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# EPA RCRA DELISTING PROGRAM GUIDANCE MANUAL FOR THE PETITIONER



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### TABLE OF CONTENTS

		<u>Page</u>
SECT	ION 1: INTRODUCTION TO REGION 6 DELISTING	1
1.1	Purpose of EPA Delisting Guidance	1
1.2	Use of the EPA Delisting Guidance	1
1.3	Availability of Additional Guidance	1
1.4	Delisting Guidance Manual Organization	2
SECT	ION 2: EPA DELISTING PROGRAM OVERVIEW	5
2.1	Regulatory Basis and Intent of Delisting	5
2.2	Regulatory Impact of a Delisting Decision	7
2.3	Petition Information	9
SECT:	ION 3: FRAMEWORK FOR DELISTING PETITIONS	15
SECT	ION 4: WASTE MANAGEMENT INFORMATION	17
4.1	Basis for Waste Listing	17
4.2	History of Waste Generation	18
4.3	Waste Volume	18
4.4	Waste Management History	19
SECT	ION 5: WASTE PROCESS INFORMATION	20
5.1	General Information	20
5.2	Special Information	23
SECTION 6: DELISTING ANALYTICAL PLAN SOP		25
6.1	Identifying Constituents of Concern and	
	Hazardous Waste Characteristics (Step 1)	26
6.2	Identifying Constituents of Concern for Special	
	Waste Categories (Step 2)	30
6.3	Conducting an Engineering Analysis to Complete	
	Your List of Constituents of Concern (Step 3)	30
6.4	Identifying Analytes (Step 4)	31

TABLE	E OF CONTENTS (cont)	<u>Paqe</u>
6.5	Demonstrating Without Laboratory Analyses that	
	Certain Constituents Are Not Present in Your	
	Waste at Hazardous Levels (Steps 5 and 6)	32
6.6	Selecting Appropriate Analytical Methods (Step 7)	33
6.7	Identifying Quality Control (QC) Protocols (Step 8)	35
6.8	Summary of Appropriate Analytical Plan Information	37
SECTI	ION 7: DELISTING SAMPLING PLAN SOP	38
7.1	Defining Sampling Objectives	38
7.2	Selection of an Appropriate Sampling Strategy	39
7.3	Sampling Plan Considerations	41
7.4	Examples of Sampling Strategies for Delisting	
	Demonstrations	43
7.5	Recommended Elements of a Written Sampling Plan	48
SECTI	ION 8: WASTE SAMPLING AND ANALYSIS INFORMATION	50
8.1	Waste Sampling Information	50
8.2	Waste Analysis Information	53
8.3	Quality Control Information	55
	LIST OF EXHIBITS	
D		
EXHIE	<u>31'T</u>	
1	STEPS TO IDENTIFY CONSTITUENTS OF CONCERN AND	
	DEVELOP AN ANALYTICAL PLAN	27
2	ANALYSES FOR CONSTITUENTS OF CONCERN AND HAZARDOUS	
	WASTE CHARACTERISTICS	28
3	CONSTITUENTS OF CONCERN FOR WASTES FROM PETROLEUM	
	PROCESSES	29

#### APPENDICES

- A. FRAMEWORK FOR EPA HAZARDOUS WASTE DELISTING PETITIONS
- B. CHECKLIST OF REQUESTED PETITION INFORMATION
- C. USE OF RANDOM NUMBER TABLE
- D. PRE-PETITION SCOPING MEETING CHECKLIST
- E. LIST OF EPA REGIONAL DELISTING CONTACTS
- F. LIST OF EPA STATE HAZARDOUS WASTE OFFICES AND STATES AUTHORIZED FOR DELISTING
- G. POLICY ISSUES
  - 1. AMENDMENT TO SUBPART C RULEMAKING PETITIONS; GROUNDWATER DATA IN DELISTING DECISIONS
  - 2. NOTICE OF DELISTING STRATEGIES AND PROCEDURES
  - 3. OIL & GREASE FEDERAL REGISTER NOTICE
  - 4. MEMO NATIONAL POLICY FOR HAZARDOUS WASTE DELISTINGS PETITIONS
- H. REGION SPECIFIC DELISTING GUIDANCE
  - 1. Region 2 Delisting Information
  - 2. Region 3 Quality Control Requirements for Delisting
  - 3. Region 6 Elements of Region 6's Delisting Evaluation Process
- I. EVALUATION FORM

# SECTION 1 INTRODUCTION TO EPA DELISTING

#### 1.1 Purpose of Delisting Guidance

This manual gives guidance to those who are interested in submitting a petition to exclude or "delist" a listed hazardous waste produced at a particular facility from the lists of hazardous wastes in 40 CFR Part 261, Subpart D. The U.S. Environmental Protection Agency (EPA) recognizes that a specific listed waste produced at a particular facility may not meet the standards for which the waste was originally listed. The 40 CFR §§ 260.20 and 260.22 contain procedures where any individual can petition the Agency for a regulatory amendment to exclude a listed waste produced at a particular facility. In general, the effect of an exclusion is to allow the management of the excluded waste as a non-hazardous solid waste.

#### 1.2 Use of the Delisting Guidance

This guidance manual will help you decide if you want to prepare a petition. It will also show you how to prepare a petition. Section 2 provides information on the regulatory basis and intent of delisting, which wastes may be eligible for a delisting, and the elements of the delisting process. Subsequent sections provide guidance in meeting the specific information needs for delisting petitions. We recommend that you read the entire manual and become fully familiar with the petition review process and information needs before going on with petition preparation.

Within this guidance manual, we often recommend other EPA-published documents for more detailed information or guidance. The review of additional documents particularly applies to identifying appropriate analytical methods for waste testing. Waste testing information can be found in the EPA publication "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846. If you choose to use a commercial laboratory experienced in analyzing waste matrices, the laboratory must be familiar with SW-846 protocols (including all updates). You may order SW-846 and other EPA publications from the Government Printing Office by calling (202) 783-3238.

Appendix A to this manual contains a "Framework for Delisting Petitions" which you may use while preparing a delisting petition. The format of the Framework follows the order of information needs addressed within the main body of the guidance manual.

## 1.3 Availability of Additional Guidance

After review, you may still need additional or more specific guidance. To get further guidance, you may write to any of the regional offices (see Appendix E). For general information regarding the hazardous waste regulations, you may contact the RCRA Hotline, toll-free at (800) 424-9346.

### 1.4 Delisting Guidance Manual Organization

The remaining sections and content of this manual are summarized below.

# Section 2.0 - DELISTING PROGRAM OVERVIEW

This section discusses the regulatory basis and intent of delisting, the impact of a delisting decision, and the elements of the delisting process.

#### Section 3.0 - FRAMEWORK FOR DELISTING PETITIONS

This section presents guidance in providing administrative information (e.g., name and address of the petitioner and names of contacts).

#### Section 4.0 - WASTE MANAGEMENT INFORMATION

This section presents guidance in describing the petitioned waste and past, present, and future waste management practices.

#### Section 5.0 - WASTE PROCESS INFORMATION

This section presents guidance in describing facility operations, processes and materials contributing to the petitioned waste, and current waste management operations.

#### Section 6.0 - DELISTING ANALYTICAL PLAN SOP

This section provides guidance in identifying the constituents of concern for the petitioned waste and developing a plan for waste testing.

#### Section 7.0 - DELISTING SAMPLING PLAN SOP

This section provides guidance in writing a plan for collecting representative samples of the petitioned waste.

#### Section 8.0 - WASTE SAMPLING AND ANALYSIS INFORMATION

This section provides guidance in supplying appropriate waste sampling and analysis information.

# Appendix A - FRAMEWORK FOR DELISTING PETITIONS

This appendix contains a delisting petition framework for use in preparing delisting documentation.

#### Appendix B - CHECKLIST OF REQUESTED PETITION INFORMATION

This appendix contains a checklist that summarizes the information requested for most delisting petitions.

Appendix C - USE OF RANDOM NUMBER TABLE

This appendix contains guidance in using a random number table to choose waste sampling points.

Appendix D - PRE-PETITION SCOPING MEETING CHECKLIST

This appendix provides a list of some of the topics and information that will be discussed in the Pre-Petition Scoping Meeting.

Appendix E - LIST OF EPA REGIONAL DELISTING CONTACTS

This appendix contains list of the EPA regional delisting contacts.

Appendix F - LIST OF STATE HAZARDOUS WASTE OFFICES AND STATES AUTHORIZED FOR DELISTING

This appendix contains a list of the state hazardous waste offices in EPA and a list of the states authorized for the delisting program.

Appendix G - POLICY ISSUES

AMENDMENT TO SUBPART C - RULEMAKING PETITIONS; USE OF GROUND-WATER DATA IN DELISTING DECISIONS

This contains a copy of a  $\underline{\text{Federal}}$   $\underline{\text{Register}}$  notice (54  $\underline{\text{FR}}$  41930, October 12, 1989) to amend the regulations and clarify Agency authority to consider ground-water monitoring data in the evaluation of delisting petitions.

NOTICE OF DELISTING STRATEGIES AND PROCEDURES

This contains a copy of a  $\underline{\text{Federal}}$   $\underline{\text{Register}}$  notice (53  $\underline{\text{FR}}$  6822, March 3, 1988), that clarifies certain delisting policies, including the policy of petition dismissal.

OIL & GREASE FEDERAL REGISTER NOTICE

This contains a copy of a  $\underline{\text{Federal}}$   $\underline{\text{Register}}$  notice (64  $\underline{\text{FR}}$   $\underline{\text{26315}}$ , May 14, 1999) to delete Method 9070 and 9071 for oil and grease and replaces it with Method 1664.

Appendix G - POLICY ISSUES (continued)

MEMO -NATIONAL POLICY FOR HAZARDOUS WASTE DELISTING PETITIONS

This contains the July 1, 1998, memo from Elizabeth Cotsworth to state the national policy as pertains to "conditional delistings" and the recommended "reopener language" for delisting petitions.

#### Appendix H - REGION SPECIFIC DELISTING GUIDANCE

This appendix contains a list of guidance that is specific to petitioners in specific regions.

#### Appendix I - EVALUATION FORM

This appendix contains an evaluation form for users of this manual to complete so that EPA may improve future guidance documents.

# SECTION 2 DELISTING PROGRAM OVERVIEW

We believe that each delisting petitioner should understand the regulatory basis and intent of the delisting process. Each petitioner should also understand the delisting program and the steps followed during the petition review and decision-making process. This understanding will help the delisting process for both the petitioner and the Agency.

This section gives an overview of the delisting program. It begins by discussing the regulatory framework and the regulatory impact of a waste exclusion. We then provide instructions on how to send a delisting petition and a description of how petitions are processed from petition receipt to final rulemaking.

#### 2.1 Regulatory Basis and Intent of Delisting

Under the regulations implementing Subtitle C of RCRA, wastes are designated as hazardous in two ways:

- (1) solid wastes that exhibit certain characteristics (those listed in 40 CFR Part 261, Subpart C) and
- (2) solid wastes that are specifically listed as hazardous (those listed in 40 CFR Part 261, Subpart D).

According to Subpart C, wastes that are characteristically hazardous remain so until they no longer exhibit the characteristic. You (the generator) are responsible to find out whether a solid waste exhibits a hazardous waste characteristic.

The Agency found that a number of wastes should be broadly listed in Subpart D as hazardous for several reasons:

- (1) the wastes may contain significant levels of toxic and/or carcinogenic constituents,
- (2) the wastes exhibit one or more of the hazardous waste characteristics., and/or
- (3) the wastes may cause specific detrimental effects on the environment.
- Subpart D identifies wastes as hazardous according to the following categories:
  - Wastes generated from non-specific sources (40 CFR §261.31). The respective EPA Hazardous Waste Numbers for such wastes are preceded by an "F" (e.g., F019 Wastewater treatment sludges from the chemical conversion coating of aluminum).

- Wastes generated from specific sources (40 CFR §261.32). The respective EPA Hazardous Waste Numbers for such wastes are preceded by a "K" (e.g., K061 Emission control dust/sludge from the primary production of steel in electric furnaces).
- Discarded commercial chemical products, off-specification species, container residues, and spill residues thereof (40 CFR §261.33)- The respective EPA Hazardous Waste Numbers for such wastes are preceded by a "P" (e.g., P056 Fluorine) or a "U" (e.g., U019 Benzene).

The three-digit number following the "F", "K", "P", or "U" code has no special significance, other than that it identifies a particular listed waste in a particular hazardous waste category.

