. The Agency requests comment on the revised data requirement, and whether there are other elements that should also be included to ensure the proper use and application of the products.

(6) *Environmental fate information.* The Agency is proposing to request any known and available measured data and supporting documentation on the persistence, bioconcentration factor, bioaccumulation factor, and biodegradability of the product and all of its components. The Agency currently has no restriction on use of persistent bio-accumulative products. The Agency considered requirements using existing testing protocols and modeling approaches to establish thresholds for listing based on bioconcentration factors (BCF) or bioaccumulation factors (BAF). These factors offer ratios of the concentration of a particular chemical in a biological tissue per concentration of that chemical in water surrounding that tissue, or in the environment respectively. The Agency considered a tiered testing approach, where the submitter would provide known BCF/BAF information for listing purposes, but would also be required to provide testing based on application rates authorized for use. That is, the lower application rates would only require existing information or the use of existing EPA models, while higher rates would require additional testing of fish, bivalves, and earthworms, as needed.

Similarly, there are no restrictions on the use of environmentally persistent products. The Agency considered requiring the submitter to use estimation techniques/models, such as the EPA model EPI Suite™, which estimates environmental fate properties (breakdown in water or air, etc.) that can indicate where a chemical will go in the environment and how long it will stay there. A tiered testing approach for larger quantity/duration spills as discussed above for bioconcentration and bioaccumulation was also considered.

EPA believes environmental fate information is necessary to inform the OSCs when authorizing these products for use, given the potential for their extended use in significant quantities. However, given that the Agency can

estimate these factors, it is only proposing to require that available information or data be submitted on the product rather than specific product testing, as specific product testing for these factors can add significantly to the testing cost for each product. The Agency requests comment on whether testing for products' bioconcentration, bioaccumulation and biodegradation should be required for listing purposes. Additionally, the Agency requests comment on whether thresholds for BCF and BAF should be established for listing a product on the Schedule.

(7) *New physical/chemical properties and removal of the incorporation by reference of the measurement standards.* The Agency is proposing to add three new elements to the physical/chemical property requirements: Physical state and appearance; vapor pressure; and particle size for solid components. The Agency believes these basic data requirements will provide added context when evaluating the products for listing determinations. These, in combination with the other general product information requirements, will assist the Agency in evaluating the expected product behavior, and the process through which it would affect the oil when used in the intended water and/or shoreline environment. Additionally, the Agency is proposing to remove the current incorporation by reference of specific standards to determine physical/chemical properties, and replacing this with a requirement for a citation of the standard methodology used to determine these values. EPA believes that citing the standard methodology used to determine the required values is sufficient in lieu of specifying commonly recognized standard methodologies. The Agency believes it is appropriate to make this change given the new requirements for accredited laboratories to conduct testing.

(8) *Chemical Abstract Service (CAS) Registry Numbers for each component in a product.* The CAS Registry is a collection of information covering a wide variety of substances identified from the scientific literature since 1957. CAS Numbers serve as an international resource for substance identifiers and are used by scientists, industry, and regulatory bodies because it can be validated quickly and reliably. This will assist the Agency in better characterizing a product's components and assessing its specific process for affecting the oil prior to listing on the Schedule.

(9) *Certification that bioremediation agents do not contain, at levels that exceed the National Ambient Water*

*Quality Criteria lowest density value, bacterial, fungal, viral or opportunistic pathogens.* While providing information on these product contaminants is currently required for bioremediation agents, there are no threshold levels for product listing; a positive result for any of the above pathogens may raise concern, but would not prevent the product from being listed on the Schedule. The proposed requirement that these contaminants not exceed the National Ambient Water Quality Criteria lowest density value is intended to provide information for listing decisions that ensure the use of bioremediation agents that will not result in exceeding established water safety levels. The Agency believes that this information is necessary to determine if a product is suitable for listing, particularly for bioremediation agents, which could potentially be used at recreational beaches. The Agency requests comments on whether it should establish listing thresholds for products based on this criteria, and whether the levels selected for certification are appropriate for this application.

(10) *Certification that the product does not contain levels that exceed the National Water Quality Standards lowest acute value for aquatic life of the following contaminants: Arsenic, cadmium, chromium, copper, lead, mercury, nickel, zinc, or any other heavy metal reasonably expected to be in the product; cyanide; chlorinated hydrocarbons; pesticides; polychlorinated biphenyls (PCBs); and polynuclear aromatic hydrocarbons (PAHs).* Providing information (i.e., upper limit/concentration, detailed analytical methods, and sample preparation) on most of these contaminants is currently required for all products, with no established threshold levels for product listing. The Agency will continue to require information on the methodology and the data and supporting documentation used to determine the levels of these contaminants in a product. The Agency, however, will not specify what analytical testing method the submitter should use to make these determinations, as it currently does for chlorinated hydrocarbons, allowing the submitter flexibility in testing their product. Additionally, the Agency is proposing to require data on several new contaminants: Pesticides, PCBs, and PAHs. The Agency's concern with pesticides as contaminants is mostly due to their potential use on organic sorbents (e.g., peat moss, corn cobs, and cellulose fibers). The concern for PCBs is for their toxicity and classification as

persistent organic pollutants, having toxic effects such as endocrine disruption. PAHs are potent atmospheric pollutants, of concern because some compounds have been identified as carcinogenic, mutagenic, and teratogenic. The proposed threshold requirements for all of these contaminants is intended to provide information for listing decisions that ensure the use of any product will not result in exceeding established water safety levels. The Agency requests comments on whether it should establish a listing threshold for products based on these criteria, and whether the levels selected for certification are the appropriate levels for this application. The Agency also requests comments on whether there are any other contaminants that should be included for evaluation of a product prior to listing.

(11) *Removal of the requirement for laboratories performing the efficacy and toxicity testing for products to have prior experience specific to the required methodology in lieu of recognized national or international accreditations.* Currently, laboratories performing testing for products to be submitted for listing consideration are required to document previous experience with the specific testing methodologies in Appendix C to part 300. The Agency believes it is more appropriate to require that laboratories be nationally or internationally accredited. Accredited laboratories are expected to be capable of following a prescribed testing protocol and good general practices, providing assurance that the test results will be reliable. Therefore, the Agency is proposing to remove the requirement for laboratories to have previous experience specific to the methodologies in Appendix C, and instead require that the laboratories hold accreditations from recognized national or international organizations. National and international accreditation organizations include, for example, the International Organization for Standardization (ISO), and the Laboratory Accreditation Bureau (recognized in the US through the National Cooperation for Laboratory Accreditation (NACLA) and the International Laboratory Accreditation Cooperation (ILAC)). The ISO 17025, Laboratory Competence standard, identifies high technical competence and management system requirements to guarantee laboratory test results and calibrations are consistently accurate. NACLA is a national voluntary organization of regulators, accreditation bodies and laboratories cooperatively

working towards standardization of laboratory accreditations throughout the industry; ILAC is the international counterpart collaboration of laboratory and inspection accreditation bodies. Established to ensure that laboratories are subject to oversight by an authoritative body, accreditation organizations have been evaluated by peers as competent and have signed arrangements to enhance the acceptance of products and services. Because interested parties can have confidence in the test results and certifications provided by accredited laboratories, the Agency is proposing to amend the laboratory requirement and believes that having no prior experience with a specific methodology should not disqualify a laboratory that has been accredited by an appropriate authoritative body. However, the Agency reserves the right to not accept particular lab data should EPA find cause to doubt the quality and integrity of the work. EPA also reserves the right to conduct its own testing of any product.

(12) *Estimated annual product production volume, average and maximum amounts that could be produced per day, and time frame needed to reach maximum production rate (days).* While there is currently no requirement for production capability information, the Agency believes it is important for the OSCs and responders to have this information. The availability of a product may impact decisions of authorization of use, depending on inventory or production capabilities. This would prove to be of key importance, for example, in the event of a major environmental disaster (e.g., a SONS event).

(13) *Recognition received from EPA's Design for the Environment (DfE).* EPA's Design for the Environment (DfE)<sup>33</sup> works in partnership with industry, environmental groups, and academia to reduce risk to people and the environment by finding ways to prevent pollution. DfE evaluates human health and environmental concerns associated with traditional and alternative chemicals and processes in a range of industries in order to select safer chemicals and technologies. DfE focuses its review of formulation ingredients on key environmental and human health characteristics of concern within functional classes. This allows formulators to use those ingredients with the lowest hazard in their functional class, while still formulating high-performing products. The DfE label means that EPA scientists have

evaluated every ingredient in the product to ensure it meets DfE's stringent criteria. Products that meet the DfE criteria are safer chemical choices. In an effort to encourage the development and use of safer technologies, the Agency is proposing a requirement for the submitter to identify products that have met and can be labeled DfE certified as part of the general information, and would include this information in the NCP Product Schedule Technical Notebook.<sup>34</sup> This Technical Notebook presents manufacturer's summary information on the conditions under which each product is recommended to be used, and is a source of information for the OSC in the event of a response.

(14) *International product certifications, testing or use data informing the performance capabilities or environmental benefits of the product.* The Agency believes that any additional data available from other countries may help identify the benefits or concerns for the listing and/or the authorization of use of a product. The Agency, however, is not proposing any specific listing criterion or threshold associated with this broad information request, as some products (particularly new formulations) may not have any of this additional data available.

#### (b) Dispersant Testing and Listing Requirements

The Agency is proposing revisions to the efficacy and toxicity testing protocols, as well as establishing new thresholds for listing dispersants on the Schedule in § 300.915(b). The Agency proposes to define dispersants as typically mixtures of solvents, surfactants, and additives that promote the formation of small droplets of oil in the water column by reducing the oil-water interfacial tension. These droplets are driven into the water column by wave action. Emergency response personnel need to know whether a dispersant or any other type of chemical or biological agent on the Schedule could have negative environmental impacts relative to the oil before decisions are made about its use in a particular oil discharge situation. Consequently, it is essential to consider comparative information about the efficacy and the toxicity of these products. The proposed revisions are in response to concerns not only for an increase in the frequency of planning for the use of these agents, but also for their potential use in large quantities, such as when responding to oil discharges from

<sup>33</sup> <http://www.epa.gov/dfe/>.

<sup>34</sup> <http://www.epa.gov/oem/docs/oil/npc/notebook.pdf>.



oil tanker accidents and offshore well blowouts, as evidenced during the Deepwater Horizon incident in 2010.

#### (1) Dispersant Efficacy

**Current Requirements:** The current NCP Subpart J requires dispersants to be tested for saltwater efficacy in order to determine listing eligibility on the Schedule. Dispersant efficacy is tested following the Swirling Flask Test (SFT) listed in Appendix C to part 300 of the NCP. This protocol was developed by Environment Canada to provide a relatively rapid and simple testing procedure for evaluating dispersant efficacy (*i.e.*, the percentage of oil that is dispersed). The procedure places seawater, oil, and a dispersant into an Erlenmeyer flask, which is then placed on a shaker table for a specified period of time. The flask is modified by the addition of a side spout attached to the bottom of the flask, which allows the analyst to pour off dispersed oil from the bottom into a collection vessel for extraction and measurement. After 20 minutes, a sample of water is poured off and chemically analyzed for dispersed oil. For products to be listed, they must attain an efficacy value of 45 percent or greater average dispersion efficacy of two different crude oils (South Louisiana Crude (SLC) and Prudhoe Bay Crude (PBC)) at room temperature (20–23 degrees Celsius (°C)).

**Proposed Revisions:** The Agency proposes to change the testing protocol for measuring efficacy and adopt the Baffled Flask Test (BFT) as the testing protocol for dispersant efficacy. The new BFT procedure incorporates a redesign of the testing flask by eliminating the side arm, incorporating baffles in the wall of the flask, and adding a stopcock at the bottom, which improves reproducibility in the hands of different operators. This protocol has undergone extensive peer review<sup>35</sup> and has been tested in several laboratories, providing reproducible and repeatable results. The Agency also proposes revisions to the efficacy listing criteria for dispersants to be listed. Specifically, the dispersant must demonstrate that the lower 95% confidence level (LCL<sub>95</sub>) of six replicate flasks meets the new proposed efficacy listing criteria at two test temperatures. New test oils representing a wider range of characteristics are also proposed for this protocol: Alaska North Slope (ANS) and

Intermediate Fuel Oil (IFO–120) were tested for this proposal. Both oils are to be tested at two temperatures: 5 °C and 25 °C. Finally, based on the ANS and IFO–120 testing, the Agency proposes that dispersants considered for listing must demonstrate that they attain all of the following dispersant effectiveness (DE) values:

- ANS at 5 °C: DE<sub>LCL95</sub> ≥70%.
- ANS at 25 °C: DE<sub>LCL95</sub> ≥75%.
- IFO–120 at 5 °C: DE<sub>LCL95</sub> ≥55%.
- IFO–120 at 25 °C: DE<sub>LCL95</sub> ≥65%.

The Agency tested eight of the dispersants currently listed on the Schedule. Three of the eight dispersants clearly differentiated themselves from the other five dispersants for having the best DE<sub>LCL95</sub> efficacy results; they would meet all the proposed thresholds. For the next best dispersant tested, the results were substantially lower than those in the top group and would not meet all the thresholds proposed today. This natural break in the results provided the Agency with the basis for the proposed threshold criteria. The Agency is also proposing to replace the SLC and PBC test oils currently used for efficacy testing with two other oils that represent a wider range of characteristics. The proposal is based on the results of tests using ANS and IFO–120 oils with the new protocol. In proposing two new oils for efficacy testing, the Agency is seeking to ensure that the efficacy testing uses oils that represent a wider range of oil gravities throughout the different geographic locations in the country where dispersants might be used. The oils selected should exhibit sufficiently different characteristics to produce results that have statistically significant variation between them. The proposal is to replace the SLC and PBC oils, both of which were light oils, with a range of light-to-medium, and heavier oils. The light-to-medium gravity ANS pipeline blend and the heavier refined product, IFO–120 were selected for testing the protocol in this proposal. The current protocol, testing 4 replicates of both the SLC and the PBC oils and combining their results, provided one threshold criterion after determining that each of the oils produced statistically similar results. In contrast, the efficacy test results between the ANS and the IFO–120 showed statistically significant differences at each temperature. The Agency believes the use of ANS and IFO–120, or similar oils that represent a wider range of oil gravities, will provide better information on the efficacy of the products when used on different types of oils.

Additionally, the Agency is proposing that efficacy testing be conducted at two different temperatures, 5 °C and 25 °C, rather than at an ambient temperature range of 20–23 °C as currently required. The Agency recognizes the current and future interest in arctic and deepwater drilling, and the continued oil production in the southern, more tropical areas of the country. Given the potential range of locations where dispersants may be used, the Agency believes it is appropriate to have products tested at temperatures that would reflect that range. These temperatures are intended to capture dispersant use scenarios in a wide range of geographic locations and under different temperatures that may occur in the same geographical location (such as, for example, the deep sea and surface water in the Gulf of Mexico, where the temperatures are typically 5 and 25 °C, respectively).

The Agency is also proposing to replace the current SFT with the BFT, which is designed to be more representative of moderately turbulent sea conditions where dispersants are more likely to be successful when used. The revised testing protocol improves test repeatability and reproducibility within and between laboratories, as well as greatly reduces both the inherent error of the method and the human error associated with the current SFT, as discussed below. In addition, reporting the test results in terms of the product's LCL<sub>95</sub> accounts for between- and within-laboratory error variability and the inherent error of the method. Only one number is reported compared to a mean and standard deviation, as the variation has already been subtracted in the reported number. Specifically:

- The new baffled trypsinizing flask design, fitted with a glass stopcock positioned at the bottom side, promotes less manipulation that could result in erroneous re-suspension of non-dispersed oil, as in the SFT. The re-suspension of oil when using the SFT test protocol was a major source of error. In the SFT, the flask has a side arm spout, requiring the analyst to pick up the flask and pour the contents out manually. Through that action, re-suspension of the dispersed oil can easily take place, leading to potentially erroneous results. In contrast, in the BFT, the flask contains a stopcock at the bottom, so the analyst need only open the stopcock to drain the contents from the flask without the potential of remixing or re-suspending the dispersed oil.

- The BFT provides more turbulent mixing and better enables more reproducible and repeatable dispersant

<sup>35</sup> Venosa, Albert D., National Risk Management Research Laboratory, US EPA; Sorial, George A., Department of Civil & Environmental Engineering, University of Cincinnati; King, Dennis W., Statking Consulting; *Round-Robin Testing of a New EPA Dispersant Effectiveness Protocol*, International Oil Spill Conference, 2001.

action than the SFT. The mixing energy within the flask is higher and, as a result of this increased mixing energy, better dispersion is possible. Turbulence is needed for the proper mixing of dispersant and oil and for producing the sheer forces needed to create the small droplet sizes with high surface area-to-volume ratio that promote the effective dispersion of the oil into the water column. The BFT provides such mixing and better enables more repeatable and reproducible dispersant effectiveness than the SFT. The BFT was tested extensively in a nine-participant round-robin inter-laboratory calibration test on six commercial dispersant products.<sup>36</sup> In addition, based on the mixing energy that can be achieved using the new baffled flask, the Agency believes a higher efficacy threshold is warranted. As a result of this increased mixing energy, better dispersion is realized under conditions more realistic of wave action in the sea.

- The variability both between analysts and within analysts is substantially lower for the BFT compared to the SFT, as measured by the coefficient of variation (CV). The CV is defined as the standard deviation divided by the mean and is expressed in terms of percent; the higher the CV, the greater the variability.<sup>37 38</sup>

The Agency considered establishing listing thresholds for efficacy based on the types of oil. The Schedule could list sub-categories of dispersants based on gravities of the oil (*i.e.*, light-medium or heavy), so that a dispersant would only be authorized for use on oils that fall within the range for which it tested effective. Similarly, the Schedule could list dispersants based on temperature, allowing a dispersant to be authorized for use only at the temperature for which it tested effective. However, determining ranges or definitions for when oil changes from light to medium or heavy, is challenging because they may be affected by a number of external factors, including temperature. Likewise, listing dispersants based on temperature would require an additional degree of complexity to the

authorization for use, as the temperatures may change drastically in some geographical regions affected by the same oil discharge. Even within more stable geographical regions, there may be variations at the time of an oil discharge that could affect the decision to use a dispersant stockpiled for a different temperature range. While these more specific listing approaches would provide more information on effective product choices, the Agency is concerned about how such specific listing approaches may contribute to OSC confusion in an emergency, the cost of stockpiling multiple types of dispersants, or the immediate availability of the appropriate dispersant in an emergency. Furthermore, the Agency is proposing revisions to Subpart J to authorize RRTs to request supplementary testing and information to provide greater flexibility in tailoring testing conditions to address area- and site-specific concerns. Given these factors, the Agency believes that it is appropriate that only those dispersants that achieve efficacy for both types of oils and at both temperatures be listed. Listing only the products that achieve efficacy for both types of oils at both temperatures would demonstrate a wide range of efficacy for the product, and a single listing would facilitate authorizing their use during a response action. The Agency requests comments on this approach.

## (2) Dispersant Toxicity

**Current Requirements:** The current NCP Subpart J requires dispersants to be tested for toxicity to saltwater species. This information is available to the OSC for their consideration in authorizing dispersant use. Dispersant toxicity is tested following the protocols in Appendix C to part 300 of the NCP. The standard toxicity test for dispersants involves exposing two saltwater species, the inland silverside fish (*Menidia beryllina* or *M. beryllina*) and the mysid shrimp (*Americamysis bahia* or *A. bahia*), to five concentrations of the test product and No. 2 fuel oil, alone and in a 1:10 mixture of product to oil. Reference toxicity tests are conducted using dodecyl sodium sulfate (DSS) as a reference toxicant. The test length is 96 hours for *M. beryllina* and 48 hours for *A. bahia*. The concentration of test product causing 50% lethality to the test organisms (LC<sub>50</sub>) is calculated at the end of the exposure period.

**Proposed Revisions:** The Agency proposes to revise the toxicity testing requirements for dispersants, including the testing protocol and the use of the test results. The proposal would require acute toxicity testing for the dispersant

alone, and the dispersant mixed with both oils used for efficacy testing; the proposal is based on the ANS and IFO-120 oils. While the Agency currently provides the toxicity testing results to the OSC to assist in authorization of use determinations, it is proposing to use these testing results to determine eligibility for listing on the Schedule.

Specifically, the Agency is proposing to require evaluations of dispersant toxicity using acute toxicity tests following existing USEPA effluent testing guidelines,<sup>39 40</sup> modified for use with dispersant product or dispersant product/oil mixtures, as detailed in Appendix C to part 300. Measured toxicity values for dispersant product and dispersant product/oil mixtures are proposed to include static acute toxicity tests using *A. bahia* and *M. beryllina*. Developmental toxicity using a sea urchin assay, and sub-chronic effects using *A. bahia* and *M. beryllina* are also proposed to be conducted, but with dispersant product only. The Agency is proposing the following toxicity thresholds for listing dispersants on the Schedule:

- Lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval for all acute toxicity tests greater than 10 ppm;
- Inhibition concentration for 50% of the test species (IC<sub>50</sub>) at the lower 95% confidence interval greater than 10 ppm;
- No Observed Effect Concentration (NOEC) equal to or greater than 1 ppm for sub-chronic toxicity.

**Test Oils and Oil Acute Toxicity Values.** The Agency proposes to replace the No. 2 fuel oil currently used for toxicity testing with two test oils that better represent a wider range of characteristics and that may be encountered during an incident. The proposal is based on ANS and IFO-120, as previously discussed. In addition to making the test oils consistent with the dispersant efficacy tests, testing oils of different gravities also provides a better estimation of dispersant/oil toxicity associated with differing oil constituencies, and ultimately a better representation of the potential overall toxicity of a product.

The Agency proposes to conduct the oil-only acute toxicity tests for the two reference oils, with both *A. bahia* and

<sup>36</sup> Venosa, A. D., D. W. King, and G. A. Sorial. 2002. The Baffled Flask Test for Dispersant Effectiveness: A Round Robin Evaluation of Reproducibility and Repeatability. *Spill Sci. & Technol. Bulletin* 7(5-6):299-308.

<sup>37</sup> Sorial, G. A., A. D. Venosa, K. M. Miller, E. Holder, and D. W. King. 2004a. Oil Spill Dispersant Effectiveness Protocol—Part I Impact of Operational Variables. *ASCE. J. Env. Eng. Div.*, 130(10):1073-1084.

<sup>38</sup> Sorial, G. A., A. D. Venosa, K. M. Miller, E. Holder, and D. W. King. 2004b. Oil Spill Dispersant Effectiveness Protocol—Part II Performance of the Revised Protocol. *ASCE. J. Env. Eng. Div.*, 130(10):1085-1093.

<sup>39</sup> USEPA 2002a Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth edition. (Acute Methods Manual), Office of Water, Cincinnati, OH. EPA-821-R-02-012.

<sup>40</sup> USEPA 2002b. Short-term Methods for Estimating the Chronic toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition. (Saltwater Chronic Methods manual), Office of Water, Cincinnati, OH. EPA-821-R-02-014.

*M. beryllina*, and provide this data for comparisons to dispersant and dispersant-oil mixture acute toxicity tests. EPA will make all of the reference oil toxicity test results available to the public on its Web site, including raw data and calculated median LC<sub>50</sub> values. By providing this information, the Agency is reducing the number of required toxicity tests the submitter would need to conduct. Alternatively, the Agency considered requiring submitters to conduct the oil acute toxicity testing as it would provide an opportunity to detect anomalies in the submitted data. The Agency is requesting comments on whether the submitter should be required to conduct the oil-only acute toxicity testing for the two test oils.

**Dispersant and Dispersant-Oil Acute Toxicity Threshold.** Using the EPA toxicity classification scheme,<sup>41</sup> LC<sub>50</sub> values ranging from 10 ppm to 100 ppm are classified as slightly toxic and above 100 ppm substances are considered acutely nontoxic to aquatic organisms. For both *M. beryllina* and *A. bahia*, the Agency is proposing as the threshold value the lower bound of the LC<sub>50</sub> 95% confidence interval (CI) greater than or equal to 10 ppm for all toxicity tests to qualify a dispersant to be listed on the Schedule. The rationale for using the lower bound of the CI is that the CI should not contain any values less than or equal to 10 ppm since theoretically, the LC<sub>50</sub> can fall anywhere within the CI. By using the lower CI, the Agency is providing a conservative decision criterion for acute toxicity, and by proposing a greater than or equal to 10 ppm threshold level, it is establishing an adequate safety margin without being overly restrictive.

**Dispersant-Oil Mixture Acute Toxicity Protocol.** The Agency is proposing to substitute the existing preparation procedure for the dispersant-oil mixture (DOM) in Appendix C to Part 300, Section 3 method for the preparation procedure for dispersant-oil mixture using a modified protocol<sup>42</sup> first described by Baron and Ka'ahue<sup>43</sup> and

fully described by Hemmer et al.<sup>44</sup> These new modified procedures for preparation of the chemically enhanced water accommodated fractions (CEWAFs) of dispersant-oil mixtures are used to make exposure solutions. Evaluations of the dispersant-oil mixture toxicity using acute toxicity tests follow existing EPA effluent testing guidelines,<sup>45,46</sup> modified for use with these exposure solutions and are detailed in Appendix C to part 300. EPA proposes to modify the variable loading preparation procedure described in Chemical Response to Oil Spills: Ecological Research Forum (CROSERF)<sup>47</sup> to a variable dilution procedure to standardize the oil-to-water ratio and provide a consistent chemical concentration in a test series. Additionally, the modified preparation procedure provides economies in analytical costs by reducing the need to analyze the composition of every tested concentration. Chemical analysis for the total petroleum hydrocarbon (TPH) concentration of the CEWAF stock solution is required.

**Sea Urchin Developmental Assay.** The Agency is proposing to require a sea urchin developmental assay (also referred to as an embryogenesis assay) to assess the potential for a dispersant product to cause adverse effects on the developmental process. During the Deep Water Horizon spill event, the possible adverse effects of oil dispersants on the developmental processes of fish and invertebrate species were identified as critical issues of concern. By incorporating the sea urchin developmental assay, the Agency is using it as a sensitive surrogate test for pelagic early life stages. This assay would employ an existing EPA test protocol<sup>48</sup> routinely used in effluent

testing. As suggested for the dispersant acute toxicity test criteria with *A. bahia* and *M. beryllina*, the Agency is proposing as the threshold value the lower bound of the developmental IC<sub>50</sub> 95% confidence interval greater than or equal to 10 ppm. For this test, the IC<sub>50</sub> is defined as the concentration of dispersant that inhibits the development of 50% of exposed embryos. As described above for acute toxicity levels, the Agency is adopting a similar rationale for IC such that IC<sub>50</sub> values ranging from 10 ppm to 100 ppm are considered to cause slight adverse effects on the developmental process, and above 100 ppm substances are considered to cause no adverse effects. By using the lower CI the Agency is providing a conservative decision criterion for developmental toxicity, and by proposing a greater than or equal to 10 ppm threshold level it is establishing an adequate safety margin without being overly restrictive.

**Sublethal/Subchronic Studies.** The Agency has limited information concerning the possible sublethal effects of dispersants currently listed on the Product Schedule. The Agency is proposing requirements for subchronic assays (duration approximately 7 days) to be performed with *A. bahia* and *M. beryllina* following established EPA short-term methods for estimating chronic toxicity of effluents.<sup>49</sup> While the Agency considered requiring longer-term tests, it believes the 7-day subchronic tests are reasonable alternatives for estimating chronic toxicity. This information would also be of value as guidance to regional responders and OSC's on possible adverse effects on survival and growth of larval fish and invertebrates caused by longer-term exposure to dispersants. The Agency is proposing a NOEC equal to or greater than 1 ppm listing threshold level for sub-chronic effects. This threshold level provides a tenfold safety factor from the proposed acute toxicity threshold values. The tenfold safety factor is a common practice in ecological risk assessment and thus the Agency believes it provides an adequate safety margin without being overly restrictive. The Agency is requesting comment on whether it should consider longer-term tests (e.g., early life stage tests), which may be more relevant to sublethal effects caused by longer

Receiving Waters to West Coast Marine and Estuarine Organisms. EPA/600/R-95/136.

<sup>49</sup> USEPA 2002b. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition. (Saltwater Chronic Methods manual). Office of Water, Cincinnati, OH. EPA-821-R-02-014.

<sup>41</sup> [http://www.epa.gov/oppefed1/ecorisk\\_ders/toera\\_analysis\\_eco.htm#Ecotox](http://www.epa.gov/oppefed1/ecorisk_ders/toera_analysis_eco.htm#Ecotox).

<sup>42</sup> The modified protocol is based on a variable dilution modification of the Chemical Response to Oil Spills: Ecological Research Forum (CROSERF) testing protocol; Aurand, D. and G. Coelho (Editors). 2005. Cooperative Aquatic Toxicity Testing of Dispersed Oil and the "Chemical Response to Oil Spills: Ecological Effects Research Forum (CROSERF)." Ecosystem Management & Associates, Inc. Lusby, MD. Technical Report 07-03.

<sup>43</sup> Barron, MG, Ka'ahue L. 2003. Critical evaluation of CROSERF test methods for oil dispersant toxicity testing under subarctic conditions. Mar Pollut Bull 46:1191-1199.

<sup>44</sup> Hemmer, MJ, Barron, MG, Greene, R. 2011. Comparative toxicity of eight oil dispersants, Louisiana sweet crude oil (LSC) and chemically dispersed LSC to two aquatic species. Environ Toxicol Chem, 30: 2244-2252.

<sup>45</sup> USEPA 2002a Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth edition. (Acute Methods Manual), Office of Water, Cincinnati, OH. EPA-821-R-02-012.

<sup>46</sup> USEPA 2002b. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition. (Saltwater Chronic Methods manual). Office of Water, Cincinnati, OH. EPA-821-R-02-014.

<sup>47</sup> CROSERF was established as a working group of industry, government, and university scientists to coordinate and disseminate research on chemical oil spill dispersants. CROSERF participants developed aquatic toxicity testing protocols during 1994 to 2000 with the foremost objective of standardizing test methods and reducing inter-laboratory variability.

<sup>48</sup> USEPA 1996. Short-term Methods for Estimating the Chronic Toxicity of Effluents and

duration exposure to low concentrations of a dispersant product.

#### Alternative Options

In addition to the criteria discussed above, the Agency considered calculating a toxicity threshold based on the ratio of the LC<sub>50</sub> of oil-alone to the LC<sub>50</sub> of the dispersant-oil mixture for *M. beryllina* and *A. bahia* to assist in comparing the relative toxicity of crude oil to chemically dispersed oil. A ratio of less than or equal to 1 would indicate the toxicity of the dispersant-oil mixture is less than or equal to the toxicity of the oil-alone. The Agency considered whether this ratio should be used as a criterion for inclusion of dispersants on the Schedule or as guidance to emergency responders. Using a less than or equal to 1 ratio as a guideline would afford an adequate safety margin without being overly restrictive. While the ratio would provide a simple, interpretable, and easily justifiable value for determining acceptable toxicity levels of dispersant-oil mixtures, it would not be specific to oils in particular discharge situations. The Agency is requesting comment as to whether such a value should be calculated, and if so, whether it should be an additional listing criterion, or provided as information for the responders to consider in authorizing dispersant use.

The Agency also considered including geographically or ecologically representative species in the testing protocol. The Agency believes, however, that this issue is addressed by the proposed revisions to the supplemental testing and information requirements in section 300.910(g), with the decisions of testing geographically and ecologically representative species left to the discretion of the RRTs. While inclusion of species from different phyla and habitats would provide useful and important information on possible adverse effects of dispersant products and dispersed oil, the proposed testing protocols would need to be modified and validated. Further, the Agency is concerned about balancing the time and cost associated with the development of these tests on the part of the submitter rather than on the end users.

The Schedule currently requires no specific toxicity or efficacy tests for subsea dispersant listing or authorization of use. While the differences in toxicity values and efficacy may be affected by application in subsea environments, the Agency believes that the proposed requirements establish an adequate baseline for listing dispersants on the Schedule and for authorizing their use by responders in

case of an incident. The Agency is addressing these concerns by proposing new provisions for dispersant monitoring for all subsea use, and requests comments on alternative testing and listing approaches to specifically address subsea concerns.

