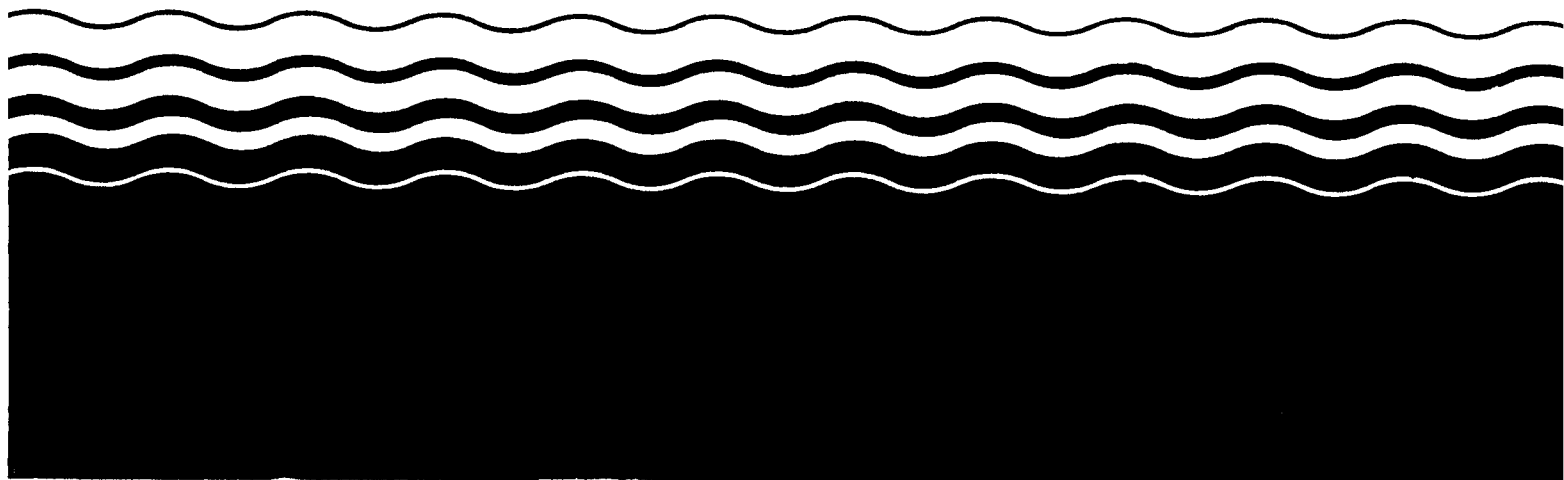




Preliminary Report of EPA Efforts to Replace Freon For The Determination of Oil and Grease



DISCLAIMER

This report has been reviewed by the Analytical Methods Staff of the EPA Office of Water. It is a preliminary report intended to provide information on the current status of this study. Evaluation of the data collected under this study is ongoing. Mention of company names, trade names or commercial products does not constitute endorsement or recommendation for use.

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- Exhibit A-1: Site Summary Freon Replacement Study
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EXECUTIVE SUMMARY

The Clean Air Act Amendments of 1990 (CAAA) require that use of Class I chlorofluorocarbons (CFCs) which deplete the ozone layer be phased out by 1996. Freon 113 (trichlorotrifluoroethane) is a Class I CFC that is required by present U.S. Environmental Protection Agency (EPA) wastewater and solid waste methods for measurement of the conventional pollutant "oil and grease". This report provides the results of Phase I of EPA's study to replace Freon 113 in the determination of oil and grease.

The objective of Phase I was to find a solvent or solvents that would produce results nearly identical to the results produced by Freon 113 in the oil and grease measurement. Solvents evaluated were *n*-hexane, *n*-hexane plus methyl tertiarybutyl ether (MTBE) in an 80/20 mixture, DuPont 123 (2,2-dichloro-1,1,1-trifluoroethane), methylene chloride, and perchloroethylene (tetrachloroethene). Solvents were evaluated by comparing results from triplicate extraction of 40 wastewaters and 28 solid/sludge wastes, from 39 industrial facilities in 24 industrial categories, to the results produced by extraction of these same samples with Freon 113.

In addition to testing the solvents listed above, sonication extraction, solid phase extraction (SPE) using cartridges and disks, and a proprietary solvent/non-dispersive infrared technique were tested on a subset of samples.

Alternative Solvents

Results were stratified according to extraction solvent and technique, and within this stratification, into three categories: all samples, petroleum-based samples, and non-petroleum based samples, depending on whether the oil and grease was of animal, vegetable, or mineral origin.

Data from solvent comparisons in the Phase I study are summarized in Exhibit 1 and show that the results produced by *n*-hexane, perchloroethylene, and the 80/20 mixture of *n*-hexane and MTBE are not statistically different from the results produced by Freon 113 (i.e., within the Acceptance Limit) for some sample strata, whereas the results produced by methylene chloride and DuPont 123 are not within this limit.

Based on these results and on the results of a preliminary study by EPA's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-Ci), EPA will retain or eliminate certain solvents and techniques from further consideration as candidates for replacement of Freon 113 in the oil and grease measurement, as follows:

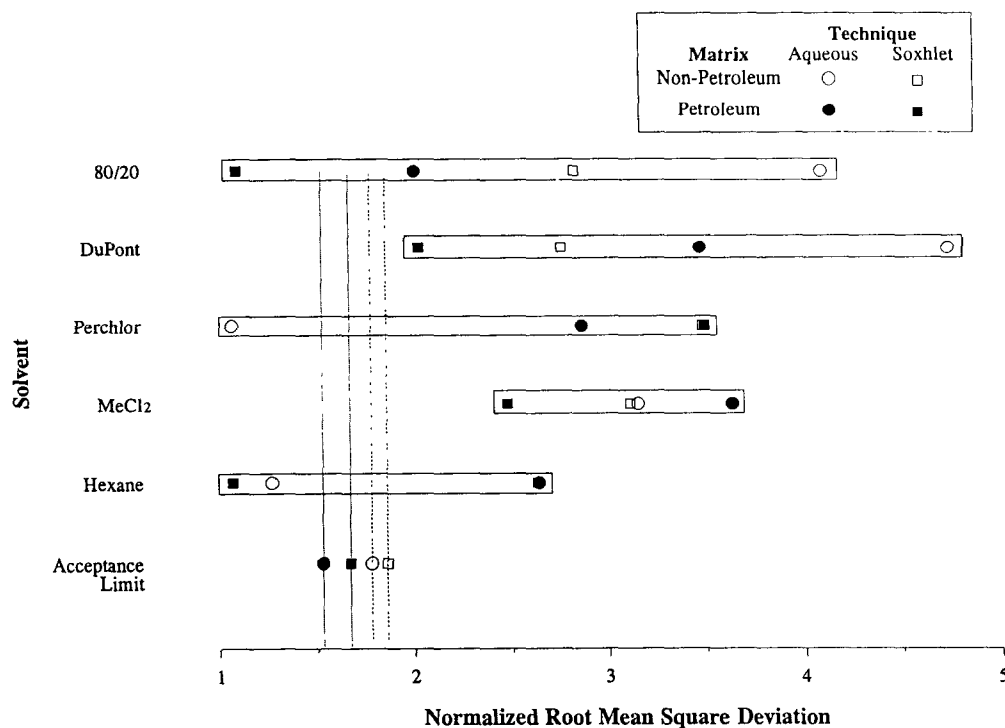
1. *n*-hexane will be retained because the results for petroleum-based solid samples and non-petroleum aqueous samples are within the Freon 113 Acceptance Limit and because *n*-hexane was used in the oil and grease measurement prior to the advent of Freon 113.
2. Perchloroethylene will be retained because the results for non-petroleum aqueous samples are within the Freon 113 Acceptance Limit and because perchloroethylene can be used in the measurement of oil and grease by infra-red techniques.

3. *n*-hexane/MTBE (80/20) will be retained because the results for petroleum-based solid samples are within the Freon 113 Acceptance Limit and because 80/20 is the solvent developed by EMSL-Ci in the preliminary study.
4. Methylene chloride will be eliminated because the results produced are far from results produced by Freon 113.
5. DuPont 123 will be eliminated because results produced using this solvent are not within the Freon 113 Acceptance Limit for any category of solid or aqueous samples and because DuPont 123 is a Class II CFC that will need to be phased out eventually.

Alternative Techniques

Of the alternative techniques evaluated in this study, only sonication extraction of non-petroleum solid samples produced results equivalent to existing techniques with Freon 113. The use of smaller solvent volumes required by solid phase extraction (SPE) and the increased sensitivity of the non-dispersive infrared technique might warrant further study.

Exhibit 1.
Solvent Root Mean Square Deviation (RMSDs) for Conventional Techniques



NOTE: Acceptance Limits for Freon 113 in each stratum (petroleum or non-petroleum) are shown by dotted vertical lines. RMSDs to the left of the respective Acceptance Limit indicate that the alternative solvent produces results equivalent to Freon 113 for that stratum, whereas RMSDs to the right of the respective Acceptance Limit indicate degrees of lesser agreement, with the least agreement being farthest to the right (highest RMSD).

SECTION 1

BACKGROUND

Chlorofluorocarbons (CFCs) have been shown to be primary contributors to the depletion of the earth's stratospheric ozone layer. The United States, as a party to the Montreal Protocol on Substances that Deplete the Ozone Layer and as required by law under the Clean Air Act Amendments of 1990 (CAAA), is committed to controlling and eventually phasing out CFCs. Under both the Montreal Protocol and the CAAA, Class I CFCs will be phased out by January 1, 1996. To be consistent with its commitment to control and phase out CFC use, the U.S. Environmental Protection Agency (EPA) is investigating the use of alternative solvents in lieu of Freon 113, a CFC that is mandated in EPA methods for the determination of "oil and grease".

Currently, three Agency methods are used for regulatory compliance monitoring determinations of oil and grease content in environmental samples. They are Method 413.1, promulgated at 40 CFR Part 136, and Methods 9070 and 9071A, promulgated at 40 CFR Parts 260-270 (EPA Publication SW-846 by reference). Method 413.1 is used in Clean Water Act (CWA) programs to determine total oil and grease content in surface and saline waters and in industrial and domestic wastes. This gravimetric method involves acidification of the sample, serial extraction of the oil and grease with Freon 113 in a separatory funnel, evaporation of the solvent from the extract, and weighing of the residue. Method 9070 is used in programs administered under the Resource Conservation and Recovery Act (RCRA) and is essentially the same as Method 413.1. RCRA Method 9071A is used to recover low levels of oil and grease from sludges, soils, other solid matrices, and some industrial wastewaters. The method involves acidification or drying, Soxhlet extraction of oil and grease with Freon 113, and weighing of the residue after evaporation of the solvent.

In all three methods described above, the result, termed "total recoverable oil and grease", is a method-defined parameter. This means that the result depends totally on how the measurement is made. Therefore, changes to the specific analytical protocols have the potential of changing the numerical value of the results for a given sample.

Clean Water Act effluent guidelines for 25 major industries include limitations on the discharge of oil and grease (40 CFR 407-471). Further, oil and grease is a regulated pollutant in over 10,000 National Pollutant Discharge Elimination System (NPDES) permits and in many RCRA operating permits. Civil penalties for violation of NPDES and RCRA permits can be severe. The measurements on which these guidelines and permits are based were made with Freon 113 or, before 1978, with *n*-hexane as the solvent. The regulated community is concerned that a change in the solvent used for oil and grease determinations could cause a change in the results and put their facilities in violation of NPDES or RCRA permits. In response to this concern, EPA initiated efforts to identify a replacement solvent or an alternative measurement technique that gives oil and grease results as close as possible to those obtained with Freon 113.

The Agency's initial efforts to find a suitable replacement solvent for Freon 113 were conducted by the Office of Research and Development's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-Ci). EMSL-Ci first used laboratory-prepared, synthetic samples containing materials that represent "oil and grease" compounds covering extremely wide

boiling ranges, such as No. 2 fuel oil, No. 6 fuel oil, Prudhoe Bay crude oil, animal lard, and wheel-bearing grease. Reagent water was spiked with these materials dissolved in an organic solvent to simulate real-world samples. These samples were then extracted using several different solvents in place of Freon 113, and the residues were determined gravimetrically.

Subsequent evaluations by EMSL-Ci used a limited number of actual industrial waste samples. The results of EMSL-Ci's work were presented in *A Study to Select a Suitable Replacement Solvent for Freon 113 in the Gravimetric Determination of Oil and Grease*, by F. K. Kawahara, October 2, 1991. This study resulted in the preparation of draft Method 413.3, a modification of 413.1 which utilizes an 80/20 mixture of *n*-hexane and methyl tertiary butyl ether (MTBE) instead of Freon 113.

On July 3, 1991, the EPA Office of Air and Radiation (OAR) proposed (56 FR 30519) to amend CWA and RCRA analytical methods for oil and grease to require the use of the 80/20 mixture in lieu of Freon 113. This proposal was based on the findings of the EMSL-Ci studies. Based on comments received on this proposal, OAR has delayed any solvent replacement to allow additional time to study alternatives. In late 1991, the Office of Water (OW) and the Office of Solid Waste (OSW) began planning a comparative study to collect data in support of OAR's efforts to replace Freon 113 in Agency oil and grease methods. The remainder of this document provides a report on the first phase of that study.

SECTION 2

PHASE I STUDY DESIGN

The first phase of the cooperative Office of Water (OW) and Office of Solid Waste (OSW) effort involved evaluation of alternative solvents and measurement techniques. The original study design is described fully in a study plan dated December 1991¹. The final Phase I study design is summarized below.

2.1 Study Objectives

The purpose of the first phase of the study was to continue EMSL-Ci's investigation of replacement solvents to identify solvents or solvent/extraction systems that provide equivalent performance to Freon 113. Specific objectives were to:

- Evaluate alternative solvent and solvent/extraction system equivalency across a range of real world effluent and solid waste samples from a variety of facility types
- Evaluate a series of solvents posing a lower potential risk to stratospheric ozone
- Provide clear direction for further study of one or two solvents or solvent/extraction systems across an even broader range of effluent and solid waste samples that are regulated under the Clean Water Act (CWA) and the Resource Conservation and Recovery Act (RCRA).

This effort was intended to be used as a screening study that would be followed by a Federal Register notice of the availability of resulting data, development of revised analytical procedures, and a confirmation study to support the recommended method revisions.

2.2 Sample Source Selection

The kinds of sample matrices included in this first phase of the study represented wastewaters and solid wastes from a variety of industrial categories and facilities. A total of 18 facilities were originally selected for sampling based on the following considerations:

- industrial category
- sample matrix (e.g., wastewater, sludge)

¹*Draft Study Plan For Sampling and Analysis Activities to Support Freon Replacement Method Study*, December 1991. Copies of this study plan are available from the Sample Control Center (operated by DynCorp Viar), 300 North Lee Street, Alexandria, Virginia 22314, (703) 557-5040.

- expected oil and grease concentration levels
- geographic location
- accessibility of waste streams
- limited co-mingling of wastes
- willingness of facilities to participate
- cooperation and assistance of facility personnel.

As the study progressed, 21 additional sites were included to expand the number of industrial categories and waste types, as budget constraints would allow. Considerations for selecting additional sites involved extending the range of oil and grease concentrations being studied, focusing on categories with existing oil and grease effluent limitations and on categories with petroleum-based waste types, and including waste streams with known analytical interference problems.

The Office of Water coordinated all facility contacts and planning for the sample collection efforts. Facilities volunteering to participate were selected primarily from EPA Regions I, II, and III to minimize the costs of travel and equipment transportation.

Aqueous and solid/sludge waste samples were collected from a total of 39 industrial and commercial facilities. These 39 facilities represent 24 industrial categories, 40 aqueous waste streams and 28 solid/sludge waste sources. A site summary is presented in Appendix A, Exhibit A-1, and shows the facility types, industrial categories, and waste streams included in the study. Appendix A, Exhibit A-2, presents additional information about the wastewater sources; it identifies petroleum-based streams and groups the samples into one of four treatment categories. Samples identified as having no treatment include raw process wastewaters. Primary treatment includes simple detention for oil/water separation or solids removal, or physical/chemical treatment designed for something other than oil removal. Secondary oil/water separation includes systems such as dissolved air flotation, dispersed gas flotation, and filtration intended for oil removal beyond simple detention. Biological treatment includes activated sludge and aerated lagoon systems.

2.3 Analytical Study Design

Initially, the study focused on evaluation of six solvents, including Freon 113, using conventional separatory funnel extraction for aqueous samples and Soxhlet and sonication extraction for solid samples, all followed by gravimetric determination. Interest in EPA's study was widespread, however, and shortly after initiating the first phase of the study EPA was approached by several manufacturers of alternative extraction devices and measurement techniques. These manufacturers agreed to analyze splits of the EPA samples by their techniques and to provide the results of their analyses to EPA at no cost to the Agency. The approaches to evaluation of each of the measurement techniques are presented below.

Separatory Funnel Extraction of Aqueous Samples

The performance of five alternative extraction solvents was compared with Freon 113 in actual sample matrices. The five solvents were:

- *n*-hexane and methyl tertiary butyl ether (MTBE) in an 80/20 mixture
- *n*-hexane alone
- DuPont 123 (2,2-dichloro-1,1,1-trifluoroethane)
- methylene chloride (dichloromethane)
- perchloroethylene (tetrachloroethene).

Wastewater samples were extracted with each of these solvents and Freon 113 using the separatory funnel techniques described in Method 413.1.² Since the densities of the solvents range from lighter than water to heavier than water, the specifics of the extraction procedures in Method 413.1 were adjusted to explicitly deal with removal of each solvent from the separatory funnel and appropriate treatment for emulsions that might form.³

Three aliquots of each sample were extracted with each solvent and were analyzed in a single laboratory only. Although the initial plan was to have all samples analyzed in one laboratory to eliminate a potential source of differences, three laboratories were ultimately required to meet schedule, cost, and quality considerations. Having multiple laboratories was thought to be a negligible source of variability because all comparisons between solvents were performed on a single sample basis.

A total of 40 triplicate aqueous sample sets were collected and sent to contract laboratories for analysis. Twenty-one triplicate sample sets were sent to Skinner and Sherman, Inc. in Waltham, MA, 13 triplicate sample sets were sent to ETS Analytical Services in Roanoke, VA and eight triplicate sample sets were sent to the Geochemical and Environmental Research Group of Texas A&M University.

Soxhlet and Sonication Extraction of Solid Samples

Sonication was tested as an alternative to the Soxhlet extraction required by Method 9071A. In addition, the performance of five alternative extraction solvents was compared with Freon 113 in actual sample matrices. The five alternative solvents tested were the same as those tested in aqueous samples using conventional separatory funnel techniques.

²Method 9070 is essentially the same as Method 413.1 and therefore was not formally evaluated.

³A total of five alternate versions of Method 413.1 were drafted for use by laboratories participating in this study. Copies of these methods are available from the Sample Control Center, 300 North Lee Street, Alexandria, Virginia 22314, (703) 557-5040.

Solid samples were extracted with each of the five alternative solvents and Freon 113 using the Soxhlet extraction techniques described in Method 9071A. Splits of the solid samples were also analyzed at the same laboratory using all six solvents and sonication procedures. The specifics of the extraction procedures in Method 9071A were adjusted to explicitly deal with the use of alternative solvents and the use of sonication extraction techniques.⁴

A total of 28 triplicate solid sample sets were collected and sent to contract laboratories for analysis. Twelve triplicate sample sets were sent to Skinner and Sherman, Inc. in Waltham, MA, nine triplicate sample sets were sent to ETS Analytical Services in Roanoke, VA and nine triplicate sample sets were sent to the Geochemical and Environmental Research Group of Texas A&M University.

Solid Phase Extraction (SPE) of Aqueous Samples

The performance of solid phase extraction (SPE) techniques was evaluated by the EPA's Central Regional Laboratory (CRL) in Annapolis, MD and by two manufacturers of SPE devices. The SPE techniques tested on aqueous samples have the advantage of using significantly less solvent than conventional separatory funnel extraction techniques.

EPA's CRL extracted aqueous samples with EnvirElute SPE columns (also known as SPE cartridges) supplied by Varian Sample Preparation Products, using Freon 113 and all five solvents listed above and gravimetric determination. Each of the samples sent to the EPA laboratory was analyzed in triplicate. Due to the high solids content in most of the aqueous samples, the EPA CRL was able to analyze only four of the eight sample sets they received.

Varian Sample Preparation Products extracted 20 aqueous samples with a newer version of the SPE columns and a refined technique that allowed testing of samples containing higher concentrations of suspended solids than the previous version tested by the EPA CRL. Varian tested Freon 113, *n*-hexane, and the 80/20 hexane/MTBE mixture as solvents and determined the results gravimetrically. Varian did not test methylene chloride or perchloroethylene as extraction solvents. Each of the samples sent to Varian was analyzed in triplicate.

The 3M Corporation extracted 28 aqueous samples, using Empore SPE disks, with the following five solvents: Freon 113, methylene chloride, *n*-hexane, 100% MTBE, and perchloroethylene. The results were determined gravimetrically. The 3M Corporation did not test the 80/20 mixture of *n*-hexane and MTBE. Splits of each sample were analyzed using 47 mm SPE disks and 90 mm SPE disks. Each of these sample splits was analyzed in triplicate. The 90 mm SPE disks required the use of a full 1-liter sample volume, which is consistent with traditional oil and grease measurement techniques and with all other techniques evaluated in this study except the 47 mm SPE disks and the infrared technique described below. Both of these techniques utilized 250 ml sample volumes.

All samples extracted with SPE devices were splits of samples analyzed by separatory funnel extraction.

⁴A total of eleven alternate versions of Method 9071A were drafted for use by laboratories participating in this study. A separate version was prepared for Soxhlet extraction of each of the five alternative solvents. Separate versions were also prepared for sonication extraction of each alternate solvent and Freon 113. Copies of these methods are available from the Sample Control Center, 300 North Lee Street, Alexandria, Virginia 22314, (703) 557-5040.

Non-Dispersive Infrared Analysis of Aqueous Samples

Horiba Instruments Inc. analyzed 36 aqueous samples by a non-dispersive infrared (NDIR) analyzer technique using S-316 (a chlorofluorocarbon also known as Flon and specifically developed for the Horiba analyzer) as a solvent. An advantage of this infrared technique is that it offers lower levels of detection than those afforded by conventional separatory funnel extraction followed by gravimetric determination. As with the SPE analyses, the Horiba results were compared statistically to the results produced on splits of the same samples with separatory funnel and Freon 113 extraction.

SECTION 3

FIELD SAMPLING

Sample collection and handling efforts were carried out by Office of Water (OW) staff, contractors, and facility staff. Activities included:

- communications and coordination with representatives of selected sampling sites and assigned laboratories
- logistics planning
- preparation of field equipment, sample containers, shipping containers, and documentation
- collection, handling, and transport of samples
- preparation of sample collection documentation
- follow-up communications with receiving laboratories for sample tracking.

For safety purposes, arrangements were made in advance to have plant personnel accompany and assist U.S. Environmental Protection Agency (EPA) and contractor representatives while on site. To minimize sampling costs where possible, EPA also asked some of the selected facilities to have plant personnel collect samples. In those cases, EPA sent prepackaged sampling kits directly to the sites with instructions for use and shipment to assigned laboratories.

Since the primary purpose of this data gathering effort was to investigate the use of alternative oil and grease extraction solvents with real world samples, and not to characterize selected wastewaters and solid wastes for regulation development, short-term grab sampling of the selected sources was deemed sufficient to meet the requirements of the study.

Sampled material was mixed in a collection vessel, and transferred by siphoning or scooping into 1-liter, wide-mouth, clear glass bottles with PTFE-lined caps. (Samples sent to 3M for extraction with the 47 mm disks and to Horiba for infrared analysis were collected in 250 ml bottles.) Aqueous samples were preserved on-site with HCl (1:1) to pH less than two, and all aqueous and solid/sludge samples were cooled to between 0° and 4°C during storage and shipment. Each sample bottle was labeled with a unique EPA sample number and identifying information, including bottle number, source location, collection date, and preservatives used. EPA sample numbers were the primary method of identifying and tracking samples. These numbers were pre-assigned and recorded to ensure proper control over samples. EPA traffic reports were completed for each site or sampling episode, and accompanied each shipment to the appropriate laboratories. Copies of these reports were used by EPA contractor and field personnel for tracking purposes.

Information regarding site-specific sampling activities was recorded in an on-site log for each sample location, and included EPA sample numbers, collection date and time, description of sample location, collection procedure, sample pH and temperature, and preservatives used. Field personnel double-checked all labels, traffic reports, and log entries to ensure accuracy and consistency.

SECTION 4

DATA VALIDATION AND STATISTICAL ANALYSIS

This section describes data validation and statistical analysis. Data validation was straightforward, as described in subsection 4.1 below. Statistical analysis was complicated by the non-uniform nature of the error distribution of the data. The ultimate measure of agreement between alternative solvents or techniques and Freon 113 was the "normalized root mean square deviation" (RMSD), as described in subsection 4.2 below. An indicator of the amount of oil and grease extracted by a given solvent relative to the amount extracted by Freon 113 is the "solvent-to-Freon ratio" which is also described in 4.2. When this ratio is averaged across samples, those with greater extraction of oil and grease (i.e., ratios of more than one) will cancel those with lesser extraction of oil and grease (i.e., ratios of less than one), yielding an average ratio close to one. Therefore, the average solvent-to-Freon ratio cannot be relied upon as a measure of agreement between the alternative solvent and Freon 113. The solvent-to-Freon ratio is presented before the RMSD in Section 4.2 because the data are not log-transformed to construct the solvent-to-Freon ratio, but are log-transformed to construct the RMSD.

