US ERA ARCHIVE DOCUMENT

Appendix E Field Sampling and Analysis

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Appendix E

Field Sampling and Analysis Program

This appendix provides a summary of the methodology used to conduct the field sampling and analysis and provides a summary of the sample analysis results. In its review of the proposed study methodology, conducted in the spring of 1997, EPA's Science Advisory Board (SAB) advised EPA to obtain monitoring data to support the study. EPA conducted field sampling and analysis of a subset of facilities that received the survey to supplement other data sources, provide "ground-truth," and fill gaps in data obtained via EPA's *Survey of Surface Impoundments* (U.S. EPA, 1999). Section E.1 describes the methodology for the field sampling and analysis program. Section E.2 describes the data quality objectives, selection of facilities, quality assurance project plan, and sampling and analysis plans. Section E.3 describes the results and conclusions and includes the analytical data from the field sampling program.

E.1 Methodology

EPA followed a systematic planning process to develop acceptance and performance criteria for the collection, evaluation, and use of data obtained from the field sampling and analysis program. Systematic planning included use of the data quality objectives (DQO) process, optimization of the sampling program to ensure completion of the sampling program within schedule and resource limits, and preparation of a detailed quality assurance project plan (QAPP). The sampling and analysis program was then implemented in accordance with the QAPP and facility-specific sampling and analysis plans (SAPs).

This section provides an overview of how EPA developed the planning documents, selected facilities for field sampling, and conducted the sampling and analysis program.

E.2 Planning and Facility Selection

E.2.1 <u>Development of Data Quality Objectives</u>

EPA developed DQOs for the field sampling and analysis program. The objective of the DQO process was to develop a sampling and analysis strategy to satisfy the data requirements of the study. The approach for developing DQOs for the Field Sampling and Analysis Program was based on the guidance presented in EPA's *Guidance for the Data Quality Objectives Process*, *EPA QA/G-4* (U.S. EPA, 2000a).

The DQO process yields qualitative and quantitative statements that

- Clarify the study objective
- Define the type, quantity, and quality of required data

- Determine the most appropriate conditions from which to collect the samples
- Specify how the data will be used.

DQOs were used to define the quality control requirements for sampling, analysis, and data assessment. These requirements were then incorporated into the QAPP and individual site-specific SAPs. The outputs of this process were documented in the *Draft DQOs Development Document* included as Attachment A of the QAPP.

E.2.2 Selection of Facilities for Field Sampling

As part of the planning process, EPA selected a subset of the 215 facilities for field sampling. This section describes the rationale for selection of facilities for field sampling and identifies those facilities at which field sampling was conducted.

E.2.2.1 <u>Rationale</u>. Selection of specific facilities for field sampling was based on criteria developed by EPA's Office of Solid Waste (OSW) and included in EPA's <u>Surface Impoundment Study Technical Plan for Human Health and Ecological Risk Assessment</u> (U.S. EPA, 2000c) and the <u>Quality Assurance Project Plan for the Surface Impoundment Study Field Sampling and Analysis Program</u> (U.S. EPA, 2000b). Specifically, EPA considered the following objectives in selecting facilities as candidates for field sampling:

- Provide chemical composition data and other information for approximately 5 to 10 percent of facilities within each industry category (i.e., within each Standard Industrial Classification (SIC) major group)
- Provide a geographically distributed set of facilities for field sampling
- "Pair" facilities geographically to optimize travel costs associated with field sampling
- Verify facility-submitted data
- Fill data gaps (e.g., check for the presence of constituents of concern for the study not reported by a facility that one might reasonably expect would be present).

E.2.2.2 <u>Facilities Selected for Sampling</u>. Table E-1 identifies each of the SIC major industry groups, the number of facilities in the sample population distributed within each group, the number and type of facilities selected for field sampling within each group, and the justification or rationale for selecting each facility.

Some facilities reported more than one waste generation process, associated waste stream, and surface impoundment in their response to the *Survey of Surface Impoundments* (U.S. EPA, 1999). Field sampling at several of these facilities included collection of samples from multiple

¹ Constituents of interest for the study are listed in Appendix 2 of the *Survey of Surface Impoundments* (U.S. EPA, 1999).