#### (3) Limitations of Use

The Agency is proposing a conditional use listing for dispersants. The proposal specifies that dispersants may only be used in saltwater environments. Dispersants are typically designed and traditionally used for responding to oil discharges in saltwater. In general, the effectiveness of dispersants decreases as the salinity of the water decreases, given the same hydrophilic-lipophilic balance, and the effectiveness is minimal in freshwater environments. Surfactants, the main active ingredient in dispersants, sustain oil droplets in water by orienting the lipophilic side of its molecule in the oil and the hydrophilic side in the water. The hydrophilic side of the surfactant is generally more soluble in waters with higher salinity values, causing the oil droplets to be more stable in saline water environments. In waters with no salinity, many dispersants have shown a very low effectiveness or are sometimes completely ineffective.<sup>50</sup>

The Agency is also concerned with using dispersants in freshwater environments because of the limited dilution typically available as compared with the open sea and because of the existence of water intakes in rivers, streams, and lakes for use in drinking water supplies. Using dispersants in freshwater has the potential for compounding the impacts caused by already discharged petroleum products, particularly near potable and non-potable subsurface water intakes. The Agency does recognize, however, that as a last resort, dispersants may have some limited applicability in freshwater. The provision for authorization of use under § 300.910(d) would allow for exceptions to the prohibition on the use of dispersants in freshwater in cases where there is an immediate threat to human life. The Agency is requesting comment on this proposed listing limitation.

#### (c) Surface Washing Agent Testing and Listing Requirements

The Agency is proposing to define Surface washing agents (SWA) as substances that separate oil from solid surfaces, such as beaches, rocks, or concrete, through a detergency

mechanism that lifts and floats oil for collection and recovery from the environment with minimal dissolution, dispersion, or transfer of oil into the water column. For this agent category, the Agency is proposing to revise the toxicity testing protocols, to establish efficacy testing protocols, and to establish both toxicity and efficacy listing thresholds in § 300.915(c). The proposed revisions respond to concerns regarding their frequent use and the potential for residual impacts after their use.

#### (1) Surface Washing Agent Efficacy

**Current Requirements:** There are currently no efficacy testing requirements in the NCP Subpart J for surface washing agents to determine listing eligibility on the Schedule.

**Proposed Revisions:** The Agency is developing a laboratory testing protocol to evaluate the efficacy of SWAs in removing crude oil from a solid substrate.<sup>51</sup> Currently available efficacy testing protocols for these agents are concerned with assessing the detergency, or cleaning power, of the agents by quantifying the physical removal of oil from non-vegetative substrates as a result of their use. The protocols typically involve the application of oil to a solid substrate (or the use of pre-oiled substrate), weathering of the oil on the substrate, application of the agent to the oiled substrate, a contact period for SWA penetration, and washing of the substrate with water. The fractions of oil removed in the wash water and/or remaining on the substrate are quantified. Oil removal efficiencies for surface washing treatments are typically compared to the washing efficiency of water without surface washing agents (untreated controls). Disadvantages or potential sources of error have been identified in the existing protocols that may not reflect field performance. Of concern are, for example, the use of substrates (e.g., stainless steel, porcelain) with different wetting and adhesion properties than natural substrates such as sand or gravel; short weathering times insufficient for oil attachment; and the absence of mixing energy during the washing step, which may lead to incomplete detachment of partially released oil.

The Agency's goal is to develop and evaluate a new bench-scale testing protocol that would provide a standard for EPA to use in SWA product

<sup>50</sup> Fingas, M., (Ed.), 2011, *Oil Spill Science and Technology*, Gulf Professional Publishing, pp. 513–518.

<sup>51</sup> Koran, K.M., Venosa, A.D., Luedeker, C.C., Dunnigan, K., Sorial, G.A., 2009. Development and Testing of a New Protocol for Evaluating the Effectiveness of Oil Spill Surface Washing Agents. *Mar. Pollut. Bull.*, 58: 1903–1908.

evaluation prior to listing on the Schedule. Changes over established protocols the Agency considered include: Using natural substrates to reflect the most commonly impacted shoreline material; establishing sufficient weathering time to permit strong attachment of the oil to the substrate surface; fully quantifiable approaches with known volumes and precise measurements to allow mass balance; and a standardized and reproducible methodology to minimize operator error. PBC oil was used as the representative standard reference oil in developing the protocol since it had previously been selected as one of two standard oils for the Agency's published protocols for dispersant and bioremediation agent testing. However, the Agency is proposing revisions to the dispersant and bioremediation testing protocols, including replacing the PBC reference oil. The protocol being developed has not been tested: With the IFO-120 or the ANS oils that are proposed to be used in the revised dispersant efficacy testing protocol; at multiple temperatures; or to include freshwater systems. The Agency expects to develop final recommendations for the testing protocol following round robin testing after adjusting for the new variables. The Agency would propose this protocol in the **Federal Register** through notice and comment before adopting it as part of Subpart J requirements.

Consequently, because of the additional work needed to finalize its protocol, the Agency is proposing to allow the use of standard recognized efficacy testing methodologies for surface washing agents. An example of such a standard recognized methodology is the American Society for Testing and Materials (ASTM) Standard Test Method for Evaluating the Effectiveness of Cleaning Agents.<sup>52</sup> Another methodology is Environment Canada's Test Method.<sup>53</sup> The capability of a particular surface washing agent depends upon the application procedures and the characteristics of the surface being cleaned, such as size, shape, and material. The ASTM test method in particular covers a procedure for evaluating the capability of the agents, providing a relatively rough

surface to which the oil can adhere. The Environment Canada method uses a stainless-steel 'trough' which is placed at a specified angle. The target oil is placed on an area on the trough. The treating agent is then applied in droplets to the surface of the oil and after 10 minutes at 5-minute intervals, rinses of water are applied to the trough. After drying, the trough is weighed and the removal calculated from the weight loss. Repeatability is within 5%. The Agency is requesting comment on available methodologies and its published protocol to incorporate as the testing protocol for these agents. The Agency also requests comment on whether the rule should identify the specific methodologies to be used until EPA develops and adopts a new testing protocol for SWA efficacy testing.

The Agency is also proposing an efficacy threshold of 30% in either fresh or salt water or both depending on the intended product use. This is based on the efficacy data published in "Surface-washing Agents or Beach Cleaners."<sup>48</sup> Numerous surface washing agent products, including several from the Schedule were tested using the Environment Canada methodology. The average efficacy of the surface washing agents tested is approximately 30%. The Agency requests comments on this approach and other efficacy test data available.

## (2) Surface Washing Agent Toxicity

**Current Requirements:** The current NCP Subpart J requires surface washing agents to be tested for saltwater species toxicity. Surface washing agent toxicity is tested following the dispersant toxicity testing protocols in Appendix C to part 300 of the NCP.

**Proposed Revisions:** The Agency is proposing revisions to the toxicity testing requirements for surface washing agents, including the testing protocol. While the Agency currently provides the toxicity testing results to the OSC to assist in authorization of use determinations, it is proposing to use these toxicity testing results to determine listing eligibility on the Schedule.

The proposed revisions to the testing protocols for surface washing agents are detailed in Appendix C to part 300. The proposed acute toxicity test protocol for surface washing agents is based on EPA's protocol, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters for Freshwater and Marine Organisms*.<sup>54</sup> The Agency

proposes to require surface washing agents be tested for acute toxicity using fresh water species *Ceriodaphnia dubia* and *Pimephales promelas*, or saltwater species *Americamysis bahia* and *Menidia beryllina*, or both, depending on the intended product use. The concentration of test product causing 50% lethality to the test organisms (LC<sub>50</sub>) and lower and upper 95% confidence intervals (LCI<sub>95</sub> and ULCI<sub>95</sub>) are calculated at the end of the exposure period. To be listed on the Schedule, surface washing agents must demonstrate an acute lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval greater than 10 ppm in either fresh or salt water, consistent with the acute toxicity thresholds proposed for dispersants. EPA's toxicity classification scheme<sup>55</sup> classifies LC<sub>50</sub> values ranging from 10 ppm to 100 ppm as slightly toxic and values above 100 ppm substances are considered practically nontoxic to aquatic organisms. By proposing this threshold level, the Agency is establishing an adequate safety margin without being overly restrictive. The Agency requests comments on the proposed acute toxicity methods (or any others available) and thresholds for surface washing agents.

The Agency is proposing provisions for conducting acute toxicity tests with dispersant-oil mixtures, but not for any other agent, including SWAs. EPA believes that non-dispersant products are not likely to be used in the same quantities or durations as dispersants and some may be recovered making a requirement to conduct toxicity tests of product/oil mixtures unnecessary. There may be concerns, however, for increased product toxicity because of their interaction with the oil. Thus, the Agency is requesting comment on the need for acute toxicity tests conducted with surface washing agents-reference oil mixtures. A protocol for preparation of product/oil mixtures for toxicity testing is available for review in the docket.

## (3) Limitations

The Agency is proposing a conditional use listing for SWAs. The proposal specifies that these products may only be used in those water environments for which the product was tested and for which it met the listing threshold criteria. The Agency recognizes that products may yield effective results in certain environments and not in others. Products that may be

<sup>52</sup> ASTM Standard Test Method for Evaluating the Effectiveness of Cleaning Agents. Designation: G122-96 (Reapproved 2008). ASTM International, 100 Barr Harbour Dr., P.O. Box C-700 West Conshohocken, Pennsylvania 19428-2959, United States

<sup>53</sup> Fingas, Merv and Fieldhouse, Ben; "Surface-washing Agents or Beach Cleaners" (2010). Chapter 21 Surface-Washing Agents or Beach Cleaners. In *Oil Spill Science and Technology* (p716). London: Gulf Professional Publishing.

<sup>54</sup> [http://water.epa.gov/scitech/methods/cwa/wet/upload/2007\\_07\\_10\\_methods\\_wet\\_disk2\\_atx1-6.pdf](http://water.epa.gov/scitech/methods/cwa/wet/upload/2007_07_10_methods_wet_disk2_atx1-6.pdf).

<sup>55</sup> USEPA 2010. [http://www.epa.gov/oppefed1/ecorisk\\_ders/toera\\_analysisisco.htm#Ecotox](http://www.epa.gov/oppefed1/ecorisk_ders/toera_analysisisco.htm#Ecotox).

effective in freshwater environments may not necessarily be so in saltwater environments, and vice versa. The Agency is proposing this limitation to allow product manufacturers the flexibility to select which environment the product is to be tested and authorized for use, either saltwater, fresh water, or both environments. Therefore, the product would be listed and could only be authorized for use in those water environments for which it was tested and for which it met both the efficacy and toxicity listing criteria.

#### (d) Bioremediation Testing and Listing Requirements

The Agency is proposing to define bioremediation agents as biological agents and/or nutrient additives deliberately introduced into a contaminated environment to increase the rate of biodegradation and mitigate any deleterious effects caused by the contaminant constituents. Bioremediation agents include microorganisms, enzymes, and nutrient additives such as fertilizers containing bioavailable forms of nitrogen, phosphorus and potassium. For this agent category, the Agency is proposing to revise the efficacy testing protocols, to establish toxicity testing protocols, and to establish both efficacy and toxicity listing thresholds in § 300.915(d).

##### (1) Bioremediation Agent Efficacy

*Current Requirements:* Bioremediation agent efficacy is currently tested following the protocols in Appendix C to part 300 of the NCP. The current bioremediation agent efficacy testing protocol is designed to determine a product's ability to biodegrade oil by quantifying changes in the oil composition resulting from biodegradation using natural seawater as the test water. The protocol tests the bioremediation agent for microbial activity and quantifies the disappearance of saturated hydrocarbons and PAHs in weathered oil; for purposes of the proposal the Agency tested the protocol using ANS 521.<sup>56</sup> The sample preparation procedure extracts the oil phase into dichloromethane (DCM), with a subsequent solvent exchange into hexane. The analytical technique uses a high-resolution gas chromatograph/mass spectrometer (GC/MS); GC/MS provides for a high degree of chemical separation and spectral resolution. Concurrently

with the chemical analysis, a microbiological analysis is also performed to determine and monitor the viability and growth of the microbial cultures being studied. For commercial products that are strictly microbial agents not containing their own nutrients, a mineral nutrient solution is provided if requested by the submitter; no further nutrients are added to commercial products containing their own nutrients. To be listed on the Schedule, bioremediation agents must demonstrate a statistically significant difference between the average degradation of both total alkanes and total aromatics in the oil achieved by the product after 28 days and the average degradation for the same compounds achieved by the non-nutrient control in the same time period.

*Proposed Revisions:* The proposal reflects a series of changes to the current efficacy testing protocol for bioremediation agents. The protocol includes freshwater testing in addition to the current saltwater-based test, and uses artificial water for both fresh and saltwater testing, replacing the natural seawater currently used. The proposed protocol also eliminates several gravimetric and microbiological analyses and testing endpoints not used in the proposed listing determinations. Additionally, the proposed protocol limits the levels at which external nutrients may be added, which allows the addition for product formulations without nutrients, or for product formulations that have nutrient concentrations at insufficient levels for the experimental setup. Finally, the methodology streamlines the statistical analysis. The proposed revisions address concerns with the existing methodology as discussed below, expanding its application to include freshwater environments, improving the consistency and comparability of the test results, and generally streamlining the protocol.

*Protocol.* The proposed efficacy testing protocol is designed to determine a bioremediation agent's ability to biodegrade weathered crude oil in the exposure water (saltwater or freshwater) by quantifying changes in the oil composition resulting from biodegradation. It consists of a series of six product and control flasks containing artificial seawater or artificial freshwater and weathered crude oil in which biodegradation of the crude oil hydrocarbons is monitored for 28 days. Product flasks at day 28 are compared to product flasks at day 0 to determine if a specified reduction both in total resolved alkanes and total

resolved aromatics was achieved in that time period. Product flasks contain exposure water, weathered oil, and product in concentrations specified by the manufacturer. Positive control flasks must contain exposure water, oil, nutrients, and the standard culture supplied by the Agency. A negative control, consisting of artificial exposure water, product, weathered crude oil, nutrients, and a sterilant is also used to ensure that the observed degradation of hydrocarbons was not caused by abiotic losses or interaction with the product. The Agency is requesting comment on whether an additional protocol specific to products containing enzymes only would be appropriate; this would consist only of exposure water, weathered oil and the enzymatic product in the concentrations specified by the manufacturer. The proposed protocol quantifies the degradation (or disappearance) of alkane hydrocarbons and aromatic hydrocarbons. The sample preparation procedure extracts the oil phase into the solvent dichloromethane (DCM; also known as methylene chloride) with a subsequent solvent exchange into hexane. The test method targets the relatively easy to degrade normal alkanes and the more resistant and toxic aromatics. The analytical technique uses a high resolution GC/MS because of its high degree of chemical separation and spectral resolution. GC/MS has long been used to study the weathering and fate of oil spilled into the environment. It gives unambiguous results for determining biodegradation efficacy. For quantitative analyses, the instrument is operated in the selected ion monitoring mode (SIM) of detection at a scan rate of > 5 scans per second to maximize the linear quantitative range and precision of the instrument.

*Fresh and Saltwater Environments.* The existing bioremediation test is exclusively for saline environments with no equivalent test for freshwater environments. Further, the existing test uses natural seawater and has been found to give variable results due to the compositional variability of seawater both chemically and microbiologically. The Agency proposes to replace the natural seawater with a standardized artificial saltwater formula called GP2, whose components and concentrations are generally recognized, and which is easily made. The use of artificial saltwater allows for better test reproducibility. Additionally, the Agency proposes to expand the requirement for efficacy testing to include freshwater, which allows for a better evaluation of the use of these agents in this environment. Similarly to

<sup>56</sup> Artificially distilled Alaska North Slope crude oil at 521 °F (272°C) to remove the low molecular weight hydrocarbons to approximate natural weathering processes that occur after a spill.

the artificial saltwater, artificial minimal salts freshwater would be used with known ingredients (Bushnell and Haas; *Journal of Bacteriology*, Vol. 41: 653, 1941). Because the Agency is adding a freshwater test, it is also allowing the submitter to test a product for freshwater only, saltwater only, or both. The Schedule listing would specify in which environment the product was tested and authorized for use. Products could be listed for use on saltwater only, on freshwater only, or for use on both environments, giving the option to the submitter to market its products accordingly.

**GC/MS Testing Endpoints.** GC/MS has long been used to study the weathering and fate of oil spilled into the environment. However, GC/MS being the primary tool used in the analysis makes this test expensive. The current protocol includes several measured variables in both the product flasks and the non-nutrient control flasks, including the gravimetric residual oil weight analysis, a Most Probable Number microbiological analysis, and the GC/MS analysis at 3 different time points, days 0, 7, and 28. The Agency is proposing to eliminate all of these analyses except the day 0 and 28 GC/MS analysis of both the non-nutrient control and the product flasks. The Agency believes this is appropriate because the other measurements are not used in determining whether a product meets the listing criterion. Additionally, the statistical analysis has been greatly simplified, and a new decision rule is proposed for listing determinations rather than relying on a statistical significance test.

• **Gravimetric Weight Analysis:** Originally, the gravimetric weight analysis was used as a preliminary test to avoid having to perform a GC/MS analysis. Products that failed to significantly reduce the gravimetric weight of the oil within 28 days were considered not effective, thereby eliminating the need to conduct the more expensive GC/MS analysis. However, numerous compounds can give rise to positive interferences with the gravimetric weight analysis, such as biomass lipids and other biogenic materials, thereby rendering the gravimetric analysis suspect. Furthermore, numerous factors can confound the interpretation of gravimetric weight changes in oil over time. This has led to confusion as some manufacturers have mistakenly concluded their product passed the testing protocol simply by complying with the gravimetric weight loss measurement. By eliminating the gravimetric analysis altogether, EPA is

streamlining the testing process, resulting in less confusion and a modest analytical cost savings for the manufacturer.

• **Most Probable Number Enumeration (Microbiological Analysis), Nutrient Control, and Day-Seven Sampling Event:** Results of these test endpoints, while currently required, would not be used in determining whether a product meets the proposed listing requirements. Therefore, the Agency is proposing to eliminate these test requirements from the proposed protocol.

• **28 day Sampling Event:** Generally, results of biodegradation may occur in less than 28 days, particularly for alkanes for which biodegradation often takes place in a much shorter time period. However, 28 days are needed to make sure that the aromatic components, which biodegrade more slowly, have been reduced significantly; testing protocols that accommodate aromatics are of particular importance because of their potential ecological toxicity. While the Agency could propose different time frames in determining whether the product met the reduction criterion for alkanes and aromatics, it believes that such an approach would be confusing. Therefore, the 28 day time period is being proposed for both fractions for simplicity and to provide sufficient time for degradation of the aromatics.

**Protocol Verification.** The Agency proposes to provide a standard culture for performing laboratories to use as a positive control benchmark. A positive control is needed as an indication that the test was performed properly. The Agency is proposing to use as the standard culture an oil-degrading bacteria isolated from Disk Island in Prince William Sound in 1990. This culture is an excellent degrader of alkanes and aromatics in saltwater and freshwater, although it performs better in saltwater, especially in degrading the aromatics. This culture has the ability to degrade ANS 521 oil, with known efficacy values for both fresh and saltwater. The proposed positive control consists of triplicate flasks containing sterile artificial seawater or freshwater, nutrients (salts of nitrogen and phosphorus), the weathered ANS 521 oil, and the standard culture. The performing laboratory must achieve the known reduction in alkane and aromatic fractions in these positive control flasks in order to certify they successfully conducted the test procedure. Additionally, data from the positive control are required to be submitted with the product test results. The Agency is not proposing, however, that

the positive control be repeated every time a product test is performed. Rather, the certifying laboratory would be required to report the results of the positive control tests conducted within a year prior to the product testing, as part of the product submission package. Additionally, this positive control culture would be used as a standard microbial inoculum to test abiotic products, such as nutrients and enzymes.

**Added Nutrients.** The current protocol allows the manufacturer to request the addition of nutrients to support a culture's ability to degrade the crude oil hydrocarbons. This addition is currently allowed for commercial products that are strictly microbial agents and do not contain any nutrients; the addition of nutrients is not allowed for commercial products containing its own nutrients. A modified salt solution of nitrogen and phosphorus is used as the mineral nutrient. All microbial cultures need nutrients to survive and grow. In the natural environment, biodegradation is not completely limited by nutrient availability given that a low level flow of nutrients is continuous. To maximize biodegradation rates, the addition of nutrients is typically recommended in the field with a bioremediation product unless nutrients are already high (*i.e.*, >5 mg/L). However, in a closed laboratory flask, the amount of nitrogen and phosphorus supplied by a typical product might easily become limiting quickly as biodegradation takes place because they are supplied only once at the beginning of the test. If nutrient limitation occurs, further reduction of hydrocarbons would likely be inhibited, and this may cause the product to fail the listing criteria. To address this problem, the Agency proposes modifying the protocol by allowing the addition of nutrients to any product containing living microbes that does not contain said nutrients in sufficient quantities to allow a fair evaluation. However, the additional quantities allowed are limited to total concentrations of nitrogen and phosphorus that do not to exceed the equivalent amounts used in the positive control. Capping the amount of nutrients that can be added to all products will yield more consistent results and more effective comparisons among products.

**Efficacy Thresholds.** The Agency is proposing to revise the listing criteria by replacing the current "statistically significant difference" test with defined values for the percent reduction of total alkane and aromatic concentrations. For a bioremediation agent to be listed on

the Schedule, the Agency proposes the percent reduction of the total alkane fraction (sum of all alkane concentrations determined by GC/MS) to be greater than 95% at day 28, and that the percent reduction of the total aromatic fraction (sum of all aromatic concentrations determined by GC/MS) must be greater than 70% at day 28, both based on the one-tailed Upper 95% Confidence Limit (UCL<sub>95</sub>) for salt water. For freshwater, the Agency proposes the percent reduction of the total alkane fraction (sum of all alkane concentrations determined by GC/MS) to be greater than 95% at day 28, and that the percent reduction of the total aromatic fraction (sum of all aromatic concentrations determined by GC/MS) must be greater than 40% at day 28, both based on the one-tailed Upper 95% Confidence Limit (UCL<sub>95</sub>).

#### *Basis of Efficacy Thresholds.*

According to Haines *et al.* (2005),<sup>57</sup> the positive control culture was able to biodegrade total alkanes in saltwater by 98.9% and total aromatics by 79.8% in 28 days, based on the UCL<sub>90</sub>. In freshwater, the percent reduction values were 97.9% and 37.8%, respectively, again based on the UCL<sub>90</sub>. Thus, the proposed listing thresholds for the percent reduction of total alkanes and total aromatics from the GC/MS analysis are based on the findings of Haines *et al.* (95% for alkanes vs. 98.9% in saltwater and 97.9% in freshwater, and 70% for aromatics in saltwater and 40% in freshwater vs. 79.8% in saltwater and 37.8% in freshwater). As for the positive control, the Agency believes a reasonable target in saltwater would be 95% for total alkanes in both types of exposure waters. For aromatics reduction, 70% is considered reasonable in saltwater and 40% in freshwater, based on Haines *et al.* EPA is using UCL<sub>95</sub> values rather than the UCL<sub>90</sub> values from Haines because EPA's latest research uses 6 replicates for the test protocol which increases precision. The proposed criteria demonstrate that the product can cause a substantial degradation of the alkane and aromatic fractions of weathered crude oil compared to a control, as determined solely by GC/MS analysis.

Of note, these proposed thresholds for listing bioremediation products on the Schedule are similar to those established for Canadian efficacy testing, which are reductions of 30% for total aliphatics and 10% for total aromatics. The 30% reduction in total

aliphatics (including all resolvable and non-resolvable but GC-detectable aliphatics) is approximately equivalent to an 80% reduction in total GC/MS-resolvable target n-alkanes, based on Canadian data. The 10% reduction in total aromatics is approximately equivalent to a 50% reduction of the 5-aromatic homologue group consisting of naphthalene, fluorene, dibenzothiophene, phenanthrene, and chrysene and their alkylated homologs. The U.S. aromatic series includes 2 other 4-ring aromatics in addition to those 5 aromatic series considered by Blenkinsopp *et al.*<sup>58</sup> Thus, the U.S. and Canadian protocols are similar in terms of decision criteria. The Agency requests comments on all the proposed changes and listing thresholds.

#### (2) Bioremediation Agent Toxicity

*Current Requirements:* The Agency currently has no bioremediation agent toxicity testing requirements for purposes of listing these agents on the Schedule. Section 5 of Appendix C is reserved for this purpose. The Agency has, however, on a case-by-case basis, requested manufacturers to test bioremediation agents for toxicity if the product contains surfactants or other ingredients that may be harmful to the environment.

*Proposed Revisions:* The Agency is proposing an acute toxicity testing protocol for bioremediation agents to include both fresh and saltwater. The Agency will use these testing results to determine listing eligibility on the Schedule. The proposed testing protocols for bioremediation agents are detailed in Appendix C. The proposed acute toxicity test protocol for bioremediation agents is based on EPA's protocol, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters for Freshwater and Marine Organisms*. The Agency proposes to require bioremediation agents be tested for acute toxicity for the product alone using fresh water species *Ceriodaphnia dubia* and *Pimephales promelas*, and saltwater species *Americamysis bahia* and *Menidia beryllina*. The concentration of test product causing 50% lethality to the test organisms (LC<sub>50</sub>) lower and upper 95% confidence intervals (LCL<sub>95</sub> and UCL<sub>95</sub>) are calculated at the end of the exposure period. To be listed on the Schedule, bioremediation agents must demonstrate

an acute lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval greater than 10 ppm in either fresh or salt water, consistent with the acute toxicity thresholds proposed for dispersants. EPA's toxicity classification scheme<sup>59</sup> classifies LC<sub>50</sub> values ranging from 10 ppm to 100 ppm as slightly toxic, and values above 100 ppm substances are considered practically nontoxic to aquatic organisms. By proposing this threshold level the Agency is establishing an adequate safety margin without being overly restrictive. The Agency requests comments on the proposed acute toxicity methods (or any others available) and thresholds for bioremediation agents.

Similarly to surface washing agents, the Agency is not proposing provisions for conducting acute toxicity tests with bioremediation agent-oil mixtures. The Agency is requesting comment on the need for acute toxicity tests conducted with bioremediation agents-reference oil mixtures. A protocol for preparation of product/oil mixtures for toxicity testing is available for review in the docket.

#### (3) Listing of Non-Proprietary Nutrients

The Agency recognizes there may be oil discharge situations where it is determined that the addition of nutrients in the form of salts of nitrogen, phosphorus and potassium (*i.e.*, fertilizers) to stimulate or enhance bioremediation may be the most effective and environmentally favorable mitigation method. However, non-proprietary commercially available formulations of nutrients are not specifically listed on the Schedule, even though as nutrient additives they are subject to Subpart J requirements. Currently, an OSC may only authorize the use of an agent not listed on the Schedule when its use is necessary to prevent or substantially reduce a hazard to human life. Further, RRTs are not currently able to preauthorize the use of nutrients unless they are listed on the Schedule. To address these concerns, the Agency proposes to include on the Schedule a specific list of the following generic non-proprietary nutrients: Ammonium nitrate, ammonium phosphate, sodium nitrate, potassium nitrate, urea, sodium triphosphate (or tripolyphosphate), sodium phosphate, potassium phosphate (mono- or dibasic), triple super phosphate, or any combination thereof. For these non-proprietary commercial nutrients, the Agency believes there is no need for submission of readily available

<sup>57</sup> Haines, J.R., E.J. Kleiner, K.A. McClellan, K.M. Koran, E.L. Holder, D.W. King, and A.D. Venosa. 2005. "Laboratory evaluation of oil spill bioremediation products in salt and freshwater systems." *J. Ind. Microbiol. Biotechnol.* 32:171-185.

<sup>58</sup> Blenkinsopp, S, G Sergy, Z Wang, MF Fingas, J Foght and DWS Westlake. 1995. Oil spill bioremediation agents-Canadian efficacy test protocols. Proceedings of the 1995 International Oil Spill Conference, Feb 27-March 2, Long Beach, CA. American Petroleum Institute, Washington, DC. pp. 91-96.

<sup>59</sup> USEPA 2010. [http://www.epa.gov/oppefed1/ecorisk\\_ders/toera\\_analysisisco.htm#Ecotox](http://www.epa.gov/oppefed1/ecorisk_ders/toera_analysisisco.htm#Ecotox).



information. Thus, the proposal requires no technical product data or further action on the part of a manufacturer prior to authorizing their use in response to a discharge event. However, this listing will be limited to products commonly formulated entirely of those mineral nutrients and synthetically-derived urea listed. For products that may contain additional proprietary components or have unique proprietary formulations, the requirement for the submitter to provide the toxicity and efficacy data under the bioremediation agent category will apply.

The Agency considered proposing a definition of "fertilizer" to specifically capture the listed bioremediation agents proposed to be exempted. However, fertilizer is a term commonly used to describe organic nutrients, which may contain substances that do not play a role in mitigating oil discharges, and that may have deleterious effects on the environment. Some non-proprietary fertilizers, for example, comprised of activated sewage sludge or manure, could contain unwanted pathogens and trace metals that could further stress the environment if used in response to an oil discharge. Thus, instead of an exemption based on a fertilizer definition, EPA proposes to restrict the exemption under the bioremediation category only to those nutrients listed above in order to avoid any confusion. The Agency requests comments on this approach.

#### (e) Solidifier Testing and Listing Requirements

The proposal defines solidifiers as substances that cause oil to become a coherent mass, preventing oil from dissolving or dispersing into the water column, and which are collected and recovered from the environment. For this agent category, the Agency proposes to revise the toxicity testing protocol and to establish a listing threshold for toxicity in § 300.915(e). Although solidifiers are intended to be removed from the environment, the proposed revisions and new toxicity listing threshold respond to concerns regarding the general increase in the use of chemical and biological agents as tools available for oil discharge responses.

#### (1) Solidifier Efficacy

There are currently no efficacy testing requirements in the NCP Subpart J for solidifiers to determine listing eligibility on the Schedule. These agents would have been captured by the current MOSCA agent category, which currently has no efficacy testing requirements, and which the Agency is proposing to eliminate. While the Agency is aware of

existing protocols to determine the effectiveness of solidifiers, its focus has been on reviewing the protocols for dispersants and bioremediation agents, given that their specific process for affecting the oil allows them to be left in the environment, whereas solidifiers are intended for removal from the environment. Therefore, the Agency is not proposing an efficacy testing methodology for solidifiers, but is requesting comment on available methodologies and/or performance criteria (e.g., buoyancy) it can consider.

#### (2) Solidifier Toxicity

**Current Requirements:** The current NCP Subpart J requires solidifiers to be tested for saltwater species toxicity. Solidifier toxicity is tested following the dispersant toxicity testing protocols in Appendix C to part 300 of the NCP.

**Proposed Revisions:** The proposal revises the toxicity testing requirements for solidifiers, including the testing protocol. While the Agency currently provides the toxicity testing results to the OSC to assist in authorization of use determinations, it is proposing to use these results to determine listing eligibility on the Schedule.

The proposed revisions to the testing protocols for solidifiers are detailed in Appendix C to part 300. The proposed acute toxicity test protocol for solidifiers is based on EPA's protocol, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters for Freshwater and Marine Organisms*. The Agency proposes to require solidifiers be tested for acute toxicity for the product alone using fresh water species *Ceriodaphnia dubia* and *Pimephales promelas*, or saltwater species *Americamysis bahia* and *Menidia beryllina*, or both, depending on the intended product use. The concentration of test product causing 50% lethality to the test organisms (LC<sub>50</sub>) and lower and upper 95% confidence intervals (LCI<sub>95</sub> and ULCI<sub>95</sub>) are calculated at the end of the exposure period. To be listed on the Schedule, solidifiers must demonstrate an acute lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval greater than 10 ppm in either fresh or salt water, consistent with the acute toxicity thresholds proposed for dispersants. EPA's toxicity classification scheme<sup>60</sup> considers LC<sub>50</sub> values ranging from 10 ppm to 100 ppm as slightly toxic and values above 100 ppm substances practically nontoxic to aquatic organisms. By proposing this threshold level, the Agency is

establishing an adequate safety margin without being overly restrictive. The Agency requests comments on the proposed acute toxicity methods (or any others available) and thresholds for solidifiers.

Similarly to surface washing agents, the Agency is not proposing provisions for conducting acute toxicity tests with solidifiers-oil mixtures. The Agency is requesting comment on the need for acute toxicity tests conducted with solidifiers-reference oil mixtures. A protocol for preparation of product/oil mixtures for toxicity testing is available for review in the docket.

