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06-AMCP-0084

DEC 29 2005

Mr. L. Michael Bogert  
Regional Administrator  
U.S. Environmental Protection Agency  
Region 10  
1200 Sixth Avenue  
Seattle, Washington 98101

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JAN 17 2006

EDMC

Dear Mr. Bogert:

EFFLUENT TREATMENT FACILITY (ETF) DELISTING EXCLUSION CONDITION 2  
REPORT

The purpose of this letter is to provide the enclosed ETF Delisting Exclusion Condition 2 Report required by Condition 2 of the Hazardous Waste Management System; Final Exclusion for Identification and Listing Hazardous Waste (70 FR 44496, August 3, 2005). A 90-day extension for submittal of the report was provided by the U.S. Environmental Protection Agency in a letter from R. Albright, dated October 19, 2005.

The report contains a proposal for data quality parameters and data acceptance criteria for sampling and analysis conducted pursuant to the requirements of the approved 200 Area ETF delisting exclusion, a detailed justification including the process used for selecting the proposed parameters and criteria, and a demonstration that proposal is appropriate with respect to the regulatory limits in the delisting exclusion. Following review and approval of this report, the proposed data quality parameters and data acceptance criteria shall become enforceable conditions of the delisting exclusion. Analytes that are found acceptable will be compared to the regulatory limit (action level) in the delisting exclusion.

If you have any questions, please contact me, or your staff may contact Mark French, of my staff on (509) 373-9863.

Sincerely,

Matthew S. McCormick, Assistant Manager  
for the Central Plateau

AMCP:RDH

Enclosure

cc: See page 2

Mr. L. Michael Bogert  
06-AMCP-0084

-2-

DEC 29 2005

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Effluent Treatment Facility Exclusion Rule Condition 2 Report:  
Date Quality Parameters and Data Acceptance Criteria

1 **INTRODUCTION**

2  
3 This report proposes data quality parameters and data acceptance criteria for sampling and  
4 analysis conducted pursuant to the requirements of the approved 200 Area Effluent Treatment  
5 Facility (ETF) delisting exclusion (*70 FR 44493, August 3, 2005*), along with a detailed  
6 justification, including the process used to select parameters and criteria, and a demonstration that  
7 they meet the requirements in the delisting exclusion. This report focuses on overall data quality,  
8 as well as parameters and criteria that can be used to identify analytical methods appropriate for  
9 generation of data demonstrating compliance with delisting exclusion limits. Following review  
10 and approval of this report, the proposed data quality parameters and data acceptance criteria  
11 shall become enforceable conditions of the delisting exclusion. The concentration of analytes  
12 whose data quality meets or exceeds the approved data quality parameters and data acceptance  
13 criteria will be compared to the regulatory limits (action levels) in the delisting exclusion.  
14

15 The process used in developing this proposal involves a step-by-step approach. First, a broad set  
16 of data quality parameters were identified, consistent with established guidance. These  
17 parameters reflect aspects of data quality associated with sampling and analysis of treated effluent  
18 under the delisting exclusion. Next, data quality parameters that are directly associated with  
19 analytical methods performance were identified as a subset of the original set of parameters.  
20 Finally, quantitative values were developed for these parameters such that analytical methods  
21 meeting the proposed values will produce results that are appropriate for comparing to the  
22 delisting exclusion regulatory limits.  
23

24 In selecting parameters and criteria, it is recognized that the performance of a particular analytical  
25 method on any particular sample may vary from typical performance. Therefore, the proposed  
26 data quality parameters and criteria are intended for purposes of selecting appropriate analytical  
27 methods, not for validating an individual result. Overall data acceptability will be evaluated in  
28 accordance with the approved project quality assurance (QA) plan. It is recognized that the  
29 parameters proposed for analytical method selection are not independent. In practice, method  
30 performance may result in analytical values whose data quality fall outside the target proposed for  
31 method selection. Target method selection parameters in Table 1 are not intended as overall data  
32 acceptance parameters, rather they are intended to distinguish between appropriate and  
33 inappropriate methods to generate exclusion compliance data. To ensure that the data quality  
34 parameters in Table 1 continue to support selection of appropriate methods, they will be reviewed  
35 periodically by the laboratory and the project.  
36

37 **GENERAL DATA QUALITY PARAMETERS**

38  
39 Data quality parameters are listed by EPA QA/G-5S *Guidance for Choosing a Sampling Design*  
40 *for Environmental Data Collection* as:

- 41 • Purpose of Data Collection (e.g., determining if a parameter exceeds a threshold level),
  - 42 • Spatial and Temporal Boundaries of Study,
  - 43 • Preliminary Estimation of Sample Support (volume that each sample represents),
  - 44 • Statistical Parameter of Interest (e.g., mean, percentile, percentage), and
  - 45 • Limits on Decision Error/Precision (e.g., false acceptance error, false rejection error).
- 46