4.1 Data Validation

All data from Phase I of the study were submitted to the U.S. Environmental Protection Agency (EPA) by May 1993. The contract laboratories and other study participants submitted data in computer-readable or hardcopy formats. The computer-readable result files were verified against the hardcopy data, then loaded into the study database. Results were entered into the study database manually if no computer-readable data were provided. Hardcopy data, including all calculations, submitted by the three contract laboratories (for separatory funnel extraction of aqueous samples and Soxhlet and sonication extraction of solid samples), were verified from the bench sheets provided.

A number of samples containing low concentrations of oil and grease yielded negative results or results below the nominal detection limit of Method 413.1 for aqueous samples (5 mg/L) or the equivalent detection limit in solid samples (approximately 1,000 mg/kg). The negative numbers resulted from either the subtraction of blanks (as required by the method) containing concentrations larger than those in the associated samples or from measurement error in the tare weights of the sample bottles (which weighed approximately 100 grams whereas the oil and grease residue was measured in milligrams). Negative results were found almost exclusively in aqueous samples and were primarily traceable to one of the contract laboratories performing the analyses. Treatment of negative values and values below the method detection limit is discussed in Section 4.2.

4.2 Statistical Analysis

Preliminary statistical analyses of gravimetric results from the separatory funnel, Soxhlet, and sonication techniques were presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy held in Atlanta, Georgia on March 10, 1993, and at the Oil and Grease

Workshop held by the Office of Water (OW) in Norfolk, VA on May 4, 1993. This report includes a revised statistical analysis of those results as well as a statistical analysis of the results generated using solid phase extraction (SPE) and infrared measurement techniques. A table containing all results for the study in unreduced form is provided in Appendix B.

Data Stratification

Initial statistical analyses of all samples within each extraction technique yielded no solvents that were within the respective RMSD Acceptance Limits. Further stratification of the study database was undertaken to determine whether results equivalent to Freon 113 could be achieved by testing subclasses of the data. Stratification by industry type was attempted, but resulted in too many small data sets. Because it was expected that oil and grease of biological (animal or vegetable) origin might behave differently than oil and grease of mineral origin (petroleum), the data were stratified into "petroleum" and "non-petroleum" samples. (If necessary, personnel from the sampled facilities were questioned as to the origin of oil and grease in their effluents or wastes). For example, the effluent from a meat packing plant was categorized as non-petroleum, whereas the effluent from a refinery was categorized as petroleum.

Solvent-to-Freon 113 Ratio

A solvent-to-Freon 113 ratio (solvent-Freon ratio) was computed to allow comparison of the average amount of oil and grease extracted by various solvents and measurement techniques with the amount extracted by the approved techniques using Freon 113.

The solvent-Freon ratio for each sample was formed by dividing the mean of the triplicate results produced with the alternative solvent or technique by the mean of the triplicate result produced with the approved technique using Freon 113. The mean, standard deviation (SD), relative standard deviation (RSD), and the median of the solvent-Freon ratios were calculated across all samples in each of the eight data sets representing an alternative solvent or technique (Exhibits 3 through 10). These results are discussed in Section 5.

Because averaging gives no indication of the variability of the data, the solvent-Freon ratio is a less powerful measure of the agreement between an alternative solvent (or technique) and Freon 113 than the normalized RMSD described below. This normalized RMSD was used as the main criterion of similarity. Median, mean, and standard deviation values of the solvent-Freon ratio were used as an aid in describing the distribution of the data.

Logarithmic Data Transformation

The triplicate analysis of each sample allows the error associated with each measurement to be modeled as a function of concentration. Analytical data normally have a proportional error structure over the calibration range of the measuring technique (i.e., as the parameter being measured increases, the standard deviation will increase in proportion to the measurement). This proportional error structure normally results in a constant relative standard deviation (RSD; coefficient of variation.)

To determine the nature of the error in this study, the RSD was plotted as a function of concentration. Exhibit 2 shows this plot for aqueous samples. Similar results occurred with solid/sludge samples. As can be seen from Exhibit 2, the plot has a proportional error structure above a concentration of approximately 35 mg/L. Below 35 mg/L, however, the RSD rises rapidly and asymptotically approaches a very large value at the detection limit of the method (5 mg/L).

The data were examined by ANOVA statistical techniques. The heteroscedasticity (non-uniform standard deviations) of these data, however, violates the statistical requirements for use of ANOVA techniques. The heteroscedasticity was overcome by modeling standard deviation as a function of concentration, as discussed in Appendix C, and then subjecting the data to an appropriate variance-stabilizing transformation according to the equation:

$$z = \log (x + c) - \log c$$

where

x is the concentration (in mg/L or mg/kg), and

c is a constant.

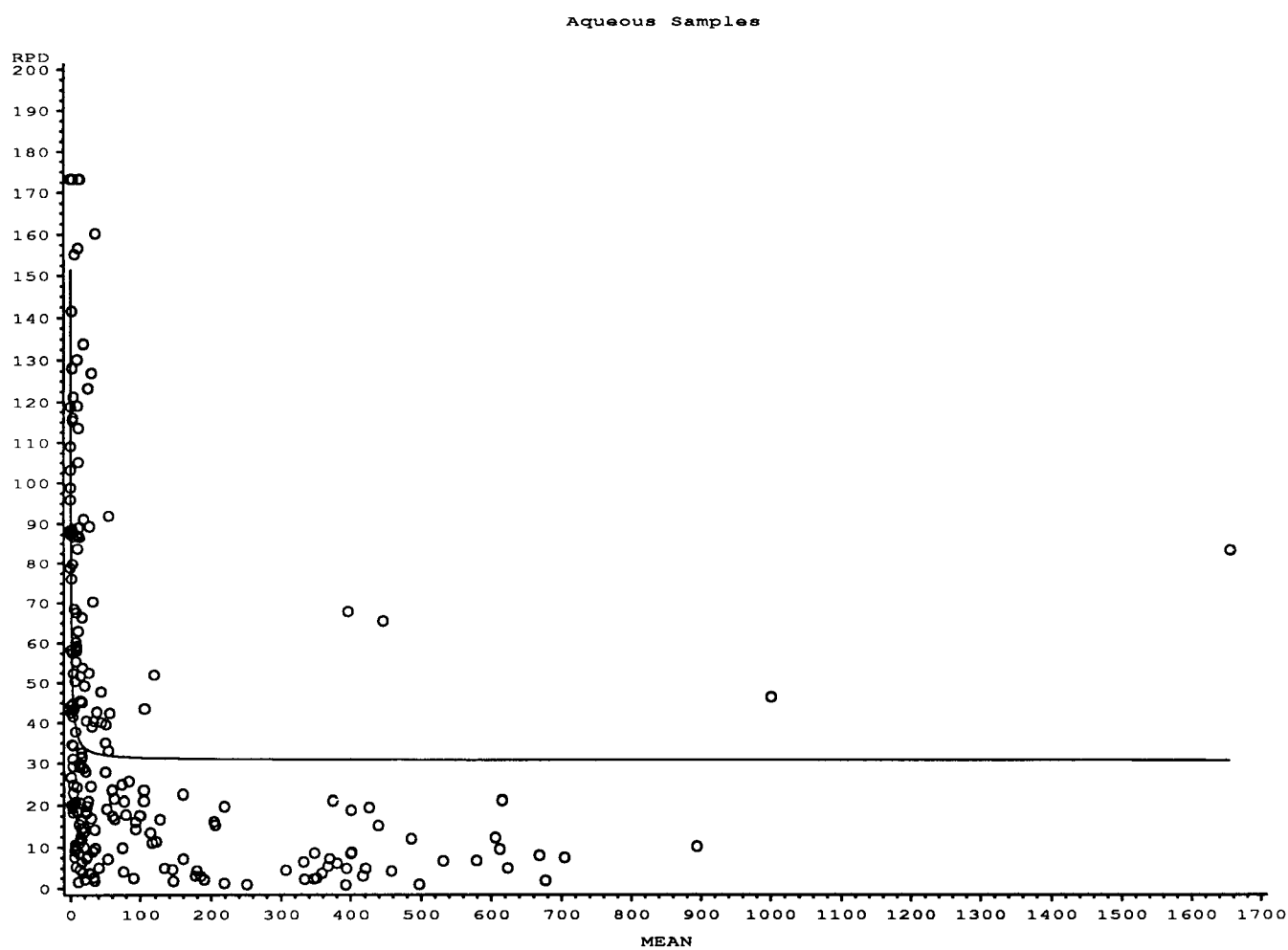
The constant (c) was used to prevent samples at low concentrations from having exaggerated influence and to allow negative values to be transformed to a logarithmic scale. The constant (c) was set to 100 mg/L for aqueous samples and 10,000 mg/kg for solid samples. The transformed data (z) were then subjected to ANOVA.

To assure that the results below the detection limit were not unduly influencing the comparison between solvents, the data were treated in three ways before transformation: (1) the results were tested "as is" (i.e., without alteration), (2) all results below the nominal detection limit were set to the detection limit, and (3) results were eliminated if they were below the nominal detection limit using the currently approved extraction technique (e.g., separatory funnel extraction with Freon 113 or Soxhlet extraction with Freon 113). After transforming the data using the equation cited above, all three treatments yielded nearly identical results: the solvents that produced results closest to Freon 113 were not changed by the three data treatments. This conclusion substantiates the belief that the transformation diminishes the effect of the results below and near the nominal detection limit. After reaching this conclusion, all data were treated with the second option for the remainder of the study. This is consistent with the EPA Engineering and Analysis Division's standard reporting of results that are below method detection limits.

Root Mean Square Deviation (RMSD)

As was noted above, the primary measure of similarity used to compare Freon 113 with each of the other solvents and techniques was the RMSD of the alternative results for each sample around the Freon 113 results. For each technique, the RMSD represented the standard deviation of the differences between the alternative solvent-determined concentrations for each sample and the Freon 113-determined concentrations using the appropriate approved technique. A smaller RMSD indicated better agreement with Freon 113.

Exhibit 2.
Relative Percent Deviation vs. Concentration



In performing the statistical analyses, the data were transformed using the equation above, and the triplicate concentration results for each sample and solvent or technique were averaged. The RMSD for each alternative procedure was calculated by taking the difference of the Freon 113 averages from those of the alternative procedure, squaring the differences, averaging these across all samples, and then taking the square root of those averages. (The details of this calculation are presented in Appendix C.)

The RMSD values were normalized by dividing each RMSD by the square root of the expected mean squared deviation based on the replicate variability (see Appendix C for details). To determine the significance of the RMSD results (where sufficient data existed), a 95 percent Acceptance Limit was calculated from each of the eight data sets (aqueous, Soxhlet, Sonication, Horiba, EPA CRL SPE, Varian SPE, 3M 47 mm SPE, and 3M 90 mm SPE) for the three sample strata: (1) all samples, (2) petroleum-based samples, and (3) non-petroleum based samples. In other words, the Acceptance Limit is the 95th percentile of the normalized RMSD that would be observed if the two methods under comparison were exactly equivalent, (i.e., if Freon 113 were compared to itself). Solvents or techniques with RMSDs smaller than the associated Acceptance Limit are not statistically different from Freon 113 extraction with the approved technique. Normalized RMSD results and Acceptance Limits for each of the alternative solvent and measurement techniques evaluated are presented in Exhibits 3 through 10.

The RMSD measures variations both above and within the amount of oil and grease extracted by Freon 113. This is important because the alternative solvent might extract less or more oil and grease than Freon 113, depending on the sample. Having the same average results across many samples is a desirable but by no means sufficient test of equivalence of the extraction performance of the alternative solvent. Average results for a solvent might be close to those obtained with Freon 113, but the results for a particular sample may be quite disparate. The RMSD is smallest, and is therefore most likely to be within the Acceptance Limit when the results for a solvent are both accurate and precise when compared to results obtained with Freon 113.

SECTION 5

DISCUSSION

The statistical results presented in Exhibits 3 through 10 are discussed in Sections 5.1 through 5.4 below. As explained in Section 4, the root mean square deviation (RMSD) was used as the main criterion of equivalence between the results obtained with established techniques (Freon 113 with separatory funnel or Soxhlet extraction) and all other solvents and techniques. Other descriptive statistics (median, mean, standard deviation [SD], and relative standard deviation [RSD] of solvent-Freon ratios) are provided in this report only to demonstrate the distribution of the data.

In these tests, some solvents and techniques extracted more oil and grease than Freon 113. Although achieving higher results than the Freon 113 methods might be seen as a method "improvement", such enhanced recovery of an operationally-defined parameter presents significant problems for the implementation of the "improved" method under the National Pollutant Discharge Elimination System (NPDES), as thousands of NPDES permits contain oil and grease limits based on the present methods. Therefore, the objective of this study was to find a solvent or technique that yields oil and grease results *equivalent to* rather than better than those obtained with Freon 113.

Exhibits 3 through 10 are arrayed to provide a comparison of each alternative solvent or technique tested, for the entire group of samples analyzed, for petroleum-based samples, and for non-petroleum samples. Solvents or techniques that have a normalized RMSD within the Acceptance Limit shown for the established method (separatory funnel or Soxhlet extraction with Freon) yield results that are not statistically different from Freon 113.

5.1 Separatory Funnel Extraction of Aqueous Samples

Results of the statistical analysis of data for separatory funnel extraction and gravimetric determination of oil and grease in aqueous samples are presented in Exhibit 3. The results are based on analysis of 25 petroleum-based samples and 13 non-petroleum samples for a total of 38 aqueous samples. (Not all of the 40 aqueous samples taken were successfully analyzed.)

Exhibit 3 shows that when all samples were examined as a group, none of the solvents tested yielded results within the Acceptance Limit. When non-petroleum and petroleum samples were examined separately, however, *n*-hexane and perchloroethylene were both within the Acceptance Limit in the non-petroleum group.

Mean solvent-Freon ratios for the separatory funnel extractions ranged from 1.02 (for petroleum-based samples extracted with *n*-hexane) to 2.61 (for non-petroleum extracted with DuPont 123). Relative standard deviations of the solvent-Freon ratios ranged from 51% (for petroleum-based samples extracted with methylene chloride) to 230% (for non-petroleum samples extracted with DuPont 123). Median solvent-Freon ratios were in the range of 0.81 to 1.38.

The data in Exhibit 3 suggest that although some alternative solvents were capable of producing average and/or median results similar to Freon 113 in each category of samples analyzed, the variability of these data was extremely high. As a result, and based on the RMSD, the only alternative solvents that yielded results equivalent to Freon 113 were *n*-hexane and perchloroethylene, and the equivalency of these solvents was demonstrated for non-petroleum samples only.

5.2 Soxhlet and Sonication Extraction of Solid Samples

Results of the statistical analysis of data for 28 solid samples are presented in Exhibits 4 and 5. Exhibit 4 presents the results of data generated using the established Soxhlet extraction and gravimetric determination with six solvents; Exhibit 5 presents the results generated using the alternative sonication and gravimetric determination with the same six solvents.

Exhibit 4 indicates that relatively good agreement between median solvent-Freon ratios was obtained using alternative solvents and Soxhlet extraction of solid samples. Means of solvent-Freon ratios ranged from 0.99 to 2.14, and RSDs ranged from 22% to 160%. The results in Exhibit 4 further indicated that none of the alternative solvents was within the Acceptance Limit when all samples were evaluated together. Similarly, none of the solvents was within the Acceptance Limit when only non-petroleum samples were examined. Two of the solvents, however, were within the Acceptance Limit when only petroleum-based samples were evaluated (*n*-hexane and 80/20.)

In Exhibit 5, all six solvents (including Freon 113) used with sonication extraction of solid samples were compared to Freon 113 using Soxhlet extraction. This comparison was made because the purpose of the study was to compare the performance of alternative solvents and techniques with currently approved solvents and techniques. As noted earlier, the Agency method for determination of oil and grease in solids (Method 9071A) specifies the Soxhlet extraction using Freon 113 as a solvent.

Exhibit 5 shows poor agreement between the mean and median results obtained with Freon 113 using the Soxhlet extraction and with all solvents using sonication. Further, variability (standard deviation and relative standard deviation) of these data was high. (Exhibit 5 shows mean solvent-Freon ratios that range from 0.49 to 3.69, with RSDs of 37% to 370% and median solvent-Freon ratios from .43 to .81.) Aside from methylene chloride, all solvents tested with sonication yielded mean and median solvent-Freon ratios within those obtained using Soxhlet extraction with Freon 113. As a result of the high variability between the sonication data and the Soxhlet data with Freon 113, only one RMSD value was observed within the Acceptance Limit in Exhibit 5. That value was for perchloroethylene when only non-petroleum samples were examined.

5.3 Solid Phase Extraction (SPE) of Aqueous Samples

Exhibits 6 through 9 present the results of the statistical analysis of data generated from aqueous samples using solid phase extraction (SPE) and gravimetric determination. The format of these tables is the same as that of Exhibits 3 through 5. As with the results of sonication extraction of solid samples, all results generated using SPE techniques were compared to the performance of approved test methods (in this case, Freon 113 using separatory funnel techniques). Results of alternative techniques were not compared with each other, nor were they compared to the performance of Freon 113 with the alternative technique.

Exhibit 6 summarizes the statistical calculations of data generated by 3M using 90 mm SPE disks. Exhibit 7 presents the results generated by 3M using 47 mm SPE disks. Nineteen of the 25 aqueous/petroleum and nine of the 13 aqueous non-petroleum samples in the study were tested using each type of disk.

Mean solvent-Freon ratios in Exhibits 6 and 7 for the SPE data using alternative solvents were all above 1.0 (they ranged from 1.09 to 2.78 for 90 mm disks and from 1.19 to 3.5 for 47 mm disks). The ratios between Freon 113 with SPE and Freon 113 with separatory funnel extraction were .94 and 1.11 for the 90 mm and the 47 mm disks, respectively. Variability with the SPE disks was generally high, with RSDs ranging from 57 to 110% for the 90 mm disks and from 60 to 130% for the 47 mm disks. Median solvent-Freon ratios using the 90 mm disks ranged from 0.94 to 1.83. The highest mean results were obtained with methylene chloride and 100% MTBE. Median solvent-Freon ratios for alternative solvents using the 47 mm disks were higher than for the 90 mm SPE disks, ranging from 1.26 to 3.65.

The RMSD values in Exhibits 6 and 7 further show that the use of separatory funnel extraction with Freon 113 and the use of either 47 mm or 90 mm disks with alternative solvents do not produce equivalent results. The only RMSD value in these tables within the Acceptance Limit is for Freon 113 using the 90 mm disks when non-petroleum samples were examined alone.

Exhibit 8 presents the results obtained by the EPA Central Regional Laboratory (CRL) using an early version of the Varian SPE column. Due to technical difficulties (primarily clogging of the columns due to high percent solids in the samples) results for only two petroleum and two non-petroleum samples were obtained. Mean and median solvent-Freon ratios were less than 1.0, and RSDs were lower than for other techniques tested. Normalized RMSD results indicate that all solvents tested were equivalent to separatory funnel extraction with Freon 113. A review of the individual results for the samples tested, however, shows that oil and grease concentrations in three of four samples is near or below the detection limit.

The setting of results below the detection limit to 5 mg/L was considered as a possible reason why the CRL SPE data showed all solvents to be equivalent to Freon 113. Tests with the three data treatments described in the section on "Logarithmic Data Transformation" in section 4.2, however, yielded equivalent results, indicating that this was not the sole reason. Because these concentrations are low, the addition of the constant in the log-transformation reduces the effect of the differences between solvents. In addition, the results are fairly precise, with RSDs ranging from 19 to 55 percent. This combination of low concentrations and precise data makes the RMSDs small and consistent, so that differences between solvents are not discernable. Although it would be possible to reduce or eliminate the constant from the equation for transforming the CRL SPE data, the net result would be that the conclusions would be based on a single sample.

Given all of the above, the final conclusion concerning the CRL SPE data is that there are insufficient data to provide a rigorous comparison between solvents.

Exhibit 9 summarizes Varian's data for fifteen of the 25 petroleum samples and five of the 13 non-petroleum samples tested using a later version of the their SPE column. As noted earlier, only Freon 113 and two alternative solvents (*n*-hexane and the 80/20 mixture) were evaluated by Varian. Mean solvent-Freon ratios ranged from 0.93 to 1.13, and the median ratios ranged from 0.93 to 1.19. Variability, as represented by the RSD of the solvent-Freon ratio, was relatively low (21% to 49%.) Neither *n*-hexane nor the 80/20 mixture yielded RMSDs within the Acceptance Limit for all samples

taken together, or for the petroleum subset. Both *n*-hexane and the 80/20 mixture had RMSDs within the Acceptance Limit in the non-petroleum category. This result must be viewed with caution due to the small number of samples involved ($n = 5$).

5.4 Non-Dispersive Infrared Analysis of Aqueous Samples

The statistical results obtained with data generated by Horiba Instruments using an infrared analyzer are presented in Exhibit 10. These results represent 23 of the 25 petroleum-based aqueous samples and all 13 of the non-petroleum aqueous samples in Phase I of the study. The mean solvent-Freon ratio found using the Horiba method was 1.2 to 2.3 times the result for separatory funnel extraction using Freon 113. Variability was high (58 to 98% RSD.) The normalized RMSDs for the Horiba results were much higher than the Acceptance Limits in all three categories analyzed, indicating that results were not equivalent to Freon 113.

5.5 Graphical Presentation of RMSD Versus Acceptance Limit Results

To give the reader a better understanding of the relative performance of all of the alternative solvents and techniques evaluated, the RMSD and Acceptance Limit data are presented graphically in Exhibit 1, which was presented in the Executive Summary of this report, and in greater detail in Exhibits 11 through 15.

In Exhibits 11 through 15, the RMSD for a particular solvent or technique is represented by a solid or hollow circle, and Acceptance Limits are indicated by horizontal lines. Where these RMSD circles fall within the relevant Acceptance Limit line, that solvent or technique is equivalent to separatory funnel or Soxhlet extraction with Freon 113, as appropriate. Where no solvents or techniques yield results equivalent to Freon 113, no circles are shown within the Acceptance Limit line.

5.6 Graphical Presentation of the Solvent-Freon Ratios

Exhibit 16 summarizes all of the solvent-Freon ratios on one graph. In this graph, the mean solvent-Freon ratio (computed prior to log-transformation of the data) is plotted on a logarithmic scale so that amounts of oil and grease greater or less than the amount extracted by Freon 113 are equidistant from 1.00.

This graph shows that the solvent-Freon ratio can range from approximately 0.3 to 2.6, depending on the solvent, technique, and kind of sample being extracted. Of interest in this graph is the influence of the solvent or technique on the average amount of material extracted. For example, Soxhlet extraction with the alternative solvents seems to extract somewhat more oil and grease than the reference Soxhlet extraction with Freon 113, whereas less oil and grease is extracted with sonication rather than with Soxhlet using Freon 113 unless methylene chloride is used in the sonication extraction. It must be remembered that these ratios represent the *average* amount of material extracted, whereas the RMSD represents the deviation from the amount extracted by Freon 113 on an individual sample basis. Therefore, although these averages are interesting, they cannot be relied upon as the final criterion for equivalence.

The use of Freon 113 as the extracting solvent in these alternative techniques allowed the variables of the solvent and the technique to be separated. The amount of oil and grease extracted by Freon 113 using cartridges and 90 mm disks was, on average, less than the amount extracted using the reference separatory funnel method whereas the average amount of material extracted by the 47 mm disks was greater. For the cartridges and the 90 mm disk, this lesser average amount extracted by Freon 113 was compensated when an alternative solvent such as MTBE, 80/20, or perchloroethylene was used. (Due to large standard deviations in the results, however, the average amount extracted may not be statistically significant.)

The variables of solvent versus technique cannot be separated for Flon-316 and NDIR because Freon 113 was not tested with the NDIR technique. Therefore it is not known whether the larger amount of oil and grease indicated by the Flon/NDIR technique results from the use of Flon-316 or results from the way in which the NDIR was calibrated.