#### (3) Limitations

The Agency is proposing a conditional use listing for solidifiers. The proposal specifies that the listing of these products is limited to use only in those water environments for which the product was tested and for which it met the listing threshold criteria. The Agency recognizes that products may yield effective results in certain environments and not in others. Products that may be effective in freshwater may not necessarily be so in saltwater, and vice versa. The Agency is proposing this limitation to allow product manufacturers the flexibility to select which environment the product is to be tested and could be authorized for use, either saltwater, fresh water, or both. Therefore, the product would be listed and may only be authorized for use in those water environments for which it was tested and for which it met the listing criteria.

#### (f) Herding Agent Testing and Listing Requirements

The proposal defines herding agents as substances that are used to control the spreading of oil across the water surface. For this agent category, the Agency proposes to revise the toxicity testing protocol and to establish a listing threshold for toxicity in § 300.915(f). While these agents are intended to be removed from the environment, the proposed revisions and new toxicity listing threshold respond to concerns regarding the general increase in the use of chemical and biological agents as tools available for oil discharge responses.

Currently, there is a test requirement for distinguishing surface collecting agents from other chemical agents (§ 300.915(c)(9), *Test to Distinguish Between Surface Collecting Agents and Other Chemical Agents*). Because the proposal eliminates surface collecting agents as a category and redefines herding agents to better reflect its specific process for affecting the oil, and

<sup>60</sup> USEPA 2010. [http://www.epa.gov/oppefed1/cecorisk\\_ders/toera\\_analysis\\_eco.htm#Ecotox](http://www.epa.gov/oppefed1/cecorisk_ders/toera_analysis_eco.htm#Ecotox).

given that the agents will need to be identified in order for the required testing to be submitted, the Agency is eliminating this requirement.

#### (1) Herding Agent Efficacy

There are currently no efficacy testing requirements in the NCP Subpart J for herding agents to determine listing eligibility on the Schedule. These agents would have been captured by the current surface collecting agent category, which currently has no efficacy testing requirements, and which the proposal eliminates. The Agency is not proposing an efficacy testing methodology for herding agents, but is requesting comment on available methodologies and/or performance criteria it can consider.

#### (2) Herding Agent Toxicity

*Current Requirements:* The current NCP Subpart J requires herding agents to be tested for saltwater species toxicity. Herding agent toxicity is tested following the dispersant toxicity testing protocols in Appendix C to part 300 of the NCP.

*Proposed Revisions:* The proposal revises the toxicity testing requirements for herding agents, including the testing protocol. While the Agency currently provides the toxicity testing results to the OSC to assist in authorization of use determinations, we are proposing to use these results to determine listing eligibility on the Schedule.

The proposed revisions to the testing protocols for herding agents follow the same procedures as described for surface washing agents and are detailed in Appendix C to part 300. The acute toxicity test protocol for herding agents is based on EPA's protocol, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters for Freshwater and Marine Organisms*. The Agency proposes to require herding agents to be tested for acute toxicity for the product alone using fresh water species *Ceriodaphnia dubia* and *Pimephales promelas*, or saltwater species *Americanysis bahia* and *Menidia beryllina*, or both, depending on the intended product use. The concentration of test product causing 50% lethality to the test organisms (LC<sub>50</sub>) and lower and upper 95% confidence intervals (LCI<sub>95</sub> and ULCI<sub>95</sub>) are calculated at the end of the exposure period. To be listed on the Schedule, herding agents must demonstrate an acute lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval greater than 10 ppm in either fresh or salt water, consistent with the acute toxicity thresholds proposed for

dispersants. EPA's toxicity classification scheme<sup>61</sup> considers LC<sub>50</sub> values ranging from 10 ppm to 100 ppm as slightly toxic and values above 100 ppm substances practically nontoxic to aquatic organisms. By proposing this threshold level, the Agency is establishing an adequate safety margin without being overly restrictive. The Agency requests comments on the proposed acute toxicity methods (or any others available) and thresholds for herding agents.

Herding agents form a monolayer of surfactants on the water surface that result in the contraction of the oil slick.<sup>62 63</sup> Herding agent composition may include hydrocarbons, fluorosurfactants and/or silicone-based surfactants, which suggests that a stock solution prepared using a WAF procedure similar to solidifiers may be appropriate. The Agency is requesting comment on the procedure for preparing the stock solution for herding agents for the acute toxicity tests proposed in Appendix C. Any alternative procedure for preparing the stock solution must include an appropriate rationale in order for the Agency to be able to consider it for final action.

Similarly to surface washing agents, the Agency is not proposing provisions for conducting acute toxicity tests with herding agents-oil mixtures. The Agency is requesting comment on the need for acute toxicity tests conducted with herding agents-reference oil mixtures. A protocol for preparation of product/oil mixtures for toxicity testing is available for review in the docket.

#### (3) Limitations

The Agency is proposing a conditional use listing for herding agents. The proposal specifies that these products may only be used in those water environments for which the product was tested and for which it met the listing threshold criteria. The Agency recognizes that products may yield effective results in certain environments and not in others. Products that may be effective in freshwater may not necessarily be so in saltwater, and vice versa. The Agency is proposing this limitation to allow product manufacturers the flexibility to select which environment the product is

to be tested and could be authorized for use, either saltwater, fresh water, or both. Therefore, the product would be listed and may only be authorized for use in those water environments for which it was tested and for which it met the listing criteria.

#### (g) Sorbent Requirements

The proposal defines sorbents as inert, insoluble substances that readily absorb and/or adsorb oil or hazardous substances. Sorbents would exclude those contaminated with substances that would interact with the environment beyond their absorption/adsorption capabilities (e.g., an invasive species). The proposed definition states that sorbents are generally collected and removed from the environment and may be used in their natural bulk form, or as manufactured products in particulate form, sheets, rolls, pillows, or booms. The list of sorbent materials in the proposed definition includes: Natural organic substances (e.g., feathers, cork, peat moss, and cellulose fibers such as bagasse, corncobs, and straw); (2) inorganic/mineral compounds (e.g., volcanic ash, perlite, vermiculite, zeolite, clay); and (3) synthetic compounds (e.g., polypropylene, polyethylene, polyurethane, polyester). The Agency proposes to identify a list of known sorbent materials, and make it publicly available so that emergency responders can be aware and make use of such sorbents on oil discharges. The Agency is also proposing a process for submitters to request to include other products as sorbents if they can certify they meet the inert, insoluble criteria.

*Current Requirements:* Sorbents are currently not listed on the Schedule, but rather a list characterizing these materials is included in section 300.915(g). The current rule allows the OSC to request written certification from the manufacturers that their particular sorbent product is comprised solely of those materials identified in the rule. When a sorbent consists of materials that are not specifically listed in the current rule, the Agency issues written certifications, and sometimes requires testing and evaluation for possible listing on the Schedule.

*Proposed Revisions:* The Agency proposes to provide a Sorbent Product List that includes certain publicly available non-proprietary sorbent materials. The Agency also proposes to allow submitters to request that other products be included in this list as sorbents if they provide the technical information required, including data to support the claim that their product meets the sorbent definition. The Agency recognizes that a sorbent

<sup>61</sup> USEPA 2010. [http://www.epa.gov/oppefed1/ecorisk\\_ders/toera\\_analysis\\_eco.htm#Ecotox](http://www.epa.gov/oppefed1/ecorisk_ders/toera_analysis_eco.htm#Ecotox).

<sup>62</sup> Buist, I., Potter, S., Nedwed, T., (2011) Herding Agents to Thicken Oil Spills in Drift Ice for In Situ Burning: New Developments, IOSC, <http://ioscproceedings.org/doi/abs/10.7901/2169-3358-2011-1-230>.

<sup>63</sup> Buist, I., Nedwed, T., (2011) Using Herders for Rapid In Situ Burning Of Oil Spills on Open Water, IOSC, <http://ioscproceedings.org/doi/abs/10.7901/2169-3358-2011-1-231>.

material may consist of one or more substances not specifically identified in the proposed non-proprietary listing. For products consisting of one or more substances not specifically identified in the proposed listing, the Agency proposes submission requirements that include general and product characterization information required under paragraphs (a)(1) through (8), and paragraph (a)(13) of proposed § 300.915. These include name and contact information, identification as manufacturer or designated agent, product name/brand/trademark(s), contact information for primary distributors or importers, product SDS, conditions for product storage, product shelf life, and product label samples. The proposal would additionally require the certification statements required under paragraphs (a)(14)(iv), (a)(15), and (a)(16) of proposed § 300.915. The submitter would be required to certify that the product does not contain specified bacterial, fungal, or viral pathogens or opportunistic pathogens above National Ambient Water Quality Criteria lowest density values; that the product does not contain specified contaminants above National Water Quality Standards levels; and that the product does not contain any of the prohibited agents. Finally, the submitter would be required to include information to support the claim that the product meets the sorbent definition, including data such as its relative solubility and non-reactivity in fresh and/or salt water. The Agency will review the submission and make a determination to include the product as a sorbent on the Sorbent Product List. The Agency requests comments on this approach.

#### Testing Options

The current Subpart J has no toxicity or efficacy testing requirements for sorbents. There are two ASTM standards for performance testing that could be applicable to sorbents: The ASTM F716–09 *Standard Test Methods for Sorbent Performance of Absorbents*, and the ASTM F726–12 *Standard Test Method for Sorbent Performance of Adsorbents*. These methods include laboratory tests that describe the performance of these products in removing oils that are not emulsified and other floating, immiscible liquids from the surface of water. While these methods are to be individually used as a basis for comparison of either absorbents or adsorbents in a consistent manner, they are not useful for a cross comparison of absorbents with adsorbents, even though according to ASTM F716–09 all absorbents exhibit

adsorbent properties. Further, these agents are comprised of inert and insoluble materials that are generally removed from the environment after use, minimizing their potential harm. Thus, the Agency is not proposing to include efficacy or toxicity testing requirements for these agents, but is requesting comments on whether testing information should be included as part of the submission requirements for other materials or products to be included as part of the Sorbent Product List.

#### 5. Submission of Confidential Business Information (CBI)

Currently, manufacturers may assert a claim of confidential business information (CBI) for any information in their product package submissions to EPA. Typically, manufacturers claim as CBI the chemical identity (*e.g.*, chemical name and chemical abstracts number [CAS]) and concentration (weight percent) of each chemical component in the product along with information about the concentrations of those components in the product. The manufacturer may also claim certain other information in the technical product data, including technical data for sorbents, as CBI. EPA safeguards CBI information under the requirements in 40 CFR part 2, subpart B. The proposal addresses the CBI provisions for product submission under Subpart J in § 300.950.

Due to the amount of dispersant used during the Deepwater Horizon incident, and the need for the public's right-to-know about chemicals intentionally discharged into the environment, EPA wanted to make public information about the chemicals in the dispersant used, the results of air and water monitoring for these chemicals, and the concerns for human and environmental impact. A number of stakeholders wanted to know the chemical makeup of not only the dispersant used during the incident, but also of all other dispersants on the Schedule. This resulted in both a number of Freedom of Information Act (FOIA) requests and a Notice of Intent to Sue for Failure to Perform Nondiscretionary Duties under the CWA, requesting that the Agency release CBI for all dispersants on the Schedule.

EPA worked with the manufacturer for the dispersant used on the Gulf in response to the Deepwater Horizon incident to make the product chemical information publicly available (see <http://www.epa.gov/bpspill/>). The Agency also compiled a comprehensive aggregate list of the chemicals used in all listed dispersants. During this process, some manufacturers recognized

the need to make information available to the public, but also argued the need to protect proprietary business information from competitors. Companies provided information on the magnitude of resources expended to develop a product and test it for listing on the Schedule; however, they argued that disclosing the chemical components, CAS numbers, and/or concentrations for their product would allow domestic and international competitors to freely duplicate or reverse engineer their product and potentially drive them out of the market. EPA is aware that over 90% of businesses on the current Schedule are small, and that for some companies the product they developed for the Schedule is their only business. While companies could protect their product via the U.S. patent process, they would be required to release components and concentrations information, which would be made public. Manufacturers voiced concern that not only others may use that information to tailor competitive products, but that the U.S. patent process would offer no protection against international competitors.

EPA believes that when chemical agents are used on oil discharges, it is critically important for the public and all other stakeholders to have information regarding the chemicals being added to the environment, along with information about their toxicity and fate. This is particularly true for major discharge events where larger quantities of chemical or biological agents may be authorized for use. Prompt and accurate information will allow the public to evaluate and understand the potential human and environmental effects of these chemical agents. While EPA understands it is essential for companies to protect their investment in developing unique products, it is proposing limitations to what submitters are allowed to claim as CBI in an effort to balance public access to information with proprietary business needs. The proposal provides that if a company submits a product for listing on the Schedule, then it will only be allowed to claim CBI for the concentrations of all chemical components, microbiological cultures, enzymes, or nutrients; all other information submitted to EPA for listing a product on the Schedule will not be considered CBI and will be made public. While providing confidential treatment for the concentrations of product components, the proposal allows public access to the identity of chemical components and relevant health and environmental effects

information. All other information required for a product submission (e.g., company data, distributors, general product properties, recommended use procedures, the product category, contaminants, production capacities, product testing data) would already be publicly available for commercial products, and would not constitute proprietary business information or provide a business advantage. The Agency requests comments on whether this approach safeguards against duplication or reverse engineering of products by competitors and whether other information in Subpart J should be considered as CBI.

Alternatively, the Agency considered maintaining the current approach of allowing CBI claims for any information in their submission. When the incident arises where affected stakeholders or the public wants access to specific information, the Agency would request that the company substantiate its claims and make a determination whether to honor the claim or release the information to the public as provided in 40 CFR part 2, subpart B. The Agency also considered modifying the current approach by making the component identity and concentration information public without further notice or action for major discharge events or SONS, or for events where a given amount of chemical or biological agent would be allowed for use. Finally, another option for modifying the current approach would be to allow manufacturers to waive CBI claims only for certain chemical components for monitoring purposes (e.g. manufacturer identifies a "marker" component as a condition for listing) was also considered. The Agency is rejecting the current approach because it does not believe that, even with the modifications considered, it offers the appropriate balance between the public interest and business needs.

EPA also considered developing an aggregate list of components used in categories of chemical and biological agents for public disclosure. For example, a list of all the chemicals used in listed dispersants, a separate list for those substances used in surface washing agents, etc. This would allow information to be disassociated with specific products and protected from reverse engineering or duplication of products by competitors, while providing public access. The concern with this approach is how to update the aggregate list for new products without potentially revealing the components added to the list for that new product. Further, in the event of a major discharge or SONS, the interest will be for information and monitoring data

specific to the product being used. Consequently, EPA is not adopting this approach, but requests comments on these and other options to handle CBI while balancing all interests.

#### 6. Addition of a Product to the Schedule

The proposal establishes the requirements for submitters to request a product be listed on the Schedule in § 300.955. It provides administrative information, such as the address where to submit the package, as well as details of the requirements for a complete submission package. Additionally, it addresses how a submitter may request a listing determination review and the requirements when there are changes in a listed product. Finally, the proposal addresses the process the Agency will follow to review all new submissions, requests for review of decisions and product changes, as well as how it will transition from the current Schedule to a new one that reflects the new and amended testing and data requirement.

*Submission.* The proposal updates the address where the package is to be submitted.

*Package contents.* The proposal specifies a complete package must include, as follows:

- A company letter certifying all testing was conducted on representative product samples at a nationally or internationally accredited laboratory, that it was conducted in accordance with all technical rule requirements, and that all test results and product technical data and information reported are true and accurate;

- A numbered Table of Contents showing all required information and data submitted;

- All required data and information (both general and product category specific) in the order the requirements appear in the rule; and

- A separate inner envelope labeled: "CONFIDENTIAL BUSINESS INFORMATION—TO BE OPENED BY THE PRODUCT SCHEDULE MANAGER ONLY", if applicable.

Because of their intended function in responding to oil discharges, products listed on the Schedule will certainly impact the environment. It is important that the information provided by the submitter is true and accurate, as it serves as the basis for evaluating those potential environmental impacts. The Agency believes that it is appropriate for the submitter to be held accountable for the technical data and information provided to make these listing determinations. Thus, the proposal requires the submitter to certify the accuracy of the information submitted, and will reject any submission that is

determined to be incomplete or non-compliant, misleading, or inaccurate.

The requirements for a Table of Contents and for the information to be organized as it appears in the regulation are intended to make the Agency review process as efficient as possible. These requirements will assist the Agency in conducting a quick and accurate review, both during the transition period, as well as for future submissions, by generally simplifying the review process.

While the Agency needs to process packages containing information claimed to be CBI with additional safeguards, it is the responsibility of the submitter to ensure that this information comes to the Agency clearly identified as such. Therefore, the proposal requires a separate and clearly marked envelope for CBI to ensure proper handling.

*EPA Review.* The proposal maintains most of the existing Agency process for reviewing product submissions. A revision to the current process increases the number of days allowed for the Agency to complete its product review from the current 60 days to 90 days from the date of receipt. This proposed change considers the additional technical data and information proposed to be required, as well as the Agency's past experience with submission packages based on the current requirements.

EPA will first review the package for completeness and compliance with all data and information requirements and will contact the submitter to verify information, or to request clarification or additional information, including a product sample, as necessary. The Agency will make product listing determinations based on a technical evaluation of all data and information submitted, any relevant information on impacts or potential impacts of the product or any of its components on human health or the environment, and on the intended use of the product. The Agency reserves the right to make a determination on whether the product will be listed, and under which category. For products that may meet more than one category (e.g., a product that meets both the bioremediation and dispersant agent criteria), the Agency requests comments on whether there should be any product listing limitations beyond those identified for each individual product category. Within the 90-day timeframe, the Agency will notify the submitter, in writing, of its decision to either list the product on the Schedule and under which category or categories, or of its decision and supporting rationale to reject the submission. Submitters may

revise submission packages to address test results, data, or information deficiencies and resubmit them. Because the Agency will need a complete set of data and technical information to make a listing determination, the 90-day review time period will start anew once a complete package is resubmitted.

*Request for review of decision.* The proposal is not substantively changing the process for a submitter to request that the Agency review its determination on a product. If the Agency rejects a product for listing on the Schedule, the proposal continues to allow for a submitter to appeal to the EPA Administrator to review its determination to reject the product listing. The proposal maintains the requirement that such a request be in writing, within 30 days of receipt of the written notification of EPA's decision not to list the product on the Schedule. The request to review the Agency's determination must include a clear and concise statement with supporting facts and technical analysis that demonstrates why the submitter believes the Agency's assessment of the product was incorrect. The proposal allows the Administrator to request additional information or a meeting opportunity. Within 60 days of receipt of any such request, or within 60 days of receipt of any requested additional information, the proposal requires the Administrator or her designee to notify the submitter in writing of the review decision, maintaining the current timeframe.

*Changes to a product listing.* The Agency proposes to revise the provisions for notification of changes to a product listing. Submitters must notify EPA in writing within 30 days of any changes to the general product information submitted for listing on the Schedule so the OSCs have timely updated information. The proposal revises the notification requirement to include details of the specific changes to information submitted under § 300.915(a)(1) through (8) and § 300.915(a)(19) through (21) for a product on the Schedule, including the reasons for such changes and the supporting data and information, and maintains the provision allowing EPA to request additional information and clarification regarding these changes. For any changes to the chemical components and/or their concentrations, the proposed revisions would require retesting of the product according to the requirements for the product category, and the resubmission of a complete new package for a new review and consideration for a listing determination by the Agency of the

reformulated product. While the Agency currently has and is retaining the option of requiring additional testing, it believes that when the chemical components or concentrations of a product change, an automatic retesting requirement is merited. The Agency believes this requirement is appropriate when the identity of the product itself changes; the only way of evaluating the potential effects of these changes on the efficacy and toxicity of a new product formulation is to retest it. The Agency considered whether it was necessary to explicitly provide the flexibility to waive this requirement under extraordinary circumstances (*i.e.*, a SONS event). However, OSCs already have broad authority to use agents in areas impacted or threatened by a release or discharge, whether the agent is identified or not on the Schedule, to prevent or substantially reduce an immediate threat to human life. In addition, the Agency considered whether there is a chemical concentration threshold that could accommodate minor adjustments to a product. For example, a producer may make a slight variation in a product formula to account for a food grade or technical grade chemical component. Such a threshold might be that if concentration changes vary by no more than 1%, no retesting is necessary. However, the Agency has no basis for such a threshold and requests comment on this approach with appropriate technical details. Because of this, and the concern for the potential impact reformulated products may have on the environment, the Agency is proposing to require retesting whenever the chemical components or concentrations of a product change. However, the Agency is requesting comments on whether it should require the retesting of all products that have a change in composition, even if the only change is of chemical components that may be considered inert.

*Transitioning Listed Products from the Current Schedule to the New Schedule.* The Agency believes it important that products on the current Schedule continue to be available during the transition period to a new Schedule that reflects the amended requirements. During this transition period, all products on the current Schedule will remain conditionally listed and the Agency will rely on it for planning and response activities. Because of the proposed revisions to test protocols and listing criteria, and because of the additional test requirements, the Agency proposes that all products currently on the Schedule

be retested, and that the new data and information be submitted to the Agency for reevaluation of the current listings within 24 months of the effective date of a final rule. The Agency believes this provides adequate time for submitters to prepare and submit new packages to EPA and for the Agency to review and make decisions on these products. For a product to be transitioned from the current Schedule to the new Schedule, manufacturers would be required to submit a new, complete package according to the amended test and listing criteria, and EPA would need to make a favorable finding to list the product on the new Schedule, either as currently listed or with modifications. Products on the current Schedule for which a new submission is not received, or that upon review of their submissions do not meet the revised listing criteria, would be removed from the Schedule at the end of the 24-month transition period. While the Agency is reviewing the new submission packages, planning and response authorities are encouraged to consider those products that based on existing data would meet the revised listing criteria. While the existing data may be limited and likely developed with different protocols, considering the new requirements in selecting products for planning and response activities would provide an increased level of environmental protection. The Agency notes, however, that this is not a requirement, and that the proposal would allow any product on the current Schedule to be authorized for use following the current protocols, until the 24-month transition period is over. The Agency is requesting comments on the practicability of this transition process, and whether the 24-month period allows sufficient time for retesting of products on the current Schedule, and for EPA to review and make listing determinations on the submissions for the new Schedule. The Agency also requests comments on potential R&D costs of modifying existing products to meet the new requirements that could be incurred during this transition.

#### 7. Mandatory Product Disclaimer

The current requirements provide that the listing of a product on the Schedule does not constitute approval or endorsement of the product. To avoid possible misinterpretation or misrepresentation, the Agency currently requires that any label, advertisement, or technical literature that refers to the placement of the product on the Schedule must either reproduce in its entirety EPA's written notification that it will add the product to the Schedule

currently in § 300.920(a)(2) or (b)(2), or include the disclaimer currently set forth in § 300.920(e). It remains the Agency's position that listing a product on the Schedule does not constitute approval or endorsement of that product, nor a recommendation of its use. The Agency continues to believe that it is important to avoid any possible misinterpretation or misrepresentation of this policy. Thus, the requirement for a disclaimer to be included on any label, advertisement, or technical literature for the product is maintained. However, the proposal removes the alternative to reproduce in its entirety EPA's written notification that it will add the product to the Schedule currently in § 300.920(a)(2) or (b)(2). The Agency believes it will be able to update the Schedule list within a reasonable timeframe given the advances in information technology, and thus the option of producing the EPA letter of notification for a product listing should no longer be necessary. The Agency is proposing to modify the disclaimer language as follows:

*[PRODUCT NAME] is listed on the National Contingency Plan (NCP) Product Schedule. This listing does NOT mean that EPA approves, recommends, licenses, or certifies the use of [PRODUCT NAME] on an oil discharge. This listing means only that data have been submitted to EPA as required by Subpart J of the NCP. Only a Federal On-Scene Coordinator (OSC) may authorize use of this product according to the NCP.*

The proposed revisions set forth in § 300.965 are intended to clarify that the use of these products is conditional to OSC authorization following the requirements set forth under the NCP regulations. The disclaimer language must continue to be conspicuously displayed in its entirety, and must be fully reproduced on all product literatures, labels, and electronic media, including Web site pages. As discussed in the next section, a product can be removed from the Schedule if the submitter does not comply with the disclaimer requirements, or makes any improper attempt to demonstrate the approval or endorsement of a product. The Agency requests comments on the proposed revisions.

#### 8. Removal of a Product From the Schedule

Products that are not properly used in the field may cause harm to human health and the environment, and may constitute violations of the CWA, and other federal, state, or local laws. Misleading, inaccurate, or incorrect statements within a product submittal package or within language that refers to the listing of a product on the Schedule

may result in their improper or incorrect use. Falsification of federal documents, unsupported toxicity or efficacy claims, submission of incorrect product composition or use information, or withholding technical product data are some examples of these acts. To minimize potential misuse of listed products, the Agency believes it is appropriate to further clarify the criteria for the removal of a product from the Schedule. In § 300.970 the proposal specifically includes, but does not limit, as causes for removal from the Schedule: Any misleading, inaccurate, or incorrect statements within the product submission to EPA or to any person or private or public entity regarding the composition or use of the product to remove or control oil discharges, including on labels, advertisements, or technical literature; any alterations to the chemical components, concentrations, or use conditions of the product without proper notification to EPA as required by § 300.955(e); the failure to print the disclaimer provided in § 300.965 on all labels, advertisements, or technical literature; or any new or previously unknown relevant information concerning the impacts or potential impacts of the product to human health or the environment. It also establishes a process for removal if the Agency obtains evidence of cause for removal. EPA would notify the submitter in writing, at the address of record, of its reasons for removal of the product from the Schedule. The proposal would allow for an appeals process similar to the one set forth for listing determinations. Appeals must be received within 30 days of receipt of EPA's removal notification and must contain a clear and concise statement with supporting facts and technical analysis demonstrating why the submitter believes EPA's decision was incorrect. Written notification from the Administrator will be sent to the submitter within 60 days of any appeal, or within 60 days of receipt of any requested additional information. However, if no appeal is received within the 30 days of receipt of EPA's removal notification, the product would be delisted without further notice. The Agency requests comments on the proposed clarification of criteria for removal of products from the Schedule, and on the associated appeals process.

#### 9. Appendix C to Part 300

The Agency is proposing to revise the current Appendix C—*Swirling Flask Dispersant Efficacy Test, Revised Standard Dispersant Toxicity Test, and Bioremediation Agent Efficacy Test* as

Appendix C—*Requirements for Product Testing Protocols and Summary Test Data: Dispersant Baffled Flask Efficacy and Toxicity Tests; Standard Acute Toxicity Test for Bioremediation Agents, Surface Washing Agents, Herding Agents, and Solidifiers; and Bioremediation Agent Efficacy Test*. The proposed revisions reflect the proposed new and revised testing protocols for listing agents on the Schedule. The details of the technical changes and rationale are discussed for each agent in section V.C.4 of this preamble—Data and Information Requirements for Product Schedule Listing. The appendix reflects the proposed technical considerations and listing requirements. The Agency is requesting comment on the protocols and their technical rationale. The Agency is also requesting comment on its organization and ease of use.

#### 10. Appendix E to Part 300

The 1994 revisions to the NCP established Appendix E, *Oil Spill Response*, which separates the oil spill response requirements of the NCP from the hazardous substance release requirements (59 FR 47414). The purpose of creating this appendix was to compile general oil discharge response requirements into one document to aid responsible parties and responders with their duties under the national response system. The Agency's intent was to provide guidance, and not to alter in any way the meaning or policy stated in other sections or subparts of the NCP. However, some minor variations between the Appendix E provisions and the analogous provisions of the NCP rule language were necessary to ensure that the appendix addressed only oil discharges; hazardous substance releases continue to be addressed in the NCP rule but are not addressed in Appendix E.

The Agency proposes to remove Appendix E. While having all of the information pertaining to oil discharges compiled in one location may offer useful guidance, it is not necessary that this guidance be codified as a regulatory appendix to the NCP. The provisions in the appendix do not alter any NCP requirement; however, they do contain variations from the main NCP requirements in order to have the appendix be relevant solely to oil discharges. While EPA carefully reviewed the appendix and the relevant sections of the NCP at the time it was established to ensure consistency in policy, instructions, guidance and requirements, there were intentional minor variations. These may result in having responses to oil discharges

subject to two sets of what appear to be potentially conflicting requirements, causing unnecessary confusion.

Because all requirements in Appendix E are part of the NCP, any revisions to the NCP necessitate revisions to this appendix. This adds burden not only for the Agency in revising and ensuring consistency, but also for the regulated community in reviewing redundant and duplicative requirements. While it may be a useful tool to have all of the oil discharge specific requirements in one location, the Agency has reconsidered its position and believes that this is more appropriately achieved through a separate guidance document, one that does not codify duplicative regulatory requirements. The Agency requests comments on the proposal to remove Appendix E from the NCP regulation, and whether it should continue to offer similar guidance through other formats.

## VI. Summary of Proposed Rule Provisions

This section summarizes the proposed changes to 40 CFR parts 110 and 300. Subpart J has been renumbered to include new, consolidated, and revised sections. Some of the rule sections have been retained, removed, or moved in their entirety. The Table below provides an overview of the existing rule and proposed rule citations for a quick reference of the proposed changes.

Section 110.4, Dispersants, would be revised to link the rule with the new and amended regulatory definitions for Subpart J product categories.

Section 300.5, Definitions, would be revised to include new, amended, and deleted definitions.

Subpart J—Use of Dispersants, Chemicals, and Bioremediations Agents, heading would be revised to reflect new and amended regulatory definitions for product categories.

Section 300.900, General, paragraphs (a) and (c) would be revised to reflect new and amended regulatory definitions for product categories.

Section 300.905, NCP Product Schedule, would be removed.

Section 300.910, Authorization of Use, would be revised and new paragraphs added to clarify planning and preauthorization responsibilities.

- Paragraph (a) would be revised to clarify the requirements, including process, responsibilities, and factors to consider for preauthorization; and add new requirements for preauthorization plan review, concurrence, and withdrawal procedures.

- Paragraph (b) would be revised to clarify the requirements for using a listed product not addressed by a

preauthorization plan and add new parameters for use considerations.

- Paragraph (c) would be revised to clarify the requirements for authorizing the use of burning agents by an OSC for authorized in-situ burns.

- Paragraph (d) would be revised to clarify the exception requirements and add specific time frames for notification of continued agent use.

- Paragraph (e) would be revised to expand the prohibition to include nonylphenol (NP) or nonylphenol ethoxylates (NPEs) as components of chemical or biological agents.

- Paragraph (f) would be revised to add new regulatory requirements for agent storage and use. Existing paragraph (f) requirements would be moved to new paragraph (g), Supplemental Testing, Monitoring, and Information.

- New paragraph (g), Supplemental Testing, Monitoring, and Information, would revise the regulatory text to clarify the requirements for supplemental testing, monitoring and information.

- New paragraph (h), Recovery of Oil and Agents from the Environment, would add regulatory requirements for recovery of oil and agents from the environment.

- New paragraph (i), Reporting of Agent Use, would add regulatory requirements for notification of agent use on an oil discharge.

New section 300.913, Monitoring the Use of Dispersants, would add regulatory requirements for monitoring certain prolonged surface and subsurface use of dispersants.

Section 300.915, Data Requirements, would be revised to consolidate general submission requirements applicable to all product categories. The section would be restructured to include new testing and listing requirements for specific product categories.

- Paragraph (a), Dispersants, would be revised to consolidate general testing and listing requirements from existing paragraphs (a), (b), (d), and (f). The paragraph would include revisions and new requirements for the identification of and testing for all product categories designated for listing. Existing paragraph (a) requirements specific to dispersants would be moved to new section 300.915(b), Dispersant Testing and Listing Requirements. The paragraph would also be revised to add new toxicity and efficacy testing requirements, limitations for use, and new criteria for listing a dispersant to the Schedule. Existing paragraph (b) would be moved to new paragraph (c), Surface Washing Agent Testing and Listing Requirements.

- Paragraph (b), Surface Washing Agents, would be moved to new paragraph (c), Surface Washing Agent Testing and Listing Requirements. The paragraph would be revised to add new toxicity and efficacy testing requirements, limitations for use, and new criteria for listing a surface washing agent to the Schedule. Existing paragraph (c), Surface Collecting Agents, would be deleted.