There are two other ways in which a solid waste can be designated as a listed hazardous waste: (1) any mixture of listed hazardous waste and solid waste is also a listed hazardous waste (40 CFR § 261.3(a)(2)(iv), the "mixture rule"); and (2) any solid waste generated from the storage, treatment, or disposal of a listed hazardous waste is itself a listed hazardous waste (40 CFR § 261.3(c)(2) (i), the "derived-from rule"). These wastes are also eligible for exclusion and remain hazardous wastes until excluded. On December 6, 1991, the U.S. Court of Appeals for the District of Columbia vacated the "mixture/derived from" rules and remanded them to the Agency on procedural grounds (Shell Oil Co. v. EPA, 950 F.2d 741 (D.C. Cir. 1991)). On March 3, 1992, EPA reinstated the mixture and derived-from rules, and solicited comments on other ways to regulate waste mixtures and residues (57 FR 7628). These rules became final on October 30, 1992. See (57 FR 49278). You should consult these references for more information regarding mixtures derived from wastes.

We recognize that a listed waste generated at a particular facility may not be hazardous. Individual wastes may vary depending on raw materials, industrial processes, and other factors. Therefore, 40 CFR §§ 260.20 and 260.22 contain a procedure whereby anyone can petition EPA to exclude or "delist" such a listed waste.

Originally, the overall intent of the delisting process was to ease the regulatory burden on handlers of listed waste improperly captured by the broad listing definitions. Delisting has since evolved to also include listed wastes that are sufficiently treated such that they no longer pose a health threat.

To be eligible for an exclusion, a listed waste must not:

- Meet the criteria for which it was listed.
- Exhibit any of the hazardous waste characteristics.

In addition, a listed waste must not exhibit any other factors (including additional constituents) that could cause the waste to be a hazardous waste, unless we find out that such factors do not warrant characterizing the waste as hazardous.

A listed waste may also exhibit a hazardous waste characteristic. For

example, a wastewater treatment sludge from electroplating operations that is listed as EPA Hazardous Waste Number F006 may exhibit the characteristic of toxicity for lead. Because it exhibits a hazardous waste characteristic, this waste (and any characteristic hazardous waste) is not eligible for delisting (40 CFR  $\S$  260.22(d)(3)).

#### 2.2 Regulatory Impact of a Delisting Decision

The submittal of a delisting petition does not automatically relieve you from any of the hazardous waste management requirements. Until a delisting decision becomes effective (i.e., on the effective date of the Final Exclusion Rule), your petitioned waste is still considered a listed hazardous waste and subject to regulation under 40 CFR Parts 260 through 268 and the permitting standards of 40 CFR Part 270. If we deny your petition, your waste remains hazardous. If we grant your petition, you will receive one of the three types of exclusions -- a standard exclusion, a conditional exclusion, or an upfront exclusion.

Standard Exclusions - We grant standard exclusions when a petition adequately demonstrates that the candidate waste meets the delisting criteria. A waste that receives a standard exclusion is no longer subject to regulation as a listed hazardous waste. Depending on the waste characteristics, the exclusion may need limited further testing of the waste. Standard exclusions generally fall into one of two categories, depending on the type of petitioned waste.

These categories are:

- ! Standard exclusions for "Source Wastes" A "source waste" is a waste that is-currently generated (typically, as a discharge from a specific point in a process) and will be generated in the future. These exclusions typically apply only to wastes generated after the effective date of the exclusion.
- "One-time" Standard Exclusions "One-time" exclusions are granted for discrete volumes of wastes, typically generated in the past, such as the waste contained in a surface impoundment. For example, an electroplating facility that treats process wastewaters in an on-site treatment system sends a petition to delist a wastewater treatment sludge, which is listed as EPA Hazardous Waste No. F006. The petitioner claims that the manufacturing and treatment processes operate continuously and the raw materials do not change and are not expected to change in the If our evaluation of the petition shows that the waste meets the future. delisting criteria, the petitioner would likely receive a standard waste exclusion.
- Conditional Exclusions We grant conditional exclusions when the petitioned waste meets the criteria for delisting, yet we believe the waste may exhibit future variability that may be of concern. Under a conditional exclusion, we set up post-exclusion testing requirements that the petitioner must meet prior to waste disposal.

For example, a pharmaceutical manufacturer produces many products from different processes at its facility. All process wastewaters, some of which are listed wastes, are combined and incinerated on site. manufacturer sends a petition to delist the bottom ash generated by the The petition shows that the composition of the influent to incinerator. the incinerator is variable although the petitioner believes the petition data sufficiently characterize this variability. Because of the many different contributing processes, however, we are concerned that the waste composition may exhibit future variability that was not sufficiently characterized. Therefore, if our evaluation shows that the waste meets the delisting criteria, the petitioner would likely receive a conditional waste exclusion, which includes testing and data verification on batches of the bottom ash prior to disposal. Only those batches that meet the conditions provided in the final exclusion could be managed as nonhazardous waste; the remainder must either be re-treated or managed as hazardous.

Upfront Exclusions - We grant upfront exclusions for wastes and/or waste residues that have not yet been generated, but will be generated in the future, based on available information (e.g., pilot-scale system data) that demonstrates that the petitioned waste will most likely meet the delisting criteria. For example, if you are considering installing a new waste treatment system, you may sends a petition for an upfront exclusion of the waste that will be generated. Our decision on your upfront petition will be based on an evaluation of the characteristics of your untreated waste, process descriptions, and data from a bench- or pilot-scale waste treatment process. If you meet the criteria for a delisting, your upfront exclusion would need testing from the full-scale treatment system to verify that, once on-line, the full-scale system is operating as described in your petition.

For example, a landfill received an F006 waste and generates a hazardous leachate. Operators would like to construct and operate a leachate treatment system to render the leachate non-hazardous. However, they do not want to initiate construction of the full-scale treatment system until they are confident that the treated leachate can be delisted. Therefore, operators performs pilot-scale studies on the treatment system and sends a petition for an upfront delisting. If we decide to grant an exclusion, the treated leachate would receive an upfront delisting with verification testing requirements, which must be met when the full-scale system becomes operational. The full-scale verification testing requirements may involve more than one round of waste characterization (e.g., testing every batch before disposal) to address any concerns regarding waste variability.

Should the full-scale process become operational and generate the petitioned waste during the review of your petition, you must also send the full-scale process information and waste analysis data.

#### 2.2.1 What an Exclusion Will Do

Once delisted, your petitioned waste is no longer considered a listed hazardous waste and, thus, you may manage it as a non-hazardous solid waste (under Subtitle D).

#### Please note:

- ! You remain obligated to find out whether your waste remains non-hazardous based on the hazardous waste characteristics.
- You must treat, store, or dispose your excluded waste in an on-site facility; or make sure that the waste is delivered to an off-site storage, treatment, or disposal facility; either of which is permitted, licensed, or registered by a state to manage municipal or industrial solid waste.
- ! We urge you to contact your State regulatory authority to find out the status of your excluded waste under State law.

#### A reminder:

- Your exclusion only applies to the waste covered by your original demonstration.
- A process change to any aspect of your manufacturing or treatment processes generating the petitioned waste may change the composition of your waste and thus your waste may not be covered by the exclusion.
- When processes are altered such that a significant change in waste composition or waste volume occurs, the new waste must be managed as hazardous until a new exclusion is granted, and you must file a new petition for the altered waste.

### 2.3 Petition Information

The requirements for delisting petitions as set forth in 40 CFR § 260.20 and 260.22 state that each petition must include certain information. Furthermore, as stated in 40 CFR § 260.22(j), we may request any additional information reasonably needed to evaluate a submitted petition.

Appendix B contains a checklist which summarizes the information that should be submitted for most delisting petitions.

You may use this checklist, in conjunction with the remaining sections of this manual, as a guide during petition development. However, delisting petition information requirements depend on the petitioned waste type and, therefore, this list is not meant to be comprehensive. Thus, all petitioners should follow the detailed guidance found in each referenced section of this manual to identify all information needs. Again, prior to sending a formal petition, you are needed to

meet in person and discuss with our staff the nature and extent of information that should be included in your petition.

If you are sending your petition to a state which is authorized to grant delistings, you should recognize that the state's delisting program may request additional information. You should contact their hazardous waste office for its specific delisting requirements.

#### 2.3.1 Submitting the Petition

As stated in 40 CFR § 260.22(a), anyone seeking to exclude (delist) a waste generated at a particular facility from the lists in Subpart D of 40 CFR Part 261 may send a petition to the Administrator. Only a waste identified as a listed waste in Subpart D of 40 CFR Part 261 can be considered for delisting.

You must send a separate petition for each distinct waste generator. submitted for wastes particular, separate petitions must be generated different facility locations, even if the contributing processes materials are similar. This requirement is necessary because an amendment to 40 CFR Part 261 for an exclusion only applies to a waste produced at a particular facility.

You also should consider whether your petition should be submitted to the State in which the petitioned waste is generated/managed, to the Federal government (i e., the EPA), or to both the State and Federal authorities. Some States are authorized to make delisting decisions without any review by the Federal government. Alternatively, a petitioner may need to get a State delisting in addition to the Federal delisting to legally manage the waste as a non-hazardous waste. In all cases, however, if the waste is transported to another state, a delisting granted by an authorized State does not exempt the waste from regulation as hazardous.

Currently, there are 19 states which have been authorized for delisting. You will find a list of those states in Appendix F. A delisting issued by an authorized State is only in effect within that State. If the petitioner wishes to also transport its waste to an <u>unauthorized</u> State and manage it as non-hazardous there, an additional EPA delisting is required. The waste remains listed in all other States. If your State is not authorized, send your petition to the appropriate Regional office. The original copy of the petition should be sent by certified mail to the regional contacts listed in Appendix E. And a copy should be provided to your State Hazardous Waste Office. (See Appendix F)

Should your petition to delist be approved, you will be needed to provide a one-time notice to each State's hazardous waste office through which/to which your petitioned waste will be transported.

You may assert a business confidentiality claim covering information submitted as part of your petition. Confidentiality claims may be asserted at any time, but should be applied only to the specific portions of your submittal which need to remain confidential. If you choose to assert a confidential

business information (CBI) claim for certain petition information, you should specify this intent in the letter transmitting your petition. In the letter, you should clearly identify which information should be treated as CBI and specify the pages of your petition containing that information. The CBI claim should be identified in red on each page containing CBI. Disclosure and C treatment of information declared as CBI will be in strict accordance with the procedures set forth in 40 CFR Part 2, Subpart B (42 FR 36902-36924, September 1, 1976, as amended by 43 FR 39997, September 8, 1978, and 44 FR 17673, March 23, 1979). All pages marked as containing CBI information are logged in with our CBI Document Control Officer, who is responsible for the security of the information.

You must include a certification statement signed by an authorized representative with your petition and with each petition addendum, including the submission of any additional information we request. This certification statement can be found in 40 CFR § 260.22(i)(12). If, at any time during the delisting petition process, any petition information becomes inaccurate, you must notify us of the new information (e.g., changes in names of contacts, facility ownership, waste management practices, waste generation rates, raw materials, the process operating cycle).

Our intent is to review petitions in chronological order of receipt. For example, a petition received in May of 1999 will typically have higher priority in the review process than a petition submitted in January of 2000. You will be notified by a letter acknowledging receipt of your petition. This letter will also identify the individual assigned to review your petition. You may contact this person regarding petition status.

The appropriate State hazardous waste office will be notified that we have received a delisting petition from a facility located in their State.

### 2.3.2 Petition Completeness Review by the Agency

During the review of a delisting petition:

- The Agency first confirms whether the petitioned waste is listed as hazardous in 40 CFR Part 261, Subpart D and, therefore, eligible for an exclusion.
- If the waste is a listed waste, then we conduct a completeness review to find out whether the petition is seriously deficient. A seriously deficient delisting petition generally lacks necessary information that needs more than 15 days to collect and send. If our review shows that a petition is seriously deficient, we will notify you by letter and phone of EPA's intent to dismiss the petition and provide an opportunity for you to respond.

If your petition is not seriously deficient:

will find out whether any additional information is necessary to conduct a complete technical review.

- In cases where available information supports petition denial, information needed to complete the petition will not be requested.
- If additional information is needed to complete our evaluation, will send you a letter outlining the requested information. You will have up to 15 days from receipt of the letter to send all of the requested information.
- If EPA does not receive a complete response to our additional information request within the designated time period, we will begin the denial process or the petitioner may request to withdraw the petition.

EPA urges you to send the requested information as soon as it is available. We recommend this because early submittal will (1) allow us to identify any remaining deficiencies related to the additional information submittal, and (2) provide you with the opportunity to address the remaining deficiencies. The additional information request letter will also offer you the option to withdraw your petition and resubmit a complete petition at a later date. When your petition is complete, we will perform a technical evaluation of all submitted information.

If your petition is found to be seriously deficient, or if you have not provided a complete response to our request for further information within the designated time period, you will be notified by letter that we intend to begin the denial process or the petitioner may request to withdraw your petition. A withdrawal will remove your petition from the review process and close your petition file. In the event of petition withdrawal, you may send a new petition after you collect all the necessary information. Resubmitted petitions received will be reviewed in the chronological order of the new submission.