Table E-1. Facilities Selected for Field Sampling

SIC Major Group	Industry Description	Number of Industry Facilities in Survey	Number of Facilities Targeted for Sampling ^a	Facility Selected for Field Sampling	Justification/Rationale for Facility Selection
20	Food and Kindred Products	19	1	Fruit processing facility SIC Code 2037	 Verify facility-submitted data. Fill data gaps due to general lack of chemical composition data from this industry group.
22	Textile Mill Products	5	0	None	NA
24	Lumber and Wood Products, Except Furniture	7	0	None	NA
26	Paper and Allied Products	31	7	Paper mill SIC Code 2621	 Verify facility-submitted data. Facility is located near another facility of interest.
				Pulp mill SIC Code 2611	 Verify facility-submitted data. Facility is located near another facility of interest.
28	Chemicals and Allied Products	38	2	Nylon manufacturing plant SIC Code 2821	 Verify facility-submitted data.
				Industrial inorganic chemical plant SIC Code 2819	 Verify facility-submitted data. Facility is located near another facility of interest.

Table E-1. (continued)

SIC Major Group	Industry Description	Number of Industry Facilities in Survey	Number of Facilities Targeted for Sampling ^a	Facility Selected for Field Sampling	Justification/Rationale for Facility Selection
29	Petroleum Refining and Related Industries	25	7	Petroleum refinery No. 1 SIC Code 2911	 Verify facility-submitted data. Facility is located near another facility of interest.
			Petroleum refinery No. 2 SIC Code 2911	 Verify facility-submitted data. 	
30	Rubber and Miscellaneous Plastics Products	6	1	Custom rubber mixing plant SIC Code 3087	 Verify facility-submitted data. Facility is located near another facility of interest.
32	Stone, Clay, Glass, and Concrete Products	20	1	Ready mixed concrete plant SIC Code 3273	 Verify facility-submitted data. Fill data gaps due to general lack of chemical composition data from this industry group.
33	Primary Metal Industries	24	2	Electrometallurgical products facility SIC Code 3313	 Verify facility-submitted data. Facility is located near another facility of interest.
			Aluminum sheet, plate, and foil manufacturing facility SIC Code 3353	 Verify facility-submitted data Facility is located near another facility of interest 	

Table E-1. (continued)

Justification/Rationale for Facility Selection			Verify facility-submitted data. Facility is located near another facility of interest.			
Ju	NA	NA	■ ■ V H ii	NA	NA	NA
Facility Selected for Field Sampling	None	None	Semiconductors and related devices manufacturing facility SIC Code 3674	None	None	None
Number of Facilities Targeted for Sampling ^a	0	0	1	0	0	0
Number of Industry Facilities in Survey	5	3	5	5	4	1
Industry Description	Fabricated Metal Products, Except Machinery and Transportation Equipment	Industrial and Commercial Machinery and Computer Equipment	Electronic and Other Electrical Equipment and Components, Except Computer Equipment	Transportation Equipment	Sewerage Systems (Except POTWs) and Refuse Systems	Industrial Supplies
SIC Major Group	34	35	36	37	49	50

Table E-1. (continued)

SIC Major Group	Industry Description	Number of Industry Facilities in Survey	Number of Facilities Targeted for Sampling ^a	Facility Selected for Field Sampling	Justification/Rationale for Facility Selection
1	Petroleum Bulk Storage Stations and Terminals	7	0	None	NA
97	National Security and International Affairs	2	0	None	NA
	Totals ^b	210	12		

NA = Not applicable.

^a Represents approximately 5 to 10 percent of the facilities in each group.

^b From U.S. EPA, 2000a. Total does not include five facilities that claimed confidential business information (CBI).

waste streams and/or multiple impoundments, thereby conserving sampling resources and providing more thorough information about an individual facility.