- Paragraph (d), Bioremediation Agents, would be revised to add new toxicity and efficacy testing requirements, limitations for use, and new criteria for listing a bioremediation agent to the Schedule. Existing paragraphs (d)(9) and (10) were moved to new paragraph (a), General Product Information.

- Paragraph (e) would be revised to add new regulatory requirements for submission and listing of a solidifier. Existing paragraph (e), Burning Agents, would be deleted.

- Paragraph (f) would be revised to add new toxicity testing requirements, limitations of use, and criteria for listing a herding agent on the Schedule. Existing paragraph (f), Miscellaneous Oil Spill Control Agents, would be deleted.

- Paragraph (g), Sorbents, would be revised to add new exceptions for listing a sorbent to the Schedule.

Section 300.920, Addition of Products to Schedule, would be moved to new section 300.955, Addition of a Product to the Schedule.

- Paragraph (a) would be revised to include submission instructions for all product categories. Existing paragraphs (a)(1) through (3) regulatory text specific to dispersant applications would be moved to new §§ 300.915(b) and 300.955(c) and (d).

- Paragraph (b) would be revised to add new regulatory text for preparation of complete submission packages. Existing paragraph (b) regulatory text would be moved to new § 300.955(c) and (d).

- Paragraph (c) would be revised to add regulatory text for EPA's review of submission packages and decision criteria for listing. Existing paragraph (c) would be moved to new § 300.950, Submission of Confidential Business Information (CBI).

- Paragraph (d) would be revised to add regulatory text for requesting a listing decision review. Existing paragraph (d) would be moved to new § 300.955(e), Changes to a Listed Product.

- Paragraph (e) would be revised to add new regulatory text for notification of changes to a listed product. Existing paragraph (e) would be moved to new

§ 300.965, Mandatory Product Disclaimer.

- New paragraph (f) would add new regulatory requirements for transitioning products on the current Schedule to the new Schedule.

New § 300.950, Confidential Business Information (CBI), would revise and clarify the allowable CBI claims in a submission package.

New § 300.965, Mandatory Product Disclaimer, would clarify the regulatory text for including a disclaimer statement on all product labels and literature.

New § 300.970, Removal of a Product from the Schedule, would add basis for removal of products from the Schedule, EPA notification of decision, and appeals process.

Revised Appendix C to Part 300—Requirements for Product Testing

Protocols and Summary Test Data: Dispersant Baffled Flask Efficacy and Toxicity Tests; Standard Acute Toxicity Test for Bioremediation Agents, Surface Washing Agents, Herding Agents, and Solidifiers; and Bioremediation Agent Efficacy Test.

Removed Appendix E to Part 300—Oil Spill Response.

40 CFR PART 100 DISCHARGE OF OIL—DISTRIBUTION TABLE

Current citation	Proposed rule citation
110.4 Dispersants .....	110.4 Chemical and Biological Agents.

40 CFR PART 300—NATIONAL OIL AND HAZARDOUS SUBSTANCES POLLUTION CONTINGENCY PLAN—DISTRIBUTION TABLE

Current citations	Proposed rule citations
§ 300.5 Definitions .....	§ 300.5 Definitions.
Subpart J—Use of Dispersants and Other Chemicals .....	Subpart J—Use of Dispersants, and Other Chemical and Biological Agents.
§ 300.900 General .....	§ 300.900 General.
§ 300.900(a) .....	§ 300.900(a)
§ 300.900(c) .....	§ 300.900(c)
§ 300.905 NCP Product Schedule .....	Deleted.
§ 300.910 Authorization of Use .....	§ 300.910 Authorization for Agent Use.
§ 300.910(a) .....	§ 300.910(a) Use of Agents Identified on the Schedule on Oil Discharges Addressed by a Preauthorization Plan.
§ 300.910(b) .....	§ 300.910(b) Use of Agents Identified on the Schedule on Oil Discharges Not Addressed by a Preauthorization Plan.
§ 300.910(c) .....	§ 300.910(c) Burning Agents.
§ 300.910(d) .....	§ 300.910(d) Exceptions.
§ 300.910(e) .....	§ 300.910(e) Prohibited Agents.
§ 300.910(f) .....	§ 300.910(g) Supplemental Testing, Monitoring, and Information.
	§ 300.910(f) Storage and Use of Agents.
	§ 300.910(h) Recovery of Oil and Agents from the Environment.
	§ 300.910(i) Reporting of Agent Use.
	§ 300.913 Monitoring the Use of Dispersants.
	§ 300.913(a).
	§ 300.913(b).
	§ 300.913(c).
	§ 300.913(d).
	§ 300.913(e).
	§ 300.913(f).
§ 300.915 Data Requirements .....	§ 300.915 Data and Information Requirements for Product Schedule Listing.
§ 300.915(a)(1) through (12) Dispersants .....	§ 300.915(a)(1) through (21) General Product Information.
§ 300.915(b) Surface Washing Agents .....	§ 300.915(b) Dispersant Testing and Listing Requirements.
	§ 300.915(a)(1) through (21) General Product Information.
	§ 300.915(c) Surface Washing Agent Testing and Listing Requirements.
	Deleted.
§ 300.915(c) Surface Collecting Agents .....	§ 300.915(a)(1) through (21) General Product Information.
§ 300.915(d) Bioremediation Agents .....	§ 300.915(d) Bioremediation Agent Testing and Listing Requirements.
	Deleted.
§ 300.915(e) Burning Agents .....	Deleted.
§ 300.915(f) Miscellaneous Oil Spill Control Agents .....	Deleted.
§ 300.915(g) Sorbents .....	§ 300.915(g) Sorbent Listing Requirements.
§ 300.915(h) Mixed Products .....	Deleted.
	§ 300.915(e) Solidifier Testing and Listing Requirements, including § 300.915(a)(1) through (21) General Product Information.
	§ 300.915(f) Herding Agent Testing and Listing Requirements, including § 300.915(a)(1) through (21) General Product Information.
§ 300.920 Addition of Products to Schedule .....	§ 300.955 Addition of a Product to the Schedule.
§ 300.920(a)(1) Dispersants .....	§ 300.955(a) Submission.
	§ 300.915(b) Dispersant Testing and Listing Requirements.
§ 300.920(a)(2) .....	§ 300.955(c) EPA Review.
§ 300.920(a)(3) .....	§ 300.955(d) Request for Review of Decision.
	§ 300.955(b) Package Contents.
§ 300.920(b)(1) Surface Washing Agents, Surface Collecting Agents, Bioremediation Agents, and Miscellaneous Oil Spill Control Agents.	§ 300.955(a) Submission.



40 CFR PART 300—NATIONAL OIL AND HAZARDOUS SUBSTANCES POLLUTION CONTINGENCY PLAN—DISTRIBUTION  
TABLE—Continued

Current citations	Proposed rule citations
§ 300.920(b)(2) .....	§ 300.955(c) EPA Review.
§ 300.920(c) .....	§ 300.950 Submission of Confidential Business Information (CBI).
§ 300.920(d) .....	§ 300.955(e) Changes to a Listed Product.
	§ 300.955(f) Transitioning Listed Products from the Current Schedule to the New Schedule.
§ 300.920(e) .....	§ 300.965 Mandatory Product Disclaimer.
	§ 300.970 Removal of a Product from the Schedule.

**VII. Statutory and Executive Order Reviews****A. Executive Order 12866: Regulatory Planning and Review and Executive Order 13563: Improving Regulation and Regulatory Review**

Under Executive Order 12866 (58 FR 51735, October 4, 1993), this action is a “significant regulatory action.” This action raises novel legal or policy issues arising out of legal mandates, the President’s priorities, or the principles set forth in the Executive Order. Accordingly, EPA submitted this action to the Office of Management and Budget (OMB) for review under Executive Orders 12866 and 13563 (76 FR 3821, January 21, 2011) and any changes made in response to OMB recommendations have been documented in the docket for this action.

In addition, EPA prepared an analysis of the potential costs and benefits associated with this action. This analysis is contained in the *Regulatory Impact Analysis for Proposed Revisions to the National Oil and Hazardous Substances Pollution Contingency Plan Regulations*. A copy of the analysis is available in the docket for this action and the analysis is briefly summarized here.

The Agency expects the proposed rule would not cause a significant economic impact on a substantial number of small businesses. The total incremental costs are estimated as \$667,610 to \$694,343 annually at 3% and 7% (\$2011) annualization rates, respectively. The benefits of this action are assessed qualitatively and include, for example, greater clarity of regulatory requirements, as well as less toxic products. The resulting ratio of compliance cost to annual sales revenue for the proposed rule for existing and new product manufacturers would be less than one percent in most instances.

**B. Paperwork Reduction Act**

The information collection requirements in this proposed rule have been submitted for approval to the Office of Management and Budget

(OMB) under the *Paperwork Reduction Act*, 44 U.S.C. 3501 *et seq.* The Information Collection Request (ICR) document prepared by EPA has been assigned EPA ICR number 1664.10. You can find a copy of the ICR in the docket for this rule, and it is briefly summarized here. The ICR supporting this proposed rule is largely self-implementing. The information collection is to ensure that: (1) The Agency has the necessary information to make Schedule listing determinations specific to the different product categories; (2) product use by owners or operators of facilities or vessels, or response personnel, in response to oil discharges is performed in accordance with all applicable requirements; and (3) the Agency can verify compliance as needed. Section 300.950 of the NCP contains provisions for confidentiality.

EPA has carefully considered the burden imposed upon the regulated community by the proposed regulations. EPA believes that the activities required are necessary and, to the extent possible, has attempted to minimize the burden imposed. The minimum requirements specified in the proposed rule are intended to ensure that, when needed, products are used properly in the field to respond to an oil discharge in a manner protective of human health and the environment.

**Respondents/affected entities:** Manufacturers of oil spill mitigating agents (products)/Oil spill responsible parties.

**Respondent’s obligation to respond:** Mandatory (40 CFR Part 300, Subpart J).

**Estimated number of respondents:** 65.

**Frequency of response:** Initially.

**Total estimated burden:** 721 hours (per year). Burden is defined at 5 CFR 1320.3(b).

**Total estimated cost:** \$584,504 (per year), includes \$575,400 operation & maintenance costs.

An agency may not conduct or sponsor, and a person is not required to respond to, a collection of information unless it displays a currently valid OMB control number. The OMB control

numbers for EPA’s regulations in 40 CFR are listed in 40 CFR part 9.

Submit your comments on the Agency’s need for this information, the accuracy of the provided burden estimates, and any suggested methods for minimizing respondent burden to the EPA using the docket identified at the beginning of this rule. You may also send your ICR-related comments to OMB’s Office of Information and Regulatory Affairs via email to [oria\\_submissions@omb.eop.gov](mailto:oria_submissions@omb.eop.gov), Attention: Desk Officer for EPA. Since OMB is required to make a decision concerning the ICR between 30 and 60 days after receipt, OMB must receive comments no later than February 23, 2015. The EPA will respond to any ICR-related comments in the final rule.

**C. Regulatory Flexibility Act (RFA)**

The Regulatory Flexibility Act (RFA) generally requires an agency to prepare a regulatory flexibility analysis of any rule subject to notice and comment rulemaking requirements under the Administrative Procedure Act or any other statute unless the agency certifies that the rule will not have a significant economic impact on a substantial number of small entities. Small entities include small businesses, small organizations, and small governmental jurisdictions.

For purposes of assessing the impacts of the proposed rule on small entities, small entity is defined as: (1) As defined by the Small Business Administration’s (SBA) regulations at 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population of less than 50,000; or (3) a small organization that is any not-for-profit enterprise that is independently owned and operated and is not dominant in its field.

After considering the economic impacts of this proposed rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. The small entities directly regulated by this proposed rule are product

manufacturers and laboratories and state and local governments that are involved in product development, testing and use for oil discharge response. EPA conducted a small business analysis consistent with the Agency's 2006 small business guidance. The Agency's analysis indicates that about 95 percent of manufacturers are small businesses. In conducting the small business analysis, the agency compared the incremental annualized compliance costs to the annual sales revenue for the smallest entities. The results indicate that 90 percent of the smallest manufacturers have annualized compliance costs that are less than 1 percent of their annual sales revenue, and that no manufacturers are expected to have incremental costs that exceed 3 percent of annual sales. The small business analysis is available for review in the Regulatory Impact Analysis (RIA). Therefore, we have determined that this proposed rule does not have a significant impact on a substantial number of small entities.

Nonetheless, EPA has tried to reduce the impact of this rule on small entities in developing the regulatory requirements that balance the costs and burden, while addressing the environmental protection concerns. We continue to be interested in the potential impacts of this proposed rule on small business entities and welcome comments on the issues related to such impacts.

#### *D. Unfunded Mandates Reform Act*

This action contains no Federal mandates under the provisions of Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), 2 U.S.C. 1531–1538 for State, local, or tribal governments or the private sector. This proposed rule imposes no new enforceable duty on any state, local or tribal governments or the private sector. UMRA excludes from the definition of "Federal intergovernmental mandate" and "Federal private sector mandate" duties that arise from conditions of Federal assistance. UMRA generally excludes from the definition of "Federal intergovernmental mandate" duties that arise from participation in a voluntary Federal program. UMRA also excludes from the definition of "Federal private sector mandate" duties that arise from participation in a voluntary Federal program. Since the decision on whether to request that a product be included on the Schedule is voluntary, the Agency has determined that this proposed rule is not subject to the requirements of sections 202 or 205 of UMRA. This action is also not subject to the requirements of section 203 of UMRA

because it contains no regulatory requirements that might significantly or uniquely affect small governments.

#### *E. Executive Order 13132: Federalism*

This action does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. This proposal does not alter the general procedures already defined in the NCP of how state, local, and federal agencies cooperate in responding to oil spills and how to consult with the OSC and RRT when considering the use of products on the Schedule. Thus, Executive Order 13132 does not apply to this action. In the spirit of Executive Order 13132, and consistent with EPA policy to promote communications between EPA and State and local governments, EPA specifically solicits comment on this proposed action from State and local officials.

#### *F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments*

Subject to Executive Order 13175 (65 FR 67249, November 9, 2000), EPA may not issue a regulation that has tribal implications, that imposes substantial direct compliance costs, and that is not required by statute, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by tribal governments, or EPA consults with tribal officials early in the process of developing the proposed regulation and develops a tribal summary impact statement.

EPA has concluded that this action may have tribal implications. However, it will neither impose substantial direct compliance costs on tribal governments, nor preempt Tribal law, similarly to the effect on states. EPA will be consulting with tribal officials as it develops this regulation to permit them to have meaningful and timely input into its development. Consultation will include conference calls, webinars, and meetings with interested tribal representatives to ensure that their concerns are addressed before the rule is finalized. In the spirit of Executive Order 13175 and consistent with EPA policy to promote communications between EPA and tribal governments, EPA specifically solicits comment on this proposed rule from tribal officials.

#### *G. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks*

EPA interprets EO 13045 (62 FR 19885, April 23, 1997) as applying only to those regulatory actions that concern health or safety risks, such that the analysis required under section 5–501 of the EO has the potential to influence the regulation. This action is not subject to EO 13045 because it does not establish environmental standards, such as limits on levels of pollutants in the water, that are intended to directly mitigate health or safety risks.

#### *H. Executive Order 13211: Actions That Significantly Affect Energy Supply, Distribution or Use*

This proposed rule is not a "significant energy action" as defined in Executive Order 13211, "Actions Concerning Regulations that Significantly Affect Energy Supply, Distribution, or Use" (66 FR 28355, May 22, 2001) because it is not likely to have a significant adverse effect on the supply, distribution, or use of energy. The proposal focuses on maintaining the availability of certain response tools that can be considered when responding to oil discharges, minimizing any potential adverse impacts from their use, and resulting in greater overall environmental protection. Thus, the proposed rule would not cause reductions in the supply or production of oil, fuel, coal, or electricity; nor would it result in increased energy prices, increased cost of energy distribution, or an increased dependence on foreign supplies of energy.

#### *I. National Technology Transfer and Advancement Act*

Section 12(d) of the National Technology Transfer and Advancement Act of 1995 ("NTTAA"), Public Law 104–113, 12(d) (15 U.S.C. 272 note) directs EPA to use voluntary consensus standards in its regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (e.g., materials specifications, test methods, sampling procedures, and business practices) that are developed or adopted by voluntary consensus standards bodies. NTTAA directs EPA to provide Congress, through OMB, explanations when the Agency decides not to use available and applicable voluntary consensus standards.

This rulemaking involves technical standards. The Agency conducted a search to identify potentially applicable

voluntary consensus standards for efficacy testing. However, we identified no such standards. Therefore, EPA developed the Baffled Flask Efficacy Test and the Bioremediation Efficacy Test required in Appendix C of this proposed rule. Voluntary consensus standards developed by ASTM are recommended for several product property data points, such as pH, flash point and pour point. The product toxicity testing relies on existing protocols that are universally accepted. EPA welcomes comments on this aspect of the proposed rulemaking and, specifically, invites the public to identify potentially-applicable voluntary consensus standards for product efficacy and to explain why such standards should be used in this regulation.

*J. Executive Order 12898:  
Environmental Justice*

Executive Order 12898, “Federal Actions to Address Environmental Justice in Minority Populations and Low-Income Populations” (59 FR 7629 (February 11, 1994)) establishes federal executive policy on environmental justice. Its main provision directs federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations and low-income populations in the United States.

EPA is committed to addressing environmental justice concerns and has assumed a leadership role in environmental justice initiatives to enhance environmental quality for all citizens of the United States. The Agency’s goals are to ensure that no segment of the population, regardless of race, color, national origin, income, or net worth bears disproportionately high and adverse human health and environmental impacts as a result of EPA’s policies, programs, and activities. In response to Executive Order 12898, EPA’s Office of Solid Waste and Emergency Response (OSWER) formed an Environmental Justice Task Force to analyze the array of environmental justice issues specific to waste programs and to develop an overall strategy to identify and address these issues (OSWER Directive No. 9200.3–17). To address this goal, EPA conducted a qualitative analysis of the environmental justice issues under this proposed rule.

Under the NCP, RRTs and ACs are required to address, as part of their

planning activities, the desirability of using appropriate chemical or biological agents, or other spill mitigating devices. In addition, the OSC, under authority granted by the NCP, must respond to an oil spill in a diligent and effective manner to protect human health and the environment. If chemical or biological agents are needed, the OSC must coordinate with the RRT and ACs before their use is authorized. In all cases, the RRT, ACs and OSC will address a broad array of oil spill response and mitigation issues, including the potential for environmental justice concerns. Historically, EPA has not found any evidence that the use of chemical or biological agents on the Schedule on oil spills in the U.S. has had any disproportionate effect on any environmental justice communities. However, EPA will continue to monitor the implementation of the rule to ensure the planned or actual use of chemical or biological agents has no disproportionate effect on any EJ communities.

EPA has determined that this proposed rule will not have disproportionately high and adverse human health or environmental effects on minority or low-income populations because it increases the level of environmental protection for all affected populations without having any disproportionately high and adverse human health or environmental effects on any population, including any minority or low-income population. Specifically, the proposed rule provides additional safeguards before any product can be listed on the Schedule, as well as allows OSCs and RRTs to request additional information to ensure that the use of any chemical or biological agent, or any other spill mitigating substance, in responding to oil discharges is protective of human health and the environment. This proposed rule is consistent with EPA’s Environmental Justice Strategy and the OSWER Environmental Justice Action Agenda.

**List of Subjects**

*40 CFR Part 110*

Environmental protection, Oil pollution, and Reporting and recordkeeping requirements.

*40 CFR Part 300*

Air pollution control, Area contingency planning, Bioremediation, Chemicals, Dispersants, Environmental protection, Hazardous materials, Hazardous substances, Intergovernmental relations, Natural resources, Oil spills, Oil spill mitigating

devices, Regional response teams, Sorbents, and Surface washing agents.

Dated: January 9, 2015.

**Gina McCarthy,**  
*Administrator.*

For the reasons set out in the preamble, the Environmental Protection Agency proposes to amend 40 CFR parts 110 and 300 to read as follows:

**PART 110—DISCHARGE OF OIL**

■ 1. The authority citation for part 110 continues to read as follows:

**Authority:** 33 U.S.C. 1321(b)(3) and (b)(4) and 1361(a); E.O. 11735, 38 FR 21243, 3 CFR parts 1971–1975 Comp., p. 793.

■ 2. Revise § 110.4 and its section heading to read as follows:

**§ 110.4 Chemical or biological agents.**

Addition of any chemical or biological agent, as defined in § 300.5, to oil to be discharged that would circumvent the provisions of this part is prohibited.

**PART 300—NATIONAL OIL AND HAZARDOUS SUBSTANCES POLLUTION CONTINGENCY PLAN**

■ 3. The authority citation for part 300 continues to read as follows:

**Authority:** 33 U.S.C. 1321(c)(2); 42 U.S.C. 9601–9657; E.O. 13626, 77 FR 56749, 3 CFR, 2013 Comp., p. 306; E.O. 12777, 56 FR 54757, 3 CFR, 1991 Comp., p. 351; E.O. 12580, 52 FR 2923, 3 CFR, 1987 Comp., p. 193.

**Subpart A—Introduction**

- 4. Amend § 300.5 by:
    - a. Adding in alphabetical order the definitions of “Bioaccumulation”, “Bioconcentration”, “Biodegradation”, “Biological agents”, and “Bioremediation”;
    - b. Revising the definitions of “Bioremediation agents”, “Burning agents”, “Chemical agents”, and “Dispersants”;
    - c. Adding in alphabetical order the definition of “Herding agents”;
    - d. Removing the definitions for “Miscellaneous oil spill control agent”;
    - e. Adding in alphabetical order the definition of “Products”;
    - f. Revising the definition of “Sinking agents”;
    - g. Adding in alphabetical order the definition of “Solidifiers”; and
    - h. Revising the definition of “Sorbents”
    - i. Removing the definition for “Surface collecting agents”.
    - j. Revising the definition title “Surface washing agent” and its definition;
- The revisions and additions read as follows:

§ 300.5 Definitions.

\* \* \* \* \*

Bioaccumulation is the process of accumulation of chemicals in the tissue of organisms through any route, including respiration, ingestion, or direct contact with the ambient or contaminated medium.

Bioconcentration is the accumulation of chemicals in the tissues of organisms from water alone.

Biodegradation is a process by which microorganisms metabolically decompose contaminants into biomass and simpler compounds such as carbon dioxide, water, and innocuous end products.

Biological agents are microorganisms (typically bacteria, fungi, or algae) or biological catalysts, such as enzymes, able to enhance the biodegradation of a contaminated environment.

Bioremediation is the process of enhancing the ability of microorganisms to convert contaminants into biomass and innocuous end products by the addition of materials into a contaminated environment to accelerate the natural biodegradation process.

Bioremediation agents are biological agents and/or nutrient additives deliberately introduced into a contaminated environment to increase the rate of biodegradation and mitigate any deleterious effects caused by the contaminant constituents.

Bioremediation agents include microorganisms, enzymes, and nutrient additives such as fertilizers containing bioavailable forms of nitrogen, phosphorus and potassium.

Burning agents are additives that improve the combustibility of the materials to which they are applied through physical or chemical means.

\* \* \* \* \*

Chemical agents are elements, compounds, or mixtures designed to facilitate the removal of oil from a contaminated environment and mitigate any deleterious effects. Chemical agent categories include burning agents, dispersants, herding agents, sinking agents, solidifiers, surface washing agents, and bioremediation agents that consist of nutrient additives.

\* \* \* \* \*

Dispersants are typically mixtures of solvents, surfactants, and additives that promote the formation of small droplets of oil in the water column by reducing the oil-water interfacial tension.

\* \* \* \* \*

Herding agents are substances that are used to control the spreading of the oil across the water surface.

\* \* \* \* \*

Products are chemical or biological agents manufactured using a unique composition or formulation.

\* \* \* \* \*

Sinking agents are substances deliberately introduced into an oil discharge for the purpose of submerging the oil to the bottom of a water body.

\* \* \* \* \*

Solidifiers are substances that through a chemical reaction cause oil to become a cohesive mass, preventing oil from dissolving or dispersing into the water column, and which are collected and recovered from the environment.

Sorbents are inert, insoluble substances that readily absorb and/or adsorb oil or hazardous substances, and that are not combined with or act as a chemical or biological agent. Sorbents are generally collected and recovered from the environment. Sorbents may be used in their natural bulk form, or as manufactured products in particulate form, sheets, rolls, pillows, or booms. Sorbents consist of:

(1) Natural organic substances (e.g., feathers, cork, peat moss, and cellulose fibers such as bagasse, corncobs, and straw);

(2) Inorganic/mineral compounds (e.g., volcanic ash, perlite, vermiculite, zeolite, clay); and

(3) Synthetic compounds (e.g., polypropylene, polyethylene, polyurethane, polyester).

\* \* \* \* \*

Surface washing agents are substances that separate oil from solid surfaces, such as beaches, rocks, metals, or concrete, through a detergency mechanism that lifts and floats oil for collection and recovery from the environment with minimal dissolution, dispersion, or transfer of oil into the water column.

\* \* \* \* \*

Subpart J—Use of Dispersants, and Other Chemical and Biological Agents

■ 5. Revise the heading of Subpart J as set out above.

■ 6. Amend § 300.900 by revising paragraphs (a) and (c), and by adding paragraph (d) to read as follows:

§ 300.900 General.

(a) Section 311(d)(2)(G) of the Clean Water Act (CWA) requires EPA to prepare a schedule identifying dispersants, other chemicals, other spill mitigating devices and substances if any, that may be used in carrying out the NCP; and the waters and quantities in which they may be used. This subpart establishes a schedule identifying chemical and biological agents, and procedures that, when taken

together, identify the waters and quantities in which such dispersants, other chemicals, or other spill mitigating devices and substances may be used.

\* \* \* \* \*

(c) This subpart applies to the use of chemical and biological agents as defined in Subpart A of this part, or other substances that may be used to remove, control, or otherwise mitigate oil discharges.

(d) [Reserved]

§ 300.905 [Removed]

■ 7. Remove § 300.905.

■ 8. Revise § 300.910 and the section heading to read as follows:

§ 300.910 Authorization for agent use.

Use of chemical or biological agents in response to oil discharges to waters of the U.S. or adjoining shorelines must be authorized by the OSC in accordance with the provisions of this section:

(a) Use of Agents Identified on the Schedule on Oil Discharges Addressed by a Preauthorization Plan. RRTs and Area Committees shall address in a preauthorization plan, as part of their planning activities, whether the use of chemical and biological agents listed on the Schedule on certain oil discharges is appropriate. RRTs and Area Committees shall, as appropriate, include applicable approved preauthorization plans in RCPs and ACPs. When a preauthorization plan is approved in advance for the use of certain agents under specified discharge situations, then the OSC may authorize the use of agents on the Schedule for their intended purpose without obtaining the incident specific concurrences described in paragraph (b) of this section.

(1) Preauthorization Plan Development. For discharge situations identified where such agents may be used, the preauthorization plan must specify limits for the quantities and the duration of use, and use parameters for water depth, distance to shoreline, and proximity to populated areas. In meeting the provisions of this paragraph, preauthorization plans should document how regional factors are addressed including likely sources and types of oil that might be discharged, various discharge scenarios, the existence and location of environmentally sensitive resources or restricted areas that might be impacted by discharged oil, and logistical factors including inventory, storage locations and manufacturing capability of available agents, availability of equipment needed for agent use, availability of adequately trained

operators, and means to monitor agent use in the environment.

(2) *Preauthorization Plan Approval.* The EPA representative to the RRT, the Department of Commerce and the Department of the Interior natural resource trustees and, as appropriate the RRT representative from the state(s) with jurisdiction over waters and adjoining shorelines within the preauthorization plan area shall review and either approve, approve with modification, or disapprove the preauthorization plans developed by the RRT and/or the Area Committees. Withdrawal of concurrence means the preauthorization plan becomes invalid and the authorization of use for chemical or biological agents must be performed according to paragraph (b) of this section. The RRTs and Area Committees shall address the withdrawal and the RRT shall notify the NRT of the final status of the preauthorization plan within 30 days from withdrawal.

(3) *Preauthorization Plans Reviews.* The RRT and/or the Area Committees must review, and revise as needed, preauthorization plans at least every 5 years; after a major discharge or after a Spill of National Significance (SONS); to address revisions of the Schedule; to reflect new listings of threatened and/or endangered species; and to address any other change that may impact the conditions under which the use of chemical and biological agents is preauthorized. The designated EPA RRT representative, the Department of Commerce and the Department of the Interior natural resource trustees, and the RRT representative from the state(s) with jurisdiction over the waters of the area to which a preauthorization plan applies shall review and either approve, approve with modification, or disapprove any revisions to the preauthorization plans.

(b) *Use of Agents Identified on the Schedule on Oil Discharges Not Addressed by a Preauthorization Plan.* For discharge situations that are not addressed by the preauthorization plan developed pursuant to paragraph (a) of this section, the OSC may authorize the use of appropriate chemical or biological agents identified on the Schedule for their intended purpose on an oil discharge with the concurrence of the designated EPA RRT representative and, as appropriate, the concurrence of the RRT representatives from the state(s) with jurisdiction over the waters and adjoining shorelines threatened by the release or discharge, and in consultation with the Department of Commerce and the Department of the Interior natural resource trustees. In meeting the

provisions of this paragraph, the OSC must consider and document the parameters for the use of agents including the quantities to be used, the duration of use, the depth of water, the distance to shoreline and proximity to populated areas, and should address factors such as environmentally sensitive resources or restricted areas that might be impacted, agent inventory and storage locations, agent manufacturing capability, availability of equipment needed for agent use, availability of adequately trained operators and appropriate means to monitor agent use in the environment.

(c) *Burning Agents.* For authorized in-situ burns, the OSC may authorize the use of burning agents.

(d) *Exception.* The OSC may authorize the use of any chemical or biological agent, whether it is identified or not on the Schedule, without obtaining the immediate concurrence of the designated EPA RRT representative and, as appropriate, the RRT representatives from the state(s) with jurisdiction over the waters and adjoining shorelines threatened by the release or discharge, when, in the judgment of the OSC, the use of the agent is necessary to prevent or substantially reduce a threat to human life. If an OSC authorizes the use of an agent pursuant to this paragraph, he or she shall immediately notify, and document the circumstances requiring and the reasons for use of the agent to the EPA RRT representative and, as appropriate, the RRT representatives from the affected state(s) and, the Department of Commerce/Department of the Interior natural resources trustees. Use of any agent beyond 48 hours under this exception shall be in accordance with paragraphs (a) or (b) of this section.

(e) *Prohibited Agents.* Notwithstanding paragraph (d) of this section, the OSC may not authorize the use of the following:

(1) Sinking agents, or any other chemical agent, biological agent, or any substance that acts as a sinking agent when mixed with oil; and

(2) Chemical or biological agents that have either nonylphenol (NP) or nonylphenol ethoxylates (NPEs) as components.

(f) *Storage and Use of Agents.* The OSC may authorize for use only products that are certified by the responsible party to have been stored under the conditions provided by the submitter under § 300.915(a)(6) and whose date of use does not exceed the expiration date listed on the container's label at the time of the incident. The responsible party must provide the OSC product documentation, developed in consultation with the submitter of the

product to the Schedule, prior to OSC authorization of product use affirming it has maintained its integrity, including no changes in its composition, efficacy, and toxicity. The owner or operator of a facility or vessel must ensure samples of the expired product lot are tested following the applicable testing protocols in Appendix C, and that they are representative of all storage conditions at any end user location. If testing demonstrates the expired product has maintained its integrity, the product may be used for an additional 5 years from the date of the testing described above. The responsible party, or its representative, must re-label the tested product lots and maintain test results and document all of the information under § 300.915(a)(17) and (a)(18) until used. The owner or operator of a facility or vessel must ensure the testing of re-labeled products every 5 years.

(g) *Supplemental Testing, Monitoring and Information.* The RRT may require supplementary toxicity and efficacy testing, or available data or information that addresses site, area, or ecosystem specific concerns relative to the use of a product for both planning and authorization of use. During a discharge incident, the RRT may request that the OSC require a responsible party to conduct additional monitoring associated with the use of a product. Such additional monitoring data may include supplementary toxicity and efficacy testing or submission of available data or information that addresses the discharge area or ecosystem specific concerns relative to the use of a product or that aids the OSC and/or the RRT in operational decisions.

(h) *Recovery of Agents from the Environment.* Depending on factors such as the safety of response personnel and harm to the environment, and as directed by the OSC, the responsible party shall ensure that any removal action adequately contains, collects, stores and disposes of agents that are intended to be recovered from the environment.