47 The parameters for the first four bullets (limits, sample points, frequency of samples, etc.) are  
48 already established in the final exclusion rule. The focus of this report is on Limits on Decision  
49 Error/Precision.  
50

1 Data quality indicators (DQIs) are proposed so as to ensure Limits on Decision Error/Precision  
2 are appropriate for purposes of using the data to demonstrate compliance with delisting exclusion  
3 limits. As stated in EPA QA/G-5, the DQIs are qualitative and quantitative descriptors used in  
4 interpreting the degree of acceptability or utility of data. The use of DQIs will provide additional  
5 flexibility in method selection. All methods which can meet these DQIs are allowed.

6  
7 The principal DQIs are precision, accuracy, representativeness, comparability, and completeness  
8 (PARCC) parameters. Secondary DQIs of importance in this effort include sensitivity and limit  
9 of quantitation. Establishing acceptance criteria for the DQIs sets quantitative goals for the  
10 quality of data generated in the analytical measurement process. Of the five principal DQIs,  
11 precision and accuracy are the quantitative measures, representativeness and comparability are  
12 qualitative, and completeness is a combination of both quantitative and qualitative measures.  
13 Accuracy comprises both random error (precision) and systematic error (bias). The DQIs and the  
14 resulting data acceptance criteria are discussed below.

### 15 16 Precision

17 Precision is a measure of agreement among replicate measurements of the same property, under  
18 prescribed similar conditions. Precision is best expressed in terms of the standard deviation or  
19 relative percent difference (RPD) for duplicate measurements. Quality assurance/quality control  
20 (QA/QC) sample types that test precision include field and laboratory duplicates and spike  
21 duplicates. The RPDs for laboratory duplicates and/or matrix spike duplicates will be routinely  
22 calculated.  
23

### 24 25 Accuracy

26 Accuracy assesses the closeness of the measured value to the true value. Accuracy of analytical  
27 results is assessed using matrix spikes. A matrix spike is the addition of a known amount of the  
28 analyte to the sample matrix being analyzed. Accuracy assessments are generally based on  
29 analysis of spiked samples rather than reference materials so that the effect of the matrix on  
30 recovery is incorporated into the assessment. Accuracy is expressed as a percent recovery of the  
31 spiked samples. The percent recovery for the laboratory control samples demonstrates that the  
32 method is working properly and gives an estimate of the method's accuracy. The percent  
33 recovery will be routinely calculated.  
34

35 Accuracy needs to be of such a quality that there is a high degree of confidence that the result is  
36 below the action level. Therefore, the closer the result is to the action level the higher the degree  
37 of accuracy needed.  
38

### 39 40 Representativeness

41 Representativeness expresses the degree to which data accurately and precisely represent selected  
42 characteristics of a population parameter at a sampling point. Because of the matrix being  
43 analyzed, dilute aqueous solution, it is not expected that representativeness will be of concern,  
44 except when there are changes to the facility influent concentrations or waste processing strategy.  
45 Sampling due to these changes is addressed in the delisting exclusion.  
46

1 **Completeness**

2  
3 Completeness is a measure of the amount of valid data obtained from a measurement system,  
4 expressed as a percentage of the number of valid measurements that should have been collected  
5 (i.e., measurements that were planned to be collected). Completeness is calculated as the number  
6 of valid (i.e., non-rejected) data points divided by the total number of data points requested. Lack  
7 of completeness is sometimes caused by loss of a sample, loss of data, or inability to collect the  
8 planned number of samples. Incompleteness also occurs when data are discarded because they  
9 are of unknown or unacceptable quality.

10  
11 Completeness is not intended to be a measure of representativeness; that is, it does not describe  
12 how closely the measured results reflect the actual concentration or distribution of the pollutant in  
13 the media sampled. Data can be complete and yet not be representative of the analyte  
14 concentrations actually present.

15  
16 **Comparability**

17  
18 Comparability is the degree of confidence with which one data set can be compared to another.  
19 Analytical procedures must provide for measurements that are consistent and representative of the  
20 media and conditions measured. All sampling procedures and analytical methods used will be  
21 consistent to provide comparability of results for samples and split samples. Comparability is the  
22 qualitative term that expresses the confidence that two data sets can contribute to a common  
23 analysis and interpolation. Comparability must be carefully evaluated to establish whether two  
24 data sets can be considered equivalent in regard to the measurement of a specific variable or  
25 groups of variables.