E.2.2.3 <u>Preparation of the Quality Assurance Project Plan.</u> Quality assurance is a system of management activities that emphasizes systems and policies to aid in the collection of data appropriate for data users and to support management decisions in a resource-efficient manner. The outputs of the DQP process and the sampling design are combined in planning documents, and one of the key planning documents required for implementation of a field sampling program is a QAPP.

The QAPP serves as a "blueprint" for identifying how the quality system of the organization performing the work is reflected in a particular project and in associated technical goals. It is critical to any environmental data collection operation because it documents project activities, including how QA and quality control (QC) will be implemented during the life cycle of a project.

To support the field sampling component of the SI Study, the Agency prepared a comprehensive QAPP in accordance with the EPA Quality Assurance Division's R-5 document on EPA Requirements for Quality Assurance Project Plans. The QAPP, Quality Assurance Project Plan for the Surface Impoundment Study Field Sampling and Analysis Program (U.S. EPA, 2000b), was used as a guide for preparing sampling plans, conducting sampling, performing all analytical work on the collected samples, and preparing all the necessary reports. The activities addressed in the QAPP cover the entire project life cycle, integrating elements of the planning, implementation, and assessment phases. The QAPP is composed of four sections of project-related information describing

- Project management
- Measurement and/or data acquisition
- Assessment and oversight
- Data validation, usability, and assessment.

E.2.2.4 <u>Development and Implementation of Facility-Specific Sampling and Analysis Plans</u>. EPA selected 12 facilities for field sampling. For each facility, the Agency prepared a sampling and analysis plan in accordance with the specifications outlined in the QAPP. Each SAP included a project description, a listing of the project organization and staff responsibilities, quality objectives and criteria for measurement data (consistent with the Agency's performance-based measurement system, or PBMS), field procedures to be used, procedures for sample custody and transport, a listing of analyses required and facility-specific QA/QC procedures (such as requirements for decontamination and use of control samples), and equipment-specific calibration procedures and frequency. Each facility-specific SAP also included a facility-specific health and safety plan.

Both the analytical plans and the sampling plans were organized so the requisite information could be obtained within the given time and resource constraints. This was achieved by narrowing down the study list of 256 constituents of concern to a list of constituents reasonably expected to be present at each facility. This approach was consistent with the

recommendations of EPA's SAB. We developed the list of target constituents for each facility-specific SAP based on industry knowledge, information obtained from National Pollutant Discharge Elimination System (NPDES) and Resource Conservation and Recovery Act (RCRA) permits, and the survey responses.

To optimize travel resources, most of the sampling trips were arranged so that two facilities located close to each other could be visited within a given week.

E.2.2.5 <u>Field Sampling Activities</u>. EPA coordinated the sampling visits in advance with each facility representative. At each facility, prior to conducting field activities, the EPA field team met with facility representatives to review the sampling strategy, receive a briefing on any facility-specific health and safety requirements, and coordinate other activities such as the collection of split samples.

At each facility, samples generally were collected at one or more surface impoundments to represent the following locations

- Influent wastewaters
- Wastewater within the impoundment
- Effluent wastewater
- Sludges in the impoundment
- Leachate or groundwater (if present).

In addition to collecting samples for chemical analysis, the field team performed other field data collection activities including measurements of pH, dissolved oxygen, conductivity, temperature, and turbidity using a portable water quality tester. At influent and effluent sampling points, the field team measured (or estimated), where possible, the flow rates.

Field sampling activities also included

- Sample labeling
- Collection and preparation of field QC samples (such as trips blanks, equipment blanks, temperature blanks)
- Chemical and physical preservation of samples
- Decontamination of field equipment
- Implementation of sample chain-of-custody procedures and documentation
- Packaging and shipping of field samples, equipment, and supplies
- Documentation of all field activities including taking photographs of field activities.