(i) *Reporting of Agent Use.* Unless already included in the OSC report required under § 300.165 of this part, within 30 days of completion of agent operations, the authorizing OSC shall provide the RRT the following information on chemical and biological agents used in response to an oil discharge: product name, quantity and concentration used, duration of use, locations, and any data collected and analysis of efficacy or environmental effects.

■ 9. Add § 300.913 to read as follows:

**§ 300.913 Monitoring the use of dispersants.**

As directed by the OSC, the responsible party must monitor any subsurface use of dispersant in response to an oil discharge, surface use of dispersant in response to oil discharges of more than 100,000 U.S. gallons occurring within 24 hours, and surface use of dispersant for more than 96 hours in response to an oil discharge, and submit a Quality Assurance Project Plan for approval to the OSC covering the collection of all environmental data. When these dispersant use conditions are met, and for the duration of dispersant operations, the responsible party shall:

(a) Document the characteristics of the source oil; best estimate of the oil discharge flow rate, periodically reevaluated as conditions dictate, including a description of the method, associated uncertainties, and materials; dispersant(s) product used, rationale for dispersant product choice(s) including the results of any efficacy and toxicity tests specific to area or site conditions, recommended dispersant-to-oil ratio (DOR); and the application method and procedures, including a description of the equipment to be used, hourly application rates, capacities, and total amount of dispersant needed. For subsurface discharges also document the best estimate of the discharge flow rate of any associated volatile petroleum hydrocarbons, periodically reevaluated as conditions dictate, including a description of the method, associated uncertainties, and materials.

(b) In areas not affected by the discharge of oil, collect a representative set of background water column samples following standard operating and quality assurance procedures, at the closest safe distance from the discharge as determined by the OSC and in any direction of likely transport considering surface and subsurface currents and oil properties for the variables listed below. In the dispersed oil plume, collect daily water column samples following standard operating and quality assurance procedures, at such depths and locations where dispersed oil is likely to be present and analyze for:

(1) In-situ oil droplet size distribution, including mass or volume mean diameter for droplet sizes ranging from 2.5 to 2,000  $\mu\text{m}$ , with the majority of data collected between the 2.5 and 100  $\mu\text{m}$  size;

(2) In-situ fluorometry and fluorescence signatures targeted to the type of oil discharged and referenced against the source oil;

(3) Dissolved oxygen (DO);

(4) Total petroleum hydrocarbons, individual resolvable constituents including volatile organic compounds, aliphatic hydrocarbons, monocyclic, polycyclic, and other aromatic hydrocarbons including alkylated homologs, and hopane and sterane biomarker compounds;

(5) Carbon dioxide (CO<sub>2</sub>) (subsurface only);

(6) Methane, if present (subsurface only);

(7) Heavy metals, including nickel and vanadium;

(8) Turbidity;

(9) Water temperature;

(10) pH; and

(11) Conductivity.

(c) In consultation with the OSC, and using best available technologies, characterize the dispersant effectiveness and oil distribution, considering the condition of oil, dispersant, and dispersed oil components from the discharge location;

(d) In consultation with the OSC, characterize the ecological receptors (e.g. aquatic species, wildlife, and/or other biological resources) and their habitats that may be present in the discharge area and their exposure pathways. Include those species that may be in sensitive life stages, transient or migratory species, breeding or breeding-related activities (e.g., embryo and larvae development), and threatened and/or endangered species that may be exposed to the oil that is not dispersed, the dispersed oil, and the dispersant alone. Estimate an acute toxicity level of concern for the dispersed oil using available dose/response information relevant to potentially exposed species.

(e) Immediately report to the OSC any:

(1) Deviation of more than 10 percent from the mean hourly dispersant use rate for subsurface application, based on the dispersant volume authorized for 24 hours use, and the reason for the deviation; and

(2) Ecological receptors, including any threatened or endangered species that may be exposed based on dispersed plume trajectory modeling and level of concern information.

(f) Report daily to the OSC water sampling and data analyses collected in § 300.913(b) and include:

(1) Specific hourly dispersant application rate and the total amount of dispersant used for the previous reporting period established by the OSC with concurrence from the EPA representative to the RRT;

(2) All collected data and analyses of those data within a timeframe necessary to make operational decisions (e.g.,

within 24 hours of collection), including documented observations, photographs, video, and any other information related to dispersant use, unless an alternate timeframe is authorized by the OSC;

(3) For analyses that take more than 24 hours due to analytical methods, provide such data and results within 5 days, unless an alternate timeframe is authorized by the OSC; and

(4) Estimates of the daily transport of dispersed and non-dispersed oil and associated volatile petroleum hydrocarbons, and dispersants, using the best available trajectory modeling. ■ 10. Revise § 300.915 and the section heading to read as follows:

**§ 300.915 Data and information requirements for Product Schedule listing.**

If you are submitting an application for listing a product to the Schedule, you must provide EPA the information required under § 300.955. Your submission must contain:

(a) *General Information for any Product Category.* (1) Your name, physical address, email, and telephone number;

(2) Your identity as the manufacturer of the product, a vendor, importer, or distributor of the product, and/or a designated agent acting on behalf of the manufacturer. Provide documentation of such identity;

(3) All name(s), brand(s), and/or trademark(s) under which the product is to be sold;

(4) Names, physical addresses, emails and telephone numbers of the primary distributors, vendors, importers, and/or designated agent acting on behalf of the manufacturer;

(5) A Safety Data Sheet (SDS) for the product;

(6) The maximum, minimum and optimum temperature, humidity and other relevant conditions for product storage and a brief description of the consequences to performance if the product is not stored within these limits;

(7) The anticipated shelf life of the product at the storage conditions noted in paragraph (a)(6) of this section and documentation for this determination;

(8) A sample product label for all name(s), brand(s), and/or trademark(s) under which the product is to be sold that includes manufacture and expiration dates, and conditions for storage. You may use an existing label provided it already contains the required dates and storage information;

(9) The chemical or biological agent category under which you want the product to be considered for listing on the Schedule, including detailed information on the specific process(es)

through which the product affects the oil, and the specific environment(s) (waters and/or adjoining shorelines) on which it is intended to be used. If your product meets the definition of more than one chemical or biological agent category and you want it considered for listing on the Schedule in more than one category, you must identify all applicable categories and provide the test data to meet the listing criteria appropriate to each category;

(10) Recommended product use procedures, including product concentrations, use ratios, types of application equipment, conditions for use, and any application restrictions. These procedures must address, as appropriate, variables such as weather, water salinity, water temperature, types and weathering states of oils or other pollutants, and product and oil containment, collection, recovery and disposal, and include supporting documentation and standard methods used to determine them;

(11) Environmental fate information, including any known measured data and supporting documentation, on the persistence, bioconcentration factor, bioaccumulation factor, and biodegradability of the product and all of its components in the environment;

(12) The physical/chemical properties of the product, as appropriate, and a citation for the standard methods used to determine them, including:

- (i) Physical state and appearance;
- (ii) Vapor pressure;
- (iii) Flash point;
- (iv) Pour point;
- (v) Viscosity;
- (vi) Specific gravity;
- (vii) Particle size for solid components; and
- (viii) pH.

(13) The identity and concentration of all components in the product, including each specific component name; corresponding Chemical Abstract Service (CAS) Registry Number; the maximum, minimum, and average weight percent of each component in the product; and the intended function of each component (e.g., solvent, surfactant);

(14) For products that contain microorganisms, enzymes and/or nutrients, provide the following along with a citation or a description of the methodology used to determine:

(i) The name of all microorganisms by current genus and species, including any reclassifications, and any physical, chemical, or biological technique used to manipulate the genetic composition and the weight percent of each genus in the product;

(ii) The name of all enzymes and their International Union of Biochemistry (I.U.B.) number(s); Enzyme Classification (EC) code numbers; the source of each enzyme; units; and specific oil-degrading activity;

(iii) The name(s), maximum, minimum, and average weight percent of the nutrients contained in the product; and

(iv) Certification, including data, methodology, and supporting documentation, indicating that the product does not contain, at levels that exceed the National Ambient Water Quality Criteria lowest density value, bacterial, fungal, or viral pathogens or opportunistic pathogens including, but not limited to: enteric bacteria such as *Salmonella*, fecal coliforms, *Shigella*, or Coagulase positive *Staphylococci*, and Beta Hemolytic *Streptococci* and enterococci.

(15) Certification, including data, methodology, and supporting documentation, indicating that the product does not contain, at levels above National Water Quality Standards lowest acute value for aquatic life:

(i) Arsenic, cadmium, chromium, copper, lead, mercury, nickel, vanadium, zinc, and any other heavy metal reasonably expected to be in the product;

- (ii) Cyanide;
- (iii) Chlorinated hydrocarbons;
- (iv) Pesticides;
- (v) Polychlorinated Biphenyls (PCBs); and
- (vi) Polynuclear aromatic hydrocarbons (PAHs).

(16) Certification, including data, methodology, and supporting documentation, indicating that the product does not contain any of the prohibited agents identified in § 300.910(e);

(17) Information about the laboratory that conducted the required tests, including:

- (i) Name of the laboratory, address, contact name, email, and phone number; and
- (ii) The national and/or international accreditations held by the laboratory.

(18) All test data and calculations, including:

- (i) Raw data and replicates, including positive controls;
- (ii) Notes and observations collected during tests;
- (iii) Calculated mean values and standard deviations;
- (iv) Reports, including a summary of stock solution preparation;
- (v) Source and preparation of test organisms;
- (vi) Test conditions; and
- (vii) Chain of custody forms.

(19) An estimate of the annual product production volume, the average and maximum amount that could be produced per day, and the time frame needed to reach that maximum production rate (days);

(20) Recognition received from EPA's Design for the Environment (DfE) if applicable; and

(21) International product testing or use data or certifications, if available, informing the performance capabilities or environmental benefits of the product.

(b) *Dispersant Testing and Listing Requirements*—(1) *Dispersant Efficacy test and listing criteria*. Test the dispersant product for efficacy using the Baffled Flask Test (BFT) method in Appendix C to part 300. To be listed on the Schedule, the dispersant must demonstrate for each oil and temperature a Dispersant Effectiveness (DE) at the 95% lower confidence level (LCL<sub>95</sub>) greater than or equal to:

- (i) 55% for Intermediate Fuel Oil 120 (IFO-120) at 5 °C;
- (ii) 65% for IFO-120 at 25 °C;
- (iii) 70% for Alaska North Slope (ANS) crude oil at 5 °C; and
- (iv) 75% for ANS at 25 °C.

(2) *Dispersant Toxicity tests and listing criteria*. Use the methods specified in Appendix C to part 300 to test the dispersant alone, the dispersant mixed with ANS, and the dispersant mixed with IFO-120 for acute toxicity, using *Americamysis bahia* and *Menidia beryllina*. Use the methods specified in Appendix C to part 300 to test the dispersant alone for developmental toxicity using a sea urchin assay and for sub-chronic effects using *Americamysis bahia* and *Menidia beryllina*. To be listed on the Schedule, the lethal concentration for 50% of the test species (LC<sub>50</sub>) at the lower 95% confidence interval for all acute toxicity tests must be greater than 10 ppm; the inhibition concentration for 50% of the test species (IC<sub>50</sub>) at the lower 95% confidence interval must be greater than 10 ppm; and the sub-chronic No Observed Effect Concentration (NOEC) must be equal to or greater than 1 ppm.

(3) *Limitations*. Product listing would be for use only in saltwater environments.

(c) *Surface Washing Agent Testing and Listing Requirements*—

(1) *Surface Washing Agent Efficacy test and listing criteria*. To be listed on the Schedule, using a recognized standard methodology, the surface washing agent must meet an efficacy of greater than or equal to 30% in either fresh or saltwater or both depending on the intended product use.

(2) *Surface Washing Agent Toxicity test and listing criteria.* Using the toxicity test methodology in Appendix C to part 300, test the surface washing agent for acute toxicity against fresh water species *Ceriodaphnia dubia* and *Pimephales promelas*, or saltwater species *Americamysis bahia* and *Menidia beryllina*, or both, depending on the intended product use. To be listed on the Schedule, the surface washing agent must demonstrate an LC<sub>50</sub> at the lower 95% confidence interval of greater than 10 ppm in either fresh or saltwater for all tested species.

(3) *Limitations.* Based on testing, product listing would be for use only in the fresh and/or saltwater environments for which it was tested and for which it met the efficacy and toxicity listing criteria.

(d) *Bioremediation Agent Testing and Listing Requirements—(1) Bioremediation Agent Efficacy test and listing criteria.* To be listed on the Schedule, a bioremediation agent must successfully degrade both alkanes and aromatics as determined by gas chromatography/mass spectrometry (GC/MS) in salt or fresh water or both, depending on the intended product use, following the test method specified in Appendix C to part 300. The percentage reduction of total alkanes (aliphatic fraction) from the GC/MS analysis must be greater than or equal to 95% at day 28, based on the ninety-fifth percentile Upper Confidence Limit (UCL<sub>95</sub>) for both salt and freshwater. The percentage reduction of total aromatics (aromatic fraction) must be greater than or equal to 70% at day 28 for saltwater and greater than or equal to 40% for freshwater based on the UCL<sub>95</sub>.

(2) *Bioremediation Agent Toxicity test and listing criteria.* The bioremediation agent must be tested for acute toxicity in saltwater, freshwater or both, depending on the intended product use, following the method specified in Appendix C to part 300. To be listed on the Schedule, the bioremediation agent must demonstrate an LC<sub>50</sub> at the lower 95% confidence interval of greater than 10 ppm in either fresh or saltwater for all tested species.

(3) *Limitations.* Based on testing, product listing would be for use only in the fresh and/or saltwater environments for which it was tested and for which it met the efficacy and toxicity listing criteria.

(4) *Exceptions.* If the product consists solely of: ammonium nitrate, ammonium phosphate, ammonium sulfate, calcium ammonium nitrate, sodium nitrate, potassium nitrate, synthetically-derived urea, sodium triphosphate (or tripolyphosphate),

sodium phosphate, potassium phosphate (mono- or dibasic), triple super phosphate, potassium sulphate, or any combination thereof, no technical product data are required, are generically listed as non-proprietary nutrients on the Schedule, and no further action is necessary.

(e) *Solidifier Testing and Listing Requirements.* (1) Solidifiers must be tested for acute toxicity in saltwater, freshwater or both, depending on the intended product use, following the method specified in Appendix C to part 300. To be listed on the Schedule, the solidifier must demonstrate an LC<sub>50</sub> at the lower 95% confidence interval of greater than 10 ppm in either fresh or saltwater for all tested species.

(2) *Limitations.* Based on testing, product listing would be for use only in the fresh and/or saltwater environments for which it was tested and for which it met the toxicity listing criteria.

(f) *Herding Agent Testing and Listing Requirements.* (1) Herding agents must be tested for acute toxicity in saltwater, freshwater, or both, depending on the intended product use, following the method specified in Appendix C to part 300. The herding agent must demonstrate an LC<sub>50</sub> at the lower 95% confidence interval of greater than 10 ppm in either fresh or saltwater for all tested species.

(2) *Limitations.* Based on testing, product listing would be for use only in fresh and/or saltwater environments for which it was tested and for which it met the toxicity listing criteria.

(g) *Sorbent Requirements.* Known sorbent materials and products will be identified on a publicly available Sorbent Product List for the use of such products when responding to an oil discharge as follows:

(1) For sorbent products that consist solely of the following materials, or any combination thereof, no technical data are required and no further action is necessary for use as a sorbent:

(i) Feathers, cork, peat moss, and cellulose fibers such as bagasse, corncobs, and straw;

(ii) Volcanic ash, perlite, vermiculite, zeolite, and clay; and

(iii) Polypropylene, polyethylene, polyurethane, and polyester.

(2) If the product consists of one or more natural organic substances, inorganic/mineral compounds, and/or synthetic compounds not specifically identified in paragraph (g)(1) of this section but you believe the product meets the definition of a sorbent then, as applicable under § 300.955(a) and (b), you must submit the following information for consideration for listing

it as a sorbent on the Sorbent Product List:

(i) The information required under paragraphs (a)(1) through (8), and paragraph (a)(13) of this section;

(ii) The certifications required under paragraphs (a)(14)(iv), (a)(15), and (a)(16) of this section; and

(iii) Information, including data, to support the claim your product meets the sorbent definition under § 300.5.

#### § 300.920 [Removed]

■ 11. Remove § 300.920.

■ 12. Add § 300.950 to subpart J to read as follows:

#### § 300.950 Submission of Confidential Business Information (CBI).

(a) Except as provided in paragraph (b) of this section, all product information submitted to EPA as required under § 300.915 will be disclosed to the public.

(b) You may only claim the concentration and the maximum, minimum, and average weight percent of each chemical component or microorganism in your product, as identified in § 300.915(a)(13) or (14), to be CBI. EPA will handle such claims in accordance with 40 CFR part 2, subpart B.

(1) You must make your CBI claim at the time you submit your information to EPA to be listed on the Schedule.

(2) You must redact the CBI from all submitted information but include the CBI separately with your submission package. Clearly identify and mark the information as “Confidential Business Information” and place it in a separate inner envelope in your submission package labeled with “CONFIDENTIAL BUSINESS INFORMATION—TO BE OPENED BY THE PRODUCT SCHEDULE MANAGER ONLY.”

■ 13. Add § 300.955 to subpart J to read as follows:

#### § 300.955 Addition of a product to the Schedule.

(a) *Submission.* Submit your complete package to: U.S. Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Mail Code: 5104A, Room 1448, William J. Clinton North, Washington, DC 20460, Attention: Product Schedule Manager.

(b) *Package contents.* Your package shall include, in this order:

(1) A cover letter on company letterhead signed and dated by you certifying that:

(i) All testing was conducted on representative product samples;

(ii) Testing was conducted at a nationally or internationally accredited laboratory in accordance with the



methods specified in Appendix C to part 300, and other applicable methods as appropriate; and

(iii) All test results and product technical data and information are true and accurate.

(2) A numbered Table of Contents showing the information and data submitted under § 300.915(a) through (g);

(3) All required data and information arranged in the same order as specified in § 300.915(a) through (g); and

(4) A separate envelope containing Confidential Business Information as specified in § 300.950(b), if applicable.

(c) *EPA Review.* EPA shall, within 90 days of receiving a submission package:

(1) Review the package for completeness and compliance with all data and information requirements in §§ 300.915, 300.950, and this section, verify information, and request clarification or additional information as necessary;

(2) Make a product listing determination based on a technical evaluation of all data and information submitted, relevant information on impacts or potential impacts of the product or any of its components on human health or the environment, and the intended use of the product. EPA reserves the right to make a determination on whether the product will be listed, and under which category; and

(3) Notify you, in writing, of its decision to list the product on the Schedule and in which category or categories, or of its decision and supporting rationale to reject the submission. If your submission is rejected:

(i) You may revise the submission package to address test results, data, or information deficiencies and resubmit it.

(ii) EPA's 90-day review will not start until a complete package is resubmitted.

(d) *Request for review of decision.* If your product is rejected for listing on the Schedule, you may request that the EPA Administrator review the determination. Your request must be in writing within 30 days of receipt of notification of EPA's decision not to list the product on the Schedule. Your request must contain a clear and concise statement with supporting facts and technical analysis demonstrating why you believe EPA's decision was incorrect.

(1) The EPA Administrator or designee may request additional information from you and may offer an opportunity for you to meet with EPA.

(2) The EPA Administrator or his designee will notify you in writing of

the decision within 60 days of receipt of your request, or within 60 days of receipt of requested additional information.

(e) *Changes to a product listing.* You must notify EPA in writing within 30 days of any changes to information submitted under § 300.915(a)(1) through (8) and § 300.915(a)(19) through (21) for a product on the Schedule. In the notification, you must detail the specific changes, the reasons for such changes and supporting data and information. EPA may request additional information and clarification regarding these changes. If you change the chemical components and/or concentrations, you must retest the reformulated product according to the requirements for the product category and submit a complete new package for a review and consideration for listing on the Schedule by EPA.

(f) *Transitioning Listed Products from the Current Schedule to the New Schedule.* All products on the current Schedule as of [EFFECTIVE DATE OF FINAL RULE] will remain conditionally listed until [DATE 24 MONTHS FROM THE EFFECTIVE DATE OF FINAL RULE] at which time all products that have not submitted and been listed in the new Schedule based on the amended test and listing criteria will be removed. Your product will be transitioned from the current Schedule to the new Schedule prior to [DATE 24 MONTHS FROM THE EFFECTIVE DATE OF FINAL RULE] after you submit a new, complete package according to the amended test and listing criteria and EPA makes a favorable finding to list the product on the new Schedule.

■ 14. Add § 300.965 to subpart J to read as follows:

**§ 300.965 Mandatory product disclaimer.**

The listing of a product on the Schedule does not constitute approval or recommendation of the product. To avoid possible misinterpretation or misrepresentation, any label, advertisement, or technical literature for the product must display in its entirety the disclaimer shown below. The disclaimer must be conspicuous and must be fully reproduced on all product literatures, labels, and electronic media including Web site pages.

*DISCLAIMER* [PRODUCT NAME] is listed on the National Contingency Plan (NCP) Product Schedule. This listing does NOT mean that EPA approves, recommends, licenses, or certifies the use of [PRODUCT NAME] on an oil discharge. This listing means only that data have been submitted to EPA as required by Subpart J of the NCP. Only

a Federal On-Scene Coordinator (OSC) may authorize use of this product according to the NCP.

■ 15. Add § 300.970 to subpart J to read as follows:

**§ 300.970 Removal of a product from the Schedule.**

(a) The EPA Administrator may remove your product from the Schedule for reasons including, but not limited to:

(1) Misleading, inaccurate, or incorrect statements within the product submission to EPA or to any person or private or public entity regarding the composition or use of the product to remove or control oil discharges, including on labels, advertisements, or technical literature; or

(2) Alterations to the chemical components, concentrations, or use conditions of the product without proper notification to EPA as required by § 300.955(e); or

(3) Failure to print the disclaimer provided in § 300.965 on all labels, advertisements, or technical literature, or

(4) New or previously unknown relevant information concerning the impacts or potential impacts of the product to human health or the environment.

(b) EPA will notify you in writing, at your address of record, of its reasons for deciding to remove the product from the Schedule. If EPA receives no appeal from you in 30 days, the product will be removed from the Schedule without further notice to you.

(c) You may appeal the decision to remove your product from the Schedule within 30 days of receipt of EPA's notification. Your appeal must contain a clear and concise statement with supporting facts and technical analysis demonstrating why you believe EPA's decision was incorrect. The EPA Administrator will notify you in writing of his decision within 60 days of your appeal, or within 60 days of receipt of any requested additional information.

■ 16. Revise Appendix C to Part 300 and appendix heading to read as follows:

**Appendix C to Part 300—Requirements for Product Testing Protocols and Summary Test Data: Dispersant Baffled Flask Efficacy and Toxicity Tests; Standard Acute Toxicity Test for Bioremediation Agents, Surface Washing Agents, Herding Agents, and Solidifiers; and Bioremediation Agent Efficacy Test.**

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#### Standard Operating Procedures Tables

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1.0 *Applicability and Scope*. This Appendix establishes laboratory protocols required under Subpart J (Use of Dispersants and Other Chemical and Biological Agents) of 40 CFR part 300 (National Oil and Hazardous Substances Pollution Contingency Plan) to make listing determinations for the Product Schedule. The protocols apply, based on product type, to dispersants, bioremediation agents, surface washing agents, herding agents, and solidifiers as defined in Subpart A (Introduction) of 40 CFR part 300.

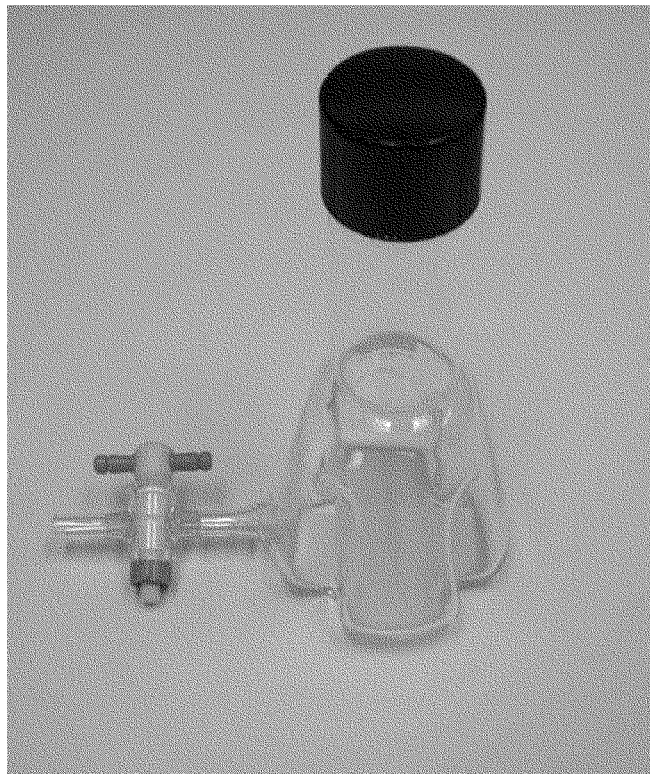
#### 2.0 Baffled Flask Dispersant Efficacy Test (BFT)

2.1 *Summary*. This laboratory protocol establishes procedures to evaluate the degree to which a product effectively disperses oil

spilled on the surface of seawater, using a modified 150-mL screw-cap trypsinizing flask (an Erlenmeyer flask with baffles) with a glass and Teflon® stopcock near the bottom to allow removal of subsurface water samples without disturbing the surface oil layer. The efficacy of a dispersant is measured using two types of oils (Intermediate Fuel Oil 120 and Alaska North Slope) at two temperatures (5 °C and 25 °C). Six replicates are required at each condition with two method blank replicates at each temperature. A layer of oil is placed on the surface of artificial seawater, and the dispersant is added to the slick at a dispersant:oil ratio (DOR) of 1:25 (4%) by volume. A standard orbital shaker table provides turbulent mixing at a speed of 250 revolutions per minute (rpm) for 10 minutes, immediately after which it is maintained stationary for 10 minutes to allow non-dispersed oil to rise to the water's surface. An undisturbed water sample is removed from the bottom of the flask through the stopcock, extracted with dichloromethane (DCM), and analyzed for oil content by UV-visible absorption spectrophotometry at wavelengths ranging between 340 and 400 nm.

2.2 *Apparatus*. All equipment must be maintained and calibrated per standard laboratory procedures.

2.2.1 *Modified Trypsinizing Flask*. A modified 150 mL glass screw-capped Erlenmeyer flask with baffles (e.g., Wheaton No. 355394 or equivalent) fitted with a 2 mm bore Teflon® stopcock and glass tubing, the center of which is no more than 1.3 cm from the bottom, as shown in Figure 1.



**Figure 1. A Baffled Trypsinizing Flask**

2.2.2 *Orbital Shaker Table.* An orbital shaker table with a variable speed control unit capable of maintaining 250 rpm. The orbital diameter must be approximately 1.0 inch (2.5 cm) +/- 0.1 inch (0.25 cm).

2.2.3 *Spectrophotometer.* A UV-visible spectrophotometer capable of measuring absorbance between 340 and 400 nm (e.g., Shimadzu UV-1800, Agilent 8453, or equivalent). Use standard transmission-matched quartz 10-mm path length rectangular cells with PTFE cover for absorbance measurements.

2.2.4 *Glassware.* Including: 25-ml graduated mixing cylinders (a graduated cylinder with a ground glass stopper); 50- and 100-ml graduated cylinders; 125-ml separatory funnels with Teflon stopcocks; 10 ml volumetric flasks; 30 ml crimp style glass serum bottles; 1-, 2-, 5-ml pipettes; other miscellaneous laboratory items.

2.2.5 *Micropipettor.* Use a micropipettor capable of dispensing 4 µL of dispersant and 100 µL of oil (e.g., Brinkmann Eppendorf repeater pipettor with 100 µL and 5 mL syringe tip attachments or equivalent).

2.2.6 *Syringes.* 25-, 100-, 250-, 1000-, 2500-, 5000- µl gas-tight syringes.

2.2.7 *Constant temperature rooms or incubators to hold the shaker at 5 °C and 25 °C.*

2.2.8 *Analytical Balance.*

2.2.9 *Chemical fume hood.*

### 2.3 Reagents

2.3.1 *Artificial seawater.* Use the artificial seawater GP2 formulation shown in Table 1 of this Appendix.

2.3.2 *Test oils.* Use the two EPA standard reference oils, Alaska North Slope oil (ANS) and Intermediate Fuel Oil 120 (IFO 120). To obtain these oils at no charge (except for a minimal shipping fee), see the instructions at <http://www.epa.gov/emergencies/content/ncp/index.htm>. Selected properties are summarized in Table 2 of this Appendix.

2.3.3 *Dichloromethane (DCM) (also known as methylene chloride), pesticide quality.*

2.3.4 *Positive Control Dispersant.* Dispersant sample with a known, reproducible efficacy. To obtain this control sample at no cost (except for shipping), see the instructions at <http://www.epa.gov/emergencies/content/ncp/index.htm>.

### 2.4 Container Handling and Storage

2.4.1 *Glassware.* If the glassware has been used with oil before, rinse with DCM to remove as much of the oil adhering to the sides of the flask as possible; waste DCM may be used. Soak in warm water with detergent and individually wash with bristled brushes. First rinse with tap water, then follow with two de-ionized water rinses. Dry either on a rack or in a 110 °C drying oven). After drying, rinse with fresh DCM (use sparingly).

2.4.2 *Serum bottles and other non-volumetric glassware.* Bake for at least 4 hours in a muffle furnace at 450 °C.

### 2.5 Calibration Curve for the UV-visible spectrophotometer

2.5.1 *Stock Standard Solution Preparation.* Stock standard solution concentrations are based on the mass measurements after each addition and density determinations of the oil/dispersant/DCM solution using a density bottle or a 1-ml gas tight syringe. An example calculation is given in Table 3 of this Appendix according to the following equation:

$$\text{theoretical concentration, } \frac{\text{mg}}{\text{mL}} = \frac{\text{mass of oil, g} \times 1000 \text{ mg/g}}{\text{total mass, g} / \rho_{\text{solution, g/mL}}} \quad (\text{Equation 1})$$

Use the reference oils and the specific dispersant being tested for a particular set of experimental test runs. Prepare the stock solution of dispersant-oil mixture in DCM, starting with 2 ml of the oil, then adding 80 µl of the dispersant followed by 18 ml of DCM. Two sets of standards are needed, one for each oil and dispersant combination.

2.5.2 *Six-point Calibration Curve.* For each reference oil, add specific volumes of its stock standard solution (given in Table 4 of this Appendix) to 30 ml of artificial seawater in a 125 ml separatory funnel. Extract the oil/water mixture with triplicate 5 ml volumes of DCM. Follow each DCM addition by 15 seconds of vigorous shaking, carefully releasing the initial pressure inside the separatory funnel by partially removing the glass stopper inside a fume hood after the first few shakes. Then, allow a 2-minute stationary period for phase separation for each extraction. Drain the extracts into a 25-ml graduated mixing cylinder. Release any entrained bubbles of DCM from the water layer by sideways shaking of the funnel. Use precaution not to drain water into the DCM extract as it can affect the absorbance readings. Adjust the final volume of the collected extracts to 20 mL in the mixing cylinder using DCM. Determine specific masses for oil concentrations in the standards as volumes of oil/dispersant solution multiplied by the concentration of the stock solution. An example calculation is given in Table 4 of this Appendix. Two calibration curves are needed, one for each oil and dispersant combination.

2.6 *Sample Preparation and Testing.* See section 2.7 of this Appendix for a detailed description of the spectrophotometer's linear calibration procedure.

2.6.1 Six replicates of each oil and test dispersant are required at each temperature plus two additional tests of method blanks (artificial seawater without oil and dispersant), one at each temperature. A completed test consists of 26 baffled flask tests (a total of six replicates for each of two reference oil/test dispersant combinations at two temperatures (5 °C and 25 °C), plus two method blanks).

2.6.2 A positive control run of 6 replicates of a dispersant with known dispersion efficacy (see 2.3.4 above for how to obtain) is prepared with both oils at both temperatures to verify the protocol is being correctly performed. The lab must certify that a positive control was successfully conducted within a year of any testing submitted for a dispersant listing.