## 2.3.3 Petition Technical Review

In order for your waste to be excluded, as stated in 40 CFR  $\S$  260.22, your petition must demonstrate that the waste:

- Does not meet the criteria for which it was listed.
- Does not exhibit any of the hazardous waste characteristics (i.e., ignitability, reactivity, corrosivity, and toxicity).
- Does not exhibit any additional factors, including additional constituents, which may cause the waste to be a hazardous waste (unless we find that such factors do not warrant characterizing the waste as hazardous).

Delisting petitions are evaluated on a waste-specific basis. In our technical evaluation, we often use appropriate fate and transport models that rely on waste-specific information (e.g., waste volume, constituent concentration data) to predict the potential environmental impact of the petitioned waste. In

selecting appropriate models, we choose a reasonable worst-case management scenario and consider plausible exposure routes for the hazardous constituents found to be present in the petitioned waste.

In some cases, we also consider ground-water monitoring data during technical evaluation. In general, you should send ground-water monitoring information if you currently manage, or ever have managed, the petitioned waste in a land-based waste management unit for which a ground-water monitoring system is required under 40 CFR Part 264 or 265 or authorized state equivalent; if collection of such information is mandated under other Federal, state, or local requirements; or if such information is otherwise available for the unit. In cases where the petitioned waste is managed in a land-based waste management unit, we assess relevant information to find out whether the petitioned waste has adversely impacted the underlying ground water. Appendix G contains a copy of a Federal Register notice to amend 40 CFR § 260.22 that clarifies our ground-water monitoring information requirements.

Once the technical evaluation has been completed, EPA will decide whether to grant or deny your petition. If we believe the waste is hazardous and thus decide to deny your petition, we will first send you a letter notifying you of our intent. In this denial letter, we will offer you the option to withdraw the petition to avoid publication of a negative finding in the <u>Federal Register</u>.

If a petition recommended for denial is not withdrawn or if we believe that a petitioned waste is non-hazardous and should be granted an exclusion, a draft <a href="#">Federal Register</a> notice is prepared. The review process then moves on to promulgation of the delisting decision.

### 2.3.4 Promulgation of Decisions

In accordance with 40 CFR § 260.20(c), Agency-proposed decisions delisting petitions are published in the Federal Register. Upon completion of the technical evaluation (as long as a petition is not withdrawn before then), <u>Federal</u> <u>Register</u> notice is drafted that presents information petitioned waste, our evaluation of the petitioned waste, and our proposed decision to grant or deny the petition. Once drafted, the delisting Federal Register notice is peer reviewed by EPA representatives. After evaluating peer review comments and revising the notice as necessary, the Region then sends the notice to its Office of Regional Council (ORC). If the ORC agrees with the decision, the notice is sent through various EPA management levels for review and Final approval for waste exclusions must be received from the Regional Administrator (or delegated representative). Final approval for petition denials must be received from the Regional Administrator (or delegated representative).

Upon publication of a proposed rule, a complete copy of the delisting petition and all supporting data are provided to the EPA RCRA docket office. EPA provides the public with an opportunity to comment before a decision is finished, in accordance with 40 CFR § 260.20(c) as mandated by the Hazardous and Solid Waste Amendments (HSWA) of 1984. The public comment period for proposed notices on delisting decisions is usually 45 days. During this time, interested persons

may send written comments on the proposed regulation.

A final notice is published in the <u>Federal Register</u> once all public comments on the proposed Notice are addressed. Final delisting rules typically become effective as of the date of publication in the <u>Federal Register</u>. HSWA amended Section 3010 of RCRA to allow rules to become effective in less than six months when the regulated community does not need the six-month period to come into compliance. In the case of a final delisting petition denial, the petitioner must to continue managing the waste as hazardous and thus a period of compliance is not necessary. In the case of a final exclusion, a six-month period generally is not necessary to come into compliance because the final rule reduces the existing waste management requirements. A final decision that excludes a petitioned waste also amends 40 CFR Part 261, Appendix IX, to include the delisted waste.

# SECTION 3 FRAMEWORK FOR DELISTING PETITIONS

This section informs you how to provide the needed administrative information in the delisting petition application. The Region needs this information to identify the petitioner, the location of the petitioned waste, sources for additional information, and the requested delisting action for the petitioned waste, as well as to get an overview of the need and basis for the delisting action. We also ask that you sign a statement certifying that information contained in the petition is not false.

The regulatory basis for most of the administrative information that should be provided is found in 40 CFR § 260.20 and § 260.22; and is identified within parentheses, when applicable. The information requested in this section corresponds with Part 1 of the "Framework for Delisting Petitions" found in Appendix A. You should include the following administrative information in your petition:

The name and mailing address of the individual or firm sending the petition (40 CFR 260.20(b)(1)).

The names, titles, addresses, and telephone numbers of people to contact for additional information pertaining to the petition. (You should notify us if these designated contacts change after petition submission.)

- The name and location of the facility responsible for generating the waste covered by the petition (40 CFR § 260.22(i)(4)), and the facility RCRA identification number. This information should also be provided for the location of the petitioned waste, if different from the generating facility.
- A description of the proposed delisting action which is supported by the remainder of the petition (40 CFR § 260.20(b)(3)). For example, a company may petition to exclude a hazardous waste from a non-specific or specific source from the lists of hazardous wastes or to amend a previously granted exclusion.
- A statement of your interest in the proposed delisting action (40 CFR § 260.20(b)(2)) and a statement of the need and justification for the proposed action (40 CFR § 260.20(b)(4)). The latter statement should explain why you need the waste delisted and why you believe it should be delisted.
- ! The following certification statement signed by the generator or an authorized representative (40 CFR § 260.22(i)(12)):

"I certify under penalty of law that I have personally examined and am familiar with the information submitted in this demonstration and all attached documents, and that, based on my inquiry of those individuals immediately responsible for getting the information, I believe that the submitted information

is true, accurate, and complete. I am aware that there are significant penalties for sending false information, including the possibility of fine and imprisonment."

An "authorized representative" is a person responsible for the overall operation of a facility or an operational unit (for example, a plant manager, superintendent, or person of equivalent responsibility) (See 40 CFR § 260.10.). Consultants or other outside parties should not sign the certification statement. The name and title of the authorized representative should be typed under the signature.

# SECTION 4 WASTE MANAGEMENT INFORMATION

This section tells you how to provide information that describes the waste and waste quantity that you are petitioning to delist. The regulatory basis for this information is found in 40 CFR § 260.22(i)(6). We need the information to fully understand the specific type and amount of waste for which a delisting is requested. The information should be complete and accurate.

This section also tells you how to provide information about the past, present, and future management of the waste. EPA requests this information to evaluate historical waste management practices and to identify the most likely management scenario for the waste if the petition is granted. The information requested in this section corresponds with Part 2 of the "Framework for Delisting Petitions" found in Appendix A.

This section is organized as follows:

- 4.1 Basis for Waste Listing
- 4.2 History of Waste Generation
- 4.3 Waste Volume
- 4.4 Waste Management History

#### 4.1 Basis for Waste Listing

You should clearly and completely describe why your waste is classified as a listed hazardous waste. The basis for listing hazardous wastes is found in 40 CFR Part 261. The lists of hazardous wastes are presented in 40 CFR  $\S$  261.31 (hazardous wastes from non-specific sources),  $\S$  261.32 (hazardous wastes from specific sources), and  $\S$  261.33 (discarded commercial chemical products, off-specification species, container residues, and spill residues thereof).

You should provide the following information, as appropriate, regarding the petitioned waste:

- ! The EPA Hazardous Waste Number(s), the appropriate hazardous waste description(s) for the petitioned waste (i.e., the appropriate waste listing description(s) found in 40 CFR Part 261) and the common name(s), if any, that may be used for the waste at the facility (e.g., Tank No. 2 sludge). If the petitioned waste is:
  - A mixture of two or more listed hazardous wastes, provide the EPA Hazardous Waste Number and appropriate hazardous waste description for each listed waste. Also identify the common name(s) of each waste, if any.
  - A mixture of one or more solid waste(s) and one or more listed hazardous wastes, as described in 40 CFR § 261.3(a)(2) (iii-iv); provide the EPA Hazardous Waste Number(s), the appropriate hazardous waste description for each listed hazardous waste, and a description

of the solid waste(s) that are mixed with the listed waste(s). Also identify the common name(s), as appropriate.

- Generated from the treatment, storage, or disposal of one or more listed hazardous wastes (or solid and listed hazardous waste mixture), as described in 40 CFR § 261.3(c)(2)(i); provide the Hazardous Waste Number and appropriate hazardous waste description for each listed hazardous waste, a description of each solid waste (if applicable), and a description of the petitioned waste (e.g., if the petition is for an ash generated from the incineration of listed Hazardous Waste Nos. F002, F004, and F005, present the requested information on all three hazardous waste numbers and then describe the ash). Identify the common name(s).
- The physical form of the petitioned waste (e.g., sludge, liquid, solid, ash). If the waste is considered a sludge or a liquid, provide an estimate (e.g., a range) of the percentage of solids in the waste (through analysis of the waste).

#### 4.2 <u>History of Waste Generation</u>

You should show in your petition which of the following applies to your waste (one or more may apply):

- The waste has been generated in the past.
- The waste is presently being generated.
  - The waste will be generated in the future.

If the waste is presently being generated or was generated in the past, we also request that you provide the year when waste generation began (and ended, if applicable) at your facility.

### 4.3 Waste Volume

You should provide an estimate of the waste volume that you are petitioning to delist. [Reminder: A waste exclusion, typically will only apply to that waste volume documented in the delisting petition.] If the petitioned waste is a fixed quantity (or will be when generated in the future), simply estimate that volume. This quantity may be obtained from information such as unit dimensions or operating records.

If the waste is (or will be) generated on a routine or continuous basis (e.g., from a process point discharge), based on your operating records (pilotor full-scale, as appropriate) estimate the following generation rates:

- Average volume generated monthly.
- Average volume generated annually.
- Maximum volume generated monthly.
  - Maximum volume generated annually.

You should also describe the method of calculation used to estimate the fixed quantity or generation rates. If volume is not a convenient measurement, you may instead provide the weight of the waste (clearly indicate the units) and a representative value for the density of the waste.

#### 4.4 Waste Management History

A description of your past, present, and future disposal methods is needed to evaluate historical waste management practices. We also need this information to identify the most likely waste management scenario should your petition be granted. Therefore, you should provide the following information:

- How the waste is managed at the present and the names, locations, and waste management methods of any off-site waste management facilities used.
- How the waste was managed in the past, if different from the present, and the names, locations, and waste management methods of any off-site waste management facilities used.
- How the waste will be managed if your delisting petition is granted; and, if known, the names, locations, and waste management methods of any off-site waste management facilities you may use.

# SECTION 5 WASTE PROCESS INFORMATION

This section tells you how to provide information on all processes and raw materials contributing to your petitioned waste. The regulatory basis for this information is found in 40 CFR § 260.22(i)(5). EPA needs this information to find out whether the petitioned waste was characterized with respect to all delisting constituents of concern potentially present due to the contributing processes.

Provide information on waste management operations if the petitioned waste (or was) managed in land-based units or derived from waste (e.g., excavated soils remaining from unit operations closure). sufficient detail to understand all processes generating and contributing to the petitioned waste, how and where in the processes the waste is (or was) formed, and how hazardous constituents may enter the waste. From this information, we will verify the waste listing and also find out whether there are likely to be any hazardous constituents in the waste other than those identified in your petition, and whether other hazardous constituents are likely to be formed in the production and/or treatment processes or during waste management. information requested in this section corresponds with Section C of "Framework for Delisting Petitions" found in Appendix A.

This section is divided into two major subsections: 5.1, "General Information" and 5.2, "Special Information." Section 5.1 explains how to provide information on processes and materials that may contribute to your waste. Section 5.2 identifies the additional information that should be provided in a petition for the delisting of a waste that is not currently generated ("upfront exclusion") and for a waste generated by a multiple waste treatment facility (MWTF).

If you wish, you may assert a confidential business information (CBI) claim for any of the information submitted. Section 2.3 of this Guidance Manual describes how to assert a CBI claim.

#### 5.1 General Information

You should provide the information noted below to fully describe general operations, manufacturing and waste treatment processes, process materials, waste management operations, and any other facility operations that might contribute to the petitioned waste. If your petition is for an upfront exclusion or for a waste generated by in MWTF, you should provide all the information requested below, as appropriate, and also provide the information described in Section 5.2, "Special Information."