Exhibit 3.
Summary Statistics For Alternative Solvents in the Determination of Oil and Grease
Aqueous Waste Stream, Separatory Funnel Extraction

All Samples (N=38)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.4*
Hexane	1.17	1.26	110	0.87	1.7
MeCl ₂	1.57	1.84	120	1.00	3.3
Perchlor	1.65	0.99	60	1.37	1.7
DuPont	1.85	3.66	200	1.08	4.5
80/20	1.38	2.17	160	0.94	3.7
Non-Petroleum (N=13)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.8*
Hexane	1.45	1.89	130	0.94	1.3**
MeCl ₂	2.33	3.00	130	1.32	3.2
Perchlor	1.65	1.01	61	1.34	1.1**
DuPont	2.61	6.00	230	1.03	4.7
80/20	2.00	3.62	180	0.98	4.1
Petroleum (N=25)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.5*
Hexane	1.02	0.77	75	0.81	2.6
MeCl ₂	1.20	0.60	51	1.00	3.6
Perchlor	1.65	1.00	61	1.38	2.9
DuPont	1.47	1.61	110	1.15	3.5
80/20	1.07	0.74	69	0.92	2.0

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 4.
Summary Statistics For Alternative Solvents in the Determination of Oil and Grease
Solid Waste Stream, Soxhlet Extraction

All Samples (N=28)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.5*
Hexane	1.18	1.07	91	0.95	2.2
MeCl ₂	1.63	1.56	95	1.06	2.9
Perchlor	1.75	2.40	140	1.20	3.5
DuPont	1.37	1.03	75	1.04	2.5
80/20	1.29	1.31	100	1.01	2.4
Non-Petroleum (N=11)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.9*
Hexane	1.48	1.69	110	0.95	2.6
MeCl ₂	2.05	2.27	110	1.11	3.1
Perchlor	2.14	3.44	160	1.22	3.5
DuPont	1.52	1.52	100	0.94	2.8
80/20	1.66	2.07	120	1.03	2.8
Petroleum (N=17)					
Solvent	Mean	SD	RSD	Median	RMSD
Freon	1.00	--	--	1.00	1.7*
Hexane	0.99	0.23	23	0.96	1.1**
MeCl ₂	1.36	0.83	61	1.04	2.5
Perchlor	1.50	1.46	98	1.18	3.5
DuPont	1.28	0.56	44	1.08	2.0
80/20	1.06	0.23	22	1.00	1.1**

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD} / \text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 5.
Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Solid Waste Stream, Sonication Extraction

All Samples (N=27)					
Solvent	Mean	SD	RSD	Median	RMSD
Soxhlet Freon	1.00	--	--	1.00	1.5*
Hexane	0.50	0.32	65	0.43	3.3
MeCl ₂	2.51	9.25	370	0.57	3.8
Perchlor	0.82	0.57	69	0.73	2.0
DuPont	0.72	0.55	77	0.52	2.6
80/20	0.61	0.61	99	0.50	3.4
Freon	0.62	0.42	68	0.50	2.4
Non-Petroleum (N=10)					
Solvent	Mean	SD	RSD	Median	RMSD
Soxhlet Freon	1.00	--	--	1.00	1.9*
Hexane	0.49	0.34	70	0.48	3.1
MeCl ₂	0.63	0.38	60	0.62	2.6
Perchlor	0.89	0.33	37	0.81	1.3**
DuPont	0.64	0.34	53	0.70	2.5
80/20	0.53	0.34	64	0.56	3.1
Freon	0.58	0.31	53	0.54	2.5
Petroleum (N=17)					
Solvent	Mean	SD	RSD	Median	RMSD
Soxhlet Freon	1.00	--	--	1.00	1.7*
Hexane	0.50	0.33	65	0.43	3.5
MeCl ₂	3.69	11.77	320	0.57	4.6
Perchlor	0.79	0.68	86	0.70	2.5
DuPont	0.76	0.66	86	0.49	2.7
80/20	0.66	0.73	110	0.47	3.7
Freon	0.64	0.49	76	0.48	2.4

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 6.

Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Aqueous Waste Stream, 90 mm Solid Phase Extraction Disk

All Samples (N=28)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.5*
Hexane	1.15	1.03	90	0.99	6.0
MeCl ₂	1.95	1.75	90	1.26	7.1
Perchlor	1.26	1.01	80	1.11	5.8
MTBE	2.71	2.81	100	1.62	7.9
Freon	0.93	0.62	66	0.94	5.0
Non-Petroleum (N=9)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	2.0*
Hexane	1.09	0.68	63	1.00	3.2
MeCl ₂	2.27	1.71	76	1.83	6.3
Perchlor	1.44	0.81	57	1.36	3.7
MTBE	2.57	2.09	81	1.67	5.4
Freon	0.94	0.35	38	1.00	1.3**
Petroleum (N=19)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.7*
Hexane	1.18	1.18	100	0.94	7.3
MeCl ₂	1.80	1.80	100	1.16	7.6
Perchlor	1.19	1.08	91	0.99	6.8
MTBE	2.78	3.15	110	1.58	9.2
Freon	0.93	0.70	76	0.87	6.3

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 7.
Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Aqueous Waste Stream, 47 mm Solid Phase Extraction Disk

All Samples (N=28)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.5*
Hexane	1.81	2.00	110	1.28	8.0
MeCl ₂	2.44	2.50	100	1.46	8.8
Perchlor	2.05	2.36	110	1.32	6.6
MTBE	3.27	3.82	120	1.75	9.2
Freon	1.70	1.75	100	1.24	5.1
Non-Petroleum (N=9)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	2.0*
Hexane	1.19	0.98	82	1.31	13.0
MeCl ₂	2.59	2.13	82	2.94	14.0
Perchlor	1.64	0.99	60	1.44	3.0
MTBE	2.78	2.26	81	3.65	14.0
Freon	1.11	0.70	63	1.06	3.7
Petroleum (N=19)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.7*
Hexane	2.11	2.30	110	1.26	7.7
MeCl ₂	2.37	2.71	110	1.44	8.5
Perchlor	2.22	2.72	120	1.30	8.5
MTBE	3.50	4.40	130	1.68	9.5
Freon	1.93	1.99	100	1.24	6.6

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 8.
Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Aqueous Waste Stream, SPE Column (old version)

All Samples (N=4)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	2.4*
Hexane	0.75	0.20	26	0.74	0.7**
MeCl ₂	0.84	0.16	19	0.77	0.3**
Perchlor	0.84	0.47	55	0.62	1.1**
DuPont	0.71	0.22	31	0.67	1.0**
80/20	0.85	0.16	19	0.80	0.3**
Freon	0.69	0.25	36	0.65	1.2**

* Acceptance Limit

** Value Within Acceptance Limit

Due to the small number of samples, these results should be interpreted with caution

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 9.
Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Aqueous Waste Streams, SPE Column (new version)

All Samples (N=20)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.7*
Hexane	0.96	0.38	39	0.95	3.5
80/20	1.13	0.49	43	1.16	4.6
Freon	0.76	0.33	44	0.78	4.5
Non-Petroleum (N=5)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	2.6*
Hexane	1.07	0.38	36	1.08	1.6**
80/20	1.13	0.24	21	1.19	1.8**
Freon	0.72	0.22	31	0.77	6.2
Petroleum (N=15)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.8*
Hexane	0.93	0.39	42	0.93	3.7
80/20	1.13	0.55	49	1.13	4.8
Freon	0.78	0.37	47	0.80	4.3

* Acceptance Limit

** Value Within Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD} / \text{Mean}$

Median = Median of Solvent to Freon Ratios

RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 10.
Summary Statistics For Alternative Techniques in the Determination of Oil and Grease
Aqueous Waste Stream, Infrared Analysis

All Samples (N=36)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.5*
Flon	1.62	1.55	96	1.01	8.3
Non-Petroleum (N=13)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	2.0*
Flon	2.33	2.29	98	2.05	9.4
Petroleum (N=23)					
Solvent	Mean	SD	RSD	Median	RMSD
Sep. Funnel Freon	1.00	--	--	1.00	1.7*
Flon	1.21	0.71	58	1.00	6.4

* Acceptance Limit

** Value Below Acceptance Limit

Mean = Mean of Solvent to Freon Ratios

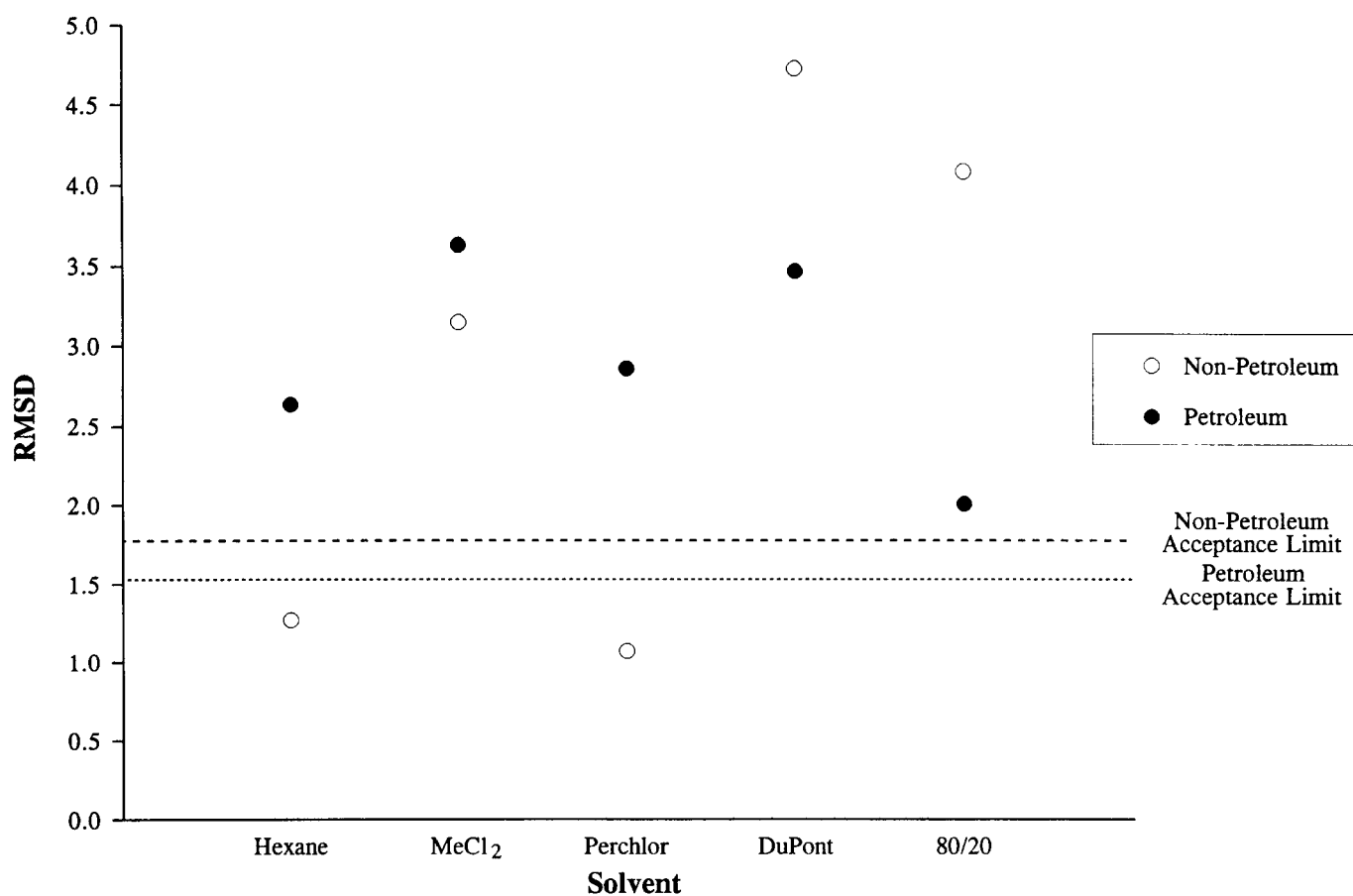
SD = Standard Deviations of Solvent to Freon Ratios

RSD = $100 \times \text{SD}/\text{Mean}$

Median = Median of Solvent to Freon Ratios

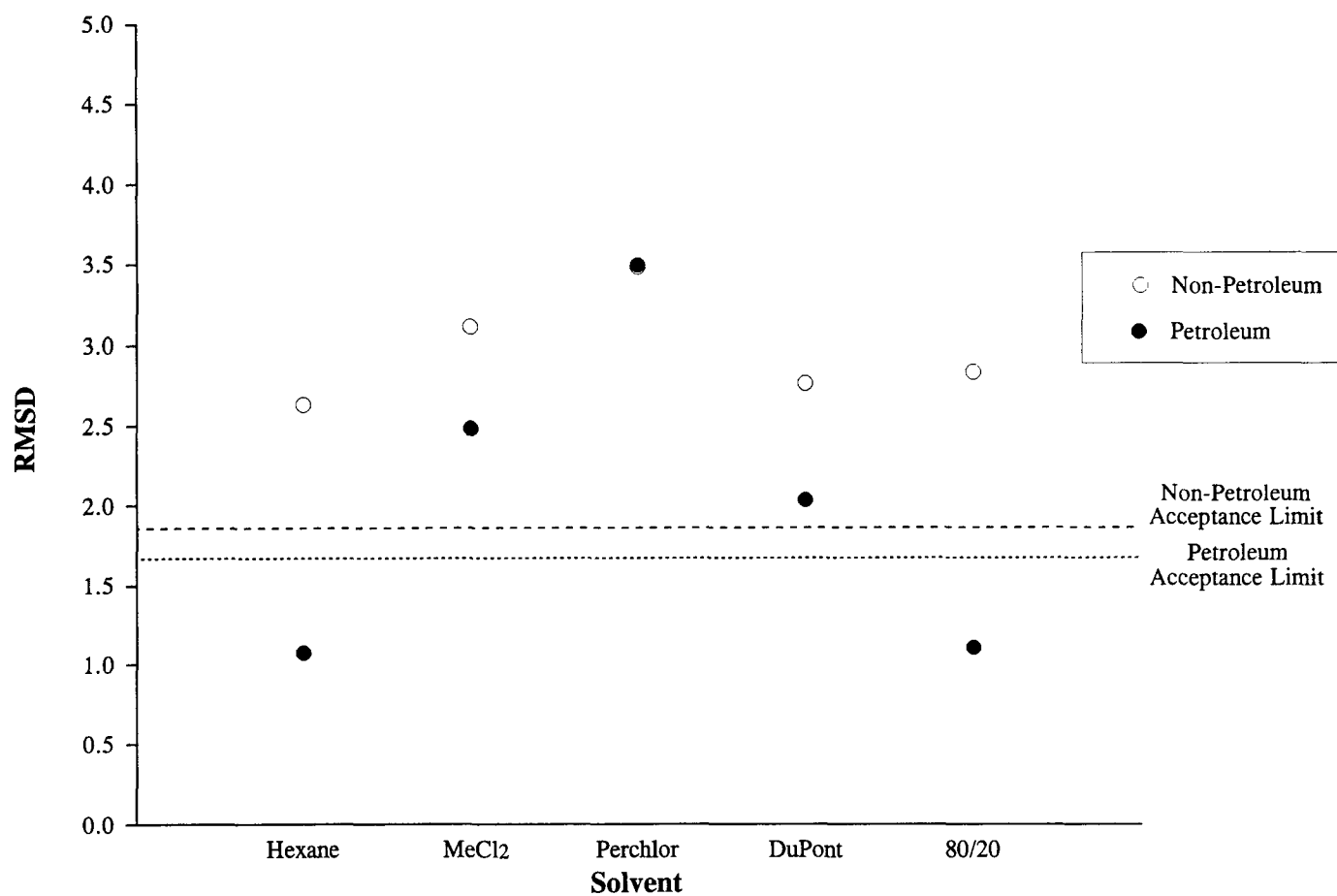
RMSD = Normalized Root Mean Square Deviation of Sample x Solvent Means

Exhibit 11.
Normalized Root Mean Square Deviations
Aqueous Waste Stream, Separatory Funnel Extraction



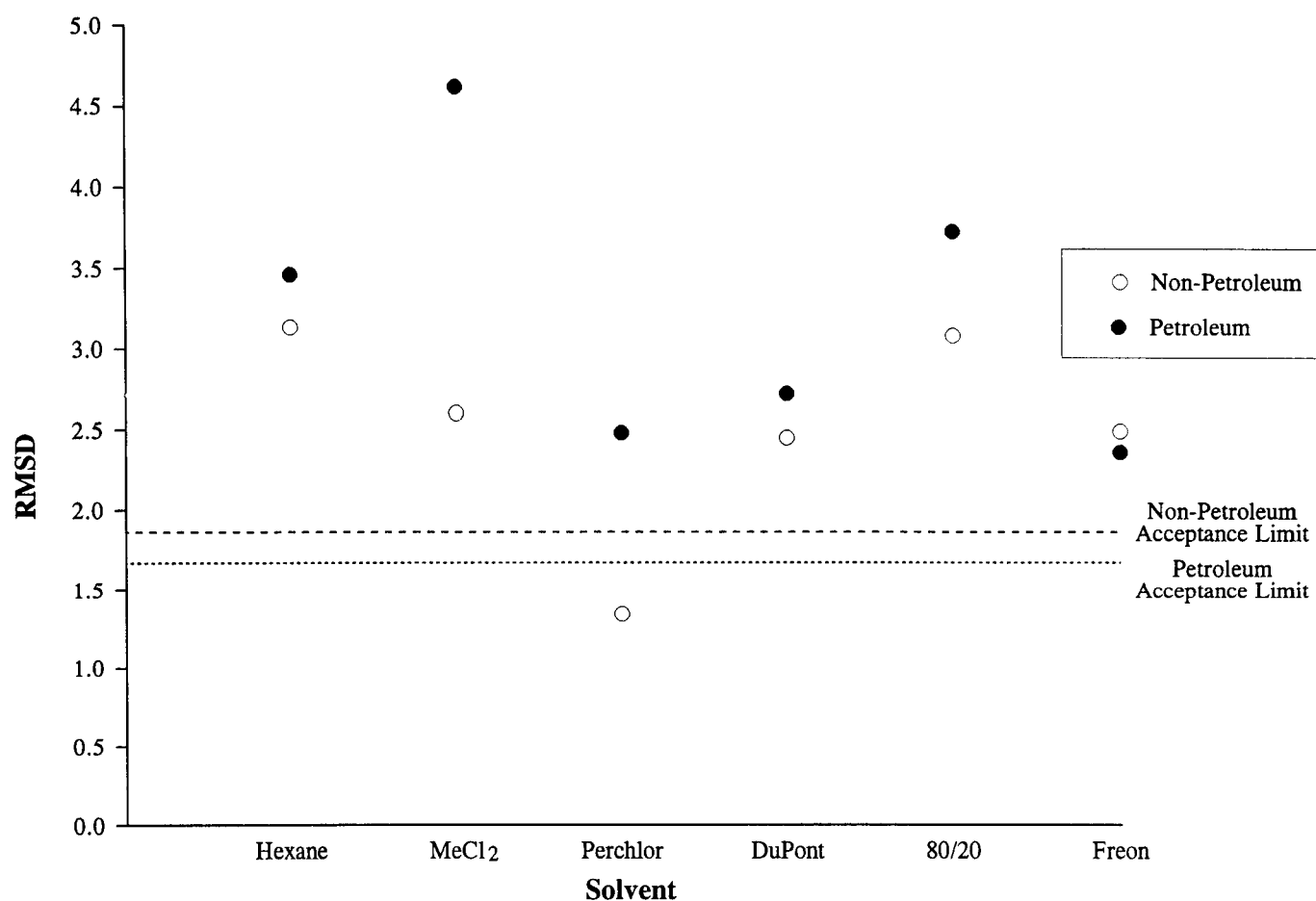
NOTE: Points below the respective Acceptance Limit are not significantly different from separatory funnel extraction with Freon

Exhibit 12.
Normalized Root Mean Square Deviations
Solid Waste Stream, Soxhlet Extraction



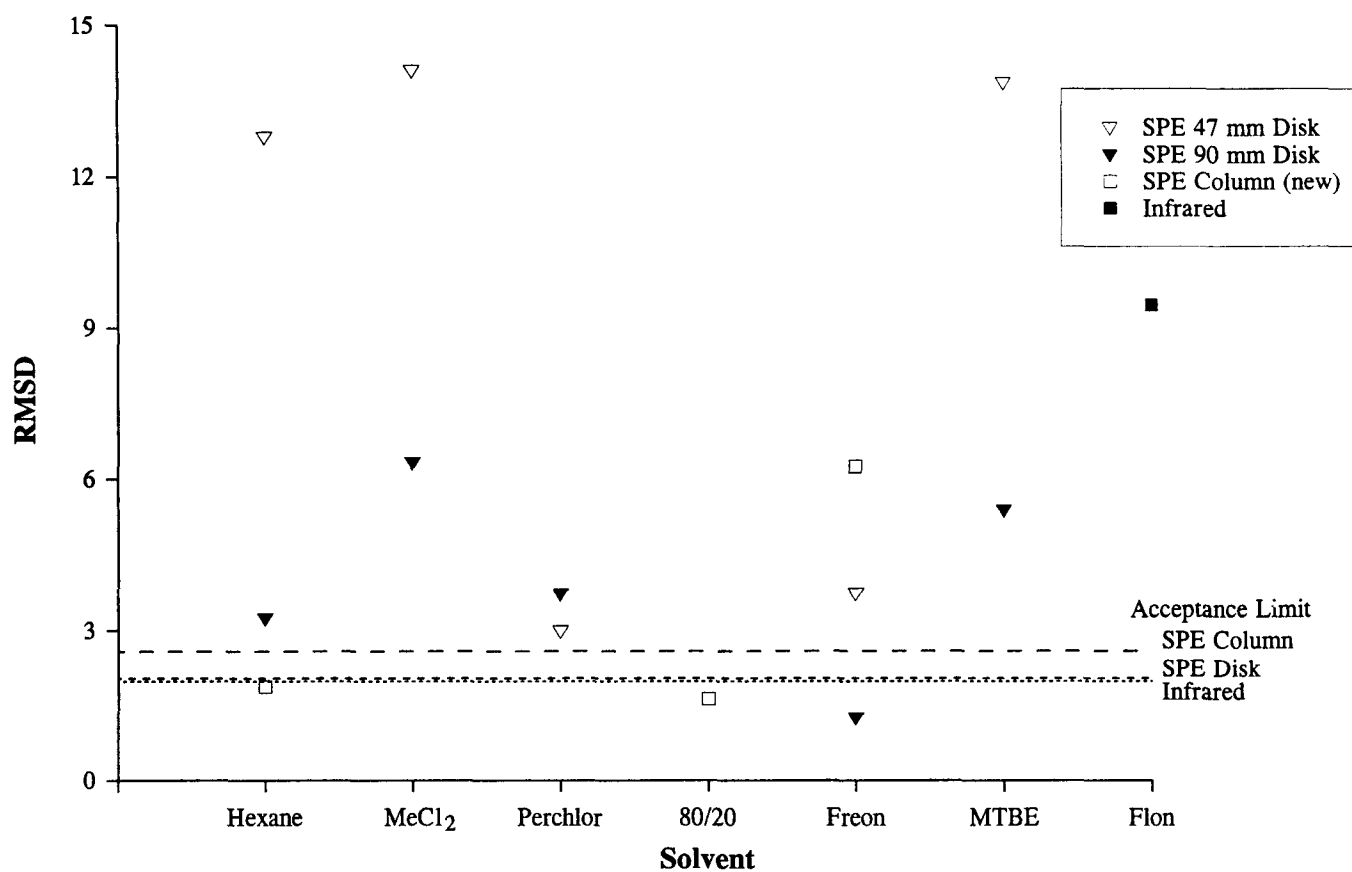
Note: Points below the respective Acceptance Limit are not significantly different from separatory funnel extraction with Freon

Exhibit 13.
Normalized Root Mean Square Deviations
Solid Waste Stream, Sonication Extraction



NOTE: Points below the respective Acceptance Limit are not significantly different from separatory funnel extraction with Freon

Exhibit 14.
Normalized Root Mean Square Deviations
Aqueous Waste Stream, Alternative Techniques
Non-Petroleum Samples



NOTE: Points below the respective Acceptance Limit are not significantly different from separatory funnel extraction with Freon

Exhibit 15.
Normalized Root Mean Square Deviations
Aqueous Waste Stream, Alternative Techniques
Petroleum Samples