E.2.2.6 <u>Sample Analysis and Data Reporting</u>. The field sampling and analysis program employed a performance-based approach for the analysis of samples. To that end, a constituent list was developed for each facility based on the list of constituents reported by the facility in their survey response, knowledge of the industry sector, and whether the constituent was reasonably expected to be present. Target quantitation limits were then set equal to or lower than the carcinogenic-risk screening factors (CRSFs) and noncarcinogenic risk screening factors (NCRSFs) for various matrices. EPA estimated the CRSF or NCRSF for each constituent using equations for the development of human health screening factors as presented in Table 2-1 of the Surface Impoundment Study Technical Plan for Human Health and Ecological Risk Assessment (U.S. EPA, 2000c). Two exposure scenarios were used: direct ingestion of water derived from the impoundment and direct ingestion of sediments or sludges. Finally, analytical methods were selected based on their capability to achieve the required quantitation limits.

Laboratories analyzed the samples in accordance with the facility-specific SAPs. In some cases, the laboratories were able to detect and report constituents of interest for the study that were not listed in the facility-specific target analyte list. This was possible because some analytical methods used are capable of detecting a range of constituents, many of which were not listed as target analytes for a given facility (but nonetheless were included in the list of 256 constituents of interest for the study).

Analytical data reports and waste characterization reports were prepared as specified in the QAPP and included the following key elements:

- Case narrative, containing a description of sample receipt, sample preparation, analysis, QA/QC, calibration, and laboratory manager certification
- Chain-of-custody and analysis request documentation
- QA/QC and calibration data and laboratory and instrument raw data
- Executive summary tables of analytical results
- Summary of field activities and observations
- Photo log
- Data reduction and analysis of the raw data
- Information about corrective action and protocol changes made during sampling and analysis.

Copies of the sampling plans and waste characterization reports are available from EPA.

E.3 Summary of Results

This section provides a summary of the sample analysis results for the Surface Impoundment (SI) Study field sampling program. Detailed tables of all sample analysis results are given in the individual waste characterization reports prepared for each facility visited for sampling.

The results are presented and discussed in terms of the original objectives.

Objective 1: Determine whether the waste characterization data provided by the facilities in their survey responses and the corresponding sample analysis results from EPA's sampling program are in reasonable agreement and within the range of values expected (i.e., do the EPA data "verify" the survey data).

Objective 2: Determine whether the field sampling and analysis program confirms the presence of constituents reported by the facilities and determine the extent to which the field data identify gaps in the industry-supplied data.

Objective 1: Are the EPA Field Data and the Survey Data in Reasonable Agreement?

Of the 12 facilities selected for field sampling, 10 provided responses in their surveys indicating concentration or other measurement values characterizing wastes in their in-scope surface impoundments. EPA's field sampling program yielded 175 measurement values that can be "paired" with a corresponding value provided in the survey responses.

Each pair was formed from a measurement value obtained from the analysis of an EPA sample and a second value provided by the facility in their survey for the corresponding impoundment, sample location, and matrix type (e.g., wastewater or sludge). Pairs formed from a detected value and a "less than" value were kept in the data set; however, pairs of "less than" values were removed from the data set. The data set was then edited as follows: If the EPA value was less than and its quantitation limit (QL) was greater than the survey value, then the EPA value was set equal to the survey value. If the EPA value was reported as less than QL and the QL was less than the survey value, then the EPA value was set equal to its QL. If the survey value was reported as "less than," then it was set equal to its reported detection or quantitation limit.

The measurement values include 24 pairs of pH data and 151 pairs of non-pH data, including various inorganics, volatile organics, semivolatile organics, total organic carbon (TOC), and total suspended solids. The pairs of data represent measurements of constituent concentrations and pH in wastewater influents, effluents, water within impoundments, sludges, and leachate. A complete listing of the paired data is presented as Attachment E-1 of this report.