#### 5.1.1 <u>General Operations</u>

You should provide the following information regarding general operations at the generating facility:

- General descriptions of facility business area(s) and operations, including all appropriate standard Industrial Classification (SIC) codes.
- A listing and description of products manufactured and wastes (including all hazardous wastes) generated at the facility.
- A description of the manufacturing and waste treatment areas and waste management units along with schematics showing the general layout at the facility.
- A description of the regulatory status of all on-site waste treatment, storage and disposal units, including a listing of all hazardous waste permits and other permits (e.g., NPDES) issued under Federal and State environmental statutes. Provide the permit numbers in this listing.

#### 5.1.2 Manufacturing Processes

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You should provide the following appropriate information on all manufacturing processes that may contribute to the petitioned waste:

- A description and a schematic of all "pre-process" steps used to prepare materials for processing before the primary manufacturing operations, including descriptions of such operations as surface and equipment preparation (e.g., machining, degreasing, cleaning, coating). Identify all pre-process material inputs and outputs in your descriptions and schematics.
- A description and schematic of each step of each manufacturing process contributing to the petitioned waste. The description and schematic should include each process step, reactions occurring, and material inputs and outputs. Identify any reaction intermediates and byproducts formed during the manufacturing process. Also describe and indicate on the schematic each waste produced and how each waste is managed.
- A description of all manufacturing process equipment, equipment functions, and the ranges of the operating parameters.
- A description of all operating cycles (e.g., batch cycles versus continuous operation; start-up, shut-down, and other process transients; and maintenance and cleaning operations) on a daily, weekly, or other period basis, as appropriate. Identify periods when process wastes are not generated (e.g., plant shutdowns or routine equipment maintenance).
- An assessment of the extent that all contributing manufacturing processes, operations, process materials, or generated wastes have varied in the past and may vary in the future, whether due to

manufacturing processes or seasonal variations in production.

#### 5.1.3 <u>Waste Treatment Processes</u>

If your waste is <u>not</u> generated directly from a manufacturing process, then you should provide the following information:

- A description and schematic of each step of each waste treatment process contributing to the petitioned waste. The description and schematic should include each process step, reactions occurring, flow rates, material inputs, and waste inputs and outputs.
- A description of the composition, rate of input (including periodic inputs), and source of all non-process wastes (e.g., sanitary wastes) entering the waste treatment processes.
- An indication in the process description and schematic of exactly where the petitioned waste is generated, if applicable.
- ! A description of all treatment process equipment, equipment functions, and the ranges of operating parameters.
- A description of all operating cycles (e.g., batch cycles versus continuous operation: start-up, shut-down, and other process transients: and maintenance and cleaning operations) on a daily, weekly, or other period basis as appropriate. Identify periods when treatment wastes are not generated (e.g., plant shutdowns or routine equipment (maintenance).
- An assessment of the extent that all contributing treatment processes, operations, process materials, or generated wastes have varied in the past and may vary in the future, whether due to manufacturing processes or seasonal variations in production.

#### 5.1.4 Process Materials

The following information should be provided by all petitioners:

- A listing of all materials used in the manufacturing and treatment processes and waste management operations (as appropriate) contributing to the petitioned waste. Examples of materials to be included are: raw materials, feed chemicals (including catalysts), treatment chemicals, oils and hydraulic fluids, and surface preparation materials (e.g., solvents, acids, cleaners).
- For each material listed, describe its function in the processes and specify the approximate quantities used annually.
- Provide Material Safety Data Sheets (MSDSs) for all non-elemental

and trade name materials, or other descriptive information that identifies the composition of the material (e.g., a listing of hazardous constituents in a material). MSDSs alone completely identify the delisting hazardous constituents of concern present in a material. MSDSs needed by the Occupational Safety and Health Administration (OSHA) only list those constituents considered hazardous by OSHA. Therefore, you should also provide composition for material <u>and</u> list any other each delisting constituents of concern which are present in the material yet not listed on the MSDS.

! Specify the source, quality (i.e., recycled or virgin), and quantity of oil, grease, and hydraulic fluids entering the processes.

#### 5.1.5 <u>Waste Management Operations</u>

If your petitioned waste is or has been managed in on-site land-based units (or in dedicated off-site land-based units), you should provide the following information:

A description of each waste management unit including construction; detailed schematic showing (as appropriate) dimensions; history; purpose and descriptions of any unit design and operating changes; estimated surface area; estimated unit capacity volume; and all waste and material inputs which have occurred throughout the life of each unit, if known.

If your petition is for an upfront waste exclusion or a waste that is generated by an MWTF, then you also should provide the information discussed below in Section 5.2, "Special Information." Otherwise, if your petition is for a waste that currently exists, then you may proceed to Section 6, "Delisting Analytical Plan SOP."

#### 5.2 Special Information

This section provides process information guidance for those petitioners requesting an upfront exclusion for a waste that is not currently generated, yet will be in the future, or for the exclusion of a waste that is generated by a multiple waste treatment facility (MWTF).

Petitioning for an upfront exclusion has the advantage of allowing the applicant to know what treatment levels for constituents should be sufficient to

render specific wastes non-hazardous, before investing in new or modified waste treatment systems. Petitions for such wastes should include all of the process and raw material information requested above, as applicable, for the bench-scale or pilot-scale process, as well as for all operating full-scale processes that will contribute to the petitioned waste. In addition, the petitioner should provide sufficient information to show that the bench-scale or pilot-scale

process is producing a waste that is similar to the waste that will be produced by the full-scale process. If the full-scale process becomes operational during the review of your petition, you should also send full-scale process information and analytical data.

If your petition is for an upfront exclusion, you should provide the following information:

- A discussion explaining why you believe that the bench-scale or pilot-scale process demonstration adequately models the proposed full-scale process.
- A detailed description of any real or potential differences between the two processes (e.g., flow rates).
- ! A description of the impact of these differences on the character of the petitioned waste.

Multiple waste treatment facilities (MWTFs) typically receive large numbers of individual waste shipments having a wide variety of compositions. <u>If your petitioned waste is generated by an MWTF.</u> you should send the following information:

- A procedure for prescreening clients and wastes and a detailed description of how this procedure will be carried outed, should your waste be excluded.
- ! A description of procedures by which you will make sure that: (1) treatment levels needed by an exclusion are maintained and (2) a hazardous waste is not disposed improperly as non-hazardous.

# SECTION 6 DELISTING ANALYTICAL PLAN SOP

This SOP tells you how to identify the delisting constituents of concern for your particular waste and how to write an analytical plan for the testing of waste samples. Analytical plan development should precede sampling plan development because sampling protocols (e.g., number and type of sample containers) will depend on the analytes evaluated.

As discussed below, your analytical plan should include the test methods generally applicable to all delisting demonstrations (see Section 1), any special test methods applicable to your particular type of waste, and any other constituents of concern identified by an engineering analysis (see Section 3). The information described in this section corresponds with Part 4 of the "Framework for Delisting Petitions" found in Appendix A.

This section is organized as follows so that each step in analytical plan development is sequentially discussed:

- 6.1 Identifying Constituents of Concern and Hazardous Waste Characteristics (Step 1)
- 6.2 Identifying Constituents of Concern for Special Waste Categories (Step 2)
- 6.3 Conducting an Engineering Analysis to Complete Your List of Constituents of Concern (Step 3)
- 6.4 Identifying Analytes (Step 4)
- 6.5 Demonstrating Without Laboratory Analyses that Certain Constituents Are Not Present in Your Waste at Hazardous Levels (Steps 5 and 6)
- 6.6 Selecting Appropriate Waste Analytical Methods (Step 7)
- 6.7 Identifying Quality Control (QC) Protocols (Step 8)
- 6.8 Summary of Appropriate Analytical Plan Information

Exhibit 2, summarizes the above steps in analytical plan development. We recommend that you review Exhibit 2, before proceeding.

The EPA publication "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 (SW-846), is an appropriate reference for the analysis and characterization of solid wastes under the Resource Conservation and Recovery Act (RCRA) Program.

# 6.1 <u>Identifying Constituents of Concern and Hazardous Waste Characteristics</u> (Step 1)

In preparing an analytical plan, you should write a complete list of the hazardous constituents of concern in your waste. For this list, you should address the full universe of delisting constituents of concern found in 40 CFR Part 261, Appendix VIII and the following constituents not found in Appendix VIII: acetone, ethylbenzene, isophorone, 4-methyl-2 pentanone, styrene, and xylenes (total). You should demonstrate that these delisting constituents of concern are not present in your waste at hazardous levels based on analytical data, mass balance demonstrations, or other appropriate information.

You should first identify the constituents of concern and hazardous waste characteristics that in general should be addressed by all delisting petitions. Specifically, information should be provided for the hazardous constituents used as the criteria for listing your petitioned waste and all of the hazardous waste characteristics defined in 40 CFR §§ 261.21 through 261.24. The regulatory basis for these requirements may be found in 40 CFR §§ 260.22(c), 260.22(d), and 260.22(e). In addition, we generally request analyses for total oil and grease, total cyanide, total sulfide, and total constituent levels of all inorganic and organic constituents of concern.

Exhibit 1 lists the analyses concerning waste constituents and characteristics that we typically request petitioners to provide. Section 6 ("Delisting Analytical Plan SOP") provides additional information regarding the appropriate SW-846 test methods.

As noted in Exhibit 2, Toxicity Characteristic Leaching Procedure (TCLP) analyses are currently requested for delisting demonstrations. The Toxicity Characteristic Rule (55  $\underline{FR}$  11798, March 29, 1990) replaced the Extraction Procedure (EP) with the TCLP as the needed procedure for testing wastes for the toxicity characteristic. The TCLP also is used for other Agency programs, and the procedure is found in SW-846 as Method 1311.

In subsequent steps, you should complete your list of constituent of concern by adding all other hazardous constituents which may be present in your petitioned waste based on: (1) the special analytical methodologies discussed below, and (2) the results of an engineering analysis of all process and materials contributing to your waste.

#### Exhibit 1

#### STEPS TO IDENTIFY CONSTITUENTS OF CONCERN AND WRITE AN ANALYTICAL PLAN

- Develop a list of constituents of concern and hazardous waste characteristics that in general should be addressed by all delisting petitions.
- 2. Identify whether your particular waste may need any special analyses and add the appropriate additional analyses to your list. For example:
  - If your waste is generated from petroleum refining operations, add the constituents of concern for petroleum refining wastes (listed in Exhibit 3).
  - If your waste is generated from stabilization processes, add the Multiple Extraction Procedure (MEP).
  - If your waste may contain dioxins, add dioxin/furan analysis using SW-846 Method 8290.
- 3. Conduct an engineering analysis to identify whether any other delisting constituents of concern may be present in your waste. Add these constituents to your list.
- 4. Based on the list developed during Steps 1, 2, and 3, identify those constituents for which testing will be conducted.
- 5. Develop, for petition inclusion, mass balance demonstrations to estimate levels in your waste of all constituents of concern on your list for which testing will not be conducted.
- 6. Develop, for petition inclusion, an explanation for why any remaining delisting constituent of concern will not appear in your waste.
- Choose appropriate analytical methods for constituents to be tested.
- 8. Identify quality control (QC) protocols for waste testing.

#### Exhibit 2

#### ANALYSES FOR CONSTITUENTS OF CONCERN AND HAZARDOUS WASTE CHARACTERISTICS

- One sample with Total constituent analyses for the entire Part 264 Appendix IX constituent list.
- Multiple pH testing. TCLP analyses using three different extraction fluids in a pH range from 3-11.
- Total oil and grease analysis.
- I Total constituent analyses for the toxicity characteristic (TC) metals currently listed under 40 CFR § 261.24, and nickel, cyanide, and sulfide.
- Toxicity Characteristic Leaching Procedure (TCLP) analyses for the TC metals, nickel, and cyanide. For cyanide analyses, deionized water should be used in place of the acid leaching medium. If the petitioned waste contains at or above one percent total oil and grease, we prefer that you conduct the Extraction Procedure for Oily Wastes (OWEP; SW-846 Method 1330) analysis and use the TCLP in place of the EP.
- ! Total constituent and TCLP (or OWEP if appropriate) analyses for any other inorganic that was used as a criterion for listing yet is not included in the above, or that is a potential constituent of concern. If the levels of total chromium in your waste exceed one percent, we recommend that you also try to analyze for the level of hexavalent chromium present.
- Analyses for the characteristics of ignitability, corrosivity, or reactivity; or provide a detailed explanation regarding why the waste does not exhibit a given characteristic. You should, however, send results from reactive sulfide and reactive cyanide testing if total sulfide and total cyanide levels exceed 500 and 250 parts per million, respectively.
- I Total constituent and TCLP analyses for the organic constituents listed under 40 CFR § 261.24.
- ! Total constituent and TCLP analyses for any other organic constituents of concern that could potentially be present in the waste. You do not need to conduct TCLP analyses if the constituent is not detected in the total constituent analysis using an appropriate SW-846 method with an adequate quantitation limit.