26  
27 In a laboratory analysis, the term comparability focuses on method type comparison, holding  
28 times, stability issues, and aspects of overall analytical quantitation. EPA QA/G-5 provides a  
29 number of issues that can affect comparability. For this sampling and analysis effort, the relevant  
30 issues are:

- 31 • similar analytic procedures and quality assurance should be used to collect data for both
- 32 data sets; time of measurements of certain characteristics (variables) should be similar for
- 33 both data sets;
- 34 • measuring devices used for both data sets should have approximately similar detection
- 35 levels;
- 36 • rules for excluding certain types of observations from both samples should be similar.

37  
38 By using standard operating procedures, the laboratory will ensure that these characteristic are  
39 met.

40  
41 **Sensitivity**

42  
43 Sensitivity is the measure of the concentration at which an analytical method can positively  
44 identify and report analytical results. Sensitivity will be assessed when issues arise with meeting  
45 the PARCC parameters. Sensitivity is determined from the value of the standard deviation at the  
46 concentration level of interest. It represents the minimum difference in concentration that can be  
47 distinguished between two samples with a high degree of confidence.

1 **ANALYTICAL METHOD DATA QUALITY PARAMETERS**

2  
3 Method performance will focus on the data quality parameters specific to the performance of  
4 analytical methods. While representativeness and completeness are essential parameters in  
5 evaluating the overall quality of data, they principally reflect sampling design, not analytical  
6 method performance. Sampling design is addressed in the exclusion rule and is outside the scope  
7 of this report. By using standard operating procedures, the ETF and the laboratory will ensure  
8 that these parameters are met. Therefore, method performance focuses on the parameters of  
9 precision, accuracy, and sensitivity.

10  
11 The following data acceptance criteria quantify the data quality parameters described above.  
12 Prior to actual analysis of samples, these data acceptance criteria may be used to define the  
13 performance of appropriate analytical methods – methods capable of achieving the specified level  
14 of performance may be considered acceptable methods. For the laboratory, analytical data  
15 generated with laboratory control samples that are within the prescribed limits are judged to be  
16 acceptable. Analytical data generated with laboratory control samples that are outside the  
17 prescribed limits are suspect.

18  
19 Table 1 lists the delisting constituents, limits, and associated data quality parameters that apply to  
20 the task of evaluating whether a particular method is acceptable. Additional information on these  
21 parameters can be found in the approved project QA/QC plan. Below is an explanation of the  
22 parameters:

23  
24 • Precision (matrix spike duplicates)

25 Matrix spike duplicates are replicates of matrix spike samples that are analyzed with  
26 every analytical batch that contains an ETF treated effluent sample. The precision of  
27 the analytical methods is estimated from the results of the matrix spike (MS) and the  
28 matrix spike duplicate (MSD) for selected analytes. The precision acceptance criteria  
29 are specified in Table 1, where:

30 [relative percent difference, RPD] =  
31 
$$\{[ \text{absolute value of: } (MS - MSD) / (\text{average of MS and MSD}) ] \times 100\}$$

32  
33 The values for precision in Table 1 are reasonable values based on previous analysis  
34 of constituents in the delisting exclusion, or similar constituents and should also  
35 provide criteria by which to select an analytical method.

36  
37 • Accuracy (matrix spikes)

38 Procedures are in place for determining the bias of the analytical method due to the  
39 matrix. These procedures include preparation and analysis of matrix spike samples.  
40 Matrix spike samples are aliquots of the sample spiked with known concentration of  
41 the target analytes and subjected to the entire analytical procedure used for the  
42 sample. A matrix spike is analyzed with every analytical batch that contains an ETF  
43 treated effluent sample to estimate method accuracy for selected analytes. The upper  
44 and lower accuracy acceptance criteria are specified in Table 1, where:

45 [percent recovery] =  
46 
$$[(\text{matrix spike sample result} - \text{sample result}) / \text{spiked amount}] \times 100$$

47  
48 The values for accuracy in Table 1 are reasonable values based on previous analysis  
49 of constituents in the delisting exclusion, or similar constituents and should also  
50 provide criteria by which to select an analytical method.

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- Sensitivity (detection level determination)

Sensitivity represents the maximum value for a detection level that will reasonably assure the results are below the delisting limits. The method selected should have a detection level below the sensitivity. The preferred detection level is the practical quantitation limit (PQL), which is lowest concentration that can be reliably measured during routine laboratory conditions. If the method PQL cannot meet the sensitivity for some constituents, the minimum concentration or attribute that can be measured by a method (method detection limit) or by an instrument (instrument detection limit) may be used. The sensitivity levels, specified in Table 1 are derived from the delisting limits and an uncertainty value which is based on the precision and accuracy.

Based on previously analysis, the detection levels of hexachlorobenzene and the aroclors cannot meet the sensitivity based on an uncertainty value. In these cases, the sensitivities specified in Table 1 are reasonable values.

Documentation is required for evaluation of data quality parameters. Documentation should allow correlation of sample results with associated QC data. Documentation should also include the source and lot numbers of standards for traceability.

Corrective actions are addressed in the project QA plan.