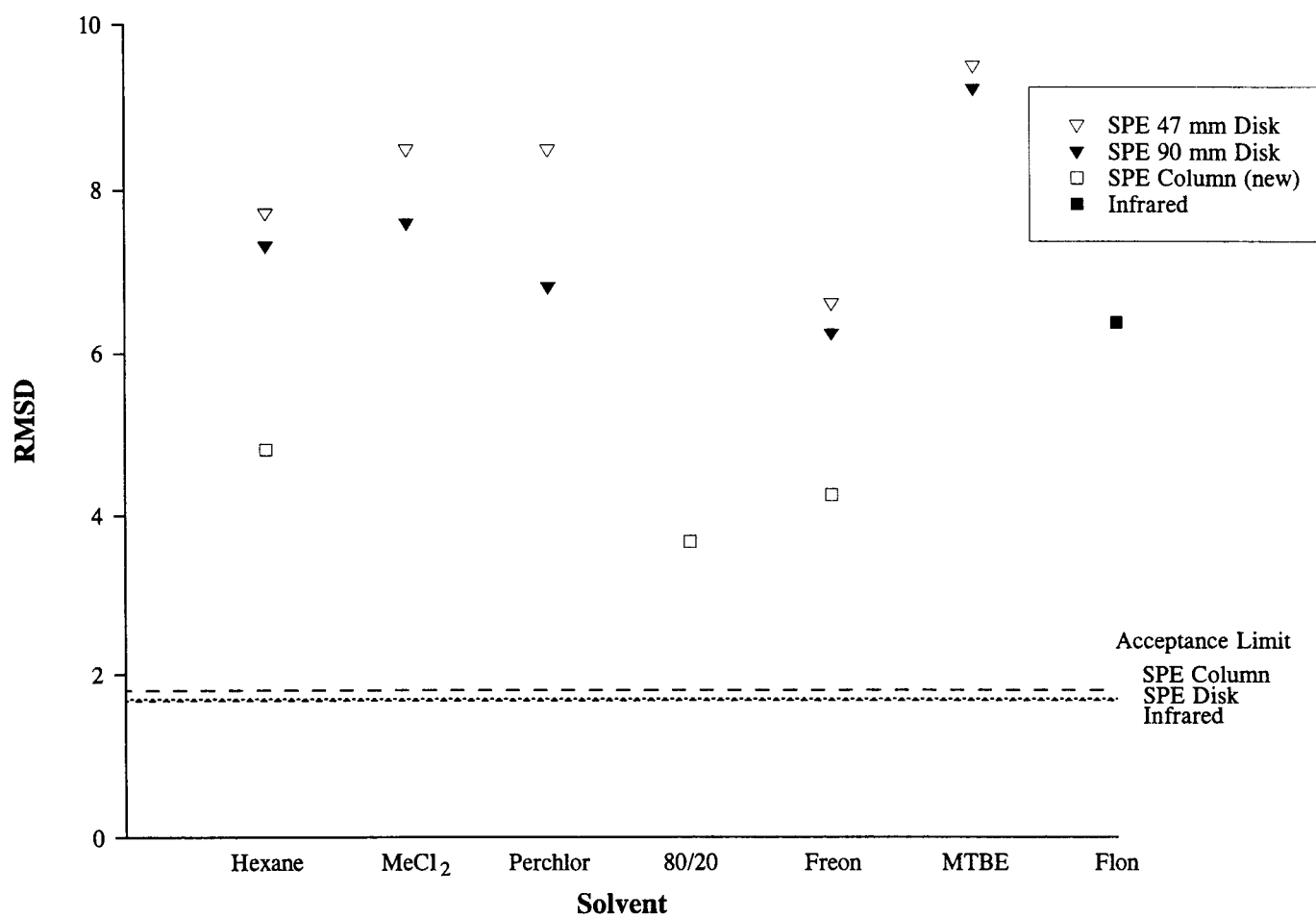
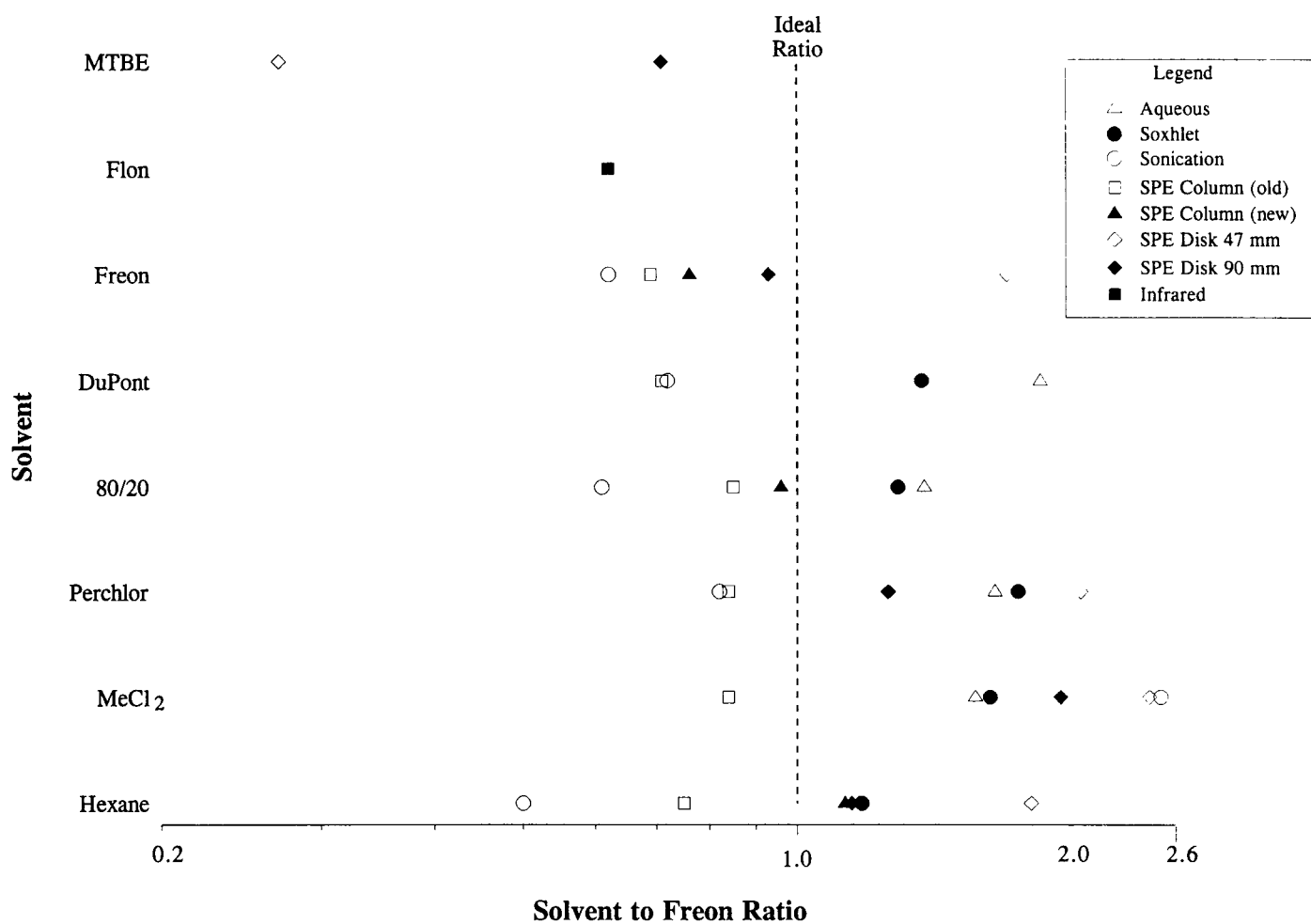


Exhibit 16.
Mean Solvent-Freon Ratios for All Techniques Tested



SECTION 6

PHASE I STUDY CONCLUSIONS

The purpose of Phase I of this study was to find an alternative solvent or extraction/measurement technique that yields results equivalent to the results produced using Freon 113 and gravimetry. No solvent or technique produced results exactly equivalent to Freon 113, but as shown in Exhibits 1 through 16, some solvents can be considered to be equivalent to Freon 113 for some sample matrices. This section summarizes some of the conclusions that can be drawn from results to date.

6.1 Alternative Solvents for Existing Methods 413.1, 9070 and 9071A

Methods 413.1, 9070 and 9071A are the three EPA oil and grease methods currently used for regulatory compliance monitoring in the NPDES and RCRA programs.

Among the five solvents tested as possible replacements for Freon 113 in Methods 413.1, 9070 and 9071A, *n*-hexane had the greatest number of results equivalent to Freon 113. Hexane had two acceptable RMSDs out of a possible four (separatory funnel extraction of non-petroleum aqueous samples and Soxhlet extraction of petroleum-based solid samples). Perchloroethylene had one acceptable RMSD (separatory funnel extraction of non-petroleum aqueous samples) as did the 80/20 mixture (Soxhlet extraction of petroleum-based solid samples). Methylene chloride and DuPont 123 had no acceptable RMSDs for the separatory funnel or Soxhlet extraction. Based on the current extraction techniques for aqueous and solid samples (separatory funnel extraction and Soxhlet extraction, respectively), *n*-hexane is the most promising candidate replacement solvent for Freon 113. Exhibit 17 below summarizes these conclusions.

Exhibit 17.
Summary of Solvents Using Conventional Techniques
with RMSDs within Acceptance Limits

Sample Stratum	Solvents with Significant RMSDs	
	Separatory Funnel Extraction (Aqueous Samples)	Soxhlet Extraction (Solid Samples)
All	None	None
Petroleum	None	<i>n</i> -hexane 80/20
Non-Petroleum	<i>n</i> -hexane perchloroethylene	None

6.2 Sonication as an Alternative Technique to Soxhlet Extraction

For sonication as an alternative extraction technique, perchloroethylene was equivalent to Soxhlet extraction with Freon 113 for petroleum samples. Therefore, perchloroethylene is a leading candidate replacement solvent if sonication is considered on an equal footing with the currently-used Soxhlet extraction technique. Perchloroethylene and *n*-hexane would both have two acceptable RMSDs out of a possible six.

6.3 Alternative Techniques for Aqueous Samples

None of the alternative techniques for aqueous samples yielded results with RMSDs within the Acceptance Limits when an adequate number of samples was tested. Some of the results using the Varian SPE columns, particularly the newer version, are promising. It remains to be seen, however, whether solid phase extraction (SPE) is applicable to samples with high dissolved solids. Without further work, none of the alternative techniques for aqueous samples can be recommended as a replacement for methods 413.1 and 9070 at this time.

6.4 Retention and Elimination of Solvents for Further Study

Based on these results and the results of the earlier study by EMSL-Ci, the following solvents will be retained or eliminated from further consideration as candidates for replacement of Freon 113 in oil and grease measurement as follows:

1. *n*-hexane will be retained because the results for petroleum-based solid samples and non-petroleum aqueous samples are within the Freon 113 Acceptance Limit and because *n*-hexane was used in the oil and grease measurement prior to the advent of Freon 113.
2. Perchloroethylene will be retained because the results for non-petroleum aqueous samples are within the Freon 113 Acceptance Limit and because perchloroethylene can be used in the measurement of oil and grease by infra-red techniques.
3. *n*-hexane/MTBE (80/20) will be retained because it is the solvent developed by EMSL-Ci in the preliminary study and because the results produced are within the Freon 113 Acceptance Limit for petroleum-based solid samples.
4. Methylene chloride will be eliminated because the results produced are far from results produced by Freon 113.
5. DuPont 123 will be eliminated because results produced using this solvent are not within the Freon 113 Acceptance Limit for any category of solid or aqueous samples and because DuPont 123 is a Class II CFC that will need to be phased out eventually.

6.5 Retention of Alternative Techniques for Further Study

Of the alternative techniques evaluated in this study, only sonication extraction of non-petroleum solid samples produced results equivalent to existing techniques with Freon 113. The use of smaller solvent volumes required by solid phase extraction (SPE) and the increased sensitivity of the non-dispersive infrared technique might warrant further study.

SECTION 7

FOLLOW-UP AND POSSIBLE PHASE II ACTIVITIES

A presentation of preliminary results was made at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy in Atlanta, Georgia on March 4, 1993, and a U.S. Environmental Protection Agency (EPA) workshop was held on May 4, 1993 in Norfolk, Virginia for all interested parties to discuss the preliminary results of Phase I and possible options for Phase II. A second workshop is scheduled to be held in Boston, Massachusetts on June 30, 1993⁵.

A notice of the availability of the results of the first phase of the study will be published in the Federal Register, with a request for public comment. This report, with subsequent revisions, if any, will be mailed to those responding to the notice. The notice may also include a study plan for the second phase of the study and a request for regulated industries to produce data using the one or two most promising alternative solvents.

If EPA proceeds with a second study phase, it will likely be designed to assess the precision, accuracy, and comparability of the one or two most promising alternative solvent/extraction systems and measurement techniques from the Phase I study results. The range of industrial effluents might also be expanded in the second phase, in part through cooperative efforts sponsored by various regulated industries. Alternatively, the second phase of the study may involve side-by-side testing of an alternative solvent versus Freon 113, so that each permittee can develop its own correction factor (see below) pending renewal of their NPDES or RCRA permit.

Options for Replacement of Freon 113 in the Oil & Grease Method

The Phase I results have shown that no solvent produces results identical to the results produced by Freon 113, but that *n*-hexane, perchloroethylene, and the 80/20 mixture of *n*-hexane and MTBE produce results equivalent to Freon 113 for some samples. These results suggest that if an immediate decision to replace Freon 113 needs to be made, one or more of these solvents should be selected, and that any Phase II effort should concentrate on these solvents. The reduction in solvent use afforded by solid phase extraction and the lowered detection limit attained with non-dispersive infrared determination, however, provide compelling reasons for further study of these techniques. EPA therefore desires to be as comprehensive as possible in exploring options for any possible Phase II study, but needs to narrow the focus of these options once they have been explored.

The range of options under consideration at this time is given below.

- File for an exemption under the Clean Air Act Amendments for the use of Freon 113 in the oil and grease method.
- Use recycled Freon. Laboratories would reuse Freon 113 recovered from earlier testing.

⁵Conference arrangements for the Oil and Grease Workshop are being coordinated by DynCorp Viar, Inc. For additional information, please contact Christopher Berry at (703)519-1387.

- Choose one new solvent.
- Choose more than one new solvent, if different solvents are found to work best on different kinds of samples.
- Use the Total Petroleum Hydrocarbon (TPH) method for petroleum-contaminated samples.
- Switch from gravimetric to infrared spectroscopy-based methods.
- Use solid phase extraction (SPE) rather than solvent extraction.
- Develop a correction factor or factors to be applied to data generated by solvents other than Freon 113.

EPA solicits comments on these options and seeks any other options for resolution of this issue. As comments and other information become available, the Agency will continue to attempt to keep all interested parties informed. Because Freon 113 will not be commercially available after January 1, 1996, and because it is desirable to phase out the use of all Class I CFCs as expeditiously as possible, the first two options listed above may not be viable.

APPENDIX A
SITE SUMMARY

Exhibit A-1.
Site Summary
Freon Replacement Study

FACILITY TYPE	INDUSTRIAL CATEGORY/ACTIVITY	WASTE STREAMS	
		AQUEOUS	SOLID/SLUDGE
Paper Mill	Pulp & Paper	Bleach Plant Effluent	Dewatered Sludge
Oil Production Site	Oil & Gas Extraction	Dispersed Gas Flotation Eff.	
POTW	POTW	Primary Effluent	Dewatered Sludge
		Secondary Effluent	
Leather Tannery	Leather Tanning	Primary Effluent	Dewatered Sludge
POTW	POTW		Digested Sludge
Petroleum Refinery	Petroleum Refining	API Separator Effluent	API Separator Sludge
		Secondary Effluent	
Industrial Laundry	Industrial Laundries	Dissolved Air Flotation Eff.	DAF Sludge
Textile Mill	Textile Manufacturing	Lagoon Effluent	
Metal Finishing Plant	Metal Finishing	Separator Effluent	
Fish Oil Plant	Soap & Detergent Manf.	Primary Effluent	Oily Sludge
Rendering Plant	Meat Products & Rendering	Primary Effluent	
Coke Plant	Iron & Steel Manufacturing	Secondary Effluent	Waste Activated Sludge
Slaughterhouse	Meat Products & Rendering	Primary Effluent	
		Secondary Effluent	
Wood Preserving Plant	Timber Products	Secondary Effluent	Solid Waste
Drilling Fluid Supplier	Oil & Gas Extraction		Used Drilling Mud
Soils	Contaminated Soils		Soil w/ Kerosene
Poultry Plant	Poultry Processing	Secondary Effluent	Waste Activated Sludge
Rolling Mill	Iron & Steel Manufacturing	Filter Effluent	Dewatered Scale Material
Oil Terminal	Petroleum Storage Facilities	Separator Effluent	
Mayonnaise Plant	Miscellaneous Foods	Filter Effluent	Oily Sludge
Seafood Plant	Seafood Processing	Secondary Effluent	Waste Sludge
Abrasives Plant	Abrasives Manufacturing	Process Wastewater	
Oil Terminal	Petroleum Storage Facilities	Separator Effluent	
Seafood Plant	Seafood Processing	Primary Effluent	Oily Sludge
			Dewatered Sludge
Poultry Plant	Poultry Processing	Dissolved Air Flotation Inf.	DAF Sludge
Meat Packing Plant	Meat Products & Rendering	Lagoon Effluent	
Railroad Yard	Transportation Facilities	Dissolved Air Flotation Eff.	Oily Sludge
Can Manufacturing Plant	Coil Coating	Primary Effluent	Filter Cake
Soup Plant	Miscellaneous Foods	Dissolved Air Flotation Eff.	DAF Sludge
Oily Water Treatment Plant	Shore Reception Facilities	Separator Effluent	Oily Sludge
Can Manufacturing Plant	Coil Coating	Dissolved Air Flotation Inf.	Filter Cake
Can Manufacturing Plant	Coil Coating	Dissolved Air Flotation Inf.	Filter Cake
		Dissolved Air Flotation Eff.	
Drum Handling Facility	Drum Reconditioning	Filter Effluent	Oily Sludge
Polymer Plant	Organic Chemicals	Secondary Effluent	Dewatered Sludge
Restaurant	Food Retail		Vegetable Oil Waste
Industrial Laundry	Industrial Laundries	Primary Effluent	
Formulating Plant	Soap & Detergent Manf.	Primary Effluent	
Leather Tannery	Leather Tanning	Primary Effluent	Waste Sludge
Petroleum Refinery	Petroleum Refining	API Separator Effluent	
		Interceptor Effluent	

Exhibit A-2.
Wastewater Sources
Freon Replacement Study

FACILITY TYPE	AQUEOUS WASTE STREAMS	BASIS	LEVEL OF TREATMENT UPSTREAM OF SAMPLE POINT			
			NONE	PRIM.	SO/W	BIOL.
Paper Mill	Bleach Plant Effluent	P	X			
Oil Production Site	Dispersed Gas Flotation Eff.	P			X	
POTW	Primary Effluent	P		X		
	Secondary Effluent	P				X
Leather Tannery	Primary Effluent	N		X		
Petroleum Refinery	API Separator Effluent	P		X		
	Secondary Effluent	P				X
Industrial Laundry	Dissolved Air Flotation Eff.	P			X	
Textile Mill	Lagoon Effluent	N				X
Metal Finishing Plant	Separator Effluent	P		X		
Fish Oil Plant	Primary Effluent	N		X		
Rendering Plant	Primary Effluent	N		X		
Coke Plant	Secondary Effluent	P				X
Slaughterhouse	Primary Effluent	N		X		
	Secondary Effluent	N				X
Wood Preserving Plant	Secondary Effluent	P				X
Poultry Plant	Secondary Effluent	N				X
Rolling Mill	Filter Effluent	P			X	
Oil Terminal	Separator Effluent	P		X		
Mayonnaise Plant	Filter Effluent	N			X	
Seafood Plant	Secondary Effluent	N				X
Abrasives Plant	Process Wastewater	P	X			
Oil Terminal	Separator Effluent	P		X		
Seafood Plant	Primary Effluent	N		X		
Poultry Plant	Dissolved Air Flotation Inf.	N		X		
Meat Packing Plant	Lagoon Effluent	N			X	
Railroad Yard	Dissolved Air Flotation Eff.	P			X	
Can Manufacturing Plant	Primary Effluent	N		X		
Soup Plant	Dissolved Air Flotation Eff.	N			X	
Daily Water Treatment Plant	Separator Effluent	P		X		
Can Manufacturing Plant	Dissolved Air Flotation Inf.	N		X		
Can Manufacturing Plant	Dissolved Air Flotation Inf.	N		X		
	Dissolved Air Flotation Eff.	N			X	
Drum Handling Facility	Filter Effluent	P			X	
Polymer Plant	Secondary Effluent	P				X
Industrial Laundry	Primary Effluent	P		X		
Formulating Plant	Primary Effluent	N		X		
Leather Tannery	Primary Effluent	N		X		
Petroleum Refinery	API Separator Effluent	P		X		
	Interceptor Effluent	P			X	

LEGEND

BASIS:

- P - Petroleum
- N - Non-Petroleum

LEVEL OF TREATMENT:

- NONE - No Treatment
- PRIM - Primary Treatment
- SO/W - Secondary Oil/Water Separation
- BIOL - Biological Treatment

APPENDIX B
DATA SUMMARY

OIL & GREASE DATA
FREON REPLACEMENT STUDY
PHASE I

June 21, 1993
Page 1 of 25

					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
1 PAPER MILL	22221	BLEACH PLANT EFF	LIQ/LIQ (A)	DUPONT123	5.0 <			5.0 <	
				FREON	5.0 <			5.0 <	
				HEXANE	5.0 <			5.0 <	
				METHYLENE CHLORIDE	5.0 <			5.0 <	
				PERCHLOR 80/20	5.1			5.1	
					5.0 <			5.0 <	
	22221	BLEACH PLANT EFF	INFRARED (A)	FLON	2.5	2.4		2.5	0.1
	22222	DEWATERED SLUDGE	SONICATION (S)	DUPONT123	3700.0	2700.0	2300.0	2900.0	679.3
				FREON		3400.0	2000.0	2700.0	1036.3
				HEXANE	1200.0	1100.0	1300.0	1200.0	101.3
				METHYLENE CHLORIDE	7800.0	320.0	8800.0	5700.0	4652.9
				PERCHLOR 80/20	4800.0	1500.0	2900.0	3100.0	1616.9
					1100.0	2400.0	1900.0	1800.0	673.1
	22222	DEWATERED SLUDGE	SOXHLET (S)	DUPONT123	11000.0	12000.0	10000.0	11000.0	909.5
				FREON	11000.0	5300.0	7900.0	8000.0	2762.2
				HEXANE	6600.0	2400.0	11000.0	6600.0	4203.1
				METHYLENE CHLORIDE	25000.0	26000.0	27000.0	26000.0	925.9
				PERCHLOR 80/20	16000.0	5000.0	7700.0	9500.0	5560.2
					11000.0	6500.0	6900.0	8000.0	2214.9
	23479	BLEACH PLANT EFF	LIQ/LIQ (A)	DUPONT123	7.4	7.7	6.3	7.1	0.8
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	8.1	8.9	8.1	8.3	0.4
				PERCHLOR 80/20	7.0	6.5	5.8	6.4	0.6
					5.0 <	5.0 <	5.8	5.3	0.4
	23479	BLEACH PLANT EFF	INFRARED (A)	FLON	5.1	4.3		4.7	0.6
	23479	BLEACH PLANT EFF	SPE DISK 90mm (A)	METHYLENE CHLORIDE	14.0	13.0		14.0	0.8
				FREON	15.0	16.0		15.0	0.2
				HEXANE	12.0	13.0		13.0	1.3
				MTBE	61.0	66.0		63.0	3.8
				PERC	11.0	11.0		11.0	0.3
	23479	BLEACH PLANT EFF	SPE DISK 47mm (A)	METHYLENE CHLORIDE	22.0	20.0		21.0	1.1
				FREON	32.0	24.0		28.0	5.4
				HEXANE	29.0	32.0		30.0	2.5
				MTBE	76.0	85.0		80.0	6.2
				PERC	28.0	26.0		27.0	1.3

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

OIL & GREASE DATA
FREON REPLACEMENT STUDY
PHASE I

June 21, 1993
Page 2 of 25

					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
2 OIL PRODUCTION SITE	23479	BLEACH PLANT EFF	SPE COLUMN II (A)	FREON	6.0	5.0 <		5.5	0.7
				HEXANE	5.0 <	5.0 <		5.0 <	
				80/20	12.0	5.0 <		8.5	4.9
	22223	DGF EFFLUENT	LIQ/LIQ (A)	DUPONT123	23.0	17.0	20.0	20.0	2.9
				FREON	5.0 <	42.0	5.0 <	17.0	21.3
				HEXANE	27.0	29.0	27.0	27.0	1.0
				METHYLENE CHLORIDE	15.0	23.0	42.0	27.0	14.0
				PERCHLOR	5.0 <	5.0 <	32.0	14.0	15.4
				80/20	44.0	31.0	19.0	31.0	12.3
	22223	DGF EFFLUENT	INFRARED (A)	FLON	15.0	15.0		15.0	0.2
3 POTW	22223	DGF EFFLUENT	SPE COLUMN I (A)	DUPONT123	11.0	11.0	12.0	11.0	0.5
				FREON	8.4	9.3	9.4	9.0	0.5
				HEXANE	14.0	18.0	13.0	15.0	2.7
				METHYLENE CHLORIDE	10.0	13.0	22.0	15.0	5.9
				PERCHLOR	10.0	16.0	12.0	13.0	2.6
				80/20	16.0	16.0	12.0	15.0	2.1
	22224	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	30.0			30.0	
				FREON	18.0			18.0	
				HEXANE	43.0			43.0	
				METHYLENE CHLORIDE	24.0			24.0	
				PERCHLOR	31.0			31.0	
				80/20	5.0 <			5.0 <	
	22224	PRIMARY EFFLUENT	INFRARED (A)	FLON	13.0	14.0		13.0	0.2
	22238	SEWAGE SLUDGE	SONICATION (S)	DUPONT123	37000.0	35000.0	37000.0	36000.0	867.0
				FREON	46000.0	40000.0	37000.0	41000.0	4211.9
				HEXANE	20000.0	27000.0	21000.0	22000.0	3894.8
				METHYLENE CHLORIDE	46000.0	45000.0	40000.0	44000.0	2996.8
				PERCHLOR	22000.0	23000.0	26000.0	24000.0	2203.7
				80/20	23000.0	19000.0	26000.0	22000.0	3487.1
	22238	SEWAGE SLUDGE	SOXHLET (S)	DUPONT123	120000.0	110000.0	110000.0	110000.0	4041.5
				FREON	98000.0	81000.0	81000.0	87000.0	9940.5
				HEXANE	110000.0	86000.0	80000.0	91000.0	13281.3
				METHYLENE CHLORIDE	120000.0	140000.0	150000.0	140000.0	14364.3
				PERCHLOR	140000.0	160000.0	110000.0	130000.0	26764.4
				80/20	100000.0	81000.0	72000.0	84000.0	14360.0