#### Exhibit 3

#### CONSTITUENTS OF CONCERN FOR WASTES FROM PETROLEUM PROCESSES

#### <u>Inorganics</u>

Antimony Lead
Arsenic Mercury
Barium Nickel
Beryllium Selenium
Cadmium Silver
Chromium Vanadium
Cyanide Zinc

#### **Organics**

Acenaphthene 2,4-Dinitrotoluene Benzene Di-n-octyl phthalate

Benzo(a)anthracene 1,4-Dioxane
Benzo(b)fluoranthene Ethylbenzene

Benzo(a)pyrene Ethylene dibromide Bis(2-ethylhexyl)phthalate Fluoranthene

Butyl benzyl phthalate Fluorene
Carbon disulfide Indeno(1,2,3-cd)pyrene
Chlorobenzene Methyl ethyl ketone

Chloroform Naphthalene Chrysene Nitrobenzene

Cresols Phenol
Dibenz(a,h)anthracene Pyrene
Di-n-butyl phthalate Pyridine
1,2-Dichlorobenzene Styrene

1,4-Dichlorobenzene Tetrachloroethylene

1,2-Dichloroethane Toluene

### 6.2 Identifying Constituents of Concern for Special Waste Categories (Step 2)

You should find out whether your waste may need special analytical methodologies because it falls into one of the following categories.

#### 6.2.1 Petroleum Industry Wastes

If petroleum industry processes have contributed to your petitioned waste, then the constituents of concern identified in Exhibit 3, should be included on your list. We identified these constituents of concern based on our knowledge of the processes and raw materials typical to the petroleum industry. You may have already included some of these constituents (e.g., toxicity characteristic constituents).

#### 6.2.2 <u>Stabilized Wastes</u>

If your petitioned waste is generated from the chemical stabilization of a listed waste, then you should quantify leachable metal concentrations using the Multiple Extraction Procedure (MEP), SW-846 Method 1320, as well as by TCLP analyses. We need MEP test results for stabilized wastes to assess the long term stability of the waste. You should change the MEP by using the TCLP in place of the EP in Method 1320.

#### 6.2.3 Dioxin Wastes

If your petitioned waste may contain dioxins, either because the chemicals may be contained in the waste before treatment or may be formed during treatment (e.g., incineration or other thermal treatment of chlorinated phenols), you should perform dioxin/furan analysis using SW-846 Method 8290. Method 8290 uses high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) and has lower detection limits than SW-846 Method 8280.

# 6.3 <u>Conducting an Engineering Analysis to Complete Your List of Constituents</u> of Concern (Step 3)

A complete engineering analysis of all processes and materials contributing to your waste should be conducted to identify other delisting constituents of concern potentially present in your waste. During the engineering analysis, you should consider all available information on contributing processes and materials, including (but not limited to) Material Safety Data Sheets (MSDSs), production data, and process rates. You should identify the constituents of all materials, intermediate products, by-products, and final products contributing processes. If the petitioned waste is or was contained in a landbased waste management unit, you should identify all past waste disposal practices and information regarding all of the processes and raw materials contributing to the disposed wastes.

As stated previously, the list of delisting constituents of concern includes those presented in 40 CFR Part 261, Appendix VIII, as well as acetone,

ethylbenzene, isophorone, 4-methyl-2-pentanone, styrene, and xylenes (total). Any of these constituents that may be present in your waste based on your engineering analysis should be considered a constituent of concern for your waste.

You may not be able to identify with certainty all of the constituents of concern for a petitioned waste because complete process and raw material information is not available. For example, this situation often applies to leachates collected from landfills which have accepted many different hazardous wastes over a long period of time and for which the waste types and waste sources are not fully documented. This situation may also apply if your facility is a multiple waste treatment facility (MWTF) and receives wastes of varying compositions from many off-site sources. In such cases, you should assume that all or most of the delisting constituents of concern may be present in your waste.

Once the engineering analysis is conducted, your list of constituents of concern is complete. This list can be sent to EPA (e.g., in conjunction with your sampling plan) for a completeness evaluation before investing in sampling and analysis and final petition preparation. If you choose this option, please also provide process, raw material composition and MSDS information and as much detail as possible from your engineering analysis so that we may better evaluate your preliminary submittal. For example, if a reactant is simply described as "technical benzene" and you do not include the MSDS, we will not be able to confirm or refute the presence of toluene and other aromatics (and their byproducts) in the waste stream. On the other hand, an MSDS describing the reactant as "90 percent benzene, 9 percent toluene, 1 percent xylenes and less than 10 parts per million (ppm) sulfur" clarifies that these constituents may be present in your waste stream.

# 6.4 <u>Identifying Analytes (Step 4)</u>

The next step is the identification of those constituents for which testing will be conducted. This step initiates development of your analytical plan. For analytical testing purposes, you should include: (1) the analyses discussed in Exhibit 3, and (2) the analyses for other constituents of concern which can be quantitated using appropriate SW-846 test methods.

If any constituent that may be present does not have an appropriate analytical method, as is the case for some 40 CFR Part 261, Appendix VIII constituents, a mass balance demonstration may be conducted to estimate constituent levels in the waste. Levels of all constituents potentially present in the waste should be quantitated, either by analytical testing or by mass balance demonstrations. The next section provides guidance in conducting mass balance demonstrations.

# 6.5 <u>Demonstrating Without Laboratory Analyses that Certain Constituents Are</u> Not Present in Your Waste at Hazardous Levels (Steps 5 and 6)

for of analytical testing those Instead (and constituents appropriate SW-846 test methods), your demonstration may be based on mass balance demonstrations (Step 5), as long as you have performed the testing outlined in This demonstration also may be made through another explanation (e.g., by showing that the constituent is not used in processes at your facility and cannot appear in your waste). If a mass balance demonstration is used instead of analytical testing to quantitate a constituent of concern, the demonstration should describe the raw materials used, the quantities used in the process(es), and the expected constituent levels in the waste.

For example, consider a wastewater generated by a process involving the production of aniline by the reduction of nitrobenzene. Based on facility data, for each 100 pounds of nitrobenzene used in the process, 72 pounds of aniline are formed. Based on the chemical reactions known to occur, therefore, 95 pounds of nitrobenzene are reacted per 100 pounds of nitrobenzene used. Furthermore, 200 gallons of wastewater result from the production of each 72 pounds of aniline.

Therefore, assuming that all of the unreacted nitrobenzene ends up in the wastewater, the maximum concentration of nitrobenzene in the wastewater will be about 3,000 ppm. The following equation demonstrates how this is estimated:

5 lbs (1 gal/8.345 lbs) (1/200 gal) =

(5 parts nitrobenzene/1,669 parts wastewater)  $(1/10^{-6}) = 3000$  ppm

Chemical reactions occurring in contributing processes should also be included in mass balance equations to predict the types and relative amounts of by-products resulting from a reaction. Unfortunately, demonstrating chemical reaction does not actually produce traces of hazardous substances can sometimes be difficult. For example, reactions involving chlorinated phenols might produce some chlorinated dibenzodioxins, which are considered hazardous at trace levels, although it is not a very probable reaction in most circumstances. For cases where a constituent is hazardous at trace levels, a mass balance equation may not be sufficient to demonstrate that the constituent is not present For these situations, analytical data should be submitted at levels of concern. instead of a mass balance demonstration.

In some instances, constructing a mass balance equation for particular substances may be difficult based on the manner in which the substances are used in the process. For example, chemicals present in mixtures used in maintenance, cleanup, and other non-process applications may be difficult to quantitate. In such cases, you should list the known constituents in each mixture and estimate the amounts entering contributing processes based on usage rates. Additionally, constructing a mass balance equation for the fate of constituents from wastes contained in a land-based waste management unit may also be very difficult. When mass balance equations are difficult or not practical, you should instead rely on laboratory analyses using appropriate methods to quantitate constituent levels

in your waste.

As noted in Exhibit 3, you may also send an explanation instead of analytical test results to show that the petitioned waste cannot exhibit a particular hazardous waste characteristic. For example, consider a petition for a sludge generated from treating electroplating process wastewaters with a liquid content of about 75 percent. An explanation discussing the high water content may be sufficient to demonstrate that the waste is not ignitable. However, if information on the manufacturing processes shows that flammable organic solvents may appear in the waste due to solvent cleaning operations, then actual testing for ignitability may be appropriate. Similarly, if the waste pH is neutral, more extensive testing for the corrosivity characteristic (i.e., to find out if the waste corrodes steel) may not be necessary.

Once you have addressed constituents of concern identified for your particular waste, you should also address why any remaining delisting constituents of concern will not appear in your waste (Step 6). If the constituent was not included in your list of constituents of concern because amounts used are expected only to appear in media that are not the subject of your petition (e.g., process effluent), you may support your hypothesis with a mass balance demonstration.

### 6.6 Selecting Appropriate Waste Analytical Methods (Step 7)

In this section, we review appropriate analyses and provide guidance in choosing appropriate test methods. Most of the test methods for the delisting constituents of concern may be found in SW-846, and its updates. The associated quality control procedures for analyzing a waste in support of a delisting petition are presented in Chapter One of SW-846 and in the specific SW-846 method.

# 6.6.1 Choosing the Appropriate Test Method

There are three main variables to consider when making this choice, namely, (1) the constituent or parameter to be quantitated, (2) the waste matrix, and (3) the minimum level at which the constituent should be quantitated. You should consider the test methods discussed above in Sections 1) and 2), and review Chapter Two in SW-846, entitled "Choosing the Correct Procedure."

The above references will help you identify the appropriate analysis for a given constituent and waste matrix. Some tests will have only one appropriate method for the given constituent or matrix, whereas other tests will have more than one appropriate method. The selected method should be appropriate for thewaste matrix. For example, SW-846 Method 7470, "Mercury in Liquid Waste" and Method 7471, "Mercury in Solid or Semisolid Waste" should each be used only when analyzing a waste with the specified matrix (liquid or solid).

Detection limits vary with analytical methods. Although method detection limits (MDLs) are important, in general, we request that petitioners use

estimated quantitation limits (EQLs) instead of MDLs.

If you think that an analytical method other than that found in SW-846 is more appropriate for quantitating a particular constituent, approval to use such a method should be sought before conducting the sampling and analysis.

## 6.6.2 <u>Summary of the Analyses Appropriate for Delisting Petitions</u>

In summary, testing methodologies appropriate for all petitions generally include:

- Total oil and grease analysis.
- One sample with Total constituent analyses for the entire Part 264
  Appendix IX constituent list.
- Total constituent analyses for the TC metals, nickel, cyanide, sulfide, any other inorganic constituent of concern for your waste.
- ! TCLP analyses (or OWEP analyses if appropriate) for the TC metals, nickel, cyanide (using deionized water as the leaching medium), and any other inorganic constituent of concern for your waste.
- ! Total constituent and TCLP analyses for the TC organic constituents and any other organic constituent of concern for your waste.
- Multiple pH testing. TCLP analyses using three different extraction fluids in a pH range from 3-11.

You also should address all of the other hazardous waste characteristics through laboratory analyses or, for some properties in particular matrices through some other demonstration.

Total oil and grease analysis finds the percent of total sample mass that is oil and grease. SW-846 Method 1664 or 9071B may be appropriate; each method involves drying the waste and then extracting oil and grease with an organic solvent (e.g.,n-hexane). See 64 FR 26315, May 14, 1999.

If the total oil and grease content of your waste is one percent or more, you should use the Extraction Procedure for Oily Wastes (OWEP), SW-846 Method 1330, to quantitate the leachable levels of all metals of concern. You should also use the TCLP in place of the EP in Method 1330. If the total oil and grease content is less than one percent, you should use TCLP analyses. You should send results from TCLP (or OWEP) and total constituent analyses for the TC metals listed in Table 1 of 40 CFR § 261.24, as well as for nickel and any other inorganic of concern for your waste. Chapter Three of SW-846 provides guidance on total constituent analyses for various metals.

You should analyze for total cyanide content in your waste, as described in Chapter Five of SW-846 using Method 9010 or 9012. If the cyanide analysis shows the presence of an interference in the waste and produces poor precision on replicate analyses, you should note and explain the interference in your petition. You also should analyze for total sulfide content in your waste, as described in Chapter Five of SW-846 using Method 9030.

Total levels and TCLP leachate concentrations of the TC organic constituents should also be quantitated, as well as analyses for any other organic constituent of concern for your waste, if appropriate methods are available. Applicable methods can be found in Chapter Four of SW-846. Typically, if an organic constituent is not found in the waste using appropriate analytical methods for total concentrations, you may not need to conduct the TCLP analysis. We will evaluate the applicability of the TCLP for organics in problem matrices (such as oily wastes) on a case-by-case basis.