Effluent Treatment Facility Exclusion Rule Condition 2 Report:  
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**TABLE 1 - DATA QUALITY PARAMETERS**

Constituent	Limit mg/L	Targets for Data Acceptance Criteria		
		Sensitivity*, Detection Level mg/L	Precision, RPD %	Accuracy, percent Recovery %
Ammonia	6.0	4.4	20	70 - 130
Barium	1.6	1.2	20	75 - 125
Beryllium	4.5E-02	3.4E-02	20	75 - 125
Nickel	4.5E-01	3.4E-01	20	75 - 125
Silver	1.1E-01	8.3E-02	20	75 - 125
Vanadium	1.6E-01	1.2E-01	20	75 - 125
Zinc	6.8	5.1	20	75 - 125
Arsenic	1.5E-02	1.1E-02	20	70 - 130
Cadmium	1.1E-02	8.0E-03	20	70 - 130
Chromium	6.8E-02	4.9E-02	20	70 - 130
Lead	9.0E-02	6.6E-02	20	70 - 130
Mercury	6.8E-03	4.9E-03	20	70 - 130
Selenium	1.1E-01	8.0E-02	20	70 - 130
Fluoride	1.2	0.88	20	70 - 130
Cyanides	4.8E-01	3.5E-01	20	70 - 130
Cresol	1.2	0.76	25	50 - 120
2,4,6-Trichlorophenol	3.6E-01	2.3E-01	25	50 - 120
Benzene	6.0E-02	4.1E-02	20	60 - 120
Chrysene	5.6E-01	3.5E-01	25	50 - 120
Hexachlorobenzene	2.0E-03	2.0E-03	25	50 - 120
Hexachlorocyclopentadiene	1.8E-01	1.1E-01	25	50 - 120
Dichloroisopropyl ether (bis(2-chloroisopropyl)ether)	6.0E-02	3.8E-02	25	50 - 120
Di-n-octyl phthalate	4.8E-01	3.0E-01	25	50 - 120
1-Butanol	2.4	1.6	20	60 - 120
Isophorone	4.2	2.6	25	50 - 120
Diphenylamine	5.6E-01	3.5E-01	25	50 - 120
p-Chloroaniline	1.2E-01	7.6E-02	25	50 - 120
Acetonitrile	1.2	0.82	20	60 - 120
Carbazole	1.8E-01	1.1E-01	25	50 - 120
N-Nitrosodimethylamine	2.0E-02	1.2E-02	25	50 - 120
Pyridine	2.4E-02	1.5E-02	25	50 - 120
Lindane (gamma-BHC)	3.0E-03	1.9E-03	25	50 - 120
Arochlor (total of Arochlors 1016, 1221, 1232, 1242, 1248, 1254, 1260)	5.0E-04	4.0E-04	25	50 - 110
Carbon tetrachloride	1.8E-02	1.2E-02	20	60 - 120
Tetrahydrofuran	5.6E-01	3.8E-01	20	60 - 120
Acetone	2.4	1.6	20	60 - 120
Carbon disulfide	2.3	1.5	20	60 - 120
Tributyl phosphate	1.2E-01	7.6E-02	25	50 - 120

\*See Appendix A for calculations to determine sensitivity.

## Appendix A: Equations to Determine Sensitivity

The goal is to establish a sensitivity level where the analytical results will be within the levels established in the delisting exclusion, while allowing for a degree of uncertainty:

$$\text{Result} + \text{Uncertainty} < \text{Limit}$$

In this case, uncertainty will be expressed as a percentage of the result:

$$\text{Result} + ([\% \text{Uncertainty} / 100] \times \text{Result}) < \text{Limit}$$

or:

$$\text{Result} < \frac{\text{Limit}}{1 + [\% \text{Uncertainty} / 100]}$$

The sensitivity will be the value for the detection level which can yield a result which is reasonably certain to be below the limit.

$$\text{Sensitivity} < \frac{\text{Limit}}{1 + [\% \text{Uncertainty} / 100]}$$

Because only one sample of the ETF effluent discharge is taken, the %Uncertainty will be determined by systematic uncertainties (precision and bias) in the analytical method.

$$\% \text{Uncertainty} = \sqrt{(\% \text{Precision})^2 + (\% \text{Bias})^2}$$

Precision is described in the text of the report. Bias is the difference between the percent accuracy and one hundred percent. For the constituents in Table 1, the lower accuracy limits are used to calculate bias, because they yield a larger bias.

$$\% \text{Bias} = 100 - \% \text{Accuracy}$$

Combining the last three equations yields:

$$\text{Sensitivity} < \frac{\text{Limit}}{1 + \left[ \frac{\sqrt{(\% \text{Precision})^2 + (100 - \% \text{Accuracy})^2}}{100} \right]}$$