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

OIL & GREASE DATA
FREON REPLACEMENT STUDY
PHASE I

June 21, 1993
Page 3 of 25

					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
4 LEATHER TANNERY	23116	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.4	5.0 <	5.0 <	5.1	0.3
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR 80/20	23.0	9.3	11.0	15.0	7.6
					5.0 <	5.0 <	5.0 <	5.0 <	
	23116	SECONDARY EFFLUENT	INFRARED (A)	FLON	0.0	0.0		0.0	
	22225	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	540.0	1200.0	3200.0	1700.0	1381.6
				FREON	89.0	80.0	53.0	74.0	18.4
				HEXANE	30.0	49.0	23.0	34.0	13.7
				METHYLENE CHLORIDE	74.0	51.0	39.0	55.0	18.1
				PERCHLOR 80/20	110.0	65.0	80.0	84.0	21.6
					920.0	580.0	1500.0	1000.0	465.8
	22225	PRIMARY EFFLUENT	INFRARED (A)	FLON	200.0	230.0		220.0	21.2
	22226	DEWATERED SLUDGE	SONICATION (S)	DUPONT123	10000.0	15000.0	15000.0	13000.0	2857.7
				FREON	10000.0	7900.0	11000.0	9600.0	1502.5
				HEXANE	11000.0	5300.0	9800.0	8700.0	2921.8
				METHYLENE CHLORIDE	8800.0	6900.0	6300.0	7400.0	1320.5
				PERCHLOR 80/20	12000.0	14000.0	20000.0	15000.0	4348.8
					6300.0	10000.0	10000.0	8900.0	2271.5
	22226	DEWATERED SLUDGE	SOXHLET (S)	DUPONT123	31000.0	27000.0	23000.0	27000.0	3998.2
				FREON	11000.0	12000.0	12000.0	12000.0	731.9
				HEXANE	21000.0	15000.0	19000.0	18000.0	3201.0
				METHYLENE CHLORIDE	62000.0	54000.0	58000.0	58000.0	4070.3
				PERCHLOR 80/20	29000.0	18000.0	23000.0	23000.0	5814.3
					23000.0	26000.0	20000.0	23000.0	2839.9
5 POTW	22227	DIGESTED SLUDGE	SONICATION (S)	DUPONT123	61000.0	50000.0	22000.0	44000.0	20031.4
				FREON	70000.0	36000.0	82000.0	63000.0	23699.5
				HEXANE	2200.0	30000.0	50000.0	28000.0	24101.2
				METHYLENE CHLORIDE	53000.0	66000.0	56000.0	58000.0	7064.5
				PERCHLOR 80/20	100000.0	63000.0	110000.0	91000.0	24297.9
					4100.0	3900.0	12000.0	6800.0	4758.3
	22227	DIGESTED SLUDGE	SOXHLET (S)	DUPONT123	67000.0	190000.0	220000.0	160000.0	80184.4
				FREON	130000.0	97000.0	66000.0	98000.0	33027.6
				HEXANE	54000.0	76000.0	48000.0	59000.0	14515.9
				METHYLENE CHLORIDE	120000.0	54000.0	97000.0	91000.0	34967.3
				PERCHLOR 80/20	160000.0	82000.0	190000.0	140000.0	55096.7
					88000.0	73000.0	67000.0	76000.0	10530.1

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					REP1	REP2	REP3	MEAN	STD. DEV.
6 PETROLEUM REFINERY	22228	API SEPARATOR EFF	LIQ/LIQ (A)	DUPONT123	35.0	31.0	25.0	30.0	5.1
				FREON	24.0	14.0	17.0	18.0	5.3
				HEXANE	32.0	35.0	29.0	32.0	2.9
				METHYLENE CHLORIDE	20.0	11.0	5.0 <	12.0	7.6
				PERCHLOR	12.0	29.0	28.0	23.0	9.5
				80/20	28.0	37.0	23.0	29.0	7.2
	22229	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	9.5	11.0	7.5	9.3	1.7
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	11.0	5.0 <	5.0 <	7.0	3.5
				80/20	5.0 <	14.0	8.0	8.9	4.4
	22229	SECONDARY EFFLUENT	INFRARED (A)	FLON	6.4	6.4		6.4	
	22229	SECONDARY EFFLUENT	SPE COLUMN I (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.6	5.6	5.4	5.5	0.1
				80/20	5.4	5.2	5.8	5.5	0.3
	22230	API SEPARATOR SLUDGE SONICATION (S)		DUPONT123	210000.0	200000.0	220000.0	210000.0	10148.9
				FREON	140000.0	190000.0	230000.0	190000.0	46177.2
				HEXANE	200000.0	220000.0	200000.0	210000.0	9609.0
				METHYLENE CHLORIDE	160000.0	160000.0	140000.0	150000.0	14153.9
				PERCHLOR	180000.0	160000.0	180000.0	170000.0	8888.2
				80/20	190000.0	200000.0	220000.0	200000.0	15534.9
	22230	API SEPARATOR SLUDGE SOXHLET (S)		DUPONT123	240000.0	310000.0	280000.0	280000.0	32187.0
				FREON	320000.0	350000.0	250000.0	310000.0	53257.2
				HEXANE	240000.0	320000.0	240000.0	270000.0	43821.6
				METHYLENE CHLORIDE	290000.0	280000.0	280000.0	280000.0	5033.2
				PERCHLOR	200000.0	280000.0	240000.0	240000.0	41036.6
				80/20	280000.0	250000.0	360000.0	300000.0	54555.8
7 INDUSTRIAL LAUNDRY	22231	DAF EFFLUENT	LIQ/LIQ (A)	DUPONT123	130.0	100.0	80.0	100.0	24.5
				FREON	62.0	74.0	94.0	77.0	16.1
				HEXANE	76.0	30.0	66.0	57.0	24.2
				METHYLENE CHLORIDE	120.0	83.0	97.0	99.0	17.4
				PERCHLOR	88.0	91.0	92.0	90.0	2.3
				80/20	67.0	46.0	39.0	51.0	14.2
	22231	DAF EFFLUENT	INFRARED (A)	FLON	150.0	160.0		150.0	4.9

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					REP1	REP2	REP3	MEAN	STD. DEV.
8 TEXTILE MILL	23463	DAF EFFLUENT	LIQ/LIQ (A)	DUPONT123	620.0	670.0	550.0	610.0	58.7
				FREON	630.0	650.0	590.0	620.0	31.1
				HEXANE	400.0	440.0	370.0	400.0	35.3
				METHYLENE CHLORIDE	440.0	340.0	500.0	430.0	82.8
				PERCHLOR	450.0	550.0	460.0	490.0	58.1
				80/20	510.0	380.0	430.0	440.0	66.6
	23463	DAF EFFLUENT	INFRARED (A)	FLON	590.0	570.0		580.0	18.4
	23463	DAF EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	450.0	430.0		440.0	12.7
				FREON	260.0	250.0		260.0	12.7
				HEXANE	290.0	290.0		290.0	
				MTBE	250.0	400.0		330.0	106.8
				PERC	390.0	490.0		440.0	68.6
	23463	DAF EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	640.0	610.0		630.0	17.7
				FREON	320.0	430.0		380.0	81.3
				HEXANE	280.0	250.0		270.0	21.2
				MTBE	750.0	410.0		580.0	234.8
				PERC	420.0	460.0		440.0	25.5
	23464	DAF SLUDGE	SONICATION (S)	DUPONT123	110000.0	140000.0	150000.0	130000.0	17175.8
				FREON	170000.0	190000.0	240000.0	200000.0	35483.4
				HEXANE	230000.0	200000.0	270000.0	240000.0	35964.3
				METHYLENE CHLORIDE	130000.0	140000.0	94000.0	120000.0	24904.3
				PERCHLOR	100000.0	120000.0	110000.0	110000.0	9558.2
				80/20	170000.0	120000.0	120000.0	140000.0	29936.9
	23464	DAF SLUDGE	SOXHLET (S)	DUPONT123	260000.0	280000.0	280000.0	270000.0	10924.7
				FREON	310000.0	310000.0	240000.0	290000.0	41716.8
				HEXANE	290000.0	360000.0	180000.0	280000.0	90818.8
				METHYLENE CHLORIDE	360000.0	140000.0	220000.0	240000.0	110232.9
				PERCHLOR	180000.0	150000.0	130000.0	150000.0	25636.6
				80/20	280000.0	280000.0	280000.0	280000.0	1035.9
	22232	LAGOON EFFLUENT	LIQ/LIQ (A)	DUPONT123	58.0	18.0	21.0	32.0	22.7
				FREON	34.0	46.0	74.0	51.0	20.3
				HEXANE	6.4	55.0	22.0	28.0	24.9
				METHYLENE CHLORIDE	140.0	160.0	49.0	120.0	61.9
				PERCHLOR	64.0	58.0	31.0	51.0	17.8
				80/20	25.0	14.0	11.0	17.0	7.5
	22232	LAGOON EFFLUENT	INFRARED (A)	FLON	110.0	110.0		110.0	0.7

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mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
9 METAL FINISHING PLANT	22233	SEPARATOR EFFLUENT	LIQ/LIQ (A)	DUPONT123	35.0	66.0	34.0	45.0	18.0
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	25.0	22.0	17.0	22.0	4.0
				METHYLENE CHLORIDE	15.0	5.0 <	13.0	11.0	5.1
				PERCHLOR	15.0	22.0	12.0	16.0	5.3
				80/20	21.0	20.0	21.0	21.0	0.5
	22233	SEPARATOR EFFLUENT	INFRARED (A)	FLON	8.9	9.9		9.4	0.7
	22233	SEPARATOR EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	18.0	28.0		23.0	7.2
				HEXANE	7.1	8.3		7.7	0.8
				MTBE	18.0	25.0		22.0	4.8
10 FISH OIL PLANT	22234	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	660.0	680.0	690.0	680.0	13.2
				FREON	570.0	530.0	500.0	530.0	35.7
				HEXANE	500.0	490.0	500.0	500.0	4.7
				METHYLENE CHLORIDE	750.0	720.0	650.0	700.0	53.1
				PERCHLOR	670.0	470.0	710.0	620.0	130.8
				80/20	580.0	540.0	620.0	580.0	39.3
	22234	PRIMARY EFFLUENT	INFRARED (A)	FLON	290.0	290.0		290.0	2.1
	22234	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	510.0	540.0		520.0	18.4
				HEXANE	460.0	430.0		450.0	21.9
				MTBE	540.0	490.0		520.0	33.9
	22235	OILY SLUDGE	SONICATION (S)	DUPONT123	280000.0	190000.0	230000.0	230000.0	45014.8
				FREON	280000.0	240000.0	210000.0	240000.0	37634.2
				HEXANE	390000.0	210000.0	420000.0	340000.0	114823.1
				METHYLENE CHLORIDE	330000.0	250000.0	340000.0	310000.0	51156.6
				PERCHLOR	880000.0	790000.0	830000.0	830000.0	44575.8
				80/20	230000.0	230000.0	390000.0	280000.0	93275.6
	22235	OILY SLUDGE	SOXHLET (S)	DUPONT123	800000.0	780000.0	770000.0	790000.0	16441.8
				FREON	890000.0	1000000.0	770000.0	890000.0	131249.1
				HEXANE	440000.0	530000.0	460000.0	480000.0	46317.7
				METHYLENE CHLORIDE	780000.0	790000.0	760000.0	780000.0	16258.3
				PERCHLOR	580000.0	350000.0	360000.0	430000.0	126950.1
				80/20	620000.0	590000.0	560000.0	590000.0	26514.1
11 RENDERING PLANT	22236	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	56.0	30.0	27.0	38.0	16.2
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	320.0	240.0	780.0	450.0	292.4
				PERCHLOR	5.1	9.7	62.0	25.0	31.4
				80/20	5.0 <	5.2	25.0	12.0	11.5

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
12 COKE PLANT	22236	PRIMARY EFFLUENT	INFRARED (A)	FLON	37.0	30.0		34.0	4.9
	22236	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	68.0	45.0		56.0	16.8
				HEXANE	7.1	8.3		7.7	0.8
				MTBE	59.0	56.0		58.0	2.5
	22239	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	25.0	5.0 <	5.0 <	12.0	11.5
				80/20	5.0 <	5.0 <	5.0 <	5.0 <	
	22239	SECONDARY EFFLUENT	INFRARED (A)	FLON	4.6	4.5		4.6	0.1
	22240	WASTE ACTIV SLUDGE	SONICATION (S)	DUPONT123	32000.0	26000.0	38000.0	32000.0	6106.9
				FREON	40000.0	30000.0	33000.0	34000.0	5598.0
				HEXANE	24000.0	14000.0	13000.0	17000.0	6280.2
				METHYLENE CHLORIDE	770000.0	1300000.0	50000.0	720000.0	650639.3
13 SLAUGHTERHOUSE				PERCHLOR	30000.0	88000.0	19000.0	46000.0	37224.4
				80/20	14000.0	75000.0	48000.0	46000.0	30839.8
	22240	WASTE ACTIV SLUDGE	SOXHLET (S)	DUPONT123	27000.0	31000.0	23000.0	27000.0	3899.9
				FREON	8300.0	8000.0	18000.0	11000.0	5505.4
				HEXANE	14000.0	19000.0	15000.0	16000.0	2732.0
				METHYLENE CHLORIDE	25000.0	29000.0	27000.0	27000.0	2141.0
				PERCHLOR	69000.0	120000.0	45000.0	78000.0	39027.1
				80/20	9800.0	15000.0	11000.0	12000.0	2738.9
	22241	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	6.8	5.0 <	5.0 <	5.6	1.1
				FREON	5.0 <	5.0 <	9.5	6.5	2.6
				HEXANE	6.4	5.5	15.0	9.0	5.3
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	7.3	15.0	5.0 <	9.2	5.4
				80/20	6.2	5.0 <	10.0	7.1	2.7
	22241	PRIMARY EFFLUENT	INFRARED (A)	FLON	46.0	45.0		46.0	0.7
	22242	SECONDARY EFFLUENT	SPE COLUMN I (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	5.0 <	5.0 <	5.2	5.1	0.1
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	9.9	10.0	10.0	10.0	0.3
				80/20	5.0 <	5.0 <	5.0 <	5.0 <	

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
14 WOOD PRESERVING PLANT	22243	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	7.0	7.4	5.0 <	6.5	1.3
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	8.8	5.0 <	5.0 <	6.3	2.2
				PERCHLOR 80/20	17.0	5.0 <	74.0	32.0	37.0
					9.9	11.0	5.0 <	8.7	3.3
	22243	SECONDARY EFFLUENT	INFRARED (A)	FLON	7.1	6.1		6.6	0.7
	22244	SOLID WASTE	SONICATION (S)	DUPONT123	87000.0	95000.0	84000.0	89000.0	5657.3
				FREON	81000.0	85000.0	83000.0	83000.0	1895.3
				HEXANE	45000.0	52000.0	59000.0	52000.0	6909.6
				METHYLENE CHLORIDE	120000.0	110000.0	110000.0	110000.0	3511.9
				PERCHLOR 80/20	120000.0	99000.0	180000.0	130000.0	42922.4
					50000.0	54000.0	61000.0	55000.0	5176.8
	22244	SOLID WASTE	SOXHLET (S)	DUPONT123	180000.0	140000.0	140000.0	150000.0	19078.8
				FREON	150000.0	140000.0	140000.0	140000.0	3511.9
				HEXANE	140000.0	130000.0	130000.0	130000.0	6557.4
				METHYLENE CHLORIDE	140000.0	160000.0	160000.0	150000.0	15373.1
				PERCHLOR 80/20	60000.0	41000.0	170000.0	89000.0	68169.0
					120000.0	140000.0	130000.0	130000.0	8888.2
15 DRILLING FLUID SUPPLIER	22237	USED DRILLING MUD	SONICATION (S)	DUPONT123	2900.0	2900.0	2900.0	2900.0	18.5
				FREON	1600.0	2200.0	1800.0	1800.0	315.3
				HEXANE	1600.0	1700.0	1500.0	1600.0	112.6
				METHYLENE CHLORIDE	4600.0	4400.0	4200.0	4400.0	191.2
				PERCHLOR 80/20	1200.0	910.0	1100.0	1100.0	143.5
					2800.0	2900.0	3500.0	3100.0	374.2
	22237	USED DRILLING MUD	SOXHLET (S)	DUPONT123	410.0	540.0	400.0	450.0	80.3
				FREON	1300.0	1600.0	1300.0	1400.0	157.0
				HEXANE	1300.0	1200.0	1600.0	1400.0	200.6
				METHYLENE CHLORIDE	800.0	950.0	1100.0	930.0	124.9
				PERCHLOR 80/20	1100.0	1200.0	1500.0	1300.0	216.6
					2000.0	1900.0	2000.0	2000.0	72.8
16 CONTAMINATED SOILS	24020	KEROSENE CONTAM SOIL	SONICATION (S)	DUPONT123	2300.0	2100.0	1900.0	2100.0	229.8
				FREON	1000.0	1600.0	1400.0	1300.0	309.3
				HEXANE	1900.0	2200.0	1700.0	1900.0	226.8
				METHYLENE CHLORIDE	3900.0	2500.0	3100.0	3100.0	689.6
				PERCHLOR 80/20	2000.0	1200.0	1600.0	1600.0	402.7
					2400.0	1400.0	1500.0	1700.0	533.1

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FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
					REP1	REP2	REP3	MEAN	STD. DEV.
17 POULTRY PLANT	24020	KEROSENE CONTAM SOIL	SOXHLET (S)	DUPONT123	1900.0	2100.0	2200.0	2100.0	148.8
				FREON	2000.0	1400.0	1900.0	1700.0	352.3
				HEXANE	2500.0	3200.0	2600.0	2800.0	409.8
				METHYLENE CHLORIDE	1200.0	1300.0	1600.0	1300.0	221.0
				PERCHLOR	1900.0	1600.0	2100.0	1900.0	232.7
				80/20	2000.0	1400.0	2000.0	1800.0	339.8
	23106	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	2.3
				FREON	8.3	12.0	8.1	9.5	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	16.2
				PERCHLOR	5.0 <	37.0	19.0	20.0	
				80/20	5.0 <	5.0 <	5.0 <	5.0 <	
	23106	SECONDARY EFFLUENT	INFRARED (A)	FLON	0.0	0.0		0.0	
	23106	SECONDARY EFFLUENT	SPE COLUMN I (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	3.9
				FREON	5.0 <	5.0 <	5.0 <	5.0 <	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	12.0	7.0	8.2	6.9
				PERCHLOR	5.0 <	5.0 <	5.0 <	5.0 <	
				80/20	17.0	5.0 <	5.0 <	9.0	
	23107	WASTE ACTIV SLUDGE	SONICATION (S)	DUPONT123	4100.0	7100.0	7500.0	6200.0	1841.6
				FREON	10000.0	19000.0	16000.0	15000.0	4438.1
				HEXANE	2300.0	1800.0	130.0	1400.0	1112.1
				METHYLENE CHLORIDE	6800.0	1200.0	8000.0	5300.0	3656.0
				PERCHLOR	130000.0	12000.0	10000.0	51000.0	69556.7
				80/20	2000.0	2100.0		2100.0	85.6
	23107	WASTE ACTIV SLUDGE	SOXHLET (S)	DUPONT123	21000.0	22000.0	13000.0	19000.0	5098.0
				FREON	38000.0	11000.0	40000.0	30000.0	16262.8
				HEXANE	5900.0	11000.0	46000.0	21000.0	21795.3
				METHYLENE CHLORIDE	39000.0	52000.0	9800.0	34000.0	21708.6
				PERCHLOR	47000.0	53000.0	19000.0	40000.0	18069.0
				80/20	10000.0	7300.0	23000.0	13000.0	8455.2
18 ROLLING MILL	23108	FILTER EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	12.2
				FREON	5.0 <	26.0	5.0 <	12.0	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	6.2
				PERCHLOR	5.0 <	17.0	13.0	12.0	
				80/20	5.0 <	5.0 <	5.0 <	5.0 <	

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

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Oil & Grease Concentrations:
mg/L in aqueous samples (A)
mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
19 OIL TERMINAL	23108	FILTER EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	5.0 <	5.0 <		5.0 <	
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	5.0 <	5.0 <		5.0 <	
				PERC	5.0 <	5.0 <		5.0 <	
	23108	FILTER EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	5.0 <	5.0 <		5.0 <	
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	5.0 <	5.0 <		5.0 <	
				PERC	8.6	7.0		7.8	1.1
	23109	DEWATERED SCALE MAT	SONICATION (S)	DUPONT123	12000.0	12000.0	13000.0	13000.0	762.7
				FREON	21000.0	16000.0	18000.0	19000.0	2600.6
				HEXANE	16000.0	17000.0	17000.0	17000.0	762.5
				METHYLENE CHLORIDE	17000.0	15000.0	14000.0	15000.0	1437.9
				PERCHLOR	18000.0	18000.0	24000.0	20000.0	3757.9
				80/20	15000.0	16000.0	15000.0	16000.0	229.1
	23109	DEWATERED SCALE MAT	SOXHLET (S)	DUPONT123	14000.0	18000.0	18000.0	16000.0	2178.0
				FREON	11000.0	14000.0	17000.0	14000.0	2884.3
				HEXANE	14000.0	14000.0	16000.0	15000.0	983.4
				METHYLENE CHLORIDE	17000.0	15000.0	18000.0	17000.0	1523.8
				PERCHLOR	18000.0	17000.0	22000.0	19000.0	2861.4
				80/20	18000.0	19000.0	16000.0	18000.0	1706.1
	23110	SEPARATOR EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	5.0 <	5.0 <	11.0	6.9	3.2
				HEXANE	5.1	5.1	5.8	5.3	0.4
				METHYLENE CHLORIDE	15.0	8.1	14.0	12.0	3.7
				PERCHLOR	20.0	21.0	5.0 <	15.0	8.9
				80/20	5.0 <	5.0 <	18.0	9.4	7.7
	23110	SEPARATOR EFFLUENT	INFRARED (A)	FLON	1.0	1.0		1.0	
	23110	SEPARATOR EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	8.0	6.6		7.3	1.0
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	15.0	14.0		15.0	0.6
				PERC	7.8	5.0 <		6.4	2.0
	23110	SEPARATOR EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	7.3	11.0		9.3	2.8
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	11.0	12.0		12.0	0.4
				PERC	5.0 <	13.0		8.9	5.4

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)					
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.	
20	MAYONNAISE PLANT	23111	FILTER EFFLUENT	LIQ/LIQ (A)	DUPONT123	17.0	5.0 <	20.0	14.0	7.9
					FREON	5.0 <	5.0 <	5.0 <	5.0 <	
					HEXANE	5.5	5.0 <	100.0	38.0	56.1
					METHYLENE CHLORIDE	21.0	14.0	8.3	15.0	6.6
					PERCHLOR 80/20	16.0	18.0	16.0	16.0	1.1
						5.0 <	8.7	5.0 <	6.2	2.1
		23111	FILTER EFFLUENT	INFRARED (A)	FLON	10.0	10.0		10.0	0.1
		23111	FILTER EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	28.0	33.0		31.0	4.0
					FREON	5.0 <	5.0 <		5.0 <	
					HEXANE	5.0 <	5.0 <		5.0 <	
					MTBE	35.0	35.0		35.0	0.1
					PERC	12.0	12.0		12.0	0.1
		23111	FILTER EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	26.0	27.0		26.0	0.8
					FREON	5.0 <	5.6		5.3	0.4
					HEXANE	5.0 <	5.0 <		5.0 <	
					MTBE	27.0	30.0		28.0	2.1
					PERC	5.0 <	6.0		5.5	0.7
		23112	OILY SLUDGE	SONICATION (S)	DUPONT123	650000.0	640000.0	740000.0	680000.0	57133.5
					FREON	670000.0	760000.0	160000.0	530000.0	319216.2
					HEXANE	480000.0	470000.0	360000.0	440000.0	68563.8
					METHYLENE CHLORIDE	560000.0	510000.0	510000.0	520000.0	29194.7
					PERCHLOR 80/20	610000.0	580000.0	460000.0	550000.0	80727.9
		23112	OILY SLUDGE	SOXHLET (S)	DUPONT123	910000.0	910000.0	940000.0	920000.0	18770.5
					FREON	880000.0	850000.0	780000.0	840000.0	50520.6
					HEXANE	590000.0	780000.0	520000.0	630000.0	132020.2
					METHYLENE CHLORIDE	890000.0	900000.0	870000.0	890000.0	15044.4
					PERCHLOR 80/20	840000.0	660000.0	670000.0	720000.0	99926.6
21	SEAFOOD PLANT	23113	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	6.9	5.0 <	5.0 <	5.6	1.1
					FREON	5.0 <	5.0 <	47.0	19.0	24.4
					HEXANE	18.0	5.0 <	27.0	17.0	11.1
					METHYLENE CHLORIDE	13.0	11.0	28.0	17.0	9.4
					PERCHLOR 80/20	14.0	23.0	13.0	17.0	5.3
						5.0 <	5.0 <	5.0 <	5.0 <	
		23113	SECONDARY EFFLUENT	INFRARED (A)	FLON	2.0	2.0		2.0	

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FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
					REP1	REP2	REP3	MEAN	STD. DEV.
22 ABRASIVES PLANT	23113	SECONDARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	6.7	6.6		6.7	0.1
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	9.3	9.8		9.6	0.4
				PERC	5.0 <	5.0 <		5.0 <	
	23113	SECONDARY EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	6.8	8.6		7.7	1.3
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	7.8	11.0		9.5	2.4
				PERC	6.4	5.0 <		5.7	1.0
	23114	WASTE SLUDGE	SONICATION (S)	DUPONT123	77000.0	5600.0	74000.0	52000.0	40342.5
				FREON	74000.0	14000.0	28000.0	39000.0	31219.4
				HEXANE	0.0	21000.0	36000.0	19000.0	17989.7
				METHYLENE CHLORIDE	59000.0	68000.0	62000.0	63000.0	4535.1
				PERCHLOR	57000.0	83000.0	62000.0	67000.0	14065.4
				80/20	46000.0	32000.0	48000.0	42000.0	8923.1
	23114	WASTE SLUDGE	SOXHLET (S)	DUPONT123	350000.0	170000.0	180000.0	230000.0	100583.3
				FREON	64000.0		53000.0	58000.0	7525.9
				HEXANE	34000.0	31000.0	27000.0	31000.0	3867.0
				METHYLENE CHLORIDE	7900.0	64000.0	39000.0	37000.0	28005.2
				PERCHLOR	35000.0	36000.0	12000.0	28000.0	13882.9
				80/20	14000.0	28000.0	94000.0	45000.0	42738.5
	23115	PROCESS WASTEWATER	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	
				FREON	9.2	27.0	5.0 <	14.0	11.6
				METHYLENE CHLORIDE	14.0	21.0	5.0 <	13.0	8.0
				PERCHLOR	5.0 <	5.0 <	5.0 <	5.0 <	
				80/20	5.0 <	5.0 <	5.7	5.2	0.4
	23115	PROCESS WASTEWATER	SPE DISK 90mm (A)	METHYLENE CHLORIDE	5.0 <	5.0 <		5.0 <	
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	5.0 <	5.0 <		5.0 <	
				PERC	5.0 <	5.0 <		5.0 <	
	23115	PROCESS WASTEWATER	SPE DISK 47mm (A)	METHYLENE CHLORIDE	5.0 <	5.0 <		5.0 <	
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	5.0 <	5.9		5.5	0.6
				PERC	5.0 <	5.0 <		5.0 <	