If the EPA field data and the corresponding facility-provided data are in agreement, then the paired values should, in theory, plot on an x-y scatter plot as a roughly elliptical pattern with points falling evenly on either side of a 45° line. Figure E-1 is a scatter plot of the 151 pairs of

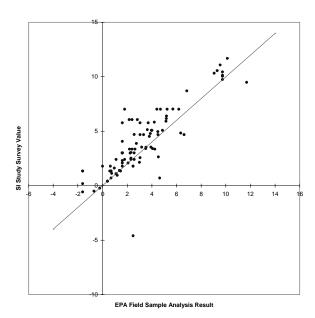


Figure E-1. EPA data versus survey data (log-log plot, without pH data).

data (not including pH).² Because the waste characterization data span several orders of magnitude, it was necessary to create the plot using the natural log-transformed values of the data sets to provide a more meaningful graphical display. The paired data plot is a roughly elliptical pattern, with points falling on both sides of a 45° line, indicating a pattern of agreement between EPA's field sample analysis results and the waste characterization data provided in the survey responses. Note that more than half of the points fall above the 45° line, indicating that the survey values generally are higher than the corresponding values obtained through the EPA field sampling effort. The plot also shows substantial deviation from agreement for a number of values. Such deviations are expected due to random variability in the waste, variability that can be introduced in the sampling and analysis processes, the fact that the two measurements in each pair were obtained at different times, and the fact that some values are represented by detection limits or they are estimates made by the facility representatives.

Another method for checking the agreement between paired values is to calculate the ratio of one value in a matched pair to the other value in the matched pair. If the ratio of the first value to the second value is 1, then there is agreement between the paired values. If the ratio of the first value to the second value is greater than 1, then the first value is greater than the second value. If the ratio is between 0 and 1, then the first value is less than the second value. Using the ratio of paired values has two advantages over the calculation of the difference between paired values: it is dimensionless, and it provides a basis for comparing multiple locations, facilities,

² pH is the negative log of the [H+] concentration and thus tends to show better agreement between paired values than does concentration data. The pH data pairs were not included in the graphic so that any pattern of agreement between paired concentration values would not be overstated.

and constituents measured in different units. Furthermore, the log base 10 of the ratio provides a simple measure of the relative relationship between paired values.

Figure E-2 is a bar chart showing the ratio of survey values to EPA sample analysis results calculated for all of the 151 pairs of non-pH data, ranked in order from the smallest to the largest. Positive values indicate survey values greater than the corresponding EPA sample analysis results. Negative values indicate survey values less than their corresponding EPA sample analysis results. The figure shows that most of the ratios are positive, indicating that the survey values generally are greater than the corresponding values obtained through the EPA field sampling effort. The study-wide pattern of agreement can be summarized as follows:

- Approximately 88 percent of paired values agree within 1 order of magnitude (i.e., one value in the pair is no more than 10 times the other value in the pair).
- Approximately 10 percent of paired values agree within 1 to 2 orders of magnitude (i.e., one value in the pair is between 10 and 100 times the other value in the pair).
- Less than 2 percent of the paired values differed by more than 2 orders of magnitude (i.e., where one value in the pair was more than 100 times the other value in the pair).

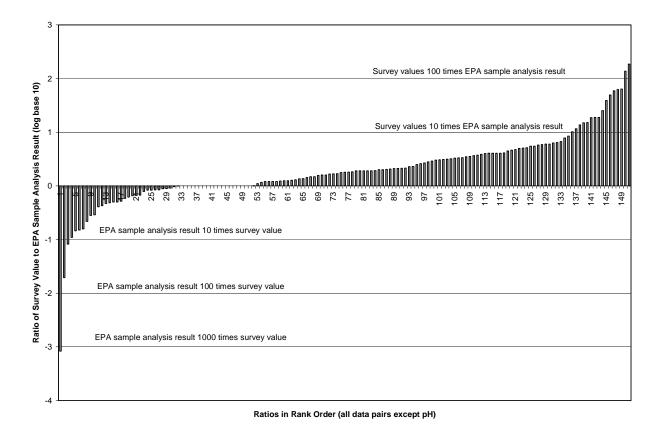


Figure E-2. Relationship between survey values and corresponding EPA measurements.

The ratios were then plotted on a facility-by-facility basis (Figure E-3) to evaluate any patterns of agreement that might exist between samples collected at a given facility. The figure shows a pattern of both negative and positive values for most facilities, similar to that which occurs on a study-wide basis.