2.6.3 Attach a 3-inch length of Teflon tubing to the stopcock of each of the 150-mL baffled flasks. Add 120 mL of artificial seawater to each flask. Put screw cap on flasks and place them at the appropriate temperature (either 5 °C or 25 °C) for equilibration.

2.6.4 Calibrate and adjust the shaker table to 250 ± 10 rpm.

2.6.5 Prepare and time separately each baffled flask. Sequentially add 100 µL of oil and 4 µL of dispersant to the flask layering them onto the center of the seawater to give a dispersant-to-oil ratio (DOR) of 1:25. Avoid any oil or dispersant splashing on the flask walls, as it may reduce efficacy or cause errors in the calculated results. Discard the sample and repeat the setup if: (1) Any oil or dispersant splashing occurs during the additions, or (2) the dispersant contacts the water first rather than the oil.

2.6.6 For the oil, fill the tip of the pipettor, using a wipe to remove any oil from

the sides of the tip. Holding the pipettor vertically, dispense several times back into the reservoir to ensure that the oil flows smoothly. Insert the syringe tip vertically into the baffled flask, and let the bottom of the pipettor rest on the neck of the flask. Slowly and carefully dispense the oil one time onto the center of the water's surface. IFO 120 takes longer to drip, and ANS will splash if dispensed too fast. The remainder of the oil can either be returned to the oil bottle or set aside for use in the next test flask. Note to 2.6.6: If a Brinkmann Eppendorf repeater pipettor is used for dispensing the oil, attach a 5-ml syringe tip, and set the dial to 1.

2.6.7 For the dispersant, use the same procedure as for the oil to dispense onto the center of the oil slick surface. As the dispersant first contacts the oil, it will usually push the oil to the sides of the flask. Replace the screw cap onto the flask.

Note to 2.6.7: If a Brinkmann Eppendorf repeater pipettor is used for dispensing the dispersant, attach a 100-µL syringe tip, and set the dial to 2.

2.6.8 Carefully place flask securely onto the shaker and agitate for 10 ± 0.25 minutes at 250 ± 10 rpm.

2.6.9 Remove the flask from the shaker table and allow a stationary, quiescent period of 10 ± 0.25 minutes to allow undispersed and/or re-coalesced oil droplets to refloat to the surface.

2.6.10 Carefully open the screw cap, then the stopcock at the bottom, and discard the first several mL of seawater into a waste beaker to remove non-mixed water-oil initially trapped in the stopcock tubing. Collect a volume slightly greater than 30-mL into a 50-mL graduated cylinder. Adjust the collected volume to the 30-mL mark by

removing excess with a disposable glass Pasteur pipette. A web-like emulsion may form at the solvent/water interface during the water sample extraction. Avoid pulling any emulsion phase into the DCM extract as it may cloud the DCM-extract leading to error.

2.6.11 Transfer the water-oil sample from the graduated cylinder into a 125-mL glass separatory funnel fitted with a Teflon stopcock.

2.6.12 Add 5 mL DCM to the separatory funnel. Start shaking, releasing pressure into the fume hood by loosening the glass stopper. Shake vigorously at least 20 times for 15 seconds.

2.6.13 Allow the funnel to remain in a stationary position for 2 minutes to allow phase separation of the water and DCM.

2.6.14 Drain the DCM layer from the separatory funnel into a 25 mL mixing cylinder. Avoid pulling any emulsion phase into the DCM extract as it may cloud the DCM extract.

2.6.15 Repeat the DCM-extraction process two or three additional times until the DCM is clear. Collect each extract in the graduated

cylinder. After the final extraction, lightly shake the separatory funnel sideways once or twice to dislodge entrained bubbles of DCM and drain.

2.6.16 Adjust the final volume to a known quantity, 20 or 25 mL, in the mixing cylinder. IFO 120 samples may require dilution when dispersed chemically because of their high absorbance properties. Using a syringe, dispense 2.5 mL or 5.0 mL of an IFO 120 sample into a 10-mL volumetric flask, and fill with DCM to make either a 1:4 or 1:2 dilution, respectively. ANS samples adjusted to 25 mL typically don't require dilution.

2.6.17 If analysis cannot be conducted immediately, store the extracted DCM samples at 4 °C until time of analysis. Glass-stoppered mixing cylinders may be used for short-term storage or prior to bringing the extracts up to volume. After bringing to volume, transfer the DCM extracts to 25–30 mL crimp-style serum vials with aluminum/Teflon seals.

2.6.18 Complete all analysis within 10 consecutive days from when the sample was collected.

### 2.7 UV-Visible Spectrophotometer Linear Stability Calibration

2.7.1 A six-point calibration of the UV-visible spectrophotometer is required at least once per day for each oil. The stability calibration criterion is determined with the six oil standards identified in Table 4 of this Appendix.

2.7.2 Turn on spectrophotometer and allow it to warm up for at least 30 minutes before beginning analysis. Blank the instrument for the wavelengths between 340 and 400 nm with DCM.

2.7.3 If refrigerated, allow all extracts, standards and samples to warm to room temperature.

2.7.4 Determine the absorbance of the six standards between the wavelengths of 340 and 400 nm. This can be done by either one of the following methods:

2.7.4.1 *Trapezoidal Rule.* Program the spectrophotometer to take readings every 5λ or 10λ and calculate the area under the curve using the Trapezoidal rule:

$$\int_{340\lambda}^{400\lambda} f(x)dx \approx \frac{H}{2} \sum_{k=1}^N (f(x_{k+1}) + f(x_k)) \quad (\text{Equation 2})$$

where N+1 = number of absorbance measurements to delineate N equally spaced sections of the curve, and H = the

distance (λ) between each reading. For H = 5, N+1 = 13 measurements, for H = 10,

N+1 = 7. The following formula illustrates readings taken every 10λ.

$$\text{Area} = \frac{(Abs_{340} + Abs_{350}) * 10}{2} + \frac{(Abs_{350} + Abs_{360}) * 10}{2} + \dots + \frac{(Abs_{390} + Abs_{400}) * 10}{2} \quad (\text{Equation 3})$$

When using readings taken every 5λ, each absorbance sum is multiplied by 5.

2.7.4.2 *Automatic Integration.* Program the spectrophotometer to automatically integrate the area under the curve between 340 nm and 400 nm.

2.7.4.3 If the wavelengths must be manually set on the spectrophotometer, the older method of only measuring at 340 λ, 370 λ, and 400 λ may be used. Then calculate using the trapezoidal rule for N + 1 = 3, H = 30. While the resulting area count with the older method is less accurate, the final

results are similar since the inaccuracy is systematic.

2.7.5 After determining the area count for each standard, determine the response factor (RF) for the oil at each concentration using the following equation:

$$RF = \frac{\text{Theoretical Concentration, } \frac{g}{mL} \text{ (Eq.1)}}{\text{area (Eq.3)}} \quad (\text{Equation 4})$$

2.7.6 Spectrophotometer stability for the initial calibration is acceptable when the RFs of the six standard extracts are less than 10%

different from the overall mean value for the six standards, as calculated in Equation 5 of

this Appendix and depicted in the example in Table 4 of this Appendix.

$$\% \text{ difference} = \frac{|RF - \overline{RF}|}{\overline{RF}} * 100 \quad (\text{Equation 5})$$

2.7.7 If this criterion is satisfied, begin analysis of sample extracts. Absorbances greater than or equal to 3.5 are not included because absorbance saturation occurs at and above this value. If any of the standard oil

extracts fails to satisfy the initial-stability criterion, the source of the problem (e.g., preparation protocol for the oil standards, spectrophotometer stability, etc.) must be

corrected before analysis of the sample extracts begins.

2.7.8 Determine the slope of the calibration points by using linear regression forced zero intercept:

$$Y(\text{area under absorbance curve}) = m(\text{slope}) * x(\text{concentration of oil}) \quad (\text{Equation 6})$$

2.8 Spectrophotometric Analysis and Calculations

2.8.1 Once a successful calibration curve for each of the two reference oils has been created and verified, measure experimental replicates for each of the reference oils at

each temperature followed by a standard check sample.

2.8.2 The same procedure is followed for the positive controls.

2.8.3 Determine the area for the absorbance values obtained for the experimental samples by using Equation 2 of

this Appendix and illustrated by Equation 3 of this Appendix.

2.8.4 Calculate the Total Oil dispersed and the percentage of oil dispersed (%OD) based on the ratio of oil dispersed in the test system to the total oil added to the system, as follows:

$$Total\ Oil\ Dispersed,\ mg = \frac{Area\ (Eq.2)}{Calibration\ Curve\ Slope} * V_{DCM} * \frac{V_{tw}}{V_{ew}} \tag{Equation 7}$$

where:

V<sub>DCM</sub> = final volume of the DCM extract (mL)

V<sub>tw</sub> = total seawater in Baffled Flask (120 mL)

V<sub>ew</sub> = volume seawater extracted (30 mL)

$$\%OD = \frac{Total\ Oil\ Dispersed}{\rho_{oil} * V_{oil}} * 100 \tag{Equation 8}$$

where:

ρ<sub>oil</sub> = density of the specific test oil, mg/mL and

V<sub>oil</sub> = Volume (mL) of oil added to test flask (100 μL = 0.1 mL)

2.8.5 The %ODs for the six replicates within a particular treatment are then subjected to an outlier test, the Grubb's Test

or Maximum Normal Residual test (6). A convenient Internet-based calculator of a Grubbs outlier may be found at: <http://www.graphpad.com/quickcalcs/Grubbs1.cfm>. If an outlier is detected (p < 0.05), analyze an additional replicate to obtain the required six replicates.

2.8.6 Report the Dispersion Efficacy value for each oil and each temperature, which is

the lower 95% confidence level of the 6 independent replicates (DE<sub>LCL95</sub>) for each oil/temperature combination. Error bars are not needed as reporting the lower confidence level computationally takes the variability of the replicates into account as shown in Equation 9 of this Appendix.

$$DE_{LCL95} = \overline{\%OD} - \left( \frac{t_{(n-1,1-\alpha)} * S}{\sqrt{n}} \right) \tag{Equation 9}$$

where  $\overline{\%OD}$  = mean percentage oil dispersed for the n = 6 replicates, S = standard deviation, and  $t_{(n-1,1-\alpha)} = 100 * (1-\alpha)^{th}$  percentile from the t-distribution with n-1 degrees of freedom. For 6 replicates,  $t_{n-1,1-\alpha} = 2.015$ , where  $\alpha = 0.05$ . An example of the calculations is given in Table 5 of this Appendix.

2.9 Performance Criterion

The dispersant product tested will remain in consideration for listing on the NCP Product Schedule if the dispersant efficacy (DE<sub>LCL95</sub>), as calculated in section 2.8.6 of this Appendix, is:

$$\%recovery = 100 * \frac{measured\ concentration\ of\ check\ standard}{theoretical\ concentration\ of\ check\ standard} \tag{Equation 10}$$

2.10.4 Calibration QC checks. Before analyzing samples, the spectrophotometer must meet an instrument stability calibration criterion using the oil standards. The instrument stability for initial calibration is acceptable when the RFs (Equation 5 of this Appendix) for each of the six standard concentration levels are less than 10% different from the overall mean value.

2.10 Quality Control (QC) Procedures for Oil Concentration Measurements

2.10.1 Absorbance readings. Perform at least 5% of all UV-visible spectrophotometric measurements in duplicate as a QC check on the analytical measurement method. The absorbance values for the duplicates must agree within ± 5% of their mean value.

2.10.2 Method blanks. Analytical method blanks involve an analysis of artificial seawater blanks (artificial seawater without oil or dispersant in a baffled flask) through testing and analytical procedures. Analyze method blanks with a frequency of at least two per completed test. Oil concentrations in method blanks must be less than detectable limits.

2.10.3 Accuracy. Determine accuracy by using a mid-point standard calibration check after each set of replicate samples analyzed. The acceptance criterion is based on a percent recovery of 90–110% using the following equation:

TABLE 1—CONSTITUENT CONCENTRATIONS FOR GP2 ARTIFICIAL SEAWATER

[Based on Spotte et al., 1984]

Constituent	Concentration (g/L)
NaCl .....	21.03
Na <sub>2</sub> SO <sub>4</sub> .....	3.52
KCl .....	0.61
KBr * .....	0.088
Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> · 10H <sub>2</sub> O * .....	0.034
MgCl <sub>2</sub> · 6H <sub>2</sub> O .....	9.50
CaCl <sub>2</sub> · 2H <sub>2</sub> O .....	1.32

TABLE 1—CONSTITUENT CONCENTRATIONS FOR GP2 ARTIFICIAL SEAWATER—Continued

[Based on Spotte et al., 1984]

Constituent	Concentration (g/L)
SrCl <sub>2</sub> · 6H <sub>2</sub> O * .....	0.02
NaHCO <sub>2</sub> * .....	0.17

\* Use Stock Solution, 1 mL/L GP2 for 100X stock solution for Bromide, Borate, and Strontium.  
10 mL/L GP2 for bicarbonate—10X stock solution as it is not soluble in a 100X solution.

Adjust to pH 8.0 prior to autoclaving.

TABLE 2—TEST OIL CHARACTERISTICS

Oil	Density, mg/ mL @ 15 °C	API gravity (deg)	Viscosity @ 15 °C, (cSt)	Category by API gravity	Category by kinematic viscosity
ANS .....	884	28.2	40	Medium .....	Light
IFO120 .....	948	17.5	1520	Heavy .....	Heavy

TABLE 3—STOCK SOLUTION PREPARATION

Item	Amount
Mass of Bottle, g .....	29.9666
Mass of Bottle + oil, g .....	31.5734
Mass of bottle + disp + oil + DCM, g .....	55.0425
Mass of oil, g .....	1.6068

TABLE 3—STOCK SOLUTION PREPARATION—Continued

Item	Amount
Mass of disp + oil + DCM, g ....	25.0759
Mass of 1 mL syringe, g .....	14.5563
Mass of 1 mL syringe + solu- tion, g .....	15.8779

TABLE 3—STOCK SOLUTION PREPARATION—Continued

Item	Amount
Density of solution, g/mL .....	1.3216
Volume of solution, mL .....	18.9740
Conc. Of stock solution, mg/mL	84.6850

Table 4. Dispersant Calibration Example for Both Oils

ANS	Stock Solution Concentration mg/mL				84.6848				
Standard – Stock vol. (uL)	Theoretical Conc.	Abs 340 nm	.....	Abs 400 nm	Calculated Trapezoid Area	RF	Avg. RF	Dev. From Avg. RF	Slope
20	0.085	0.231	.....	0.09	8.55	0.0099	0.0101	2.27	99.11
40	0.169	0.452	.....	0.175	16.68	0.0102		0.15	
80	0.339	0.864	.....	0.334	31.91	0.0106		4.72	
120	0.484	1.307	.....	0.503	48.16	0.0100		0.88	
160	0.677	1.818	.....	0.711	67.38	0.0101		0.82	
200	0.847	2.251	.....	0.888	84.30	0.0100		0.91	
IFO120	Stock Solution Concentration mg/mL				95.7542				
Standard – Stock vol. (uL)	Theoretical Conc.	Abs 340 nm	.....	Abs 400 nm	Calculated Trapezoid Area	RF	Avg. RF	Dev. From Avg. RF	Slope
10	0.048	0.278	.....	0.109	10.33	0.0046	0.0048	3.64	205.67
20	0.096	0.536	.....	0.209	19.87	0.0048		0.22	
40	0.192	1.066	.....	0.414	39.39	0.0049		1.10	
60	0.287	1.608	.....	0.628	59.72	0.0048		0.04	
80	0.383	2.154	.....	0.836	79.73	0.0048		0.10	
100	0.479	2.649	.....	1.021	97.24	0.0049		2.39	

Table 5. Sample Calculation with ANS															
Rep	Abs 340nm	..	Abs 400nm	Area from spec	Dilution Factor	Extract Volume (ml)	Area	Conc, mg/m L.	Mass in 30 mL, mg	Total Oil Dispersed, mg	Efficiency %	Avg.	Std. Dev.	Coeff. Of Var	LCL 95
1	2.096	..	0.814	77.51	1	25	77.59	0.72	18.12	72.49	82.00	81.01	3.17	3.91	78.40
2	1.985	..	0.774	73.72	1	25	73.70	0.69	17.21	68.86	77.89				
3	2.012	..	0.781	74.50	1	25	74.54	0.70	17.41	69.64	78.78				
4	2.18	..	0.841	80.25	1	25	80.19	0.75	18.73	74.93	84.76				
5	1.999	..	0.785	73.87	1	25	73.93	0.69	17.27	69.07	78.13				
6	2.173	..	0.841	79.86	1	25	79.96	0.75	18.68	74.71	84.51				
dup	2.162	..	0.841	80.143	1	25	80.16	0.75	18.72	74.89	84.72				
	2.18	..	0.841	80.245	1	25	80.19	0.75	18.73	74.93	84.76				

2.11 *References for Section 2.0*  
 (1) U.S. Environmental Protection Agency (1994), "Swirling Flask Dispersant Effectiveness Test," *Title 40 Code of Federal Regulations*, Pt. 300, Appendix C, pp 47458–47461.  
 (2) Sorial, G.A., A.D. Venosa, K.M. Koran, E. Holder, and D.W. King. 2004. "Oil spill dispersant effectiveness protocol: I. Impact of operational variables." *ASCE J. Env. Eng.* 130(10):1073–1084.  
 (3) Sorial, G.A., A.D. Venosa, K.M. Koran, E. Holder, and D.W. King. 2004. "Oil spill dispersant effectiveness protocol: II. Performance of revised protocol." *ASCE J. Env. Eng.* 130(10):1085–1093.

(4) Venosa, A.D., D.W. King, and G.A. Sorial. 2002. "The baffled flask test for dispersant effectiveness: A round robin evaluation of reproducibility and repeatability." *Spill Sci. & Technol. Bulletin* 7(5–6):299–308.  
 (5) Spotte, S., G. Adams, and P.M. Bubucis. 1984. "GP2 medium is an synthetic seawater for culture or maintenance of marine organisms," *Zoo Biol.* 3:229–240.  
 (6) Grubbs, F. 1969. "Sample Criteria for Testing Outlying Observations," *Annals of Mathematical Statistics*, pp. 27–58.

**3.0 Dispersant Toxicity Testing**

3.1 *Summary.* This laboratory protocol includes testing for: (1) Dispersant standard static acute toxicity tests for the mysid shrimp, *Americamysis bahia* (48-hr duration) and the inland silverside, *Menidia beryllina* (96-hr duration); (2) dispersant/oil mixture static acute toxicity tests for *Americamysis bahia* and *Menidia beryllina* (48-hr and 96-hr duration, respectively); (3) dispersant developmental assay for the purple sea urchin, *Strongylocentrotus purpuratus*, (72-hr duration); and (4) dispersant 7-day static subchronic tests with *Americamysis bahia* and *Menidia beryllina* (Table 6 of this Appendix).

TABLE 6—TOXICITY TESTING REQUIREMENTS FOR DISPERSANTS

Test substance	Test procedure			
	96-hr static acute: <i>Menidia beryllina</i>	48-hr static acute: <i>Americamysis Bahia</i>	72-hr sea urchin Developmental Assay	7-day subchronic: <i>M. beryllina</i> & <i>A. bahia</i>
<i>Dispersant only</i> .....	yes .....	yes .....	yes .....	yes.
<i>Dispersant/Reference Oil Mixture</i> .....	yes .....	yes .....	no .....	no.

3.2 *Preparation of Stock Solutions*

3.2.1 *Dispersant.* Prepare a 1000 µL/L primary stock solution prior to test initiation by adding 1.1 mL of dispersant to 1100 mL of dilution water consisting of salinity adjusted uncontaminated natural or artificial seawater, in a glass vessel. Using a laboratory top stirrer equipped with a stainless steel blade, center the stirrer blade in the mixing vessel one inch off the bottom. Initially mix the resulting stock solution for approximately five seconds at speeds of < 10,000 rpm to avoid foaming. Thereafter, set the speed to provide a 70% vortex. Using a glass pipette, remove appropriate aliquots of stock solution from between the mixing vessel wall and edge of the vortex and place directly into the dilution water within an exposure vessel. Suspend mixing of the stock solution after the removal of each aliquot. Base the preparation of exposure solutions on the nominal concentration of the stock solution and follow procedures outlined in sections 3.5 and 3.6 of this Appendix.

3.2.2 *Dispersant-Reference Oil(s) Mixtures.* Use IFO 120 and ANS oils. To obtain these oils at no charge (except for a minimal shipping fee) see <http://www.epa.gov/emergencies/content/ncp/index.htm>. Assessment of dispersant-reference oil mixture (DOM) toxicity is determined for each reference oil using the aqueous phase of a chemically enhanced-water accommodated fraction (CE-WAF). Repeat the following procedure for each reference oil tested. Fit a glass aspirator bottle (approximately 23L) equipped with a hose bib at the base with a length of silicon tubing containing a hose clamp. Fill the bottle with 19L of seawater leaving a 20% headspace above the liquid, place on a magnetic stir plate then add and center a stir bar. Add the respective crude oil at 25g/L using a silicon tube attached to a glass funnel that reaches just below the water surface. Using this method reduces the production of

air bubbles on the oil surface slick. Adjust the stir plate to obtain an oil vortex of 25% of the total volume of the seawater, then add the dispersant to be tested at a ratio of 1:10 dispersant:oil (2.5 g/L). Securely seal the bottle to reduce the loss of volatiles using a silicon stopper and wraps of Parafilm and stir for 18 hours, then allow the solution to settle for 6 hours. Maintain the temperature at 25 °C during stirring and settling. Purge the hose at the base of the bottle of any material followed by removal of the CE-WAF (aqueous phase) into a clean glass container without disturbing the surface oil slick. The CE-WAF should be remixed and 1–2 L removed for chemical analysis of total petroleum hydrocarbons (TPH) following the procedures outlined in section 3.4 of this Appendix. The remaining volume will be used for the preparation of exposure solutions following procedures outlined in section 3.3 of this Appendix. To reduce time and cost, mix sufficient amounts of dispersant product-reference oil mixture CE-WAF to allow preparation of exposure solutions for conducting simultaneous acute tests with both *Americamysis bahia* and *Menidia beryllina*.

3.3 *Preparation of Exposure Concentrations*

3.3.1 *Concentration Selection.* Preliminary rangefinder tests may be necessary using a series of logarithmic concentrations (e.g. 0.1, 1, 10, 100 µl dispersant product/L or mg TPH/L) to determine the appropriate exposure concentration range necessary to determine LC<sub>50</sub> values and 95% confidence intervals. For definitive tests, conduct a minimum of five test concentrations using a geometric ratio between 1.5 and 2.0 (e.g. 2, 4, 8, 16, and 32). Note that when testing only the dispersant product, the highest test concentration must not exceed the dispersant's self-dispersibility limit.

3.3.2 *Exposure Concentrations.* Exposure solutions are prepared by adding the appropriate amount of stock solution directly to dilution water in each test chamber. Mix each exposure solution using five rotations in one direction followed by five rotations in the opposite direction using a solid glass stir rod.

3.3.3 *Reference Toxicants.* Separate toxicity tests must be performed with a reference toxicant for each species tested. Conduct additional reference toxicity tests any time a change in the population or source of a test species occurs. Use sodium dodecyl sulfate (SDS), also known as dodecyl sodium sulfate (DSS), and sodium lauryl sulfate (SLS) as the reference toxicant for exposures conducted with *Menidia beryllina* and *Americamysis bahia*. Use copper chloride as the reference toxicant for exposures conducted with the sea urchin developmental test. Use reagent grade quality SDS and copper chloride for tests. Information on procedures for conducting reference toxicant tests with these species can be found in the specific EPA methods documents cited in sections 3.5.1, 3.6.1 and 3.7.1 of this Appendix.

3.4 *Chemical Analysis of Stock Solutions.* Add the 1L sample of CE-WAF (Section 3.2.2 of this Appendix) solutions directly to amber glass bottles with Teflon®-lined cap. Collect a replicate sample in the event of accidental loss or if reanalysis of the stock solution becomes necessary. Adjust sample to a pH=2 using 50% hydrochloric acid, immediately refrigerate and analyze within 48 hours of collection. Analyze samples for C9–C32 TPH by gas chromatography-flame ionization detection (GC-FID) following EPA SW-846, Method 8015B–DRO (4). Report TPH concentration of stock solutions as milligrams TPH/L and use in the calculation of exposure concentrations for all toxicity tests conducted with CE-WAF.



### 3.5 *Static Acute Tests With M. beryllina and A. bahia*

3.5.1 *General.* Use EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012) (1) for testing each species separately with dispersant product or a mixture of dispersant product and reference oil (DOM).

3.5.2 *Test Solutions.* Modify procedures in EPA-821-R-02-012 specifically dealing with the handling and toxicity testing of effluents or receiving water samples as follows: Prepare stock solutions following section 3.2 of this Appendix and exposure concentrations following section 3.3 of this Appendix.

3.5.3 *Number of Treatments, Replicates and Organisms.* Conduct a minimum of three replicates of at least five exposure treatments plus a minimum of three replicate dilution water controls. Expose ten organisms per replicate treatment.

3.5.4 *Exposure Period.* Test duration is 48-hr for *Americamysis bahia* and 96-hr for *Menidia beryllina*. Mortality must be recorded at each 24 hour period of each test.

3.5.5 *Test Acceptability.* For each test performed, survival of control animals must be >90% and test results must allow determination of statistically valid LC<sub>50</sub> and 95% confidence interval values except in cases where the LC<sub>50</sub> is >1000 µl/L or is determined to be greater than the limits of water solubility of dispersibility.

3.5.6 *Static Acute Test Summary.* A summary of required test conditions is provided in Table 7 of this Appendix.

### 3.6 *Sea Urchin Developmental Test With Dispersant Product*

3.6.1 *General.* Use Section 15, "Purple Urchin, *Strongylocentrotus purpuratus* and Sand Dollar, *Dendraster excentricus* Larval Development Test Method" of EPA's *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms* (EPA/600/R-95-136) (2).

3.6.2 *Test Organism.* Tests of dispersant products are to follow methods for the purple urchin only. Tests with the sand dollar are not required.

3.6.3 *Test Solutions.* Modify procedures in EPA/600/R-95-136, Section 15 specifically dealing with the handling and toxicity testing of effluents or receiving water samples as follows: Prepare stock solutions following section 3.2.1 of this Appendix and exposure concentrations following section 3.3 of this Appendix.

3.6.4 *Number of Treatments and Replicates.* Conduct a minimum of four replicates of five exposure treatments plus a minimum of four replicate dilution water controls.

3.6.5 *Exposure Duration and Test Endpoint.* Examine the effects of the dispersant product on normal development of sea urchin embryos over a period of 72 hours. An IC<sub>50</sub> (the exposure concentration at which normal development is inhibited in

50% of the embryos) with 95% confidence intervals are to be determined in place of an IC<sub>25</sub>. The concentration of dispersant causing inhibition of development in 50% of exposed embryos (IC<sub>50</sub>) with the lower and upper 95% confidence intervals (LCI<sub>95</sub> and ULCI<sub>95</sub>) must be calculated at the end of the exposure period. Mortality determinations are not required.

3.6.6 *Test Acceptability.* Requirements of the assay are: (i) ≥ 80% normal larval development in the control treatment, (ii) the minimum significant difference (MSD) that can be statically detected relative to the control is ≤25%, (iii) test results which support the determination of a statistically valid IC<sub>50</sub> and 95% confidence interval unless the LC<sub>50</sub> is >1000 µl/L or is greater than the limits of water solubility of dispersibility.

3.6.7 *Urchin Developmental Test Summary.* A summary of required test conditions is provided in Table 7 of this Appendix.

### 3.7 *Seven-Day Subchronic Tests With M. beryllina and A. bahia*

3.7.1 *General.* Use Section 13, Method 1006.0, "Inland Silverside (*Menidia beryllina*) Larval Survival and Growth Method," and Section 14, Method 1007.0, "Mysid (*Mysidopsis* [renamed *Americamysis bahia*] Survival, Growth, and Fecundity Method" of EPA's *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms* (EPA-821-R-02-014) (3) for testing of dispersant product.

3.7.2 *Test Solutions.* Modify procedures in EPA-821-R-02-014, sections 13 and 14 specifically dealing with the handling and toxicity testing of effluents or receiving water samples as follows: Prepare stock solutions following section 3.2.1 of this Appendix and exposure concentrations following section 3.3 of this Appendix. Exposure solutions should be renewed every 24 hr for the duration of the test.

3.7.3 *Number of Treatments, Replicates and Organisms.* (i) *Menidia beryllina*: Conduct a minimum of four replicates of at least five exposure treatments plus a minimum of four replicate dilution water controls. Expose ten *M. beryllina* per replicate treatment. (ii) *Americamysis bahia*: Conduct a minimum of eight replicates of at least five exposure treatments plus a minimum of eight replicate dilution water controls. Expose five *A. bahia* per replicate treatment.

3.7.4 *Exposure Duration and Test Endpoint.* The test duration is seven days for both species. Test endpoints for *Menidia beryllina* are survival and growth (dry weight) and for *Americamysis bahia* is survival, growth (dry weight) and fecundity. Calculate an LC<sub>50</sub> and 95% confidence interval for survival and IC<sub>25</sub> and IC<sub>50</sub> with 95% confidence intervals for growth (and fecundity for *A. bahia* only). Report the lowest observed effect concentration (LOEC) and no observed effect concentration (NOEC) for each endpoint.

3.7.5 *Test Acceptability.* Requirements of the assay are: (i) ≥80% survival in the control treatment for each species, (ii) dry weights must meet the specific requirements as stipulated in Method 1006.0 for *Menidia beryllina* and Method 1007.0 for *Americamysis bahia*, (iii) egg production must occur in 50% of female *Americamysis bahia* in the replicate control treatments.

3.7.6 *Subchronic Test Summary.* A summary of required test conditions for each species is provided in Table 7 of this Appendix.

3.8 *Laboratory Report.* The laboratory must include, for each toxicity test report, all applicable information, data and analyses as follows:

3.8.1 *Test Objective:* Protocol title and source, endpoint(s);

3.8.2 *Product Information:* Product name, manufacturer contact information, lot number, production date, date received/chain of custody;

3.8.3 *Contract Facility:* Contact information;

3.8.4 *Dilution Water:* Source, pretreatment, physical and chemical characteristics (pH, salinity);

3.8.5 *Test Conditions:* Date and time of test (start and end), test chambers type and volume, volume of solution per chamber, number of organisms per chamber, number of replicate chambers per treatment, feeding frequency, amount and type of food, test concentrations, test temperature (mean and range), test salinity (mean and range);

3.8.6 *Test Organisms:* Common and scientific name, source contact information, age and date purchased, acclimation conditions (e.g., temperature, salinity, both mean and range), age at test start;

3.8.7 *Reference toxicant:* Date received, lot number, date of most recent test, results and current Cumulative Sum Chart, dilution water used, physical and chemical methods used;

3.8.8 *Quality Assurance:* Verification of laboratory accreditation, including subcontractor facilities;

3.8.9 *Test Results:* Raw data in tabular and graphical form, daily records of affected organisms in each concentration replicate and controls, table of required endpoints (i.e., LC<sub>50</sub> with 95% confidence interval (CI), IC<sub>25</sub> and IC<sub>50</sub> with 95% CI, LOEC and NOEC), statistical methods used to calculate endpoints, summary tables of test conditions and QA data;

3.8.10 *Analytical Results:* Method summary including Limit of Detection (LOD)/Limit of Quantitation (LOQ), deviations and reasons if any, sample summary, results including chromatograms and data qualifiers, QA summary including calibration curves, method blank and surrogate recovery, analytical results summary; and

3.8.11 *Conclusions:* Relationship between test endpoints and threshold limit.