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
23 OIL TERMINAL	23120	SEPARATOR EFFLUENT	LIQ/LIQ (A)	DUPONT123	7.0	6.0	9.0	7.3	1.5
				FREON	5.0 <	5.0 <	6.0	5.3	0.6
				HEXANE	6.0	5.0 <	5.0 <	5.3	0.6
				METHYLENE CHLORIDE	12.0	13.0	9.0	11.0	2.1
				PERCHLOR 80/20	8.0	14.0	5.0 <	9.0	4.6
	23120	SEPARATOR EFFLUENT	INFRARED (A)	FLON	5.0 <	5.0 <	5.0 <	5.0 <	
					3.2	3.3		3.3	0.1
	23120	SEPARATOR EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	11.0	9.2		10.0	1.3
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				MTBE	17.0	17.0		17.0	0.2
				PERC	5.7	5.5		5.6	0.1
	23120	SEPARATOR EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	19.0	17.0		18.0	1.4
				FREON	7.3	9.2		8.3	1.3
				HEXANE	5.0 <	5.5		5.3	0.4
				MTBE	23.0	23.0		23.0	0.2
				PERC	13.0	12.0		13.0	0.4
	23120	SEPARATOR EFFLUENT	SPE COLUMN II (A)	FREON	5.0 <	7.0		6.0	1.4
				HEXANE	8.0	8.0		8.0	
				80/20	7.0	5.0 <		6.0	1.4
24 SEAFOOD PLANT	23121	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	16.0	17.0	13.0	15.0	2.2
				FREON	14.0	14.0	13.0	14.0	0.6
				HEXANE	21.0	20.0	17.0	19.0	1.9
				METHYLENE CHLORIDE	37.0	29.0	38.0	35.0	4.9
				PERCHLOR 80/20	28.0	24.0	19.0	24.0	4.7
	23121	PRIMARY EFFLUENT	INFRARED (A)	FLON	11.0	11.0	11.0	11.0	0.2
					40.0	33.0		36.0	4.7
	23121	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	44.0	43.0		44.0	0.7
				FREON	11.0	10.0		11.0	0.5
				HEXANE	17.0	23.0		20.0	4.0
				MTBE	57.0	56.0		56.0	1.1
				PERC	20.0	21.0		20.0	0.4
	23121	PRIMARY EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	60.0	58.0		59.0	1.8
				FREON	11.0	13.0		12.0	1.3
				HEXANE	18.0	19.0		18.0	0.9
				MTBE	63.0	65.0		64.0	1.3
				PERC	24.0	25.0		24.0	0.4

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
25 POULTRY PLANT	23121	PRIMARY EFFLUENT	SPE COLUMN II (A)	FREON	13.0	11.0		12.0	1.4
				HEXANE	7.0	8.0		7.5	0.7
				80/20	13.0	7.0		10.0	4.2
	23122	OILY SLUDGE	SONICATION (S)	DUPONT123	490000.0	420000.0	410000.0	440000.0	42673.2
				FREON	340000.0	310000.0	300000.0	320000.0	20108.0
				HEXANE	300000.0	330000.0	300000.0	310000.0	17616.3
				METHYLENE CHLORIDE	420000.0	400000.0	410000.0	410000.0	6806.9
				PERCHLOR	470000.0	530000.0	410000.0	470000.0	63500.7
				80/20	320000.0	340000.0	330000.0	330000.0	8621.7
	23122	OILY SLUDGE	SOXHLET (S)	DUPONT123	360000.0	400000.0	320000.0	360000.0	37527.8
				FREON	400000.0	410000.0	430000.0	410000.0	16370.7
				HEXANE	400000.0	390000.0	390000.0	400000.0	7094.6
				METHYLENE CHLORIDE	520000.0	570000.0	620000.0	570000.0	48507.7
				PERCHLOR	400000.0	390000.0	300000.0	370000.0	57873.4
				80/20	440000.0	420000.0	420000.0	430000.0	10116.0
	23123	DEWATERED SLUDGE	SOXHLET (S)	DUPONT123	24000.0	27000.0	23000.0	25000.0	2022.4
				FREON	31000.0	27000.0	31000.0	29000.0	2285.5
				HEXANE	220000.0	150000.0	200000.0	190000.0	36295.1
				METHYLENE CHLORIDE	220000.0	260000.0	210000.0	230000.0	25534.3
				PERCHLOR	380000.0	280000.0	440000.0	370000.0	79567.6
				80/20	250000.0	220000.0	210000.0	230000.0	20792.6
	23457	DAF INFLUENT	LIQ/LIQ (A)	DUPONT123	340.0	340.0	330.0	330.0	7.6
				FREON	730.0	630.0	650.0	670.0	54.1
				HEXANE	530.0	600.0	680.0	610.0	74.7
				METHYLENE CHLORIDE	420.0	400.0	430.0	420.0	12.8
				PERCHLOR	820.0	990.0	870.0	890.0	90.7
				80/20	400.0	440.0	290.0	370.0	79.0
	23457	DAF INFLUENT	INFRARED (A)	FLON	24.0	27.0		25.0	2.2
	23457	DAF INFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	1700.0	1700.0		1700.0	0.7
				FREON	700.0	840.0		770.0	99.7
				HEXANE	700.0	770.0		740.0	48.1
				MTBE	850.0	830.0		840.0	14.1
				PERC	870.0	950.0		910.0	57.3
	23457	DAF INFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	1000.0	970.0		980.0	24.0
				FREON	900.0	910.0		900.0	3.5
				HEXANE	890.0	850.0		870.0	26.9
				MTBE	830.0	1000.0		920.0	125.2
				PERC	1000.0	910.0		960.0	72.8

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Oil & Grease Concentrations:
mg/L in aqueous samples (A)
mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
26 MEAT PACKING PLANT	23458	DAF SLUDGE	SONICATION (S)	DUPONT123	170000.0	210000.0	240000.0	200000.0	36721.9
				FREON	190000.0	160000.0	190000.0	180000.0	14298.1
				HEXANE	130000.0	85000.0	92000.0	100000.0	22482.0
				METHYLENE CHLORIDE	190000.0	150000.0	170000.0	170000.0	21072.8
				PERCHLOR	320000.0	390000.0	330000.0	350000.0	39362.0
				80/20	140000.0	140000.0	70000.0	120000.0	40578.7
	23458	DAF SLUDGE	SOXHLET (S)	DUPONT123	590000.0	570000.0	580000.0	580000.0	7019.6
				FREON	670000.0	600000.0	570000.0	610000.0	49549.3
				HEXANE	530000.0	530000.0	530000.0	530000.0	2449.0
				METHYLENE CHLORIDE	580000.0	540000.0	530000.0	550000.0	22853.1
				PERCHLOR	690000.0	450000.0	530000.0	560000.0	120291.7
				80/20	610000.0	430000.0	550000.0	530000.0	88521.9
	23124	LAGOON EFFLUENT	LIQ/LIQ (A)	DUPONT123	12.0	16.0	17.0	15.0	2.6
				FREON	11.0	13.0	14.0	13.0	1.4
				HEXANE	15.0	12.0	15.0	14.0	1.8
				METHYLENE CHLORIDE	39.0	43.0	39.0	40.0	2.0
				PERCHLOR	51.0	52.0	58.0	54.0	3.9
				80/20	110.0	25.0	26.0	55.0	50.7
	23124	LAGOON EFFLUENT	INFRARED (A)	FLON	87.0	88.0		87.0	0.8
	23124	LAGOON EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	17.0	11.0		14.0	4.2
				FREON	11.0	11.0		11.0	
				HEXANE	13.0	13.0		13.0	0.3
				MTBE	21.0	22.0		22.0	0.8
				PERC	9.3	10.0		9.8	0.7
	23124	LAGOON EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	39.0	38.0		38.0	0.9
				FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	28.0	26.0		27.0	1.5
				MTBE	61.0	66.0		63.0	3.7
				PERC	13.0	13.0		13.0	0.5
	23124	LAGOON EFFLUENT	SPE COLUMN II (A)	FREON	7.0	5.0 <		6.0	1.4
				HEXANE	20.0	13.0		17.0	4.9
				80/20	14.0	17.0		16.0	2.1
27 RAILROAD YARD	23459	DAF EFFLUENT	LIQ/LIQ (A)	DUPONT123	34.0	30.0	69.0	44.0	21.2
				FREON	170.0	120.0	190.0	160.0	36.1
				HEXANE	91.0	110.0	81.0	93.0	13.3
				METHYLENE CHLORIDE	54.0	62.0	42.0	53.0	10.1
				PERCHLOR	170.0	240.0	250.0	220.0	43.3
				80/20	140.0	150.0	150.0	140.0	6.7

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
	23459	DAF EFFLUENT	INFRARED (A)	FLON	400.0	380.0		390.0	12.7
	23459	DAF EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	80.0	96.0		88.0	11.5
				FREON	83.0	48.0		66.0	24.7
				HEXANE	57.0	39.0		48.0	13.1
				MTBE	150.0	160.0		160.0	8.5
				PERC	53.0	58.0		56.0	3.6
	23459	DAF EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	330.0	360.0		340.0	17.0
				FREON	66.0	88.0		77.0	15.1
				HEXANE	210.0	230.0		220.0	15.6
				MTBE	430.0	440.0		440.0	6.4
				PERC	91.0	95.0		93.0	2.5
	23460	OILY SLUDGE	SONICATION (S)	DUPONT123	290000.0	480000.0	520000.0	430000.0	123939.1
				FREON	380000.0	380000.0	450000.0	400000.0	37435.9
				HEXANE	250000.0	110000.0	200000.0	190000.0	68359.5
				METHYLENE CHLORIDE	350000.0	320000.0	190000.0	290000.0	83973.2
				PERCHLOR	400000.0	410000.0	410000.0	410000.0	3281.3
				80/20	160000.0	310000.0	310000.0	260000.0	85200.8
	23460	OILY SLUDGE	SOXHLET (S)	DUPONT123	790000.0	790000.0	670000.0	750000.0	68154.5
				FREON	870000.0	920000.0	870000.0	890000.0	27906.0
				HEXANE	850000.0	840000.0	830000.0	840000.0	6884.3
				METHYLENE CHLORIDE	800000.0	800000.0	800000.0	800000.0	3722.0
				PERCHLOR	1000000.0	1000000.0	930000.0	990000.0	52451.3
				80/20	860000.0	860000.0	750000.0	820000.0	60549.2
28 CAN MANUFACT PLANT	23461	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	400.0	390.0	350.0	380.0	23.4
				FREON	220.0	220.0	220.0	220.0	2.9
				HEXANE	180.0	180.0	170.0	180.0	5.5
				METHYLENE CHLORIDE	340.0	360.0	370.0	360.0	13.4
				PERCHLOR	400.0	390.0	390.0	390.0	3.2
				80/20	190.0	190.0	190.0	190.0	4.1
	23461	PRIMARY EFFLUENT	INFRARED (A)	FLON	230.0	230.0		230.0	1.4
	23461	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	390.0	360.0		380.0	19.1
				FREON	370.0	370.0		370.0	0.7
				HEXANE	250.0	300.0		270.0	37.5
				MTBE	420.0	430.0		420.0	7.8
				PERC	360.0	360.0		360.0	2.1

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					Oil & Grease Concentrations:				
					mg/L in aqueous samples (A)				
					mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
29 SOUP PLANT	23461	PRIMARY EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	500.0	450.0		470.0	37.5
				FREON	410.0	360.0		380.0	29.7
				HEXANE	370.0	370.0		370.0	3.5
				MTBE	510.0	500.0		500.0	3.5
				PERC	450.0	470.0		460.0	14.1
	23461	PRIMARY EFFLUENT	SPE COLUMN II (A)	FREON	290.0	270.0		280.0	14.1
				HEXANE	220.0	190.0		200.0	16.3
				80/20	320.0	340.0		330.0	15.6
	23462	FILTER CAKE	SONICATION (S)	DUPONT123	43000.0	25000.0	42000.0	37000.0	10167.0
				FREON	42000.0	38000.0	34000.0	38000.0	4175.8
				HEXANE	31000.0	36000.0	38000.0	35000.0	3749.6
				METHYLENE CHLORIDE	51000.0	48000.0	46000.0	48000.0	2486.4
				PERCHLOR	61000.0	72000.0	44000.0	59000.0	14050.6
				80/20	27000.0	30000.0	39000.0	32000.0	6014.5
	23462	FILTER CAKE	SOXHLET (S)	DUPONT123	40000.0	61000.0	60000.0	53000.0	11630.8
				FREON	62000.0	62000.0	60000.0	61000.0	976.2
				HEXANE	69000.0	64000.0	66000.0	66000.0	2615.1
				METHYLENE CHLORIDE	74000.0	72000.0	74000.0	73000.0	927.7
				PERCHLOR	71000.0	85000.0	68000.0	75000.0	9239.2
				80/20	67000.0	65000.0	69000.0	67000.0	1791.8
	23466	DAF EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	6.7	5.0 <	5.6	1.0
				FREON	5.4	5.0 <	5.0 <	5.1	0.2
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	5.1	5.0 <	5.0 <	5.0 <	0.1
				80/20	5.0 <	5.0 <	5.0 <	5.0 <	
	23466	DAF EFFLUENT	INFRARED (A)	FLON	3.2	2.9		3.1	0.2
	23466	DAF EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	9.8	9.0		9.4	0.6
				FREON	6.0	7.3		6.7	0.9
				HEXANE	7.8	7.1		7.5	0.5
				MTBE	11.0	10.0		10.0	0.6
				PERC	6.4	6.9		6.7	0.4
	23466	DAF EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	17.0	21.0		19.0	2.5
				FREON	6.9	8.9		7.9	1.4
				HEXANE	6.8	9.4		8.1	1.8
				MTRE	23.0	15.0		19.0	5.8
				PERC	13.0	17.0		15.0	2.9

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
30 OILY WATER TREAT PLANT	23466	DAF EFFLUENT	SPE COLUMN II (A)	FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	11.0	5.0 <		8.0	4.2
				80/20	6.0	7.0		6.5	0.7
	23467	DAF SLUDGE	SONICATION (S)	DUPONT123	580000.0	650000.0	620000.0	610000.0	34703.5
				FREON	580000.0	630000.0	650000.0	620000.0	33709.5
				HEXANE	560000.0	550000.0	540000.0	550000.0	13576.9
				METHYLENE CHLORIDE	650000.0	630000.0	620000.0	630000.0	16258.3
				PERCHLOR	550000.0	620000.0	590000.0	580000.0	36115.6
				80/20	580000.0	580000.0	590000.0	590000.0	6245.0
	23467	DAF SLUDGE	SOXHLET (S)	DUPONT123	620000.0	610000.0	600000.0	610000.0	8504.9
				FREON	600000.0	590000.0	610000.0	600000.0	10066.4
				HEXANE	580000.0	520000.0	600000.0	570000.0	40360.9
				METHYLENE CHLORIDE	650000.0	630000.0	720000.0	660000.0	47318.1
				PERCHLOR	700000.0	740000.0	740000.0	730000.0	24433.6
				80/20	640000.0	670000.0	650000.0	650000.0	15099.7
	23468	SEPARATOR EFFLUENT	LIQ/LIQ (A)	DUPONT123	23.0	26.0	25.0	24.0	1.9
				FREON	29.0	27.0	28.0	28.0	1.0
				HEXANE	16.0	21.0	29.0	22.0	6.2
				METHYLENE CHLORIDE	22.0	24.0	21.0	22.0	1.6
				PERCHLOR	16.0	32.0	13.0	20.0	10.0
				80/20	11.0	15.0	16.0	14.0	2.8
	23468	SEPARATOR EFFLUENT	INFRARED (A)	FLON	24.0	27.0		25.0	2.2
	23468	SEPARATOR EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	17.0	14.0		16.0	1.8
				FREON	9.0	9.1		9.1	0.1
				HEXANE	11.0	8.3		9.4	1.6
				MTBE	30.0	22.0		26.0	5.7
				PERC	13.0	8.3		11.0	3.5
	23468	SEPARATOR EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	39.0	44.0		42.0	3.9
				FREON	34.0	36.0		35.0	1.7
				HEXANE	38.0	42.0		40.0	2.8
				MTBE	18.0	15.0		16.0	1.9
				PERC	46.0	40.0		43.0	4.2
	23468	SEPARATOR EFFLUENT	SPE COLUMN II (A)	FREON	9.0	9.0		9.0	
				HEXANE	23.0	29.0		26.0	4.2
				80/20	14.0	16.0		15.0	1.4

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Oil & Grease Concentrations:
mg/L in aqueous samples (A)
mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
31 CAN MANUFACT PLANT	23469	OILY SLUDGE	SONICATION (S)	DUPONT123	43000.0	58000.0	51000.0	51000.0	7650.6
				FREON	60000.0	52000.0	43000.0	51000.0	8431.0
				HEXANE	16000.0	32000.0	37000.0	28000.0	11093.8
				METHYLENE CHLORIDE	43000.0	63000.0	60000.0	55000.0	10772.3
				PERCHLOR	52000.0	62000.0	23000.0	46000.0	20211.1
				80/20	65000.0	46000.0	58000.0	56000.0	9559.5
	23469	OILY SLUDGE	SOXHLET (S)	DUPONT123	91000.0	99000.0	110000.0	100000.0	10159.8
				FREON	76000.0	75000.0	70000.0	74000.0	3214.6
				HEXANE	77000.0	60000.0	79000.0	72000.0	10712.9
				METHYLENE CHLORIDE	77000.0	79000.0	76000.0	77000.0	1414.8
				PERCHLOR	85000.0	91000.0	100000.0	92000.0	7886.5
				80/20	82000.0	86000.0	84000.0	84000.0	2099.4
	23470	DAF INFLUENT	LIQ/LIQ (A)	DUPONT123	350.0	360.0	400.0	370.0	26.7
				FREON	360.0	340.0	350.0	350.0	8.9
				HEXANE	320.0	410.0	470.0	400.0	75.5
				METHYLENE CHLORIDE	370.0	390.0	350.0	370.0	19.9
				PERCHLOR	380.0	390.0	420.0	390.0	19.2
				80/20	370.0	360.0	320.0	350.0	29.8
	23470	DAF INFLUENT	INFRARED (A)	FLON	290.0	280.0		290.0	7.8
	23470	DAF INFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	450.0	410.0		430.0	21.9
				FREON	410.0	400.0		410.0	8.5
				HEXANE	370.0	350.0		360.0	12.7
				MTBE	440.0	440.0		440.0	4.9
				PERC	430.0	410.0		420.0	12.0
	23470	DAF INFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	480.0	450.0		470.0	18.4
				FREON	300.0	420.0		360.0	81.3
				HEXANE	410.0	410.0		410.0	0.7
				MTBE	510.0	460.0		480.0	36.1
				PERC	460.0	470.0		460.0	6.4
	23471	OILY SLUDGE	SONICATION (S)	DUPONT123	64000.0	61000.0	65000.0	63000.0	2086.2
				FREON	59000.0	69000.0	74000.0	67000.0	7407.7
				HEXANE	67000.0	45000.0	46000.0	53000.0	12097.0
				METHYLENE CHLORIDE	43000.0	23000.0	35000.0	34000.0	10135.4
				PERCHLOR	62000.0	64000.0	70000.0	65000.0	4477.1
				80/20	70000.0	51000.0	67000.0	62000.0	10181.4

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Oil & Grease Concentrations:
mg/L in aqueous samples (A)
mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
32 CAN MANUFACT PLANT	23471	OILY SLUDGE	SOXHLET (S)	DUPONT123	92000.0	95000.0	89000.0	92000.0	2998.2
				FREON	94000.0	88000.0	94000.0	92000.0	3291.2
				HEXANE	80000.0	90000.0	83000.0	85000.0	4991.8
				METHYLENE CHLORIDE	96000.0	110000.0	92000.0	98000.0	7734.3
				PERCHLOR	120000.0	120000.0	100000.0	120000.0	12649.4
				80/20	110000.0	110000.0	100000.0	110000.0	1719.8
	23472	DAF EFFLUENT	LIQ/LIQ (A)	DUPONT123	34.0	33.0	35.0	34.0	0.9
				FREON	9.0	16.0	16.0	14.0	3.9
				HEXANE	5.0 <	5.0 <	5.5	5.2	0.3
				METHYLENE CHLORIDE	35.0	34.0	34.0	34.0	0.7
				PERCHLOR	19.0	27.0	24.0	23.0	4.2
				80/20	6.2	6.7	7.5	6.8	0.7
		DAF EFFLUENT	INFRARED (A)	FLON	8.3	8.8		8.6	0.4
		DAF EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	65.0	68.0		66.0	1.8
				FREON	9.2	12.0		11.0	2.2
				HEXANE	8.1	6.0		7.1	1.5
				MTBE	58.0	63.0		60.0	4.0
				PERC	22.0	21.0		21.0	0.4
	23472	DAF EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	110.0	100.0		110.0	4.2
				FREON	35.0	34.0		35.0	1.0
				HEXANE	38.0	43.0		41.0	3.5
				MTBE	65.0	71.0		68.0	3.9
				PERC	51.0	58.0		55.0	4.6
	23472	DAF EFFLUENT	SPE COLUMN II (A)	FREON	7.0	9.0		8.0	1.4
				HEXANE	5.0 <	7.0		6.0	1.4
				80/20	30.0	27.0		29.0	2.1
	23473	DAF INFLUENT	LIQ/LIQ (A)	DUPONT123	480.0	440.0	450.0	460.0	19.3
				FREON	430.0	410.0	360.0	400.0	33.8
				HEXANE	310.0	340.0	350.0	330.0	21.5
				METHYLENE CHLORIDE	420.0	440.0	400.0	420.0	20.6
				PERCHLOR	570.0	560.0	460.0	530.0	59.0
				80/20	360.0	340.0	350.0	350.0	8.1
	23473	DAF INFLUENT	INFRARED (A)	FLON	150.0	140.0		150.0	5.7

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					Oil & Grease Concentrations:				
					mg/L in aqueous samples (A)				
					mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
33 DRUM HANDLING FACILITY	23473	DAF INFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	470.0	460.0		470.0	8.5
				FREON	420.0	410.0		420.0	7.8
				HEXANE	430.0	410.0		420.0	11.3
				MTBE	470.0	500.0		480.0	21.9
				PERC	520.0	520.0		520.0	4.2
	23473	DAF INFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	630.0	520.0		580.0	80.6
				FREON	490.0	480.0		480.0	10.6
				HEXANE	510.0	500.0		510.0	2.1
				MTBE	510.0	490.0		500.0	16.3
				PERC	560.0	560.0		560.0	1.4
	23473	DAF INFLUENT	SPE COLUMN II (A)	FREON	470.0	250.0		360.0	157.7
				HEXANE	470.0	460.0		470.0	7.1
				80/20	500.0	450.0		480.0	31.8
	23474	FILTER CAKE	SONICATION (S)	DUPONT123	96000.0	120000.0	81000.0	100000.0	21559.9
				FREON	120000.0	110000.0	120000.0	120000.0	7177.7
				HEXANE	190000.0	200000.0	180000.0	190000.0	10923.2
				METHYLENE CHLORIDE	86000.0	130000.0	110000.0	110000.0	20643.2
				PERCHLOR	200000.0	190000.0	230000.0	210000.0	18781.7
				80/20	180000.0	190000.0	180000.0	180000.0	6847.2
	23474	FILTER CAKE	SOXHLET (S)	DUPONT123	310000.0	310000.0	310000.0	310000.0	3104.1
				FREON	290000.0	290000.0	300000.0	290000.0	6217.5
				HEXANE	290000.0	290000.0	290000.0	290000.0	2029.0
				METHYLENE CHLORIDE	310000.0	320000.0	270000.0	300000.0	25951.7
				PERCHLOR	260000.0	390000.0	300000.0	320000.0	65215.3
				80/20	300000.0	290000.0	290000.0	290000.0	6203.2
	23475	FILTER EFFLUENT	LIQ/LIQ (A)	DUPONT123	230.0	220.0	170.0	210.0	31.4
				FREON	140.0	130.0	130.0	130.0	6.6
				HEXANE	110.0	98.0	130.0	110.0	15.3
				METHYLENE CHLORIDE	310.0	290.0	320.0	310.0	13.7
				PERCHLOR	180.0	180.0	190.0	180.0	5.5
				80/20	140.0	140.0	150.0	150.0	2.6
	23475	FILTER EFFLUENT	INFRARED (A)	FLON	110.0	110.0		110.0	5.7
	23475	FILTER EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	150.0	130.0		140.0	9.9
				FREON	36.0	43.0		40.0	4.7
				HEXANE	57.0	48.0		53.0	5.9
				MTBE	110.0	110.0		110.0	4.2
				PERC	62.0	66.0		64.0	2.7