Finally, the ratios were plotted by constituent group (Figure E-4) to evaluate any patterns of agreement that might exist between samples analyzed for a given constituent or parameter. For the purpose of this analysis, constituents were grouped into inorganics, volatile organics, and semivolatile organics, plus total organic carbon (TOC), total suspended solids (TSS), and pH. Again, the figure shows a pattern of both negative and positive values for all constituent groups, similar to that which occurs on a study-wide basis.

In summary, there is a pattern of agreement between the waste characterization data provided in the surveys and EPA's sample analysis results for the corresponding impoundments, sample locations, and parameters of interest. While there is a range of differences between paired data, the differences are not strongly associated with a particular facility or a particular constituent group.

Objective 2: To What Extent Do the EPA Data Confirm the Survey Data and Identify Data Gaps?

As an indicator of the extent to which the EPA field sampling data confirm the SI Survey responses and identify gaps in their responses, three activities were performed:

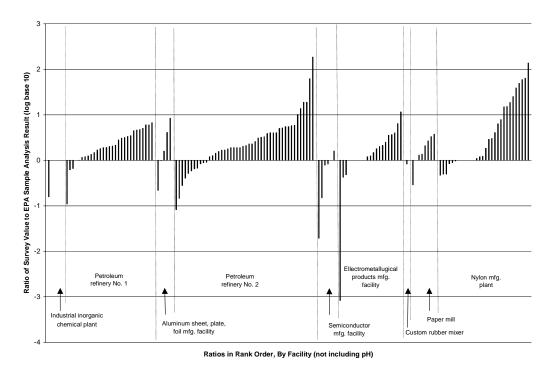


Figure E-3. Relationship between survey value and corresponding EPA measurement, by facility.

E-13

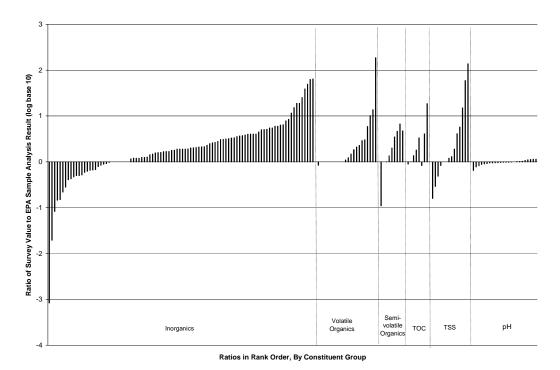


Figure 4. Relationship between survey values and corresponding EPA measurements, by constituent group.

- Determined the number of constituents reported by each facility and sample location (i.e., influent, wastewater, effluent, or sludge) in their survey as being present in their waste stream. A constituent was considered present in the waste stream if it was (1) reported as a concentration value in their survey, (2) reported as "present, but quantity unknown," or (3) reported as less than (<) a detection or quantitation limit.
- Counted the number of those **same** constituents (from above) that were also detected in samples taken by EPA.
- Counted the number of **additional** constituents detected in EPA samples that were not reported by the facility in their survey response. The count of additional constituents provided an indication of the "completeness" of the survey responses regarding constituents present and indicated the extent to which EPA data might fill "gaps" in data reported by the facilities.

These data are summarized on Table E-2. For most of the facilities sampled, the EPA field sampling confirmed the presence of constituents reported by the facilities.³ The EPA field sampling also confirmed the presence of a number of additional constituents not reported by the facilities. Quantitation of these additional constituents serves to fill gaps in data provided by the

³ In Table 2, "Number of constituents" does not include parameters such as pH, TOC, TS, and TSS.