Finally, you are needed to demonstrate that your waste does not exhibit any of the four hazardous waste characteristics of ignitability, corrosivity, reactivity, and toxicity. This demonstration may take the form of a detailed explanation regarding why the waste does not exhibit a characteristic property. 40 CFR § 261.20 through 261.24 explain what basis finds that a waste exhibits one of the characteristics.

If you cannot demonstrate in a written explanation that your waste does not exhibit a characteristic, then you should instead test the waste appropriately. Applicable methods for the testing of ignitability, corrosivity, and reactivity can be found in Chapters 7 and 8 of SW-846. As noted above, you should, at a minimum, test for the toxicity characteristic constituents using the TCLP (or OWEP if appropriate), and address whether the toxicity characteristic would be exhibited for any of the TC constituents. SW-846 Methods 9010/9012 and 9030 measure reactive cyanide and sulfide, respectively. In addition, the Defense Hazard Classification Agency's Explosives Logistics Procedures (DLAR prescribe a blasting-cap test for characterizing explosiveness. If you dealing with a waste listed because it is explosive, you may want to contact the EPA for further guidance on test methods.

## 6.7 <u>Identifying Quality Control (QC) Protocols (Step 8)</u>

Laboratory quality control information demonstrates the validity of the analytical data submitted with your petition. Certain specific quality control information should be generated simultaneously with each analytical method. If you do not have the necessary personnel or laboratory resources in-house to conduct the testing program using the appropriate QC protocols, you should contract with a commercial laboratory or a consulting analytical chemist with the necessary resources and experience.

Chapter One of SW-846 contains a general discussion on the QC protocols for waste analysis. The general procedures, however, do not replace the specific QC methodologies described under each SW-846 test method which should also be

followed. Chapter One of SW-846 discusses planning for a Quality Assurance and Control Program to make sure that all data are of known quality, to show when corrective action is needed, and to outline the procedure to correct out-of-control situations. In addition, the chapter discusses certain general quality control data appropriate for all analytical methods. Examples of these methodologies are given below.

## 6.7.1 Matrix Spike and Matrix Spike Duplicate Analyses

These analyses are measures of analytical accuracy and precision performed by splitting a sample spiked with known quantities of stock solutions of the analyte in question into duplicates, conducting the appropriate analysis, and calculating the percent recovery of each spiked analyte. These percent recoveries show the accuracy of the analysis. The relative percent difference between the samples shows the precision of analysis for the analyte in the specific matrix. The frequency at which the matrix spike and matrix spike duplicate analyses should be performed is discussed within most SW-846 analytical methods.

## 6.7.2 Method Blank Analyses

These analyses are artificial, laboratory-made samples carried through the entire analytical procedure that are used to document any contamination resulting from the analytical procedures. A method blank is often a sample of analyte-free water or solvent.

## 6.7.3 <u>Surrogate Spikes</u>

These analyses are needed for most organic analyses to measure and evaluate analytical accuracy. Surrogate compounds, which are spiked into each sample, are chosen on the basis of similar chemical structure to the substance of interest, with consideration for physical properties as well. (Frequently, isotopic isomers are selected as surrogate compounds.) The analysis procedures found in SW-846 will indicate which surrogates are to be used. Percent recovery of the surrogate spike is then calculated and used to evaluate analytical accuracy.

# 6.7.4 Field QC Analyses

These analyses monitor the introduction of any variables during the sampling process. Field QC analyses may include trip blanks and equipment blanks. Trip blanks are samples of analyte-free water or solvent brought to the field in sealed containers and transported unopened back to the laboratory along with the actual samples. Equipment blanks are used to document adequate decontamination of sampling equipment and are actually passed through the sampling equipment before sampling.

Trip blanks document contamination attributable to sample shipment and handling procedures.

This section provided a brief discussion of some of the Quality Control methodologies appropriate for all analyses. We recommend that you review Chapter One of SW-846, in addition to the specific QC procedures for each method, before performing your waste testing. If these methodologies are not met, we may not consider your analytical data valid or your petition complete. In addition, refer to the Quality Assurance (QA) SOP for guidance on developing a QA Plan for your Delisting Petition.

### 6.8 <u>Summary of Appropriate Analytical Plan Information</u>

In summary, you should send with your petition:

- A complete list of the constituents of concern identified for your petitioned waste. Identify which of these constituents were quantitated by laboratory analysis in support of your petition.
- Mass balance demonstrations for those constituents of concern in your list for which analyses were not conducted.
- A discussion explaining why any other delisting constituent of concern is not on the constituent of concern list for your petitioned waste.
- ! A discussion explaining why your petitioned waste does not exhibit any hazardous waste characteristic for which analysis was not conducted.

# SECTION 7 DELISTING SAMPLING PLAN SOP

This section tells you how to write a plan for sampling the petitioned waste. Analytical data must be from a sufficient number of representative samples, but in no case less than four, collected over a period of time sufficient to represent the uniformity of the petitioned waste. The regulatory basis for this requirement is found in 40 CFR §260.22(h). Information in this Standard Operating Procedure (SOP) will help you write a sampling plan that makes sure that samples are representative of the petitioned waste and that sample integrity is maintained until analysis.

As noted in the SOP for Analytical Plan Development, the EPA publication "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, (SW-846) is the reference for the analyses of solid wastes under the Resource Conservation and Recovery Act (RCRA) Program. SW-846 also contains guidance in developing plans for solid waste sampling. This section of the SOP summarizes and refers to certain sampling plan development considerations discussed in SW-846 and addresses sampling plan considerations and needs specific to delisting demonstrations.

Guidance in sampling plan development may also be obtained from other EPA-published guidance manuals or other standard reference sources, such as documents published by the American Society for Testing and Materials (ASTM). The development of a scientifically credible sampling plan is a critical element unique to each delisting petition demonstration. Please contact EPA (See Section 2) if you need more guidance than given in this manual or additional references. The information described in this section corresponds with Part 5 of the "Framework for Delisting Petitions" found in Appendix A.

This SOP is organized as follows:

- 1) Defining Sampling Objectives
- 2) Selection of an Appropriate Sampling Strategy
- 3) Sampling Plan Considerations
- 4) Examples of Sampling Strategies for Delisting Demonstrations
- 5) Recommended Elements of a Written Sampling Plan

The "Waste Sampling and Analysis Information," SOP, will provide specific guidance on what information concerning your sampling procedures should be submitted with your delisting petition.

### 7.1 <u>Defining Sampling Objectives</u>

You should define the objectives of your sampling effort as the first step in sampling plan development. Once defined, these objectives should be considered throughout the development of the sampling strategy.

The primary objective of any waste sampling effort is to get valid information that can be used to describe the chemical and physical nature of a waste. Specific objectives should be defined based on such considerations as:

- 1. Compliance with the regulatory requirement for representativeness found in 40 CFR § 260.22(h) (i.e., characterizing temporal and spatial constituent variability in the waste).
- 2. Meeting the data quality assurance objectives (e.g., accuracy and precision) defined in SW-846.
- Familiarity with the analytical parameters (as identified during analytical plan development).
- 4. Whether the waste is continuously generated, not yet generated (e.g., pilot-scale process for future wastes), or located in a container or in a land-based waste management unit.

You can refer to SW-846 for additional guidance regarding specific sampling objectives. All personnel involved in sampling plan development and the actual sampling effort should be familiar with and understand the basis for the defined objectives. This understanding by your personnel will maintain sampling program consistency and quality.

## 7.2 <u>Selection of an Appropriate Sampling Strategy</u>

It is important to collect <u>representative</u> samples for your delisting petition because the resulting analytical data will be used to characterize the entire waste stream. These samples should be non-biased and "sufficient to represent" your waste. Otherwise, re-sampling may be needed.

As stated in 40 CFR § 260.22(h), you must collect and analyze at least four representative samples of the petitioned waste. The actual number of samples sufficient to represent the variability or uniformity of your waste will likely be more than four; the number of samples must be determined on a waste- and unit-specific basis.

With respect to most wastes. We also request that each sample be a composite of independent grab samples collected over a certain period of time (e.g., for continuously generated wastes) or area (e.g., for wastes held in a container or unit). The number of grab samples adequate to make up each composite sample will be waste- and unit-specific.

Additionally, the analytical testing to be performed may impact sample collection. Specifically volatile organic analysis is typically performed on individual grab samples rather than on composite samples. This practice eliminates the possibility or volatile organic loss due to sample compositing. In some cases, compositing of samples under laboratory conditions may be appropriate. In any case, the volatile organic sample aliquot should be

appropriately packaged and sent to the laboratory as soon as feasible to avoid exceedance of the analytical holding time. Generally, other types of analysis can be performed on aliquots from the composite sample.

Waste variability can be categorized as spatial (area) or temporal (in and both types can act separately or together to influence The following sections provide general guidance composition. in selecting that may exhibit sampling strategies for wastes temporal and/or variability. Section 4 provides more specific delisting guidance regarding sampling strategy considerations and requirements for various scenarios (e.g., process discharge points, drums, and surface impoundments).

## 7.2.1 <u>Temporal Variability</u>

Temporal (relating to time) variability occurs when a periodic fluctuation in process or raw material also causes a fluctuation in physical or chemical output (e.g., a change in waste characteristics). Temporal variability is often exhibited in wastes generated from process point discharges, such as characteristics that periodically vary due to changes in product Temporal variation may depend on whether the generating processes are continuous or batch operations. Continuous processes are more likely to generate a fairly homogeneous waste than batch operations. Batch operations involve processes that may be frequently stopped and started or involve changes in raw materials or Batch operations, therefore, usually generate product lines. waste with temporal variability. To account for this greater variability, a waste generated by batch operations needs more frequent sampling over time than a waste generated by continuous operations.

Temporal composite sampling is usually needed to get representative samples from either continuous or batch process wastes that may exhibit variability over time. This method involves collecting a number of independent grab samples of equal volume at predetermined time intervals (e.g., every two hours) over a certain time period of waste generation (e.g., during one 8-hour operating shift). The grab samples are composited in equal proportions and a sample aliquot is collected from the composite at the end of the time period. The time intervals for grab sampling and compositing are predetermined based on known process operating shifts and known discharge variations over time.

If your waste exhibits temporal variability and you use this strategy to get representative samples, you should collect and analyze enough composite samples to sufficiently characterize expected waste variability over the life of the process. For a delisting demonstration petitioners typically collect and analyze at least four composite samples over a period of at least one month. You should, however, be able to defend your entire sampling strategy and actual sampling activities.

The frequency of grab sample collection and the number of composite samples will ultimately depend on the process and its operating schedule and known periodic variations in waste composition. The greater the potential for waste

composition variability, the greater the number of composite samples needed to characterize the waste.

The schedule for sampling should be developed by personnel who are familiar with the process and understand the importance of collecting a representative sample. The needed volume of waste per sample must also be found out beforehand. The needed volume, the preservation techniques and number of different types of containers (for a single sample) are dictated by the analyses to be performed and the waste matrix. Sample volume, therefore, should be identified by the individuals selected to perform waste analyses.

## 7.2.2 Spatial Variability

Spatial (relating to space) variability may be vertical or horizontal and is often exhibited by a waste contained in a tank, drum, pile, landfill, or surface impoundment. Contained wastes usually vary more in a vertical than a horizontal direction due to the settling of solids, variations in densities of liquid phases, and periodic (temporal) variations in the composition of the waste entering the container or management unit. However, horizontal variation can also occur when the routine for waste deposition is altered (e.g., the relocation of influent points at a surface impoundment or the one-time discharge of a volume of waste). Factors such as waste unit design, waste influent locations, and mixing device location and usage rates can also affect the waste deposition patterns and spatial variability within the unit.

You should characterize the waste's spatial variability in both the horizontal and vertical directions. This consideration usually needs collecting a complete vertical (i.e., "full-depth" core) sample of the waste. Actual sample locations can be found through randomly selected points on an imaginary horizontal grid overlaying the waste unit. The actual number of samples collected will depend on the area of the containment unit and the expected degree of both vertical and/or horizontal spatial variability.

### 7.3 <u>Sampling Plan Considerations</u>

During the selection of an appropriate sampling strategy and development of the waste sampling plan, you should consider a number of factors. These factors include waste properties, site factors (e.g., processes), sampling equipment, sample containers and preservatives, personnel health and safety, quality control (QC), and sample handling and documentation. Each of these factors interact and should be fully understood and addressed before finalizing a sampling strategy and initiating sample collection. To account for all factors, personnel designing the sampling plan should include: (1) an analytical chemist, (2) a facility engineer familiar with contributing processes, waste properties, and site factors, (3) a quality control representative, and (4) experienced field samplers.