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

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Oil & Grease Concentrations:
mg/L in aqueous samples (A)
mg/kg in solid/sludge samples (S)

FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
34 POLYMER PLANT	23475	FILTER EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	160.0	150.0		150.0	6.4
				FREON	42.0	40.0		41.0	1.3
				HEXANE	67.0	68.0		68.0	0.9
				MTBE	120.0	150.0		130.0	16.3
				PERC	110.0	89.0		99.0	14.4
	23475	FILTER EFFLUENT	SPE COLUMN II (A)	FREON	65.0	46.0		56.0	13.4
				HEXANE	150.0	84.0		120.0	48.8
				80/20	230.0	250.0		240.0	12.0
	23476	OILY SLUDGE	SONICATION (S)	FREON	1000000.0	1000000.0	970000.0	1000000.0	71174.7
				HEXANE	860000.0	820000.0	960000.0	880000.0	73646.2
				PERCHLOR	1200000.0	1200000.0	1100000.0	1200000.0	38349.5
				80/20	890000.0	730000.0	810000.0	810000.0	79642.6
	23476	OILY SLUDGE	SOXHLET (S)	DUPONT123	1200000.0	980000.0	1100000.0	1100000.0	100976.9
				FREON	1200000.0	1100000.0	1200000.0	1200000.0	57735.0
				HEXANE	990000.0	1000000.0	980000.0	1000000.0	27319.1
				METHYLENE CHLORIDE	1000000.0	1200000.0	1000000.0	1100000.0	94516.3
				PERCHLOR	810000.0	850000.0	830000.0	830000.0	24062.4
				80/20	800000.0	840000.0	780000.0	810000.0	28160.2
	23477	SECONDARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	5.0 <	5.0 <	5.0 <	5.0 <	4.6
				FREON	6.4	5.0 <	14.0	8.3	
				HEXANE	5.0 <	5.0 <	5.0 <	5.0 <	
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	5.0 <	5.0 <	5.0 <	5.0 <	1.4
				80/20	5.0 <	7.5	5.0 <	5.8	
	23477	SECONDARY EFFLUENT	INFRARED (A)	FLON	4.0	4.0		4.0	
	23477	SECONDARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	7.4	6.4		6.9	0.7
				FREON	16.0	7.6		12.0	6.2
				HEXANE	14.0	14.0		14.0	0.2
				MTBE	22.0	25.0		24.0	2.2
				PERC	7.6	7.7		7.7	0.1
	23477	SECONDARY EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	16.0	14.0		15.0	1.4
				FREON	57.0	54.0		55.0	2.3
				HEXANE	40.0	49.0		44.0	6.6
				MTBE	86.0	91.0		88.0	3.6
				PERC	30.0	29.0		30.0	0.4
	23477	SECONDARY EFFLUENT	SPE COLUMN II (A)	FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				80/20	5.0 <	5.0 <		5.0 <	

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
35 RESTAURANT	23478	DEWATERED SLUDGE	SONICATION (S)	DUPONT123	28000.0	23000.0	23000.0	25000.0	2849.2
				FREON	7100.0	3400.0	3500.0	4700.0	2089.3
				HEXANE	1800.0	1900.0	2500.0	2100.0	366.0
				METHYLENE CHLORIDE	13000.0	21000.0	23000.0	19000.0	4998.5
				PERCHLOR	23000.0	15000.0	17000.0	18000.0	4251.7
				80/20	4200.0	5500.0	3700.0	4500.0	959.8
	23478	DEWATERED SLUDGE	SOXHLET (S)	DUPONT123	31000.0	33000.0	30000.0	32000.0	1826.6
				FREON	13000.0	12000.0	8200.0	11000.0	2523.7
				HEXANE	8400.0	6900.0	9100.0	8100.0	1121.9
				METHYLENE CHLORIDE	36000.0	34000.0	43000.0	38000.0	5115.1
				PERCHLOR	26000.0	28000.0	31000.0	28000.0	2373.8
				80/20	15000.0	21000.0	19000.0	19000.0	2917.7
	23125	VEGETABLE OIL WASTE	SONICATION (S)	DUPONT123	790000.0	840000.0	790000.0	800000.0	27039.5
				FREON	740000.0	960000.0	1000000.0	900000.0	148256.9
				HEXANE	930000.0	950000.0	820000.0	900000.0	70035.6
				METHYLENE CHLORIDE	740000.0	810000.0	920000.0	820000.0	89963.1
				PERCHLOR	850000.0	950000.0	970000.0	920000.0	64103.0
				80/20	940000.0	800000.0	780000.0	840000.0	87470.4
	23125	VEGETABLE OIL WASTE	SOXHLET (S)	DUPONT123	930000.0	960000.0	960000.0	950000.0	16464.8
				FREON	760000.0	610000.0	780000.0	720000.0	92059.7
				HEXANE	1100000.0	980000.0	980000.0	1000000.0	80063.8
				METHYLENE CHLORIDE	960000.0	920000.0	1000000.0	970000.0	57444.9
				PERCHLOR	1000000.0	1000000.0	1100000.0	1000000.0	27967.4
				80/20	970000.0	1000000.0	1000000.0	990000.0	18063.3
36 INDUSTRIAL LAUNDRY	23480	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	250.0	250.0	250.0	250.0	2.5
				FREON	180.0	190.0	170.0	180.0	7.9
				HEXANE	120.0	120.0	79.0	100.0	22.0
				METHYLENE CHLORIDE	170.0	150.0	160.0	160.0	11.6
				PERCHLOR	230.0	170.0	220.0	200.0	32.8
				80/20	110.0	130.0	130.0	120.0	13.8
	23480	PRIMARY EFFLUENT	INFRARED (A)	FLON	190.0	190.0		190.0	2.8
	23480	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	230.0	240.0		240.0	7.8
				FREON	190.0	190.0		190.0	5.7
				HEXANE	190.0	190.0		190.0	3.5
				MTBE	270.0	260.0		260.0	2.1
				PERC	200.0	200.0		200.0	0.7

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
37 FORMULATING PLANT	23480	PRIMARY EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	270.0	250.0		260.0	9.9
				FREON	220.0	220.0		220.0	0.7
				HEXANE	210.0	210.0		210.0	3.5
				MTBE	330.0	320.0		330.0	3.5
				PERC	210.0	210.0		210.0	2.1
	23481	PRIMARY EFFLUENT	LIQ/LIQ (A)	DUPONT123	64.0	82.0	92.0	79.0	14.2
				FREON	73.0	78.0	78.0	76.0	3.1
				HEXANE	66.0	67.0	48.0	60.0	10.5
				METHYLENE CHLORIDE	72.0	70.0	84.0	75.0	7.4
				PERCHLOR	110.0	130.0	110.0	120.0	12.8
	23481	PRIMARY EFFLUENT	SPE DISK 90mm (A)	80/20	71.0	68.0	52.0	64.0	10.7
				FLON	260.0	280.0		270.0	19.1
				METHYLENE CHLORIDE	500.0	570.0		540.0	52.3
				HEXANE	430.0	400.0		410.0	20.5
				MTBE	670.0	710.0		690.0	30.4
38 LEATHER TANNERY	23481	PRIMARY EFFLUENT	SPE DISK 47mm (A)	PERC	420.0	350.0		390.0	46.7
				METHYLENE CHLORIDE	1100.0	630.0		840.0	297.7
				FREON	370.0	540.0		450.0	123.7
				HEXANE	640.0	750.0		690.0	80.6
				MTBE	910.0	870.0		890.0	25.5
	23482	PRIMARY EFFLUENT	LIQ/LIQ (A)	PERC	1000.0	750.0		890.0	188.8
				DUPONT123	47.0	73.0	68.0	63.0	13.5
				FREON	48.0	58.0	76.0	61.0	14.3
				HEXANE	100.0	150.0	130.0	130.0	21.2
				METHYLENE CHLORIDE	55.0	120.0	150.0	110.0	46.1
	23482	PRIMARY EFFLUENT	SPE DISK 90mm (A)	PERCHLOR	88.0	82.0	110.0	94.0	14.9
				80/20	49.0	76.0	54.0	60.0	14.1
				FLON	200.0	220.0		210.0	13.4
				METHYLENE CHLORIDE	160.0	180.0		170.0	12.0
				FREON	83.0	64.0		74.0	13.4
	23482	PRIMARY EFFLUENT	SPE DISK 47mm (A)	HEXANE	160.0	140.0		150.0	7.8
				MTBE	270.0	220.0		240.0	33.2
				PERC	150.0	160.0		150.0	11.3
	23482	PRIMARY EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	300.0	320.0		310.0	13.4
				FREON	170.0	110.0		140.0	43.1
				HEXANE	200.0	160.0		180.0	27.6
				MTBE	260.0	260.0		260.0	0.7
				PERC	150.0	210.0		180.0	36.1

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					Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	REP1	REP2	REP3	MEAN	STD. DEV.
39 PETROLEUM REFINERY	23483	WASTE SLUDGE	SONICATION (S)	DUPONT123	190000.0	140000.0	160000.0	160000.0	24084.7
				FREON	130000.0	140000.0	120000.0	130000.0	9423.1
				HEXANE	140000.0	160000.0	130000.0	150000.0	12447.0
				METHYLENE CHLORIDE	220000.0	190000.0	240000.0	220000.0	21264.5
				PERCHLOR	210000.0	170000.0	220000.0	200000.0	25168.8
				80/20	120000.0	150000.0	130000.0	130000.0	13055.7
	23483	WASTE SLUDGE	SOXHLET (S)	DUPONT123	170000.0	170000.0	180000.0	170000.0	5487.6
				FREON	180000.0	220000.0	190000.0	190000.0	22140.3
				HEXANE	240000.0	270000.0	210000.0	240000.0	31176.8
				METHYLENE CHLORIDE	180000.0	150000.0	210000.0	180000.0	29053.2
				PERCHLOR	270000.0	240000.0	220000.0	240000.0	28710.4
				80/20	160000.0	190000.0	150000.0	170000.0	18624.2
	23484	API SEPARATOR EFF	LIQ/LIQ (A)	DUPONT123	20.0	23.0	18.0	20.0	2.7
				FREON	11.0	15.0	11.0	12.0	1.9
				HEXANE	17.0	18.0	17.0	18.0	0.7
				METHYLENE CHLORIDE	5.0 <	5.0 <	36.0	15.0	18.0
				PERCHLOR	34.0	40.0	33.0	36.0	3.5
				80/20	14.0	17.0	18.0	16.0	1.9
		API SEPARATOR EFF	INFRARED (A)	FLON	23.0	24.0		24.0	0.7
		API SEPARATOR EFF	SPE DISK 90mm (A)	METHYLENE CHLORIDE	16.0	16.0		16.0	0.4
				FREON	16.0	17.0		16.0	0.9
				HEXANE	8.7	14.0		11.0	3.9
				MTBE	18.0	21.0		20.0	2.1
				PERC	17.0	10.0		14.0	4.9
	23484	API SEPARATOR EFF	SPE DISK 47mm (A)	METHYLENE CHLORIDE	13.0	12.0		13.0	0.3
				FREON	17.0	16.0		16.0	0.8
				HEXANE	27.0	26.0		27.0	0.5
				MTBE	19.0	20.0		19.0	0.6
				PERC	9.2	9.2		9.2	
	23484	API SEPARATOR EFF	SPE COLUMN II (A)	FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	14.0	10.0		12.0	2.8
				80/20	10.0	12.0		11.0	1.4
	23485	INTERCEPTOR EFFLUENT LIQ/LIQ (A)		DUPONT123	22.0	14.0	13.0	16.0	5.1
				FREON	14.0	12.0	12.0	12.0	1.0
				HEXANE	5.0 <	9.3	8.8	7.7	2.3
				METHYLENE CHLORIDE	5.0 <	5.0 <	5.0 <	5.0 <	
				PERCHLOR	30.0	20.0	28.0	26.0	5.4
				80/20	5.0 <	6.8	5.7	5.8	0.9

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

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FACILITY	SCC#	WASTE STREAM	TECHNIQUE	SOLVENT	Oil & Grease Concentrations: mg/L in aqueous samples (A) mg/kg in solid/sludge samples (S)				
					REP1	REP2	REP3	MEAN	STD. DEV.
	23485	INTERCEPTOR EFFLUENT	INFRARED (A)	FLON	15.0	13.0		14.0	1.4
	23485	INTERCEPTOR EFFLUENT	SPE DISK 90mm (A)	METHYLENE CHLORIDE	9.0	9.9		9.5	0.6
				FREON	12.0	18.0		15.0	4.1
				HEXANE	16.0	20.0		18.0	3.0
				MTBE	32.0	34.0		33.0	1.6
				PERC	8.0	8.7		8.4	0.5
	23485	INTERCEPTOR EFFLUENT	SPE DISK 47mm (A)	METHYLENE CHLORIDE	6.7	8.0		7.4	0.9
				FREON	21.0	24.0		23.0	2.5
				HEXANE	22.0	24.0		23.0	1.6
				MTBE	21.0	24.0		23.0	2.5
				PERC	5.0 <	6.2		5.6	0.8
	23485	INTERCEPTOR EFFLUENT	SPE COLUMN II (A)	FREON	5.0 <	5.0 <		5.0 <	
				HEXANE	5.0 <	5.0 <		5.0 <	
				80/20	7.0	8.0		7.5	0.7

SPE Disk Values are not blank-corrected since blanks were not analyzed for two solvents, and the remaining solvents had blank values < 1 mg/L.

APPENDIX C
DISCUSSION OF STATISTICAL TECHNIQUES
USED IN THE PRELIMINARY DATA EVALUATION

Data Transformations

The standard deviations of the measured sample concentrations varied greatly and were roughly proportional to the mean sample concentrations, as is common with analytical data. This heteroscedasticity violates the basic ANOVA assumptions, and was taken into account in the statistical analysis.

The association between the standard deviations of sample concentrations and the mean sample concentrations was easily demonstrated in this data set by plotting the replicate standard deviations versus the mean of the replicate analyses for each sample/solvent combination, as is shown in Attachment A. Linear regressions of the standard deviation of concentration versus the mean concentration showed a very significant trend. Secondary predictors of the standard error including solvent, industrial category, sampling point, sample kingdom, and laboratory, were checked by including them as predictors along with the mean concentration. The only other factor that showed a relationship with standard error is laboratory (which is described in greater detail below.)

In order to allow the use of standard statistical tools, the concentration data were transformed so that the data would have constant standard deviations. If the standard deviations were exactly proportional to concentration, i.e., if there were no intercept in the regressions, then a logarithmic transformation would have produced constant standard deviations [1]. Since there was a positive intercept in each of the standard deviation regressions, the data were transformed using the equation:

$$z = \log(x + c) - \log(c),$$

where c serves the dual purpose of adjusting for the non-zero intercept of the standard deviation curve and allowing for a well-defined transformation that is robust to the negative concentration estimates found in this data. Examination of the data suggested a value of $c = 100$ for the aqueous analyses and $c = 10,000$ for both types of solid sample extractions. These values are on the same order of magnitude as the negative of the x -intercept ($= -\text{intercept}/\text{slope}$) of each regression, and they are of sufficient magnitude to keep even the most negative reported concentrations (-56 for aqueous, -1931 for sonication, -7556 for Soxhlet) a reasonable distance away from the pole at 0 in the log transformation. This distance prevents these points from gaining disproportionate leverage due to being transformed far out to the left of the data. Following this transformation, the data no longer showed a significant association between standard deviation and mean concentration. Therefore, the transformation was suitable to the ANOVA analysis described below.

The results on the transformed variable were translated back to statements about concentration by inverting the transformation, such that

$$x = \exp(z + \log(c)) - c$$

Results could then be expressed as approximate proportionate results, in concentration terms, if the inverse calculations were applied at a representative level of concentration, such as the mean of the transformed variable. For example, a limit value z_1 and a mean z_0 could be presented as a proportionate limit in concentration terms by calculating

$$\frac{\exp(z_1 + \log(c)) - \exp(z_0 + \log(c))}{\exp(z_0 + \log(c)) - c}$$

The analyses described in the remainder of this discussion should not be highly sensitive to the particular values of c used, within the constraints described above. A sensitivity check could be done to demonstrate this by testing alternative values of c .

Negative Concentrations

While it is counterintuitive to find negative measured concentrations, it can be expected to occur, at least occasionally, due to analytical variability whenever very small concentrations are measured via methods involving blank-subtraction. It is possible that some sample/solvent combinations could be removed from this study by setting up statistical limits on whether the sample can statistically be shown to contain a non-zero amount of oil and grease, for instance simply computing a one-sided hypothesis (at say 5%) that the mean concentration for a sample/solvent combination is greater than zero using the three replicate measurements (either with or without the data transformation). However, it is desirable that the study remain balanced across solvents, and since some solvents show concentrations clearly greater than zero while other solvents do not, such a rule may not be useful. Since the data transformation method described above can cope with negative measured concentrations, such samples were left in the study for the moment.

ANOVAs

The ANOVA model deemed best for this analysis was

$$y = a + b_i + g_j + d_{ij} + e_{ijk}$$

where $i = 1 \dots I$ solvents ($1 = \text{Freon}$), $j = 1 \dots J$ samples, and $k = 1 \dots K$ (3) replicate analyses. Here a , b_i , g_j , and d_{ij} are all fixed effects that were estimated⁶, and e_{ijk} is the random measurement error, with mean 0 and variance σ^2 . This model is a standard ANOVA model, and was fitted to obtain estimates of each of these parameters and standard errors associated with each estimate.

Upon testing this model (see Attachment B), it was clear that the interaction term is significant, (i.e., the effect of solvents depends on the sample matrix).

⁶With $\sum b_i = 0$, $\sum g_j = 0$, and $\sum d_{ij} = 0$ over i for fixed j and over j for fixed i

Evaluation of Solvents

The evaluation of individual sample-by-sample confidence limits on the difference between each alternative solvent and Freon would have resulted in too many outcomes to consider and would not have produced an overall picture of the performance of each alternative solvent. Instead, an overall index of performance that summarizes the similarity of each alternative solvent to Freon, while taking into account the differences in the outcomes for each solvent on different samples was considered desirable. A natural measure for this is the root mean square deviation across samples between the alternative solvent and the Freon results. In terms of the model above, the mean result for each solvent for a sample is,⁷

$$a + b_i + g_j + d_{ij},$$

so the root mean square deviation for solvent i is

$$\begin{aligned} RMSD_i &= \sqrt{\sum_j (((a + b_i + g_j + d_{ij}) - (a + b_1 + g_j + d_{1j}))^2) / J} \\ &= \sqrt{\sum_j (((b_i + d_{ij}) - (b_1 + d_{1j}))^2) / J} \\ &= \sqrt{(b_i - b_1)^2 + \sum_j (d_{ij} - d_{1j})^2} / J. \end{aligned}$$

This can also be computed as the root mean square deviation between the sample*solvent cell means for the alternative solvent and Freon. The smaller this measure, the more closely the results using the alternative solvent approximate the results using Freon. RMSD computes the squared deviation of the average analytical results using alternative solvents on a sample from that of Freon on the same sample, and accumulates these over all samples to provide an overall measure of agreement. The data show significant interaction between solvent and sample in the statistical model, that is, whether alternative solvents extract more or less oil and grease than Freon varies according to the sample matrix. RMSD measures variations both above and below the Freon results, because we must capture in our statistic the possibility that an alternative solvent extracts significantly less oil and grease than Freon on some samples, and more on other samples. Having the same average results as Freon across multiple samples is a desirable, but by no means sufficient, test of equivalence of the solvents. As a rule of thumb, the RMSD that would be expected by chance alone, for instance if Freon were tested in this protocol and compared with itself using separate analyses, can be computed. Under the null hypothesis that there is no actual difference in the procedures, the square of RMSD, appropriately normalized by the residual error estimate, will have an F distribution. Therefore,

⁷ For typographical simplicity, the carats over each parameter are omitted from this point on even though the formulae refer to the sample estimates rather than the theoretical model values.

$$\frac{K(RMSD)^2}{2s^2} \sim F_{J, IJ(K-1)}$$

where s is the root mean square error (RMSE) of the model, and I , J , and K are as above.

A 95% acceptance region for the equality of the test is

$$RMSD < \sqrt{\frac{2s^2}{K} F_{J, IJ(K-1)}^{-1}(.95)}$$

or, in terms of the normalized RMSD,

$$\frac{RMSD}{\sqrt{2s^2/K}} < \sqrt{F_{J, IJ(K-1)}^{-1}(.95)}$$

Examples of these analyses are shown in Attachment C.

Laboratory Comparisons

To test the sensitivity of the rankings to the effect of each laboratory, the above rankings were rerun, dropping out each laboratory in turn. These tests yielded no significant changes in the comparability of the solvents to Freon.

Laboratory differences in the analytical standard deviation were also explored. This was done by an ANOVA of the within-sample*solvent standard deviation of the non-transformed concentration versus laboratory. One laboratory showed significantly greater RSDs than the other two on soxhlet and sonication extractions of solid samples. All labs had statistically similar RSDs on separatory funnel extraction of aqueous samples.

Analysis by Type of Sample

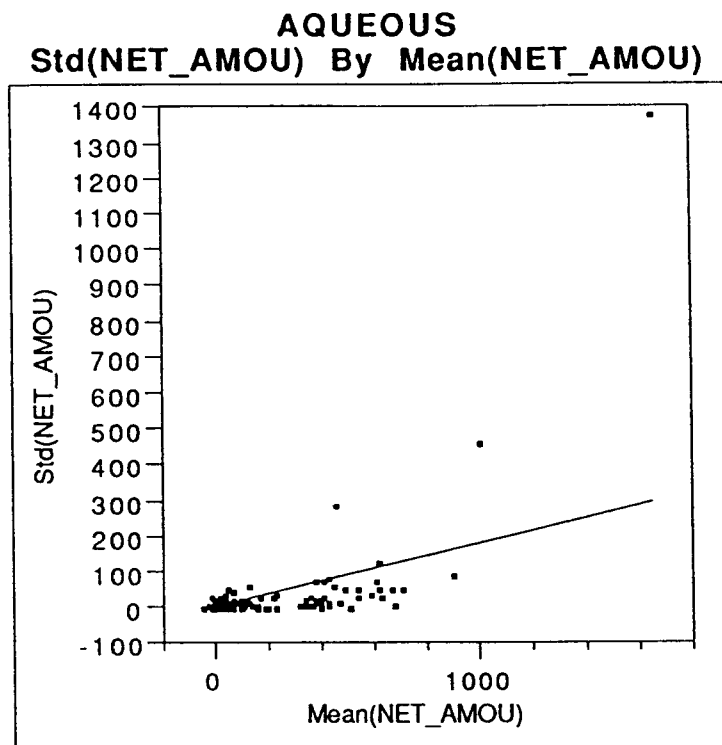
Rankings were also run over subsets of the samples, including divisions by industrial source, sampling point, kingdom, etc. to look for situations where the ranking changes or where all solvents are markedly different from Freon. The results of these rankings as divided by industrial source (petroleum vs. non-petroleum) are discussed in the report of preliminary findings.

Alternative solvents which rank well overall could be examined for their deviation on individual samples in order to spot sample types showing larger deviations, which may indicate areas for method improvement. Since none of the alternative solvents ranked well overall in the preliminary analyses, no such examination of individual sample deviation was performed.

Review Reference

1. N.R. Draper and H. Smith, Jr.. Applied Regression Analysis, Second Edition. John Wiley & Sons, New York, 1981, pp 238.