Table E-2. Summary of Number of Constituents Reported By Facilities and Detected in EPA Samples

Facility	Location	No. of Constituents Reported by Facility in Survey ^a	No. of Same Constituents Detected by EPA ^b	Additional Constituents Detected by EPA ^c
Fruit processing facility	Influents	0	0	9
SIC Code 2037	Wastewaters	0	0	10
	Effluents	0	0	10
	Sludges	0	0	11
	Aggregate of all locations	0	0	11
Paper mill	Influents	14	6	9
SIC Code 2621	Wastewaters	14	5	10
	Effluents	13	3	6
	Sludges	10	3	16
	Aggregate of all locations	15	8	18
Pulp mill	Influents	8	7	17
SIC Code 2611	Wastewaters	8	5	13
	Effluents	8	5	15
	Sludges	10	8	27
	Aggregate of all locations	11	10	30
Nylon manufacturing plant	Influents	8	5	11
SIC Code 2821	Wastewaters	8	6	9
	Effluents	8	3	8
	Sludges	8	6	10
	Aggregate of all locations	8	6	18
Industrial inorganic chemical plant SIC Code 2819	Influents	0	0	11
	Wastewaters	0	0	9
	Effluents	6 (2)	1	5
	Sludges	0	0	15
	Aggregate of all locations	6	4	13

Table E-2. (continued)

Facility	Location	No. of Constituents Reported by Facility in Survey ^a	No. of Same Constituents Detected by EPA ^b	Additional Constituents Detected by EPA ^c
Petroleum refinery No. 1	Influents	53 (4)	10 (1)	4
SIC Code 2911	Wastewaters	53 (3)	10	3
	Effluents	54 (4)	11	4
	Sludges	53 (5)	13	7
	Aggregate of all locations	55	17	7
Petroleum refinery No. 2 SIC Code 2911	Influents	10	9	7
	Wastewaters	11	10	4
	Effluents	11	7	3
	Sludges	0	0	20
	Aggregate of all locations	11	11	13
Custom rubber mixing plant	Influents	10	4	0
SIC Code 3087	Wastewaters	10	3	0
	Effluents	10	2	0
	Sludges	10	5	3
	Aggregate of all locations	10	5	3
Ready-mixed concrete plant SIC Code 3273	Influents	0	0	5
	Wastewaters	0	0	5
	Effluents	0	0	4
	Sludges	0	0	9
	Aggregate of All locations	0	0	10
Electro-metallurgical products facility SIC Code No. 3313	Influents	14	12	16
	Wastewaters	17	12	7
	Effluents	16	11	5
	Sludges	14	12	15
	Aggregate of all locations	17	15	13

Table E-2. (continued)

Facility	Location	No. of Constituents Reported by Facility in Survey ^a	No. of Same Constituents Detected by EPA ^b	Additional Constituents Detected by EPA ^c
Aluminum sheet, plate, and foil manufacturing facility SIC Code No. 3353	Influents	5	4	5
	Wastewaters	5	4	3
	Effluents	7	5	1
	Sludges	5	5	11
	Aggregate of all locations	7	7	11
Semiconductors and related devices manufacturing facility, SIC Code No. 3674	Influents	0	0	8
	Wastewaters	0	0	7
	Effluents	4	3	3
	Sludges	0	0	13
	Aggregate of all locations	4	4	9

^a Value in () is the number of constituents reported by the facility as a "nondetect" value or "<."

facilities sampled. These additional data may be used by EPA to evaluate the veracity of survey data used to conduct the human health and ecological risk assessments.

E.4 Conclusions

The results of the field sampling indicate that there is a general pattern of agreement between the EPA field data and the reported survey data on a sample-by-sample basis. As expected, however, some extreme departures from agreement (i.e., differences greater than 2 orders of magnitude) are observed for a relatively small (<2 percent) percentage of the data. This pattern of agreement also is found when the data are grouped by facility and by constituent categories.

The Agency's field sampling program also confirmed the presence of constituents reported by the facilities and found additional constituents not reported by the facilities. The number of additional constituent identified at each facility provides evidence that facilities may have incomplete knowledge of their waste composition. Quantitation of these additional

^b Value in () is the number of constituents reported as nondetect by the facility but detected in the corresponding EPA sample.

^c A complete listing of all constituents detected in EPA samples and not reported in the surveys is provided in Attachment E-2.

constituents provides supplemental data for possible use in the uncertainty analysis of the SI Study on human health and ecological risk assessment.

References

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