TABLE 7—SUMMARY OF TEST CONDITIONS—DISPERSANT TOXICITY

	Acute <i>M. beryllina</i>	Acute <i>A. bahia</i>	Subchronic <i>M. beryllina</i>	Subchronic <i>A. bahia</i>	Development <i>S. purpuratus</i>
Test type .....	Static non-renewal .....	Static non-renewal ..	Static renewal (daily)	Static renewal (daily)	Static non-renewal.
Test duration .....	96 hours .....	48 hours .....	7 days .....	7 days .....	72 ± 2 hours.
Salinity .....	20 ± 2‰ .....	20 ± 2‰ .....	20 ± 2‰ .....	20 ± 2‰ .....	34 ± 2‰.
Temperature .....	25 ± 1 °C. Test temperatures must not deviate (maximum minus minimum temperature) by for than 3 °C during the test.				15 ± 1 °C.
Light quality .....	Ambient laboratory illumination				
Light intensity .....	10–20 µE/m <sup>2</sup> /s				
Photoperiod .....	16 h light, 8 h darkness, with phase in/out period recommended				
Test chamber size <sup>1</sup>	250 mL .....	250 mL .....	600 mL–1 L .....	400 mL .....	30 mL.
Test solution volume <sup>1</sup> .	200 mL .....	200 mL .....	500–750 mL .....	150 mL .....	10 mL.
Age of test organism <sup>2</sup> .	9–14 days .....	1–5 days .....	7–11 days .....	7 days .....	1 hr old fertilized eggs.
No. organisms per test chamber.	10 .....	10 .....	10 .....	5 .....	25 embryos per mL.
No. of replicate chambers per concentration.	3 .....	3 .....	4 .....	8 .....	4.
Feeding regime .....	Refer to specific feeding procedures provided in each test method				None.
Aeration .....	None, unless DO falls below 4.0 mg/L, then aerate all chambers. Rate:<100 bubbles/minute				
Test concentrations ..	5 exposure concentrations and a control (minimum required)				
Test acceptability (required).	≥90% survival in controls.	≥90% survival in controls.	For controls: ≥80% survival; average dry weight ≥0.5mg where test starts with 7 day old larvae, or ≥ 0.43 mg for larvae preserved for ≤7days.	For controls: ≥80% survival; average dry weight ≥0.20 mg; ≥ 50% of control females produce eggs.	≥80% normal shell development in controls.

<sup>1</sup> Recommended minimum value.

<sup>2</sup> Less than or equal to 24-hr range in age.

3.9 References for Section 3.0

- (1) U.S. EPA. 2002. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. Fifth Edition. U.S. Environmental Protection Agency, Washington, DC (EPA-821-R-02-012).
- (2) U.S. EPA. 1995. *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms*. First Edition. U.S. Environmental Protection Agency, Washington, DC (EPA/600/R-95-136)

- (3) U.S. EPA. 2002. *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms* U.S. Environmental Protection Agency, Washington, DC (EPA-821-R-02-014).
- (4) U.S. EPA. 2008. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* U.S. Environmental Protection Agency, Washington, DC (SW-846) <http://www.epa.gov/osw/hazard/testmethods/sw846/online/index.htm>.

4.0 Standard Acute Toxicity Testing of Surface Washing Agents, Bioremediation Agents, Herding Agents, and Solidifiers.

4.1 Summary. This laboratory protocol includes testing for: (1) Saltwater standard static acute toxicity tests for test products with the mysid shrimp, *Americamysis bahia* (48-hr duration) and the inland silverside, *Menidia beryllina* (96-hr duration); and (2) freshwater standard static acute toxicity tests for test products with the daphnid, *Ceriodaphnia dubia* (48-hr duration) and the fathead minnow, *Pimephales promelas* (96-hr duration) (see Table 8 of this Appendix).

TABLE 8—TOXICITY TESTING REQUIREMENTS FOR SURFACE WASHING AGENTS, HERDING AGENTS, BIOREMEDIATION AGENTS AND SOLIDIFIERS

Application environment	Test procedure			
	96-hr static acute: <i>Menidia beryllina</i>	48-hr static acute: <i>Americamysis bahia</i>	96-hr static acute: <i>Pimephales promelas</i>	48-hr static acute: <i>Ceriodaphnia dubia</i>
Saltwater only .....	yes .....	yes .....	no .....	no.
Freshwater only .....	no .....	no .....	yes .....	yes.
Freshwater and saltwater use .....	yes .....	yes .....	yes .....	yes.

4.2 *Dilution Water.* Use Section 7 of EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012) [1] for preparation of the appropriate dilution water for each species tested. Use of clean natural or synthetic seawater for tests conducted with saltwater species is acceptable.

#### 4.3 *Preparation of Stock Solutions*

4.3.1 *Liquid Surface Washing Agents and/or Herding Agents.* Prepare a 1000 µL/L stock solution prior to test initiation by adding 1.1 mL of test product to 1100 mL of dilution water in a glass vessel. Place on a magnetic stir plate then add and center a stir bar and adjust the stir plate to obtain a vortex of 25% of the total volume of the liquid. Mix the resulting stock solution for approximately five minutes at room temperature. Using a glass pipette, remove appropriate aliquots of stock solution from between the mixing vessel wall and edge of the vortex and place directly into the dilution water within an exposure vessel. Base the preparation of exposure solutions on the nominal concentration of the stock solution and follow procedures outlined in sections 4.6 and/or 4.7 of this Appendix, as appropriate.

4.3.2 *Bioremediation Agents.* For products consisting of two or more liquid and/or solid components, prepare the product following the manufacturers recommended procedure and ensure the test product mixture is completely blended. Prepare a 1000 µL/L stock solution prior to test initiation by adding 1.1 mL of the test product mixture to 1100 mL of dilution water in a glass vessel. Place on a magnetic stir plate then add and center a stir bar and adjust the stir plate to obtain a vortex of 25% of the total volume of the liquid. Mix the resulting stock solution for approximately five minutes at room temperature. Using a glass pipette, remove appropriate aliquots of stock solution from between the mixing vessel wall and edge of the vortex and place directly into the dilution water within an exposure vessel. Base the preparation of exposure solutions on the nominal concentration of the stock solution and follow procedures outlined in sections 4.5 and/or 4.6 of this Appendix, as appropriate.

4.3.3 *Solid Phase Products.* Assessment of the toxicity of solidifiers and other solid phase products are determined using the aqueous phase of water-accommodated fractions (WAFs) of the test product. Fit a glass aspirator bottle (approximately 23L) equipped with a hose bib at the base with a length of silicon tubing containing a hose clamp. Fill the bottle with 19L of dilution water leaving a 20% headspace above the liquid, place on a magnetic stir plate then add and center a stir bar. Add the test product at 25 g/L and securely seal the bottle using a silicon stopper and wraps of parafilm. Adjust the stir plate to obtain a vortex of 25% of the total fluid volume, stir for 18 hours then settle for 6 hours. Maintain the temperature at 25 °C during stirring and settling. Purge the hose at the base of the bottle of any material followed by removal of the WAF (aqueous phase) into a clean glass container without disturbing the product on the surface. The WAF should be remixed and

used for the preparation of exposure solutions following procedures outlined in section 4.4 of this Appendix.

#### 4.4 *Preparation of Exposure Concentrations*

4.4.1 *Concentration Selection.* Preliminary rangefinder tests may be necessary using a series of logarithmic concentrations (e.g. 0.1, 1, 10, 100 µl test product/L) to determine the appropriate exposure concentration range necessary to determine LC<sub>50</sub> values and 95% confidence intervals. For definitive tests, conduct a minimum of five test concentrations using a geometric ratio between 1.5 and 2.0 (e.g. 2, 4, 8, 16, and 32). Note that when testing the product, the highest test concentration should not exceed the test product's self-dispersibility limit.

4.4.2 *Exposure Concentrations.* Exposure solutions are prepared by adding the appropriate amount of stock solution directly to dilution water in each test chamber. Mix each exposure solution using five rotations in one direction followed by five rotations in the opposite direction using a solid glass stir rod.

4.4.3 *Reference Toxicants.* Separate toxicity tests must be performed with a reference toxicant for each species tested. Conduct additional reference toxicity tests any time a change in the culture population or source of a test species occurs. Use reagent grade quality sodium dodecyl sulfate (SDS), also known as dodecyl sodium sulfate (DSS), and sodium lauryl sulfate (SLS) as the reference toxicant. Information on procedures for conducting reference toxicant tests with these species can be found in section 4 of EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012) (3).

#### 4.5 *Saltwater Static Acute Tests With *Menidia beryllina* and *Americamysis bahia**

4.5.1 *General.* Use EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012) (1) for testing each species separately with the test product.

4.5.2 *Test Solutions.* Modify procedures in EPA-821-R-02-012 specifically dealing with the handling and toxicity testing of effluents or receiving water samples as follows: Prepare stock solutions following the appropriate sections (4.3.1, 4.3.2, or 4.3.3) of this Appendix and exposure concentrations following section 4.4 of this Appendix.

4.5.3 *Number of Treatments, Replicates and Organisms.* Conduct a minimum of three replicates of at least five exposure treatments plus a minimum of three replicate dilution water controls. Expose ten organisms per replicate treatment.

4.5.4 *Exposure Period.* Test duration is 48-hr for *A. bahia* and 96-hr for *M. beryllina*. Mortality must be recorded at each 24 hour period of each test.

4.5.5 *Test Acceptability.* For each test performed, survival of control animals must be > 90% and test results must allow determination of statistically valid LC<sub>50</sub> and 95% confidence interval values except in cases where the LC<sub>50</sub> is >1000 µl/L or is determined to be greater than the limits of water solubility or dispersibility.

4.5.6 *Static Acute Test Summary.* A summary of required test conditions is provided in Table 9 of this Appendix.

#### 4.6 *Freshwater Static Acute Tests With *Pimephales promelas* and *Ceriodaphnia dubia**

4.6.1 *General.* Use EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012) (1) for testing each species separately with the test product.

4.6.2 *Test Solutions.* Modify procedures in EPA-821-R-02-012 specifically dealing with the handling and toxicity testing of effluents or receiving water samples as follows: Prepare stock solutions following the appropriate sections (4.3.1, 4.3.2, or 4.3.3) of this Appendix and exposure concentrations following section 4.4 of this Appendix.

4.6.3 *Number of Treatments, Replicates and Organisms.* *P. promelas:* Conduct a minimum of three replicates of at least five exposure treatments plus a minimum of three replicate dilution water controls. Expose ten organisms per replicate treatment. *C. dubia:* Conduct a minimum of four replicates of at least five exposure treatments plus a minimum of four replicate dilution water controls. Expose five organisms per replicate treatment.

4.6.4 *Exposure Period.* Test duration is 48-hr for *C. dubia* and 96-hr for *P. promelas*. Mortality must be recorded at each 24 hour period of each test.

4.6.5 *Test Acceptability.* For each test performed, survival of control animals must be > 90% and test results must allow determination of statistically valid LC<sub>50</sub> and 95% confidence interval values except in cases where the LC<sub>50</sub> is >1000 µl/L or is determined to be greater than the limits of water solubility or dispersibility.

4.6.6 *Static Acute Test Summary.* A summary of required test conditions is provided in Table 9 of this Appendix.

#### 4.7 *Laboratory Report*

The laboratory must include, for each toxicity test report, all applicable information, data and analyses as follows:

4.7.1 *Test Objective:* Protocol title and source, endpoint(s);

4.7.2 *Product Information:* Product name, manufacturer contact information, lot number, production date, date received/chain of custody;

4.7.3 *Contract Facility:* Contact information;

4.7.4 *Dilution Water:* Source, pretreatment, physical and chemical characteristics (pH, salinity);

4.7.5 *Test Conditions:* Date and time of test (start and end), test chambers type and volume, volume of solution per chamber, number of organisms per chamber, number of replicate chambers per treatment, feeding frequency, amount and type of food, test concentrations, test temperature (mean and range), test salinity (mean and range);

4.7.6 *Test Organisms:* Common and scientific name, source contact information, age and date purchased, acclimation conditions (e.g., temperature, salinity, both mean and range), age at test start;

4.7.7 *Reference toxicant:* Date received, lot number, date of most recent test, results

and current Cumulative Sum Chart, dilution water used, physical and chemical methods used;

4.7.8 *Quality Assurance*: Verification of laboratory accreditation, including subcontractor facilities;

4.7.9 *Test Results*: Raw data in tabular and graphical form, daily records of affected organisms in each concentration replicate and controls, table of required endpoints (*i.e.*, LC<sub>50</sub>, 95% CI, inhibited concentration for 50% of the species (IC<sub>50</sub>), lower observed effect concentration (LOEC) and no observed

effect concentration (NOEC)), statistical methods used to calculate endpoints, summary tables of test conditions and QA data; and

4.7.10 *Conclusions*: Relationship between test endpoints and threshold limit.

TABLE 9—SUMMARY OF TEST CONDITIONS—SURFACE WASHING AGENTS, HERDING AGENTS, BIOREMEDIATION AGENTS AND SOLIDIFIERS TOXICITY

	Saltwater Acute <i>M. beryllina</i>	Saltwater Acute <i>A. bahia</i>	Freshwater Acute <i>P. promelas</i>	Freshwater Acute <i>C. dubia</i>
Test type .....	Static non-renewal .....	Static non-renewal .....	Static non-renewal .....	Static non-renewal .....
Test duration .....	96 hours .....	48 hours .....	96 hours .....	48 hours.
Salinity .....	20 ± 2‰ .....	20 ± 2‰ .....	NA .....	NA.
Temperature .....	25 ± 1 °C. Test temperatures must not deviate (maximum minus minimum temperature) by more than 3 °C during the test.			
Light quality .....	Ambient laboratory illumination			
Light intensity .....	10–20 µE/m <sup>2</sup> /s			
Photoperiod .....	16 h light, 8 h darkness, with phase in/out period recommended			
Test chamber size <sup>1</sup> .....	250 mL .....	250 mL .....	250 mL .....	30 mL.
Test solution volume <sup>1</sup> .....	200 mL .....	200 mL .....	200 mL .....	15 mL.
Age of test organism <sup>2</sup> .....	9–14 days .....	1–5 days .....	1–14 days .....	<24 hours.
No. organisms per test chamber .....	10 .....	10 .....	10 .....	5.
No. of replicate chambers per concentration (minimum) .....	3 .....	3 .....	3 .....	4.
Feeding regime .....	Refer to specific feeding procedures provided in each test method			
Aeration .....	None, unless DO falls below 4.0 mg/L, then aerate all chambers. Rate: <100 bubbles/minute			
Test concentrations .....	5 exposure concentrations and a control (minimum required)			
Test acceptability (required) .....	≥90% survival in controls			

<sup>1</sup> Recommended minimum value.

<sup>2</sup> Less than or equal to 24-hr range in age.

4.8 *References for Section 4*

(1) U.S. EPA. 2002. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. Fifth Edition. U.S. Environmental Protection Agency, Washington, DC (EPA-821-R-02-012).

5.0 *Bioremediation Agent Efficacy Test Protocol*

5.1 *Summary*. This protocol quantifies changes in weathered Alaska North Slope (ANS) crude oil composition of alkanes and aromatics resulting from the use of a bioremediation agent in either artificial seawater or freshwater. The manufacturer may test either one or both freshwater or saltwater, depending on the product's intended use. Biodegradation of the alkanes and aromatics is monitored for 28 days at 20–23 °C. Product flasks at Day 28 are compared to Day 0 flasks to determine reductions in alkanes and aromatics. A positive control of a known oil-degrading bacterial culture supplied by EPA is tested. A negative, sterile control is also set up containing exposure water, weathered crude oil, product, and a sterilant, sodium azide. The purpose of the negative, killed control is to make sure the disappearance of the oil constituents at day

28 is due to biodegradation and not some physical loss such as volatilization. The day 28 GC/MS results from the killed control must not be less than 90% of the day 0 results. The sample preparation procedure extracts the oil phase into the solvent dichloromethane (DCM) (also known as methylene chloride) with a subsequent solvent exchange into hexane. The hexane extracts are analyzed by a high resolution gas chromatograph/mass spectrometer (GC/MS) operated in the selected ion monitoring mode (SIM) at a scan rate of >5 scans per second. Note to 5.1: Artificially distilled at 521 °F (272 °C) to remove the low molecular weight hydrocarbons to approximate natural weathering processes that occur after a spill.

5.2 *Apparatus*. All equipment must be maintained and calibrated per standard laboratory procedures.

- 5.2.1 Assorted flasks and other glassware;
- 5.2.2 Graduated cylinders (100 mL);
- 5.2.3 Deionized water; 250 mL borosilicate glass Erlenmeyer flasks;
- 5.2.4 Pasteur pipettes;
- 5.2.5 Multichannel pipettor (5–50 mL and 50–200 mL);
- 5.2.6 Autoclave; environmental room or incubator;

- 5.2.7 Balance accurate to 0.1 mg;
- 5.2.8 Orbital shaker table with clamps sized to hold flasks securely;
- 5.2.9 GC/MS instrument equipped with a DB-5 capillary column (30 m, 0.25 mm ID, and 0.25 mm film thickness) or equivalent, and a split/splitless injection port operating in the splitless mode, such as a Agilent 6890 GC/5973 MS (or equivalent) equipped with an auto-sampler for testing multiple samples; and
- 5.2.10 Fixed Rotor Centrifuge.

5.3 *Reagents and Culture Medium*

5.3.1 *Stock Seawater Preparation*. Prepare the artificial seawater GP2 (Spotte *et al.*, 1984) following the procedures in section 2.3 of this Appendix, to obtain the final concentration of the salts listed in Table 1 of this Appendix, except for the sodium bicarbonate (NaHCO<sub>3</sub>) which is prepared separately. Autoclave the artificial seawater. Filter sterilize the concentrated solution of sodium bicarbonate through a 0.45 µm membrane filter and add to the autoclaved and cooled artificial seawater GP2 to obtain the final concentration listed in Table 1 of this Appendix.

5.3.2 *Seawater for the positive control flasks*. Prepare sodium triphosphate (a.k.a.,

sodium tripolyphosphate) (Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>), potassium nitrate (KNO<sub>3</sub>), and ferric chloride hexahydrate (FeCl<sub>3</sub> · 6H<sub>2</sub>O) as a concentrated solution. Filter sterilize through a 0.45 μm membrane filter and add to autoclaved artificial seawater to obtain the final nutrient concentrations listed in Table 10 of this Appendix. Calibrate the pH meter at room temperature (approximately 20–23 °C) using commercial buffers of pH 4.0, 7.0, and 10.0, as appropriate, prior to use. Adjust the pH of the artificial seawater with concentrated hydrochloric acid (HCl) or 10 normality sodium hydroxide (10 N NaOH), as appropriate.

TABLE 10—ARTIFICIAL SEAWATER NUTRIENT CONCENTRATIONS

Constituent	Final concentration, g/L
* FeCl <sub>3</sub> · 6H <sub>2</sub> O .....	0.050
KNO <sub>3</sub> .....	2.890
* Na <sub>5</sub> P <sub>3</sub> O <sub>10</sub> .....	0.297

\* Added aseptically after the GP2 has been autoclaved to limit phosphorus and iron precipitation.

5.3.3 *Seawater for bioremediation agents that do not include nutrients.* If a bioremediation agent contains living microorganisms but not nutrients (or limiting concentrations of nutrients), then nutrients may be added by the manufacturer. However, the total concentration of the nutrients added to the bioremediation agent must not exceed the final concentrations listed in Table 11 of this Appendix.

TABLE 11—ARTIFICIAL SEAWATER NUTRIENT CONCENTRATIONS FOR BIOREMEDIATION AGENTS HAVING NO NUTRIENTS INCLUDED

Constituent	Final concentration, g/L
as Iron (Fe) .....	0.010
as Nitrogen (N) .....	0.400
as Phosphorus (P) .....	0.075

If nutrients are supplied by the product manufacturer, the specific composition and concentration used in the efficacy testing must be submitted.

5.3.4 *Freshwater Preparation.* The artificial freshwater, which is a modification of Bushnell-Haas medium (Haines *et al.*, 2005), is prepared following the concentrations listed in Table 12 of this Appendix and then autoclaved. The pH is adjusted to 7.4 before autoclaving. Constituents removed from the original formulation are KNO<sub>3</sub>, K<sub>2</sub>HPO<sub>4</sub> and KH<sub>2</sub>PO<sub>4</sub>.

TABLE 12—CONSTITUENT CONCENTRATIONS FOR ARTIFICIAL FRESHWATER [Bushnell-Haas]

Constituent	Final concentration (mg/L)
MgSO <sub>4</sub> · 7H <sub>2</sub> O .....	200
CaCl <sub>2</sub> · 2H <sub>2</sub> O .....	20
FeCl <sub>3</sub> · 6H <sub>2</sub> O .....	50
MnSO <sub>4</sub> · H <sub>2</sub> O .....	0.0302
H <sub>3</sub> BO <sub>3</sub> .....	0.0572
ZnSO <sub>4</sub> · 7H <sub>2</sub> O .....	0.0428
(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>2</sub> .....	0.0347

5.3.5 *Freshwater for the positive control.* To prepare the freshwater for the positive controls, prepare the nutrients potassium phosphate monobasic (KH<sub>2</sub>PO<sub>4</sub>), potassium phosphate dibasic (K<sub>2</sub>HPO<sub>4</sub>) and potassium nitrate (KNO<sub>3</sub>) as a concentrated solution. Filter sterilize and add to autoclaved artificial freshwater to obtain the final concentrations given in Table 13 of this Appendix. Calibrate the pH meter at room temperature (approximately 20–23 °C) using commercial buffers of pH 4.0, 7.0, and 10.0, as appropriate, prior to use. Adjust the pH of the artificial freshwater to 7.4 with 1 N HCl or 1 N NaOH, as appropriate.

TABLE 13—FRESHWATER NUTRIENT CONCENTRATIONS

Constituent	Final concentration (g/L) <sup>1</sup>
KNO <sub>3</sub> .....	2.89
KH <sub>2</sub> PO <sub>4</sub> .....	1.00
K <sub>2</sub> HPO <sub>4</sub> .....	1.00

<sup>1</sup> Adjust pH to 7.4 prior to autoclaving

5.3.6 *Freshwater for bioremediation agents that contain living microorganisms but not nutrients or limiting concentrations of nutrients.* If a bioremediation agent does not include nutrients, then nutrients may be added. However, the total concentration of the nutrients added to the bioremediation agent must not exceed the final concentrations provided in Table 14 of this Appendix.

TABLE 14—ARTIFICIAL FRESHWATER NUTRIENT CONCENTRATIONS FOR BIOREMEDIATION AGENTS HAVING NO NUTRIENTS INCLUDED

Constituent	Final concentration, g/L <sup>1</sup>
as Iron (Fe) .....	not added since iron is already in the freshwater solution.
as Nitrogen (N) .....	0.400.
as Phosphorus (P) .....	0.400.

<sup>1</sup> Adjust to pH 7.4 prior to autoclaving.

If nutrients are supplied by the product vendor, the specific composition and concentration used in the efficacy testing must be submitted.

5.3.7 *Oil Preparation.* The test oil, weathered ANS521 crude oil, can be obtained from EPA at no charge (except for a minimal shipping fee). See <http://www.epa.gov/emergencies/content/ncp/index.htm> for more information.

5.3.8 *Sodium azide sterilant.* Prepare a stock solution of NaN<sub>3</sub> for addition to the negative killed control. The final concentration in the killed controls will be 0.5 g/L.

5.4 *Experimental Setup and Procedure*

5.4.1 Autoclave clean borosilicate glass Erlenmeyer flasks (250 mL) for 20 minutes at 121 °C at 15 psig.

5.4.2 Label flasks with the appropriate code (negative control, positive control, or product; day to be sampled (0 or 28); letter indicating replicate number) to reflect the following treatment design in Table 15 of this Appendix:

TABLE 15—BIOREMEDIATION EFFICACY TEST—SUMMARY OF EXPERIMENTAL SETUP

Treatment	Number of replicates at sampling times		Analysis
	Day 0	Day 28	
Negative (killed) Control (oil + exposure water + product + EPA culture + NaN <sub>3</sub> sterilant) .....	0	3	GC/MS
*Positive control (oil + exposure water + nutrients + EPA culture) .....	6	6	GC/MS
<i>Test Type 1:</i> Product containing living microorganisms (oil + exposure water + living product + supplemented nutrients (if necessary)).	6	6	GC/MS
<i>Test Type 2:</i> Product containing proprietary nutrients but no live microorganisms (oil + exposure water + product + EPA culture).	6	6	GC/MS
<i>Test Type 3:</i> Product (such as an enzyme) containing no live microorganisms and no nutrients (oil + exposure water + product).	6	6	GC/MS

\* The laboratory must report positive control test results conducted within the year of any test results for bioremediation products, for one or both types of water as applicable.

5.4.3 Aseptically dispense 100 mL of pre-sterilized artificial exposure water (seawater or freshwater) into each flask. For the positive control flasks, use exposure water containing nutrients. Alternatively, the artificial exposure water can be dispensed into clean flasks and then autoclaved.

5.4.4 Tare the labeled flasks containing exposure water and other additions, as necessary, on the balance with a minimum accuracy of 0.01 g. Add drop-wise 0.50 g oil (this results in a final oil concentration of 5 g/L) using a sterile Pasteur pipette to the center of the flask taking care to avoid splashing the oil onto the sides of the flasks. Record the precise weight. ANS521 may be previously warmed in a hot water bath to facilitate its flow. Take precautions when handling and charging the flasks to minimize the likelihood of contamination by exogenous microbes, including using a new sterile pipette for each series of flasks.

5.4.5 Preparation of the EPA culture for both the positive control flasks and the flasks containing non-living bio-stimulation products. Use two vials containing approximately 5 mL of the known EPA culture frozen in glycerol. Thaw both vials at room temperature, transfer the contents of both thawed vials to a single sterile centrifuge tube, rinse tubes with two volumes each of sterile exposure water, centrifuge at between 6,000 and 7,000  $\times$ s gravity (6,000–7,000  $\times$  g) for 15 minutes using a fixed rotor to fully pellet the cells.

5.4.6 Positive control flasks contain exposure water, oil, nutrients, and the EPA culture.

5.4.7 Negative killed control flasks for all products shall contain exposure water, oil, product, the EPA culture for products not containing a living culture, and the sodium azide sterilant at a final concentration of 0.5 g/L. Add the sodium azide sterilant prior to adding any product or EPA culture. For the negative killed control flasks and product flasks, prepare and add the product to the flasks in a concentration specified by the manufacturer or vendor.

5.4.8 For non-living products that contain nutrient only, use the EPA culture as the inoculum.

5.4.9 For other non-living products (e.g., enzymes), do not add nutrients or the EPA culture as the inoculum as they are not needed.

5.4.10 For products containing microbial cultures, prepare 6 flasks the same way as in Steps a-d, but without the EPA culture. A product that contains its own nutrients must not be amended with nutrients, unless the product contains insufficient nutrients. Since this is a closed flask test, nutrients could be limiting if they are at the same concentration as used in the field. This could cause the product to fail the test. Thus, the manufacturer has the option to supplement its product with a higher concentration of nutrients than that contained in the product. Any nutrient supplements to a product must

be reported and must not exceed the concentration limits in Table 10 (for seawater) and 13 (for freshwater) of this Appendix, as applicable.

5.4.11 Cap all flasks either with cotton stoppers or loosely applied aluminum foil to allow gas exchange with the atmosphere. Set aside the T=0 flasks for immediate extraction and analysis. Place the rest of the flasks onto the orbital shaker table. Do not tip the flasks excessively to avoid stranding oil above the mixing area of the flask. Set the orbital shaker to 200 rpm and shake the flasks for 28 days at 20–23° C.

5.4.12 Submit all information on added cultures and nutrients for testing in the data report.

## 5.5 Sampling and Chemical Analysis

5.5.1 *Summary.* At each sampling event (Days 0 and 28), product and control flasks are sacrificed for analysis of residual oil concentrations (SOP 4 of this Appendix). Record all physical observations for each flask (such as degree of emulsification, whether the oil has coagulated into tar balls, wall growth, color, etc.) at each sampling. The analytical procedure is summarized in Table 16 of this Appendix. Dichloromethane (DCM) is the solvent used for the initial extraction. Solvent-exchange the extract into hexane prior to injection into the gas chromatograph. The solvent exchange is done to prevent asphaltenes from contaminating the column.

TABLE 16—BIOREMEDIATION EFFICACY—SUMMARY OF ANALYTICAL PROCEDURES

Matrix	Measurement	Sampling/measurement method	Analysis method	Sample container/quantity of sample	Preservation/storage	Holding times
DCM .....	N/A .....	Solvent Exchange to Hexane. SOP 4 .....	N/A .....	Capped Vial with Teflon septa, 30 mL.	4 °C .....	6 months.
Hexane .....	Hydrocarbon Concentration	SOP 4 .....	GC/MS .....	Capped Vial with Teflon septa, 10 mL.	4 °C .....	6 months.

5.5.2 *Hydrocarbon Extraction.* To measure extraction efficiency, 500 mL of the surrogate recovery standard (compounds and concentrations described in SOP 1 in this Appendix) is added to each flask. Add 50 mL DCM to each flask. Use magnetic stir bars to stir the flasks for 20 minutes to transfer the oil into the DCM phase. Allow the flasks to stand one hour for phase separation. If an emulsion remains after one hour, centrifuge the emulsion in Teflon® centrifuge tubes for at least ten minutes in a low speed centrifuge at 3,000  $\times$  gravity (3,000  $\times$  g) to break the emulsion and recover the DCM phase. Pass the DCM extract through a funnel plugged with glass wool and containing approximately 20 g anhydrous, granular sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) to remove water. Collect the extract in 30 mL serum vials, capped with Teflon lined septa and aluminum crimp seals, and store at 4 °C for up to 6 months.

5.5.3 *Solvent Exchange.* Perform a solvent exchange (DCM to hexane) prior to GC/MS analysis to prevent injection of asphaltenes into the GC/MS column. Using a Class A volumetric pipette, transfer 10 mL of DCM extract to 16  $\times$  125 mm test tubes with Teflon

lined screw caps. Place the test tubes in a 37° C water bath under a stream of dry nitrogen gas. Reduce the sample to 1 mL and return to volume with hexane. Repeat the blow down and re-suspension twice to exchange the hydrocarbons from DCM into the hexane.

5.5.4 *Hydrocarbon Analysis.* Quantify the concentrations of 25 alkanes (including hopane) and 31 aromatics (SOP 4, Table SOP 4.1 of this Appendix) using an Agilent 6890 GC/5973 MS or equivalent equipped with a 30-m  $\times$  0.25-mm ID  $\times$  0.25- $\mu$ m film thickness DB-5 or equivalent fused silica column. To prepare the samples, transfer 1.0 mL of the hexane extract into a 2 mL autosampler vial with Teflon lined cap. Add 20  $\mu$ L of internal standard solution to each vial with a syringe or positive displacement pipettor. SOP 2 of this Appendix outlines the procedure for preparing the internal standard solution. Load vials onto the autosampler tray and analyze in selected ion monitoring mode (SIM). Sum the individual alkane concentrations for the total alkane concentration and the individual aromatic concentrations for total aromatic concentrations in each flask.

## 5.6 Quality Assurance/Quality Control (QA/QC)

5.6.1 *Objectives.* The critical variables to be analyzed for each set of experimental conditions are the individual petroleum hydrocarbons, i.e., the alkanes ranging in carbon number from nC-14 to nC-35, plus pristane and phytane, and the 2- to 4-ring polycyclic aromatic hydrocarbons (PAHs) and their alkylated homologs as listed in SOP 4 of this Appendix. The quality assurance objectives for precision, accuracy, and detection limits are  $\pm$  20%, 75–125% recovery, and 22.5  $\mu$ g/L, respectively. For more details, refer to the SOPs of this Appendix.

5.6.2 *Precision Objectives.* Precision is presented as relative percent difference (RPD) for duplicate measurements and as relative standard deviation (RSD, or coefficient of variance) for triplicate measurements, applicable to replication of treatments as separate samples.

5.6.3 *Accuracy Objectives.* These are based on the check standards and standard oil samples run concurrently with the sample analyses for GC/MS analysis of critical

compounds. Critical compounds in the check standards and in the oil standards must fall within 75–125% of expected values for the analysis to be valid. Six surrogate compounds (SOP 1 of this Appendix) added to each sample before extraction can also serve as a surrogate for determining accuracy. The measured surrogate concentrations must fall within 75–125% of expected values.

5.6.4 *Calibration Range.* Conduct all measurements within the linear calibration range of the instrument. The calibrated concentration range for GC/MS analysis is 1 mg/L to 30 mg/L. If the measured concentration of any critical compound is above the calibration range, dilute the sample and re-analyze to quantify that particular

compound within the linear calibration range.

5.6.5 *Quality Control.* Table 17 of this Appendix summarizes the QC checks for each measurement. See the corresponding SOP in this Appendix for detailed descriptions of QC checks, frequency, acceptance criteria, and corrective actions.