# 7.3.1 <u>Waste Properties</u>

The physical and chemical properties of your waste are the most important factors to be addressed in a sampling plan because waste properties affect many aspects of your sampling effort. For example, waste properties (e.g., liquid or solid) will determine the type of sampling equipment that has to be used and whether spatial variation has to be addressed in the sampling strategy.

Sampling equipment should be selected based upon whether the waste is a viscous or a free-flowing liquid or a hard, soft, powdery, or monolithic solid. The waste's physical state will also affect the type of sample container needed. Wide-mouth containers are used for most solids, sludges, or liquids with large amounts of suspended matter and narrow-mouth containers are usually used for liquid and flowing samples. If the physical state and composition of the waste allows random heterogeneity or stratification of liquid phases or solids, then your sampling strategy for getting representative samples should address these characteristics. The number of samples collected should be dictated by the volume of sample waste (e.g., based on the area and depth of a waste management unit).

## 7.3.2 Site Factors

Site factors include such items as waste accessibility, waste generation and handling practices, transitory (changing) process events (e.g., process start-up and shut-down), and hazards. These factors will influence the sampling strategy and personnel health and safety. Site factors should be thoroughly examined during sampling plan development to ensure a successful sampling effort. You should, of course, be familiar with the waste generation, waste handling and/or management practices, and transitory process events to ensure collection of representative samples.

# 7.3.3 <u>Sampling Equipment</u>

When choosing the sampling devices, you should consider waste properties and site factors, as noted above. Sampling devices should also be selected based on the analytes to be quantitated. For example, if the waste will be analyzed for volatile organics, you should choose a sampling device that will minimize sample agitation. You should also consider ease of use under the site conditions and the degree of hazard associated with using a given device.

Commonly used sampling devices include dippers, weighted bottles, coliwasas (composite liquid waste samplers), thiefs, triers, and augers. SW-846 contains descriptions of these and other sampling equipment.

Disposable sampling devices should be used when practical. Examples of disposal sampling equipment include trowels, thiefs, coliwasas, and buckets. If use of disposable devices is not feasible, then the sampling device should be decontaminated after collecting each sample. Containers used for sample compositing, such as buckets, should also be decontaminated before sampling and disposed or decontaminated after each compositing event.

Besides sampling devices, you also will use other types of equipment during

the sampling effort. We recommend that you write a detailed list of all types of equipment that may be needed during the sampling effort.

# 7.3.4 <u>Sample Containers and Preservatives</u>

All samples should be placed in containers of a size and construction appropriate for the planned analyses. SW-846 identifies appropriate containers and preservatives for SW-846 analytical methods.

Containers for collecting and storing waste samples before analysis are usually made of plastic or glass. Sample containers should be compatible with the waste and not interfere with sample integrity. A list of appropriate containers is presented in SW-846. Your laboratory should be responsible for guaranteeing that the appropriate containers are available for both waste and quality control (QC) samples based on the planned analysis.

### 7.3.5 Quality Control and Sample Handling and Documentation

Quality assurance procedures are employed to measure how well quality control (QC) objectives are met. The analytical QC methodologies for a delisting demonstration are discussed in the Analytical Plan SOP. As noted in that SOP, trip and equipment sample blanks should be collected to monitor the introduction of any variables during sampling. SW-846 contains guidance regarding the collection frequency of these sample blanks.

In addition to collecting field QC samples, quality control procedures include sample handling and documentation protocols. Waste sample containers should be properly handled and stored to make sure that the samples are chemically and physically representative of the sampled waste. In addition, proper sample labeling and documentation of sample collection, preservation, and chain-of-custody procedures should be followed to maintain sample integrity. Chain-of-custody procedures include documenting sample collection activities, sample shipment, and laboratory processing to make sure that sample results are fully traceable back to sample collection. All persons having custody of the samples should follow the procedures discussed in SW-846.

# 7.4 Examples of Sampling Strategies for Delisting Demonstrations

In this section, examples of sampling strategies are presented for the following sampling locations: pipes and other process discharges, drums, landfills, and surface impoundments. We also describe approaches for dealing with "hot spots" within a unit and collecting representative samples at MWTFs. Diagrams and descriptions of sampling equipment mentioned in this section can be found in SW-846.

The variety of waste properties and the uniqueness of each sampling situation necessitates a thorough examination of options for acquiring representative samples. We believe the procedures discussed here represent the best approaches to sampling under each respective waste scenario. However, the most appropriate sampling strategy for your waste may be somewhat different and

should be devised by personnel familiar with the specific waste properties, site factors, and sampling objectives.

#### 7.4.1 Pipes and Other Process Discharges

Representative samples from pipes and other process discharges are best obtained through time-composite sampling, which is described in Section 2). Composite sampling may be performed manually or through the use of automatic compositers. The following discussion assumes that manual compositing is performed.

There are two basic types of process discharges: (1) liquid or slurry discharges, and (2) solid or semi-solid discharges. Liquids or slurries are generally sampled from pipes, valves, or sluiceways. Solid process discharges are often sampled from conveyor belts or filter presses.

For liquids and free-flowing slurries, a dipper-type sampler can be used. The size of the dipper beaker should be directly related to the stream flow rate. A dipper should be used to sample only one waste stream; any sampling equipment which comes in contact with the waste should be decontaminated or replaced before collecting the next sample.

Before collection of an actual grab sample, the dipper should be first rinsed in the waste stream to be sampled. The dipper should then be passed in one sweeping motion through the discharge stream so that the beaker is filled in one pass. The whole stream flow should be sampled, not just the edge of the stream. If the cross-sectional area of the stream is large compared to the size of the beaker, more than one pass may be necessary to sweep the entire cross section of the stream and fill the beaker. Each grab sample should be of about the same volume and emptied into a larger container for compositing. After collecting all of the grab samples, the entire contents in the compositing container should be mixed well before collecting a sample aliquot for analysis. (Typically, aliquots for volatile analysis are collected from a grab sample and not from the composite sample.)

Solid or semi-solid wastes on a conveyer belt can be sampled with a trowel, scoop, or shovel. A shovel should be chosen or fabricated to match the width and general contour of the belt as closely as possible. The grab samples can be taken at any point along the belt, as long as the entire width of the belt is sampled. Any fines or liquid present on the belt at the sampling point should be included in the sample.

Regardless of the waste type, the frequency of sampling and the number of independent grab samples combined to form a composite sample depend on the variability of the waste over time and on the time span which the sample is meant to represent. If you are sampling point-discharges from a fully continuous operation, independent grab samples can be collected periodically (e.g., every four hours during a 24-hour period of operation) and combined to form a daily composite. As an alternative, one independent grab sample can be collected from

the continuous process per work day for a 5-day work week and the daily grab samples combined into a weekly composite. You are needed to collect at least four composite samples over a period adequate to gather representative waste samples (typically not less than one month).

The sampling of a waste generated by a process that is not fully continuous (e.g., transient operating cycles or batch operations) usually needs a greater number of composite samples, composed of fewer grab samples, to sufficiently characterize waste variability. Again, the sampling period and number of composite samples will vary for each petitioned waste and will depend on which scenario is best for getting the most representative samples. Therefore, it is important that the individuals designing the sampling strategy are familiar with the known temporal variability of the waste stream.

Solid discharges, such as filter cakes, often fall from a press or plateand-frame filter into a hopper or storage area. In this case, a composite sample representative of a certain waste generation period may be obtained by sampling the waste from the storage container after a period of accumulation. example, filter cake can be allowed to accumulate in a storage container for 24 hours, and then random core grab samples can be taken using a simple The sampling of filter cake from a storage dimensional random sampling strategy. container needs the use of a thief or a trier, depending on the total liquid content of the material. A coliwasa could also be used if the solids content is The core samples can be combined to form a composite representative of a This procedure should be repeated enough 24-hour period of waste generation. times to get the required number of samples (e.g., four composite samples over one month).

If phase separation of the waste has occurred, such as exhibited by the appearance of a liquid in the storage container, the waste should be mixed well before drawing the samples. Excessive agitation of the waste, however, must be avoided if volatile analysis will be performed.

#### 7.4.2 <u>Drums</u>

If the petitioned waste is contained in drums, then each drum should be sampled. If there are too many drums for this to be practical, then a representative number of individual drums must be randomly selected for sampling. Random selection is easily done by consecutively assigning a number to each drum and then using a random number table to choose the drums to be sampled. Guidance in using a random number table may be found in Appendix C of this manual.

For each drum, a single core sample representative of the entire depth of the drum along its axis is sufficient. Equipment used for sampling waste in drums includes weighted bottles, coliwasas, thiefs, and triers. Coliwasas and weighted bottles are best suited for sampling liquids and free-flowing slurries; if the sample is comprised of dry granules or a powder, a drum thief should be used. A trier should be used to sample moist or sticky solids.

## 7.4.3 <u>Landfills and Surface Impoundments</u>

Before sampling landfills or surface impoundments, the complete history of waste discharge to the unit must be reviewed and understood. Such a review can provide a better understanding of whether any waste stratification and spatial variability has occurred. Regardless of whether variability has occurred, it is important to get vertical samples representative of the entire unit depth, including the bottom. Hollow-stem augers combined with split-spoon samplers are frequently used for sampling landfills. Triers or modified triers may also be used.

Generally, on-site and dedicated units contain wastes generated and managed by fairly continuous processes and methods. As long as no process changes or one-time discharges have occurred during the life of the waste units, random number sampling procedures or fixed transect and offset sampling procedures are suitable. These procedures are summarized below:

## 7.4.4 Random Sampling Procedure

- 1. On a diagram, divide the unit to be sampled into sections of equal area. If the unit area is under 40,000 square feet, then divide the unit into four equal quadrants. If the unit area is over 40,000 square feet, then divide the unit into equal sections of not more than 10,000 square feet each.
- 2. Divide each quadrant or section into an imaginary  $10 \times 10$  grid to get 100 rectangles of equal size. Number the grid lines in each dimension from 1 to 9.
- 3. For each section, find out the number of sampling points necessary (e.g., five grab samples per section composite sample) to characterize the waste. This number will depend on the degree of known spatial variability within the unit.
- 4. Select a two-digit number using a random number table. Repeat this procedure until you have one two-digit number for each sampling point in the section. (A random number table is provided in Appendix C of this manual.)
- 5. Locate those grid intersections with coordinates corresponding to each of the two-digit random numbers. These intersections are the locations of the randomly selected sampling points.
- 6. Collect a full-core sample at each sampling point (i.e., samples should be collected across the entire vertical depth, from top to bottom of the unit).
- 7. Combine and mix the samples from each section to form a homogeneous composite sample representative of each section. Do not, however, combine samples from different Sections.

For example, a surface impoundment of 10,000 square feet should be divided

into four quadrants of 2,500 square feet each. In each quadrant, five full-core samples should be collected and composited, resulting in four composite samples, one from each quadrant. Similarly, a landfill of 60,000 square feet would be divided into six sections of 10,000 square feet each, yielding six composite samples. At a minimum, we need the collection and analyses of four representative samples per unit.

## 7.4.5 Fixed Transect and Offset Sampling Procedure

Waste units without a well documented history of waste management should be sampled more extensively (e.g., greater number of samples per unit and fewer grab samples for each composite sample) than units with complete documentation. More extensive sampling is appropriate to fully characterize the potential variability of the petitioned waste.

These types of units also are best sampled using a fixed transect method with an offset sampling procedure rather than a random sampling procedure. In this method, a grid is formed similar to that for the random sampling procedure. The samples, however, are collected at different points (non-random) along a grid line with a specified offset. In cases where the waste deposition pattern has varied in a regular manner, the sampling plan should be modified to account for any waste deposition changes that are known to exist.

# 7.4.6 Hot Spots

Sometimes a localized area of contamination (a "hot spot") is known or expected to exist within a contained waste that otherwise exhibits comparatively low concentrations of constituents. Hot spots are often caused by the discharge of a concentrated material (dumping) at a specific location. The waste discharged in the specific area should be characterized separately from the rest of the contained waste to determine the boundaries of the hot spot.

Hot spots are sampled using procedures and equipment similar to that described for sampling landfills and surface impoundments. For contained wastes known or expected to have hot spots, it is best to use the fixed transect and offset sampling procedure. We usually request more than the minimum of four samples to characterize a petitioned waste expected to contain hot spots, regardless of the unit area. In such cases, additional samples (e.g., grab samples without compositing) should be collected to verify the locations and exact boundaries of all hot spots. If necessary, you may contact us for further guidance regarding the sampling of wastes expected to have hot spots.