TABLE 17—QA/QC CHECKS

Sample matrix	Measurement	QA/QC check	Frequency	Acceptance criteria	Corrective action
DCM .....	GC/MS hydrocarbon analysis.	Blanks .....	Once per calibrated run	Peak area of interfering peaks <10% of lowest standard peak area.	Flush with solvent, clean injection port, and/or bake column.
DCM .....	GC/MS hydrocarbon analysis.	DFTPP Check Standard	Once per calibrated run	Must pass all DFTPP criteria.	If any criteria fail, retune and rerun DFTPP check standard.
DCM .....	GC/MS hydrocarbon analysis.	Initial Calibration Samples.	Once per calibrated run	Response Factor RSD ≤25%.	If RSD for any one compound >25%, recalibrate.
DCM .....	GC/MS hydrocarbon analysis.	Calibration Check Standards.	Every 10–15 samples ...	± 25% of expected values.	If >5 compounds are out of range, recalibrate and rerun samples.
Hexane ....	GC/MS hydrocarbon analysis.	Surrogates .....	Every Sample .....	± 30% of expected values.	Re-inject.
Hexane ....	GC/MS hydrocarbon analysis.	Biomarker Concentration.	Every Sample .....	± 25% of average values.	Re-inject.

5.7 *Pass/Fail Criteria*

5.7.1 Calculate the mean and standard deviation of the hopane-normalized total aromatics (sum of all resolved aromatics) and hopane-normalized total alkane

concentrations (sum of all resolved alkanes) from the 6 independent replicates at days 0 and 28. To normalize, divide the sum of the alkane analytes and the sum of the aromatic analytes in each replicate by the hopane concentration in the corresponding replicate.

5.7.2 From those data, calculate the 95% Upper Confidence Level (UCL<sub>95</sub>) at days 0 and 28 using the following formula (Equation 11 of this Appendix):

$$UCL_{95} = \bar{x}_{t(0\text{ and }28)} + \left( \frac{t_{95,5df} \times \sigma}{\sqrt{n}} \right) \tag{Equation 11}$$

Where:

x<sub>t28</sub> = total hopane-normalized alkane or total hopane-normalized aromatic mean of 6 replicates at days 0 and 28,

t<sub>95, 5 df</sub> = the 95% one-tailed t-value with 5 degrees of freedom (2.015),  
 σ = the standard deviation of the 6 replicates at day 0 and 28, and  
 n = no. of replicates = 6.

5.7.3 Using Equation 12 of this Appendix, calculate the % reduction of each oil fraction from day 0 to day 28, using the day 0 and 28 UCL<sub>95</sub> hopane-normalized values for each fraction:

$$\% \text{ reduction} = 100 \times \left[ 1 - \left( \frac{t_{28(UCL95)}}{t_{0(UCL95)}} \right) \right] \tag{Equation 12}$$

Where:

t<sub>28(UCL95)</sub> = UCL<sub>95</sub> of the hopane-normalized total alkane or total aromatic mean of 6 replicates on day 28, and  
 t<sub>0(UCL95)</sub> = UCL<sub>95</sub> of the hopane-normalized total alkane or total aromatic mean of 6 replicates on day 0.

5.7.4 A product is successful in saltwater if the % reduction of total alkanes (aliphatic fraction) from the GC/MS analysis is greater than or equal to 95% and the % reduction of total aromatics (aromatic fraction) is greater than or equal to 70% at day 28 based on the UCL<sub>95</sub> (Equation 12 of this Appendix).

For freshwater, a product is successful if the % reduction of total alkanes and total aromatics is greater than or equal to 95% and 40%, respectively. The benchmark reduction ranges in aliphatic and aromatic fractions for the positive control are the same as for the products specified above. The average concentration of the biomarker hopane at day 28 must not differ from the average concentration at day 0 by more than 12% in the positive control. If the conditions for the positive control are not met, the entire procedure must be repeated.

5.8 *Data Verification and Reporting.* GC/MS data files are generated by MS ChemStation software (the Agilent standard software for GC/MS) or equivalent for each injection. Data files contain summed ion chromatograms and selected ion chromatograms. Calibration curves are generated within MS ChemStation software, and all data files are calculated against the calibration curve by MS ChemStation. Data verification would be done by crosschecking between analysts for 10% of the raw data and its reduction process.

5.9 *Laboratory Report.* The summary of findings from a product test must include the data listings for each analyte that was analyzed (*i.e.*, all individual alkanes and aromatics in the list of required analytes). Express all concentrations as mg analyte/L exposure water.

#### 5.10 *Standard Operating Procedures (SOPs) 1–4*

##### 5.10.1 *SOP 1. Preparation of Surrogate Recovery Standards*

###### 5.10.1.1 *Preparation:*

5.10.1.1.1 Solvents: Dichloromethane (DCM), Optima (Fisher) grade or equivalent.

5.10.1.1.2 Reagents: D36-Heptadecane (C17), D50-Tetracosane (C24), D66-Dotriacontane (C32), D10-1-Methylnaphthalene, D10-Phenanthrene, D10-Pyrene, 5-beta-cholestane (coprostane).

Note: Deuterated reagents are available from Cambridge Isotope Laboratories, Andover, MA.

5.10.1.1.3 Equipment: Micro-spatula, Small beakers, Glass funnel, Analytical balance (0.0001g), Vials with Teflon-lined caps, Teflon wash bottle with Optima grade DCM, Volumetric flask (250 mL), class A, Pasteur pipettes.

###### 5.10.1.2 *Procedure:*

5.10.1.2.1 Using a calibrated analytical balance, weigh 100 mg (0.100 g) of each reagent into separate 10–25 mL beakers.

5.10.1.2.2 Dissolve the reagents in their beakers by adding 10 mL DCM. Use a Pasteur pipette to transfer the solutions to a single 250 mL volumetric flask.

5.10.1.2.3 Wash the beakers 3 or 4 times with DCM. Use a Pasteur pipette to transfer each of the washings to the 250 mL volumetric flask.

5.10.1.2.4 Dilute the solution to the 250 mL volume mark on the volumetric flask with DCM.

5.10.1.2.5 Use a glass stopper to seal the flask and homogenize the solution by inverting the flask 5 or more times. The final concentration of this solution is 400 mg/L for each of the reagents.

5.10.1.2.6 Transfer the solution into 40 mL storage vials and cap with Teflon-lined caps and label each with the date of preparation, operator, sample names, and concentrations.

5.10.1.2.7 Weigh each vial and record its weight on the label. This weight is used to monitor possible evaporation during storage.

5.10.1.2.8 Store these vials at 0 °C or lower.

5.10.1.2.9 Before using, allow the solution to come to room temperature, and then shake it well.

5.10.1.2.10 Weigh the vial before using it, and compare the weight with the last weight recorded on the vial.

5.10.1.2.11 If the weights are consistent, the integrity of the solution can be assumed. If not, investigate and resolve the cause. Prepare a new solution if the integrity has been compromised.

5.10.1.3 *Quality Control:* Inject 20 µL of the surrogate stock solution into 1 mL DCM. Add 20 µL of the internal standard solution (SOP 2 of this Appendix). Analyze this solution by GC/MS using a calibrated method (SOPs 3 and 4 of this Appendix). The expected concentration of each of the corresponding surrogate compounds is  $8 \pm 2$  mg/L. If the measured value does not fall within this range, prepare and measure another independent surrogate solution. If the measured concentration of the second surrogate solution is within the allowable tolerance range, the calibration and instrument conditions are acceptable; properly discard the first surrogate solution. If the concentration of the second surrogate solution is also out of range, then clean and recalibrate the instrument until the problem is resolved.

##### 5.10.2 *SOP 2. Preparation of Internal Standard Solution*

###### 5.10.2.1 *Preparation*

5.10.2.1.1 Solvents: Dichloromethane (DCM), Optima (Fisher) grade or equivalent.

5.10.2.1.2 Reagents: D34 n-Hexadecane (C16), D42 n-Eicosane (C20), D62 n-Triacontane (C30), D8-Naphthalene, D10-Anthracene, D12-Chrysene, 5-alpha-Androstane.

Note: Deuterated reagents are available from Cambridge Isotope Laboratories, Andover, MA.

5.10.2.1.3 Equipment: Micro-spatula, Small beakers, Glass funnel, Analytical balance (0.0001g), calibrated and checked for accuracy, Amber vials with Teflon-lined caps, labeled, Teflon wash bottle with DCM, Volumetric flask (200 mL), class A, Pasteur pipettes.

###### 5.10.2.2 *Procedure:*

5.10.2.2.1 Using a calibrated analytical balance, weigh 100 mg (0.100 g) of each of the reagents into separate small beakers.

5.10.2.2.2 Dissolve the reagents in their beakers by adding 10 mL DCM; using a Pasteur pipette, transfer the solutions to a single 200 mL volumetric flask.

5.10.2.2.3 Wash the beakers 3 or 4 times with DCM; use a Pasteur pipette to transfer each of the washings to the 200 mL volume mark on the volumetric flask.

5.10.2.2.4 Dilute the solution with DCM to the 200 mL volume.

5.10.2.2.5 Seal the flask with a glass stopper and homogenize the solution by inverting the flask a minimum of 5 times. The final concentration of this solution is 500 mg/L of each reagent.

5.10.2.2.6 Transfer the solution into 40 mL storage vials and cap with Teflon-lined caps. Label each vial with the date of preparation, operator, sample names, and concentrations.

5.10.2.2.7 Weigh each vial, and record its weight on the label. This weight is used to monitor possible evaporation during storage.

5.10.2.2.8 Store this solution at 0 °C or lower.

5.10.2.2.9 Before using, allow the solution to come to room temperature, and then shake it well.

5.10.2.2.10 Weigh the vial before using it, and compare the weight with the last weight recorded on the vial.

5.10.2.2.11 If the weights are consistent, the integrity of the solution can be assumed. If not, investigate and resolve the cause. Prepare a new solution if the integrity has been compromised.

5.10.2.3 *Quality Control:* Inject 20 µL of the internal standard solution into 1 mL DCM. Analyze this solution by GC/MS. The only peaks corresponding to the internal standards must appear. If other peaks appear, particularly close to the internal standard peaks, discard the internal standard solution and prepare a new solution.

##### 5.10.3 *SOP 3. Preparation of Working Standards, Check Standards, and Oil Standards for GC/MS Consistency.*

###### 5.10.3.1 *Preparation:*

5.10.3.1.1 Solvent: Dichloromethane (DCM), Optima (Fisher) grade or equivalent.

###### 5.10.3.1.2 *Stock solutions:*

5.10.3.1.2.1 Oil analysis standard: 44 compounds, 100 mg/L in hexane/DCM (9:1), four, 1-mL vials required. Available from Absolute Standards, Inc., Hamden, CT, Part # 90311.

5.10.3.1.2.2 Nine compound PAH standard: 1,000 mg/L in DCM, one vial. Available from Absolute Standards, Inc., Hamden, CT, Part # 90822.

5.10.3.1.2.3 1,2-Benzodiphenylene sulfide, (synonym for naphthobenzothiophene). Prepare a 2 mg/mL stock solution. Available from Sigma-Aldrich Co., Part # 255122, purity 99%.

5.10.3.1.2.4 Hopane solution (17 β (H), 21β (H), 0.1 mg/mL in isoctane. Available from Sigma-Aldrich Co. Part # 07562.

5.10.3.1.2.5 Surrogate solution: 400 mg/L of each reagent in DCM (see SOP 1 of this Appendix).

5.10.3.1.2.6 Internal standard solution, 500 mg/L in DCM (see SOP 2 of this Appendix).

5.10.3.1.3 Alaska North Slope Crude Oil 521 (ANS521).

###### 5.10.3.1.4 *Equipment:*

5.10.3.1.4.1 Glass storage vials with Teflon-lined caps (2 mL and 40 mL capacity);

5.10.3.1.4.2 Volumetric flasks, Class A, 5 mL, 10 mL, and 100 mL.

5.10.3.1.4.3 Glass syringes capable of dispensing 25–500 mL with an accuracy and precision of ± 1%, or equivalent.

5.10.3.1.4.4 Wheaton repetitive dispenser, Model 411 STEP-PETTE or equivalent.

5.10.3.1.4.5 Teflon wash bottle filled with Optima grade DCM or equivalent grade DCM.

5.10.3.1.4.6 Pasteur pipettes.

The volumes of stock solutions required to make the working standards are listed in Table SOP 3.1 of this Appendix.



TABLE SOP 3.1—AMOUNT OF STOCK SOLUTIONS REQUIRED TO MAKE THE WORKING STANDARDS

Stock standards	A	B	C	D	E		F	
Working standards concentration, mg/L	Oil Analysis Mix (44 compounds, 100 mg/L) $\mu$ L	Aromatics Mix (9 compounds, 1000 mg/L) $\mu$ L	1,2-Benzo-diphenylene sulfide (NBT) (2 mg/mL) $\mu$ L	Surrogate solution (100 mg/L) $\mu$ L	Hopane solution (100 mg/L) $\mu$ L	Volumetric Flask Volume mL	ISTD (500 mg/L) $\mu$ L	
STD 30 (no hopane) .....	1500	150	75	375	0	5 .....	100	
STD 20 (5 mg/L hopane) .....	1000	100	50	250	250	5 .....	100	
STD 10 (2.5 mg/L hopane) .....	500	50	25	125	125	5 .....	100	
STD 5* (1 mg/L hopane) .....	500	50	25	125	100	10 .....	200	
STD 5-Utility (1 mg/L hopane) .....	500	50	25	125	100	10 (used for preparation of STD 2.5 & STD 1).	0	
STD 2.5 (0.5 mg/L hopane) .....	Use 5 mL of STD 5-Utility and dilute to 10 mL							200
STD 1 (0.2 mg/L hopane) .....	Use 2 mL of STD 5-Utility and dilute to 10 mL							200

\* Make extra STD 5 for use as check standard.

5.10.3.2 Procedure for Working Standards and Check Standards:

5.10.3.2.1 Label three 5 mL volumetric flasks as STD30, STD20, STD10, and two 10 mL volumetric flasks as STD5, and STD5-utility.

5.10.3.2.2 Add 1–2 mL of DCM to each volumetric flask.

5.10.3.2.3 Using glass syringes, add the appropriate volume of stock solution A (as listed in Table SOP 3.1 of this Appendix) to the flasks labeled STD30, STD20, STD10, STD5, and STD5-utility.

5.10.3.2.4 Wash the walls of the inner neck of the flasks with several drops of DCM to rinse off the residue of the stock solution into the flasks.

5.10.3.2.5 Repeat Step 3 and Step 4 to dispense stock solutions B–E (do not add stock solution F, internal standard solution, at this step).

5.10.3.2.6 Dilute to volume with DCM for all the above flasks, seal with glass stoppers, and invert several times to homogenize the solutions.

5.10.3.2.7 Label two additional 10 mL volumetric flasks as STD2.5 and STD1. Wet with 1–2 mL DCM.

5.10.3.2.8 Dispense 5 mL of STD5-utility solution into flask STD2.5, and 2 mL of STD5-utility solution into flask STD1.

5.10.3.2.9 Dilute to volume with DCM, seal with glass stoppers, and invert several times to homogenize the solutions.

5.10.3.2.10 Using a 100  $\mu$ L glass syringe, dispense 100  $\mu$ L of internal standard solution into flasks STD30, STD20, and STD10. Dispense 200  $\mu$ L into flasks STD5, STD2.5, and STD1 to give a final concentration of 10 mg/L internal standard.

5.10.3.2.11 Seal with glass stoppers, and invert the flasks several times to homogenize the solutions.

5.10.3.2.12 Transfer the solutions into 2 mL storage vials, and cap with Teflon-lined caps.

5.10.3.2.13 Label each vial with date of preparation, analyst, sample names, and concentrations.

5.10.3.2.14 Weigh each storage vial and record its weight on the label. This weight is used to monitor possible evaporation during storage.

5.10.3.2.15 Store this solution at 0 °C or below.

5.10.3.2.16 Before using, allow the solution to come to room temperature, and shake it well.

5.10.3.2.17 Weigh the vial before opening, and compare the weight with the last weight recorded on the vial. If the weights are consistent, the integrity of the solution can be assumed. If not, investigate and resolve the cause. Do not use the solution if the integrity has been compromised.

5.10.3.3 Procedure for Oil Standard. In a 100 mL volumetric flask, weigh 0.500 g of the standard ANS521 crude oil, add 2 mL of surrogate solution (see SOP 1 of this Appendix), and bring to volume with DCM. Add 2 mL of internal standard solution (see SOP 2 of this Appendix). Follow steps 5.10.3.2.11 through 5.10.3.2.17 of this SOP, substituting 40 mL storage vials for the 2 mL vials.

5.10.3.4 Quality Control/Quality Assurance:

5.10.3.4.1 Run the six standard solutions using the GC/MS method (SOP 4) on a tuned GC/MS. Use the EnviroQuant software or equivalent to calculate the average Relative Response Factor (RRF) and the relative standard deviation (RSD) of the RRFs for each analyte over the six concentrations. The RRF is defined as:

$$RRF = \frac{\text{area analyte}}{\text{area internal standard}} \times \frac{\text{concentration of internal standard}}{\text{concentration of analyte}} \quad \text{(Equation 13)}$$

5.10.3.4.2 The RSD of the RRFs for all analytes must be 25% or less according to EPA Method 525.2.

5.10.4 SOP 4. GC/MS Method for the Analysis of Crude Oil Samples.

5.10.4.1 Instrument Specifications:

5.10.4.1.1 Use an Agilent 6890 GC coupled with an Agilent 5973 mass selective detector (MSD) and an Agilent 6890 series auto sampler or equivalent, equipped with a DB–5 capillary column (30 m, 0.25 mm I.D., and 0.25  $\mu$ m film thickness) or equivalent, and a split/splitless injection port operating in the splitless mode. Data acquisition occurs

either in the SIM (selected ion monitoring) mode for quantitative analysis or in SCAN mode for qualitative analysis. In SIM mode, the dwell time of each ion is set to be 10 milliseconds and the ions are split up into groups by retention time. One way to divide the ions is by retention time grouping as shown in Table SOP 4–1 of this Appendix. The number of ions in each ion group must be constant, yielding the same scan rate for each group.

TABLE SOP 4–1—IONS ASSOCIATED WITH RETENTION TIME GROUPS

Group	Ions
1 .....	57, 66, 128, 136, 142, 152, 156, 166, 170, 184
2 .....	57, 66, 166, 170, 178, 180, 184, 188, 192, 194, 198, 208
3 .....	57, 66, 178, 184, 188, 192, 194, 198, 202, 206, 208, 212, 220, 226

TABLE SOP 4-1—Ions Associated With Retention Time Groups—Continued

Group	Ions
4	57, 66, 192, 198, 202, 206, 208, 212, 216, 220, 226, 230, 234, 245
5	57, 66, 191, 217, 228, 240, 242, 248, 256, 262, 264, 270, 276, 284

5.10.4.1.2 Table SOP 4.2 of this Appendix summarizes the instrumental conditions for crude oil analysis. Use only ultra-high purity helium (99.999% pure) as the carrier gas. In series, connect a moisture trap, an oxygen trap, and an organic trap to the carrier gas line before it enters the column.

TABLE SOP 4.2—INSTRUMENTAL CONDITIONS FOR CRUDE OIL ANALYSIS

Instrument	Agilent 6890 Series II Gas Chromatograph (GC) with an Agilent 5973MSD and an Agilent 6890 auto sampler, or equivalent.
Column	DB-5 capillary column (30 m, 0.25 mm I.D., and 0.25-µm film thickness) or equivalent.
Carrier Gas	Helium, ultra-high purity grade (99.999%).
Inlet Temperature	300 °C.
Transfer Line (detector) Temperature	310 °C.
Oven Temperature Program	50 °C for 4 minutes, then 7 °C/min to 310 °C, hold for 18 minutes.

TABLE SOP 4.2—INSTRUMENTAL CONDITIONS FOR CRUDE OIL ANALYSIS—Continued

Flow Rate	Constant flow at 1mL/min. Linear velocity: 36.2 cm/sec.
Injection Volume	1 µL.
Split/Splitless Mode	Splitless.
Total Run Time	59.18 minutes.

5.10.4.2 Procedure for preparing the instrument:

5.10.4.2.1 Lower the injection port temperature and the oven temperature to 50° C or less to avoid oxidation of the column.

5.10.4.2.2 Replace the liner with a clean, silanized liner. Do not touch the liner with bare fingers. A small piece of muffled glass wool may be inserted to protect the column.

5.10.4.2.3 Return the injection port and oven to the appropriate temperatures.

5.10.4.2.4 Wait five minutes after the temperature equilibrates before using the instrument.

5.10.4.3 Procedure for tuning the MSD:

5.10.4.3.1 Perform an air/water check. The value reported for the relative abundance of water (m/z 18), nitrogen (m/z 28), oxygen (m/z 32), or carbon dioxide (m/z 44) shall be less than 5% of the base peak for the system to be considered leak free.

5.10.4.3.2 Tune the MSD using the Standard Autotune program and the decafluorotriphenylphosphine (DFTPP) Tune program to reduce instrument variability. The Autotune report file is referenced by the instrument when performing an air/water check and thus must be run at least once per month. Run standards and samples using DFTPP Tune parameters, and retune the instrument using DFTPP Tune at least once per week. The tune programs use three fragment ions of perfluorotributylamine (PFTBA) as a standard for tuning: m/z 69, 219, and 502. Tune reports must meet the following criteria:

5.10.4.3.2.1 Symmetrical peaks;

5.10.4.3.2.2 Mass assignments within ± 0.2 amu's from 69, 219, and 502;

5.10.4.3.2.3 Peak widths within 0.5 ± 0.1 amu's;

5.10.4.3.2.4 Relative abundance is 100% for ion 69, at least 35% for ion 219, and at least 1% for ion 502;

5.10.4.3.2.5 Relative abundances for isotope masses 70, 220, and 503 ± 0.2 amu's are 0.5–1.5%, 2–8%, and 5–15%, respectively; and

5.10.4.3.2.6 Air and water peaks at m/z = 18, 28, 32, and 44 amu's must be very small and consistent with historical values.

5.10.4.4 Maintaining a log book. Maintain an instrument logbook, and make entries for each use. Include the following information in the logbook: operator name, helium cylinder tank pressure and outlet pressure, vacuum gauge reading, any maintenance performed on the instrument (such as changing the injection port liner, gold seal, guard column, source cleaning), sequence name, data path, samples in order of injection, method information, GC column number, and the Standard Auto Tune report and DFTPP Tune report.

5.10.4.5 Running a Solvent Blank: Following a liner change or at the start of a new run, run an injection of a pure solvent to confirm that the system is free of excessive or interfering contamination. Analyze the solvent in SCAN mode using the same temperature program used for sample analysis. If contamination is present, analyze additional samples of fresh solvent until the interfering contamination is removed.

5.10.4.6 Checking the DFTPP Tune: Prior to running the first calibration standard, verify the instrument tune conditions by running a 10 ng/µL DFTPP check standard to check the mass measuring accuracy of the MS, the resolution sensitivity, the baseline threshold, and the ion abundance ranges. Run the standard using the DFTPP method provided with the instrument. Each of the criteria identified in Table SOP 4.2 of this Appendix must be met before using the instrument for analysis:

TABLE SOP 4.3—Ion Abundance Criteria for DFTPP

Mass, M/z	Relative to mass	Relative abundance criteria	Purpose of checkpoint
51	442	10–80% of the base peak	Low mass sensitivity.
68	69	<2% of mass 69	Low mass resolution.
70	69	<2% of mass 69	Low mass resolution.
127	442	10–80% of the base peak	Low-mid mass sensitivity.
197	198	<2% of mass 198	Mid mass resolution.
198	442	Base peak or >50% of 442	Mid mass resolution and sensitivity.
199	198	5–9% of mass 198	Mid mass resolution and isotope ratio.
275	442	10–60% of the base peak	Mid-high mass sensitivity.
365	442	>1% of the base peak	Baseline threshold.
441	443	Present and < mass 443	High mass resolution.
442	442	Base peak or >50% of 198	High mass resolution and sensitivity.
443	442	15–24% of mass 442	High mass resolution and isotopic ratio.

5.10.4.7 Calibrating with a Multiple-Point Calibration Curve. A 5- or 6-point calibration curve is obtained by running 5 or 6 working standards (see SOP 3) on the tuned GC/MS instrument. Calculate the relative response factor (RRF) for each compound relative to its

corresponding deuterated internal standard as indicated in Table SOP 4.3 of this Appendix. The relative standard deviation (RSD) of the RRFs for each compound must be less than 25%. Run an independently prepared check standard immediately after

the calibration standards to validate the accuracy of the calibration curve.

5.10.4.8 Running Samples. Once the calibration curve has been validated, samples can be analyzed. Dispense 1,000 µL of sample extract into labeled auto-sampler vials. Add

20 µL of the internal standard solution (see SOP 2 of this Appendix) to the extract using a syringe or a positive displacement pipettor. Run a check standard every 10 samples to ensure the consistency of the instrument. The RRF for each compound in the check

standard must be within 25% of the average RRF obtained in the initial calibration.

5.10.4.9 *Quantification*: Once a calibration table has been generated, quantify each data file using the “Calculate and Generate” function in the MS ChemStation

software, or equivalent software. Review individual peak integration manually to ensure proper baseline integration. The quantification of a compound is based on the peak area of the primary ion (Q Ion) indicated in Table SOP 4.4 of this Appendix.

TABLE SOP 4.4—TARGET COMPOUND LIST

Compound name	Quantitation ion	Reference compound for response factor	Internal standard for quantitation
N D34 C16 .....	66	N D34 C16 .....	D34 n C16 Q Ion 66.
n-C14 .....	57	n C14.	
n-C15 .....	57	n C15.	
n-C16 .....	57	n C16.	
N D34 C17 .....	66	N D34 C17.	
n-C17 .....	57	n C17.	
Pristane .....	57	Pristane.	
n-C18 .....	57	n C18.	
Phytane .....	57	Phytane.	
n C19 .....	57	n C19.	
N D42 C20 .....	66	N D42 C20 .....	D42 n C20 Q Ion 66.
n C20 .....	57	n C20.	
n C21 .....	57	n C21.	
n C22 .....	57	n C22.	
n C23 .....	57	n C23.	
N D50 C 24 .....	66	N D50 C 24.	
n C24 .....	57	n C24.	
n C25 .....	57	n C25.	
n C26 .....	57	n C26.	
n C27 .....	57	n C27.	
n C28 .....	57	n C28.	
n C29 .....	57	n C29.	
N D62 C30 .....	66	N D62 C30 .....	D62 n C30Q Ion 66.
n C30 .....	57	n C30.	
n C31 .....	57	n C31.	
N D66 C32 .....	57	N D66 C32.	
n C32 .....	57	n C32.	
n C33 .....	57	n C33.	
n C34 .....	57	n C34.	
n C35 .....	57	n C35.	
D8 Naphthalene .....	136	D8 Naphthalene .....	D8 Naphthalene Q Ion 136.
Naphthalene .....	128	Naphthalene.	
D10 1-Methylnaphthalene .....	152	D10 1-Methylnaphthalene.	
C1 Naphthalene* .....	142	C1 Naphthalene.	
C2 Naphthalene* .....	156	C2 Naphthalene.	
C3 Naphthalene* .....	170	C3 Naphthalene.	
C4 Naphthalene* .....	184	C4 Naphthalene.	
D10 Anthracene .....	188	D10 Anthracene .....	D10 Anthracene Q Ion 188.
D10 Phenanthrene .....	188	D10 Phenanthrene.	
Phenanthrene .....	178	Phenanthrene.	
C1 Phenanthrene* .....	192	C1 Phenanthrene.	
C2 Phenanthrene* .....	206	C2 Phenanthrene.	
C3 Phenanthrene* .....	220	C2 Phenanthrene.	
C4 Phenanthrene* .....	234	C2 Phenanthrene.	
Fluorene .....	166	Fluorene.	
C1 Fluorene* .....	180	Fluorene.	
C2 Fluorene* .....	194	Fluorene.	
C3 Fluorene* .....	208	Fluorene.	
Dibenzothiophene .....	184	Dibenzothiophene.	
C1 Dibenzothiophene* .....	198	Dibenzothiophene.	
C2 Dibenzothiophene* .....	212	Dibenzothiophene.	
C3 Dibenzothiophene* .....	226	Dibenzothiophene.	
Naphthobenzothiophene (NBT) .....	234	Naphthobenzothiophene.	
C1 NBT* .....	248	Naphthobenzothiophene.	
C2 NBT* .....	262	Naphthobenzothiophene.	
C3 NBT* .....	276	Naphthobenzothiophene.	
Fluoranthene .....	202	Fluoranthene.	
D10 Pyrene .....	212	D10 Pyrene.	
Pyrene .....	202	Pyrene.	
C1 Pyrene* .....	216	Pyrene.	
C2 Pyrene* .....	230	Pyrene.	
D12 Chrysene .....	240	D12 Chrysene .....	D12 Chrysene Q Ion 240.
Benzo(a)anthracene/Chrysene* .....	228	Chrysene.	
C1 Chrysene* .....	242	Chrysene.	

TABLE SOP 4.4—TARGET COMPOUND LIST—Continued

Compound name	Quantitation ion	Reference compound for response factor	Internal standard for quantitation
C2 Chrysene *	256	Chrysene.	5 $\alpha$ -androstane Q Ion 245
C3 Chrysene *	270	Chrysene.	
C4 Chrysene *	284	Chrysene.	
5 $\alpha$ -androstane	245	5 $\alpha$ -androstane	
Coprostane	219	Coprostane.	
Hopane	191	Hopane.	

\* Summed compounds; draw an integration line underneath all peaks with selected ion.

5.10.4.10 Equation 14 of this Appendix is used to calculate the concentration of analytes in units of  $\mu\text{g/g}$  oil added:

$$\text{Concentration of analyte } (\mu\text{g/g oil}) = \frac{100 \times A_{\text{analyte}} \times C_{\text{istd}}}{A_{\text{istd}} \times \text{RRF}} \quad (\text{Equation 14})$$

where:

$A_{\text{analyte}}$  = the peak area of the analyte,  
 $C_{\text{istd}}$  = the concentration of the internal standard,

$A_{\text{istd}}$  = the area of the internal standard,  
 RRF = the relative response factor, and  
 100 is the conversion factor to convert mg/L DCM to  $\mu\text{g/g}$  oil added.

5.10.4.11 If some analytes are not commercially available, the RRFs of other compounds (usually the parent compound) are used to quantify those analytes. For example, the RRF of C3-naphthalene may be used to calculate the concentrations of C3- and C4-naphthalenes. See Table SOP 4.4 of this Appendix for details. The quantification of these alkylated PAHs is relative because it is assumed that the molecular ions of the alkylated PAHs have the same RRFs as the parent compound ions. Nevertheless, these relative concentrations are useful for monitoring the fate of these compounds during the course of any analysis, as long as

their concentrations are measured in a consistent way throughout the analysis.

5.10.4.12 Concentration calculations for all target compounds are performed using EnviroQuant software or equivalent. Data for each sample can be printed directly using a customized report template. Data can also be automatically entered into a spreadsheet within the EnviroQuant software.

5.10.5 *Quality Assurance/Quality Control*. The following criteria must be met before any samples are analyzed:

5.10.5.1 Air/water check to verify the system is leak free.

5.10.5.2 AutoTune and DFTPP Tune pass all criteria.

5.10.5.3 DFTPP check standard passes all criteria.

5.10.5.4 Solvent blank scan indicates the GC/MS system is free of interfering contamination.

5.10.5.5 Prepare and monitor a control chart of a standard oil analysis.

Concentrations of the analytes in the control chart must be no more than 25% different from their historical averages.

5.10.5.6 Relative response factors for analytes in the check standards inserted between every 10 samples must be no more than 25 percent different from the average RRF of those same analytes in the calibration curve. Peak shapes must be symmetrical.

#### 5.11 *References for Section 5*

- (1) Haines, J.R., E.J. Kleiner, K.A. McClellan, K.M. Koran, E.L. Holder, D.W. King, and A.D. Venosa. 2005. "Laboratory evaluation of oil spill bioremediation products in salt and freshwater systems." *J. Ind. Microbiol. Biotech* 32: 171–185.

Appendix E to Part 300 [Removed]

#### ■ 17. Remove Appendix E to Part 300.

[FR Doc. 2015–00544 Filed 1–21–15; 8:45 am]

**BILLING CODE 6560–50–P**