## 7.4.7 <u>Multiple Waste Treatment Facilities</u>

Multiple waste treatment facilities (MWTFs) typically receive large numbers of individual waste shipments from a variety of off-site processes. This practice results in greater variability of constituent concentrations and thus makes it more challenging to collect representative samples. Therefore, the MWTF petitioner has fewer options for collecting samples that adequately demonstrate

that the petitioned waste is not hazardous. To get representative samples, the following guidelines are suggested:

- ! Collect and analyze as many samples as are deemed necessary to characterize the known variability in constituent concentrations in the treatment residue generated over one year (e.g., eight weekly composite samples collected over a period of two months).
- Find out what percentage of waste treated annually was represented by the sampling period. You should try to represent wastes from a majority of your clients.
- Explain in your petition why the remaining percentage of the waste is not expected to contain any other hazardous constituents of concern, different levels of constituents of concern, or other different characteristics than that represented by the sampling period.

## 7.5 Recommended Elements of a Written Sampling Plan

A written sampling plan makes sure that proper planning has taken place and that planned procedures are followed during the actual sampling effort. A good sampling plan should contain the following information:

- Objectives of the sampling effort.
- Description of the waste to be sampled.
- Description of the sampling strategy.
- Names and professional qualifications of all sampling team members.
- Description of all planned sampling and QC procedures, including location of sampling points, sample numbering, on-site sample preservation methods, sample containers, sample shipment, and documentation.
- Decontamination and health and safety procedures.
- Analytical lab to be used and analyses to be performed.
- Health and safety plan.
- Sampling equipment list.

A formal, written sampling plan is not necessary as part of a delisting petition. You may send your written sampling plan to EPA for review before sample collection and submittal of a formal delisting petition. If you choose this option, you should also send the process and waste management information

requested in Sections 4 and 5 of this Guidance Manual, and a complete list of analytes. Based on this information, we will review your sampling plan and provide guidance before you dedicate additional resources to sample collection and analysis.

# SECTION 8 WASTE SAMPLING AND ANALYSIS INFORMATION

This section tells you how to provide waste sampling and analysis information. You should send this sampling and analysis information to support your contention that the petitioned waste should be removed from the lists of hazardous wastes found in 40 CFR Part 261. The regulatory basis for requesting most of this information is found in 40 CFR § 260.22(i).

The information requested in this section corresponds with Part 5 of the "Framework for Delisting Petitions" found in Appendix A. This section is divided into two major subsections: 8.1, "Waste Sampling Information" and 8.2, "Waste Analysis Information."

As noted in Section 6 ("Delisting Analytical Plan SOP") and Section 7 ("Delisting Sampling Plan SOP"), you have the option of sending, for our review, a draft plan outlining future sample collection and analysis efforts before committing resources to actual sample analysis and petition preparation. If you choose this option, then you should note and document that you did so when sending your formal petition.

## 8.1 Waste Sampling Information

We will review information on waste sampling efforts to find out whether:

- Collected samples sufficiently represent the variability or uniformity of the petitioned waste.
- Waste sampling was conducted by qualified personnel.
- Sample integrity was maintained through analyses.

To demonstrate that collected samples are sufficiently representative of the petitioned waste, you should include the following information in your petition:

- Identification of which process point discharges, containment areas (e.g., lagoons), or other areas (e.g., soils) were sampled and why these areas were selected for sample collection.
- A description of the techniques and guidelines used to select waste sampling points (e.g., random sampling procedure or fixed transect and offset sampling procedure).
- A general description of the sampling and subsampling (i.e., transferring aliquots of a sample to containers specific to certain analyses) procedures used during the sample collection

process, including the particular days and times selected for sample collection, the number of grab samples collected for each composite sample, and why these procedures were used.

- ! A description of the sampling devices used for sample collection and the basis for selecting the devices.
- ! Identification of any deviations from your original sampling plan and strategy and the impact of these deviations on waste characterization.
- A detailed discussion explaining why you believe the samples collected are non-biased and sufficiently represent the petitioned waste. This explanation should fully address the potential for waste uniformity or spatial and temporal variability and how the strategy ensured collection of representative samples.

You should demonstrate that waste sampling was conducted by qualified personnel. Therefore, you should provide the following information:

- ! The name and address of the organization(s) or company(s) responsible for designing the sampling strategy and collecting the samples, if sampling assistance was obtained from outside the petitioning facility.
- The names, affiliations, titles, and qualifications (a resume will suffice) of all personnel (in-house and otherwise) who designed the sampling plan and the quality control plan and those individuals responsible for sample collection.

We also request sample-specific information to show that sample integrity was maintained. Therefore, you should include the following information for each sample collected for analysis:

- The sample identification number, as it appears in your field logbook and other records.
- Whether or not the sample is a waste sample or a quality control sample.
- A detailed description of how the sample was collected and its point of collection from the petitioned waste including whether the sample is a composite of grabs, the number of grab samples collected for the composite sample, the sampling location for <a href="each grab sample">each grab sample</a>, the volume of each grab sample, and the volume of the composite sample. The general sampling location (e.g., which quadrant of a surface impoundment) and the specific sampling point (e.g., specific location in the quadrant) should be provided. You may refer to numbered sampling points shown in a diagram.

- A description of how the sample was composited (e.g., equipment used and manner of mixing).
- A physical description of the sample at time of collect on (e.g., color, odor, whether phase separation occurred soon after collection).
- ! The time and date when each grab sample was collected and the time when the sample was composited, as applicable.
- ! The handling and preparation techniques used for each sample (including types of containers used and techniques employed for container preparation) and types and amounts of preservatives used.

Petitioners should also include some of the more general information about sample collection so that we may evaluate sample representativeness and integrity. This information includes:

- An indication as to whether any facility activities separate from sampling occurred at the same time and might have affected sample representativeness (such as fossil-fueled motors being used nearby).
- A description of weather conditions during sample collection (if sampling is conducted outdoors).
- A description of sampling device decontamination between samples.

  Alternatively, note when new devices were used for each sample.
- A description of the quality control procedures and documentation system used to track sample location and maintain sample integrity during transportation to the laboratory. You may simply state that you followed the chain-of-custody system specified in SW-846, if you have done so. You may also provide copies of the chain-of-custody forms, but this is not a requirement.

If you have collected samples to characterize a localized area of contamination (a "hot spot," see Section 7.4) within the petitioned waste, then you should also provide the following information:

- Your basis for believing a hot spot may or does exist (e.g., records of a one-time discharge of a concentrated material at a specific location).
- ! The known or predicted location (on a diagram) and the dimensions (e.g., depth, width and length) of the hot spot.
- ! Identification of the samples specifically collected to characterize the hot spot.

! An explanation for why samples adequate represent the hot spot.

If you have collected samples to characterize a waste generated by a multiple waste treatment facility (MWTF), then you should also provide the following information:

- ! A listing and description (based on your knowledge) of the untreated wastes that were treated and are represented by the treatment residue samples collected during the sampling period.
- ! The percentage of wastes treated annually that was represented by the sampling period.
- A listing and brief description of the untreated wastes that also are treated at the facility but were not represented by the sampling period.
- A discussion explaining why the wastes not represented by the sampling period are not expected to contain any other hazardous constituents of concern, different-levels of constituents of concern, or other different characteristics than those represented by the sampling period.

## 8.2 Waste Analysis Information

Based on the analytical results reported in your petition, we will find out whether your waste exhibits a hazardous waste characteristic (if you chose analysis for the demonstration) and whether any of the tested constituents are present at levels of regulatory concern. Besides analytical results, you should also provide sufficient information to demonstrate whether:

- Waste sampling was conducted by qualified personnel.
- the data are valid (e.g., results from quality control analyses supporting data validity)
- ! Sample integrity was maintained through analyses.

Some of the information requested below is routinely supplied on reports from an analytical laboratory. However, we suggest that you inform your laboratory of the specific information we prefer for delisting petitions to ensure complete data submittals.

You should demonstrate that laboratory analyses were conducted by qualified personnel. Therefore, you should provide the following information:

! The name and address of the organization(s) or company(s) responsible for sample analyses, if analytical assistance from outside the petitioning facility was gotten.

! The names, affiliations, titles, and qualifications (a resume will suffice) of all personnel (in-house and otherwise) who conducted analyses or were responsible for data reduction, validation, and laboratory quality control.

Your petition should clearly specify analytical information for each analytical test conducted, including testing for the hazardous waste characteristics, total oil and grease, inorganic constituents (leaching procedure and total results), and organic constituents (leaching procedure and total concentration results).

For each sample, you should report the following information:

- Sample identification number as logged during collection and as assigned by the laboratory.
- Type of sample (e.g., waste sample, waste sample replicate, equipment blank, trip blank).
- Date of sample receipt by the laboratory.

For  $\underline{each}$  analysis performed on each sample, you should report the following information:

- ! The sample preparation method and reference for the method (e.g., SW-846 Method 3500).
- The date of sample workup or preparation.
- Initials (or name) of the person conducting each analysis.
- The date each extraction and analysis was performed.
- The test method used and the source of the test method (e.g., SW-846 Method 8020).
- The specific constituent, parameter, or hazard for which analysis was conducted.
- The test results, expressed in appropriate units (e.g., mg/L, mg/kg).
- Basis for each analysis (e.g., wet weight, dry weight). We prefer that this basis is the state in which the waste will be disposed, if allowed by the test.
- In the estimated quantitation limits (EQLs). While method detection limits (MDLs) are of concern, we also evaluate EQLs for delisting

purposes. EQLs are provided in SW-846 and should be discussed in all petitions.

- ! The names and model numbers of all equipment used during analysis.
- ! All other information necessary to fully interpret the test procedures or results (e.g., percentage solids in the waste, solvent(s) used during oil and grease extraction, extraction method used during semi-volatile analysis).

# 8.3 Quality Control Information

We also request that laboratory quality control (QC) procedures be performed and the results reported. You should report the methods taken to make sure the quality of sample analysis so that we can fully and accurately evaluate the validity of the waste sample results.

Chapter One of SW-846 is an excellent source for general guidance regarding quality control procedures. Each SW-846 test method has specific QC requirements that should also be followed.

For each QC analysis, you should report the analytical information listed above, as appropriate (e.g., date of analysis, person conducting the analysis, sample identification number, units, method source). You should

also report the information specific to each type of QC analysis that is listed below. You should present your own acceptance criteria, which should be at least as stringent as the criteria specified for each method in SW-846. Section 6.7 of this guidance manual, "Identifying Quality Control (QC) Protocols (Step 8)," provides more guidance regarding QC procedures. You should follow the QC procedures and criteria of each analysis method as documented in the method source (e.g., SW-846), otherwise, we may not consider your analytical data valid or your petition complete.

Each type of analysis should have a method blank, matrix spike, and a matrix duplicate or matrix spike duplicate. The appropriate frequencies for performing these QC analyses are discussed in SW-846. For each method blank, you should report the concentration of each analyte in the sample. For each matrix spike, you should report:

- The name of the matrix spike analyte added.
- Concentration of the matrix spike analyte in the unspiked sample.
- The amount of the matrix spike analyte added.
- Concentration of the matrix spike in the spiked sample.
- ! The calculated percent recovery of the matrix spike and method of calculation.

! The acceptance criterion for recovery of the matrix spike.

In addition to the information listed above, for each matrix spike duplicate pair, you should report:

- ! The measured amount of the matrix spike in both spiked samples.
- Interelative percent difference (RPD) between the two analytical results. The calculation for the RPD is:

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(X1 - X2/X)100), where X1 = first value, X2 = second value, and X = mean value = (X1 + X2)/2.
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The acceptance criterion for RPD of each matrix spike compound.

Most organic analyses need specific <u>surrogate spikes</u>, depending on the method. <u>Surrogates</u> are specific constituents that are similar in chemical structure and physical properties to a substance of interest, but are not in the waste itself. (Deuterated compounds, in which deuterium replaces hydrogen in a given compound, are popular surrogates.) We request that the surrogates listed with the method in SW-846 be used. You should report the same type of information as listed above for matrix spikes .

Finally, you should provide the following information:

- Were waste analytical data corrected based on quality control results (e.g., spike recoveries) and, if so, how was any correction made?
- Explain any inconsistencies or deviations found in the reported analytical results. This discussion should include any observed analytical interferences and what actions were taken to resolve the problems.

## Data Validation

The petitioner should demonstrate that the submitted data is valid. all data should be compared against specific criteria. Data should be qualified according to guidance and any bias (high or low) should be identified. In addition to SW-846, EPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis (EPA/540/R/94/083) and EPA Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis (EPA/540/R/94/082) should be referred to for guidance. The petition should thoroughly evaluate all data for validity using the QA/QC procedures and criteria outlined here.

Although not initially needed, the inclusion of a third party validation report within the delisting petition would be beneficial to the review process. An independent validation report or resampling could be need if the

evaluation of the data shows sampling or analysis problems that could render

the data unusable for delisting purposes. Due to review time constraints, the petition could be denied if an additional report or resampling is needed.

The petition could also be denied should some of the data be found to be unacceptable, rendering less than the needed minimum number of samples (4). The EPA suggests that the petitioner consider this possibility when preparing a sampling plan.