Attachment A



Fitting
— Linear Fit

Linear Fit Summary of Fit

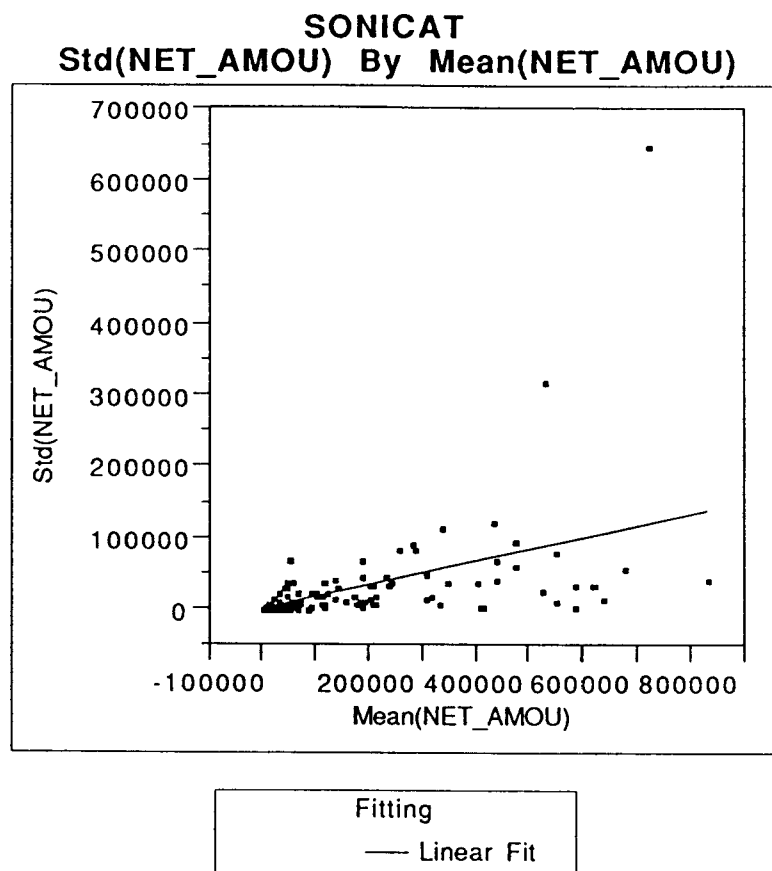
Rsquare	0.30436
Root Mean Square Error	24.46967
Mean of Response	10.53657
Observations (or Sum Wgts)	139.2707

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	49775.18	49775.2	83.1297
Error	190	113765.33	598.8	Prob>F
C Total	191	163540.51		0.0000

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	3.9038199	2.19739	1.78	0.0772
Mean(NET_AMOU)	0.1751866	0.01921	9.12	0.0000



Linear Fit Summary of Fit

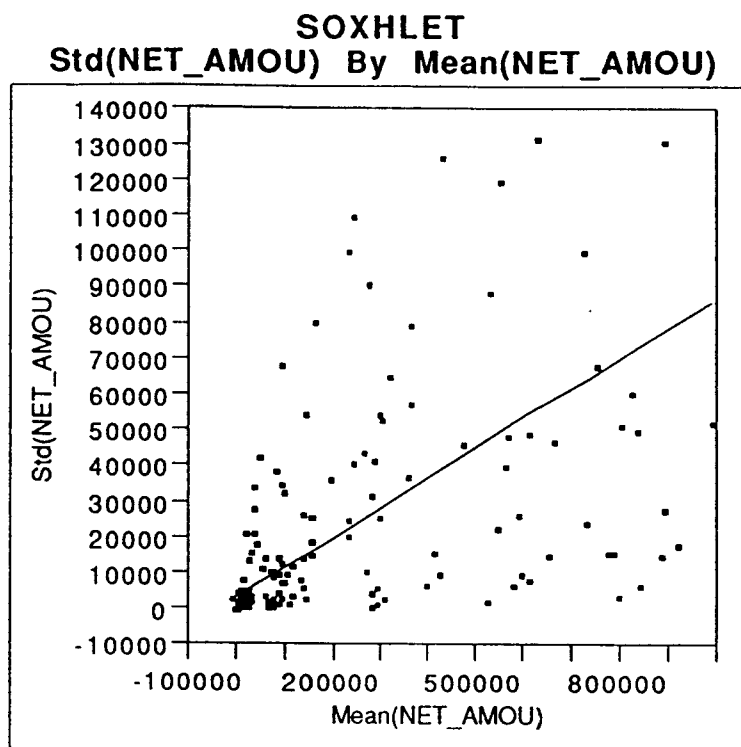
Rsquare	0.353493
Root Mean Square Error	7629.233
Mean of Response	5671.682
Observations (or Sum Wgts)	40.93224

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	4519150093	5e+9	77.6417
Error	142	8265136937	58205190	Prob>F
C Total	143	1.28e+10		0.0000

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	1578.4888	1279.76	1.23	0.2195
Mean(NET_AMOU)	0.1625567	0.01845	8.81	0.0000



Fitting
— Linear Fit

Linear Fit Summary of Fit

Rsquare	0.341522
Root Mean Square Error	5148.764
Mean of Response	5813.688
Observations (or Sum Wgts)	35.17674

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	2034907888	2e+9	76.7607
Error	148	3923446381	26509773	Prob>F
C Total	149	5958354269		0.0000

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	3095.6208	921.88	3.36	0.0010
Mean(NET_AMOU)	0.0832695	0.0095	8.76	0.0000

Attachment B

AQUEOUS

Response: LNP_AMT

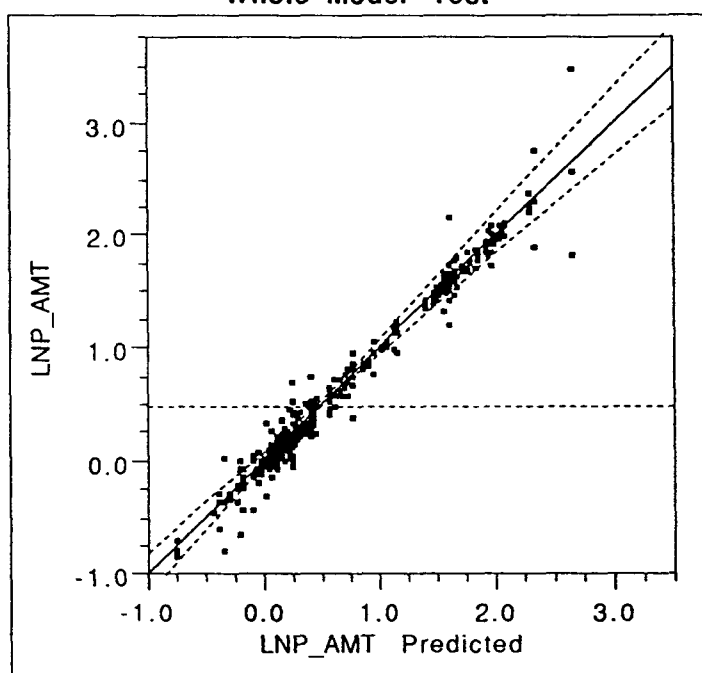
Summary of Fit

Rsquare 0.97779
 Root Mean Square Error 0.126681
 Mean of Response 0.4824
 Observations (or Sum Wgts) 587

Effect Test

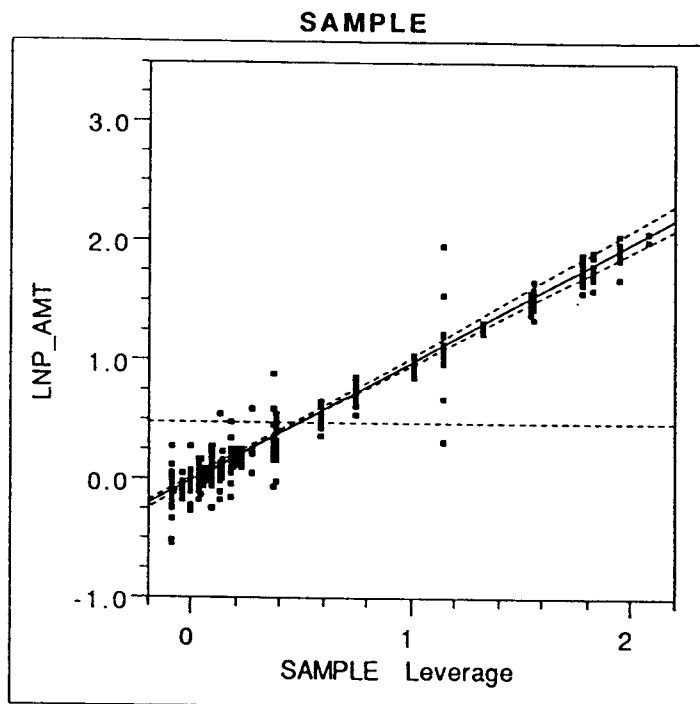
Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
SAMPLE	33	33	235.42051	444.5370	0.0000
SOLVENT	5	5	1.16299	14.4939	0.0000
SAMPLE*SOLVENT	165	165	33.66981	12.7155	0.0000

Whole-Model Test



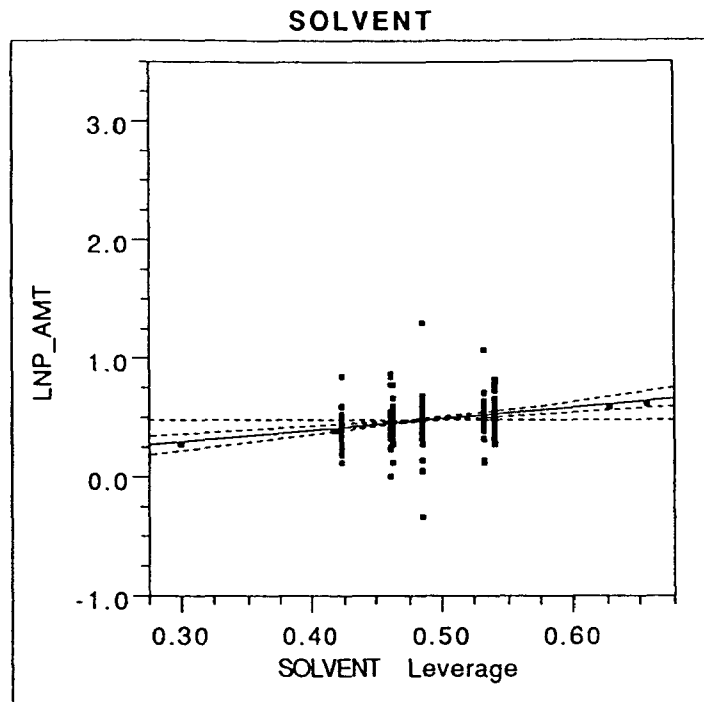
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob>F
Model	203	270.59227	1.33297	83.0609	0.0000
Error	383	6.14641	0.01605		
C Total	586	276.73868			

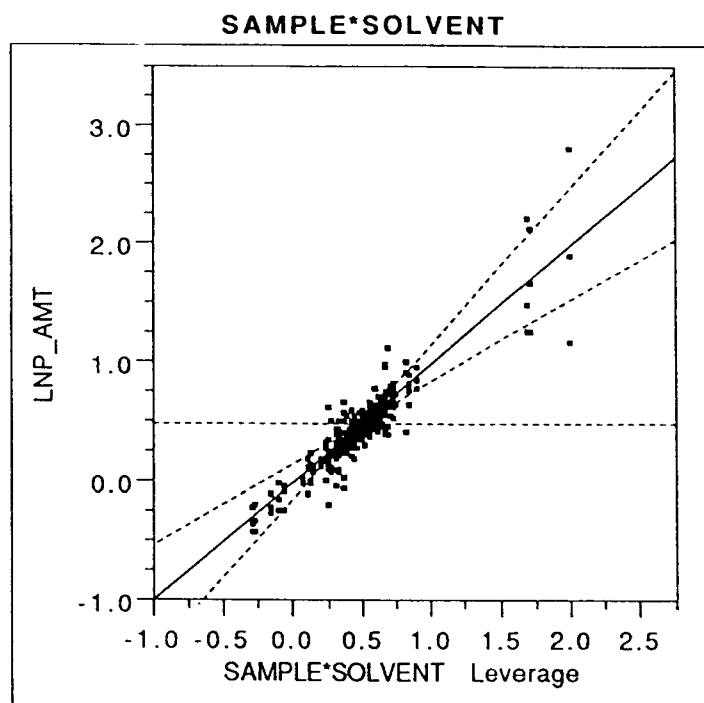


Sum of Squares		Effect Test		
235.42051		F Ratio	DF	Prob>F
		444.5370	33	0.0000

Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
22221	-0.081927652	0.0517172715	-0.08193
22223	0.166153922	0.0298589806	0.16615
22224	0.213805049	0.0517172715	0.21381
22225	1.143809535	0.0298589806	1.14381
22228	0.211932028	0.0298589806	0.21193
22229	0.045608445	0.0298589806	0.04561
22231	0.576576027	0.0298589806	0.57658
22232	0.370878501	0.0298589806	0.37088
22233	0.129629655	0.0298589806	0.12963
22234	1.939240086	0.0298589806	1.93924
22236	0.358980370	0.0298589806	0.35898
22239	0.025785114	0.0298589806	0.02579
22241	0.060454805	0.0298589806	0.06045
22243	-0.008480414	0.0298589806	-0.00848
23106	0.040835588	0.0298589806	0.04084
23108	-0.052768754	0.0298589806	-0.05277
23110	0.026058421	0.0298589806	0.02606
23111	0.114974854	0.0298589806	0.11497
23113	0.082593153	0.0298589806	0.08259
23115	-0.091542461	0.0298589806	-0.09154
23116	-0.101100310	0.0298589806	-0.10110
23120	0.063497202	0.0298589806	0.06350
23121	0.177725090	0.0298589806	0.17773
23124	0.262299843	0.0298589806	0.26230
23457	1.823537819	0.0298589806	1.82354
23459	0.739219598	0.0298589806	0.73922
23461	1.321908021	0.0298589806	1.32191
23463	1.774738573	0.0298589806	1.77474
23466	0.021799847	0.0298589806	0.02180
23468	0.196272754	0.0298589806	0.19627
23470	1.549298928	0.0298589806	1.54930
23472	0.171667433	0.0298589806	0.17167
23473	1.630797456	0.0310782124	1.62519
23475	1.011166706	0.0298589806	1.01117



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
1.1629909	14.4939	5	0.0000
Least Squares Means			
Level	Least Sq Mean	Std Error	Mean
80/20	0.4429666373	0.0132606094	0.460088
DUPONT123	0.4689320069	0.0133475652	0.471056
FREON	0.4441710175	0.0132606094	0.460378
HEXANE	0.3996973354	0.0132606094	0.417870
METHYLENE CHLORI	0.5210694150	0.0132606094	0.537898
PERCHLOR	0.5317680406	0.0132606094	0.546996



Effect Test			
Sum of Squares	F Ratio	DF	Prob>F
33.669811	12.7155	165	0.0000

Least Squares Means		
Level	Least Sq Mean	Std Error
22221,80/20	0.030529205	0.1266809262
22221,DUPONT123	-0.052346480	0.1266809262
22221,FREON	-0.073108472	0.1266809262
22221,HEXANE	-0.446287103	0.1266809262
22221,METHYLENE CHLORI	-0.000000000	0.1266809262
22221,PERCHLOR	0.049646940	0.1266809262
22223,80/20	0.270663947	0.0731392668
22223,DUPONT123	0.183009609	0.0731392668
22223,FREON	0.008045125	0.0731392668
22223,HEXANE	0.242453319	0.0731392668
22223,METHYLENE CHLORI	0.232777770	0.0731392668
22223,PERCHLOR	0.059973765	0.0731392668
22224,80/20	0.016463726	0.1266809262
22224,DUPONT123	0.258587912	0.1266809262
22224,FREON	0.167292518	0.1266809262
22224,HEXANE	0.355223892	0.1266809262
22224,METHYLENE CHLORI	0.217527813	0.1266809262
22224,PERCHLOR	0.267734435	0.1266809262
22225,80/20	2.339072219	0.0731392668
22225,DUPONT123	2.647357710	0.0731392668
22225,FREON	0.549248083	0.0731392668
22225,HEXANE	0.288558265	0.0731392668
22225,METHYLENE CHLORI	0.432379193	0.0731392668
22225,PERCHLOR	0.606241738	0.0731392668
22228,80/20	0.255844448	0.0731392668
22228,DUPONT123	0.262488136	0.0731392668
22228,FREON	0.168376570	0.0731392668
22228,HEXANE	0.276765722	0.0731392668
22228,METHYLENE CHLORI	0.100717481	0.0731392668
22228,PERCHLOR	0.207399809	0.0731392668
22229,80/20	0.080562062	0.0731392668
22229,DUPONT123	0.088903341	0.0731392668
22229,FREON	0.007156074	0.0731392668
22229,HEXANE	0.028209478	0.0731392668
22229,METHYLENE CHLORI	0.039780510	0.0731392668
22229,PERCHLOR	0.029039203	0.0731392668
22231,80/20	0.407664714	0.0731392668
22231,DUPONT123	0.710641029	0.0731392668
22231,FREON	0.568079927	0.0731392668
22231,HEXANE	0.442544918	0.0731392668
22231,METHYLENE CHLORI	0.686321980	0.0731392668
22231,PERCHLOR	0.644203593	0.0731392668
22232,80/20	0.153214764	0.0731392668
22232,DUPONT123	0.270130900	0.0731392668
22232,FREON	0.408605785	0.0731392668
22232,HEXANE	0.233756184	0.0731392668
22232,METHYLENE CHLORI	0.754308091	0.0731392668

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22232,PERCHLOR	0.405255284	0.0731392668
22233,80/20	0.188988959	0.0731392668
22233,DUPONT123	0.365724967	0.0731392668
22233,FREON	-0.201028849	0.0731392668
22233,HEXANE	0.196219393	0.0731392668
22233,METHYLENE CHLORI	0.076333861	0.0731392668
22233,PERCHLOR	0.151539602	0.0731392668
22234,80/20	1.915012680	0.0731392668
22234,DUPONT123	2.050117391	0.0731392668
22234,FREON	1.842390883	0.0731392668
22234,HEXANE	1.787820143	0.0731392668
22234,METHYLENE CHLORI	2.083455076	0.0731392668
22234,PERCHLOR	1.956644340	0.0731392668
22236,80/20	0.091455233	0.0731392668
22236,DUPONT123	-0.079757881	0.0731392668
22236,FREON	0.315852264	0.0731392668
22236,HEXANE	0.008113392	0.0731392668
22236,METHYLENE CHLORI	1.610903084	0.0731392668
22236,PERCHLOR	0.207316127	0.0731392668
22239,80/20	0.021363971	0.0731392668
22239,DUPONT123	0.006132858	0.0731392668
22239,FREON	0.035865335	0.0731392668
22239,HEXANE	-0.027923760	0.0731392668
22239,METHYLENE CHLORI	0.023174515	0.0731392668
22239,PERCHLOR	0.096097765	0.0731392668
22241,80/20	0.063867979	0.0731392668
22241,DUPONT123	0.043815594	0.0731392668
22241,FREON	0.051997954	0.0731392668
22241,HEXANE	0.085105647	0.0731392668
22241,METHYLENE CHLORI	0.034613868	0.0731392668
22241,PERCHLOR	0.083327785	0.0731392668
22243,80/20	0.074858149	0.0731392668
22243,DUPONT123	-0.397055696	0.0731392668
22243,FREON	0.041624164	0.0731392668
22243,HEXANE	-0.065607314	0.0731392668
22243,METHYLENE CHLORI	0.056849687	0.0731392668
22243,PERCHLOR	0.238448526	0.0731392668
23106,80/20	-0.037921896	0.0731392668
23106,DUPONT123	0.009991076	0.0731392668
23106,FREON	0.090729899	0.0731392668
23106,HEXANE	0.013890435	0.0731392668
23106,METHYLENE CHLORI	-0.001218016	0.0731392668
23106,PERCHLOR	0.169542026	0.0731392668
23108,80/20	-0.304263947	0.0731392668
23108,DUPONT123	0.012451833	0.0731392668
23108,FREON	0.107839250	0.0731392668
23108,HEXANE	0.010516094	0.0731392668
23108,METHYLENE CHLORI	-0.248037118	0.0731392668
23108,PERCHLOR	0.104881365	0.0731392668
23110,80/20	0.054514033	0.0731392668
23110,DUPONT123	-0.112464383	0.0731392668
23110,FREON	-0.057731793	0.0731392668
23110,HEXANE	0.051891377	0.0731392668
23110,METHYLENE CHLORI	0.115524202	0.0731392668
23110,PERCHLOR	0.104617092	0.0731392668
23111,80/20	0.032430537	0.0731392668
23111,DUPONT123	0.113213271	0.0731392668
23111,FREON	0.010865913	0.0731392668
23111,HEXANE	0.246631887	0.0731392668
23111,METHYLENE CHLORI	0.134357581	0.0731392668
23111,PERCHLOR	0.152349936	0.0731392668
23113,80/20	-0.012864876	0.0731392668
23113,DUPONT123	-0.112727899	0.0731392668
23113,FREON	0.156503533	0.0731392668
23113,HEXANE	0.152200758	0.0731392668
23113,METHYLENE CHLORI	0.158711876	0.0731392668
23113,PERCHLOR	0.153735529	0.0731392668
23115,80/20	-0.353191414	0.0731392668
23115,DUPONT123	-0.215308359	0.0731392668
23115,FREON	0.105828916	0.0731392668
23115,HEXANE	-0.000000000	0.0731392668
23115,METHYLENE CHLORI	0.107651494	0.0731392668
23115,PERCHLOR	-0.194235402	0.0731392668
23116,80/20	-0.031491895	0.0731392668
23116,DUPONT123	-0.755272277	0.0731392668
23116,FREON	0.014470841	0.0731392668
23116,HEXANE	0.033139273	0.0731392668
23116,METHYLENE CHLORI	-0.002536661	0.0731392668
23116,PERCHLOR	0.135088860	0.0731392668
23120,80/20	0.035937835	0.0731392668
23120,DUPONT123	0.070701751	0.0731392668
23120,FREON	0.039128838	0.0731392668
23120,HEXANE	0.045570111	0.0731392668
23120,METHYLENE CHLORI	0.107241338	0.0731392668
23120,PERCHLOR	0.082403339	0.0731392668
23121,80/20	0.106309145	0.0731392668
23121,DUPONT123	0.142302937	0.0731392668
23121,FREON	0.128118963	0.0731392668
23121,HEXANE	0.177416900	0.0731392668
23121,METHYLENE CHLORI	0.298465221	0.0731392668
23121,PERCHLOR	0.213737374	0.0731392668
23124,80/20	0.406740348	0.0731392668

23124,DUPONT123	0.142777666	0.0731392668
23124,FREON	0.122163816	0.0731392668
23124,HEXANE	0.133983982	0.0731392668
23124,METHYLENE CHLORI	0.337924452	0.0731392668
23124,PERCHLOR	0.430208791	0.0731392668
23457,80/20	1.548051893	0.0731392668
23457,DUPONT123	1.467895079	0.0731392668
23457,FREON	2.037291907	0.0731392668
23457,HEXANE	1.950458735	0.0731392668
23457,METHYLENE CHLORI	1.643079228	0.0731392668
23457,PERCHLOR	2.294450072	0.0731392668
23459,80/20	0.893192151	0.0731392668
23459,DUPONT123	0.360133237	0.0731392668
23459,FREON	0.948661900	0.0731392668
23459,HEXANE	0.657482979	0.0731392668
23459,METHYLENE CHLORI	0.420806466	0.0731392668
23459,PERCHLOR	1.155040855	0.0731392668
23461,80/20	1.065287664	0.0731392668
23461,DUPONT123	1.569397541	0.0731392668
23461,FREON	1.158550214	0.0731392668
23461,HEXANE	1.019989934	0.0731392668
23461,METHYLENE CHLORI	1.522559692	0.0731392668
23461,PERCHLOR	1.595663081	0.0731392668
23463,80/20	1.679632893	0.0731392668
23463,DUPONT123	1.961525653	0.0731392668
23463,FREON	1.978669660	0.0731392668
23463,HEXANE	1.610839908	0.0731392668
23463,METHYLENE CHLORI	1.651522296	0.0731392668
23463,PERCHLOR	1.766241026	0.0731392668
23466,80/20	-0.020279108	0.0731392668
23466,DUPONT123	0.032455987	0.0731392668
23466,FREON	0.035542717	0.0731392668
23466,HEXANE	0.014449181	0.0731392668
23466,METHYLENE CHLORI	0.029515790	0.0731392668
23466,PERCHLOR	0.039114518	0.0731392668
23468,80/20	0.128916135	0.0731392668
23468,DUPONT123	0.218894130	0.0731392668
23468,FREON	0.245849721	0.0731392668
23468,HEXANE	0.198197495	0.0731392668
23468,METHYLENE CHLORI	0.202826321	0.0731392668
23468,PERCHLOR	0.182952721	0.0731392668
23470,80/20	1.499087286	0.0731392668
23470,DUPONT123	1.546585294	0.0731392668
23470,FREON	1.507133089	0.0731392668
23470,HEXANE	1.603586213	0.0731392668
23470,METHYLENE CHLORI	1.541638107	0.0731392668
23470,PERCHLOR	1.597763579	0.0731392668
23472,80/20	0.065431592	0.0731392668
23472,DUPONT123	0.292055667	0.0731392668
23472,FREON	0.126754795	0.0731392668
23472,HEXANE	0.042734632	0.0731392668
23472,METHYLENE CHLORI	0.293780572	0.0731392668
23472,PERCHLOR	0.209247342	0.0731392668
23473,80/20	1.497531799	0.0731392668
23473,DUPONT123	1.726140760	0.0895769419
23473,FREON	1.609112920	0.0731392668
23473,HEXANE	1.464292065	0.0731392668
23473,METHYLENE CHLORI	1.650423816	0.0731392668
23473,PERCHLOR	1.837283378	0.0731392668
23475,80/20	0.898253438	0.0731392668
23475,DUPONT123	1.115189881	0.0731392668
23475,FREON	0.845932136	0.0731392668
23475,HEXANE	0.757485270	0.0731392668
23475,METHYLENE CHLORI	1.402980525	0.0731392668
23475,PERCHLOR	1.047158987	0.0731392668

Attachment C

AQUEOUS SAMPLE	Sample * Solvent Means					
	FREON	80/20	DUPONT123	HEXANE	METHYLEN	PERCHLOR
22223	0.008	0.271	0.183	0.242	0.233	0.060
22225	0.549	2.339	2.647	0.289	0.432	0.606
22228	0.168	0.256	0.262	0.277	0.101	0.207
22229	0.007	0.081	0.089	0.028	0.040	0.029
22231	0.568	0.408	0.711	0.443	0.686	0.644
22232	0.409	0.153	0.270	0.234	0.754	0.405
22233	-0.201	0.189	0.366	0.196	0.076	0.152
22234	1.842	1.915	2.050	1.788	2.083	1.957
22236	0.316	0.091	-0.080	0.008	1.611	0.207
22239	0.036	0.021	0.006	-0.028	0.023	0.096
22241	0.052	0.064	0.044	0.085	0.035	0.083
22243	0.042	0.075	-0.397	-0.066	0.057	0.238
23106	0.091	-0.038	0.010	0.014	-0.001	0.170
23108	0.108	-0.304	0.012	0.011	-0.248	0.105
23110	-0.058	0.055	-0.112	0.052	0.116	0.105
23111	0.011	0.032	0.113	0.247	0.134	0.152
23113	0.157	-0.013	-0.113	0.152	0.159	0.154
23115	0.106	-0.353	-0.215	0.000	0.108	-0.194
23116	0.014	-0.031	-0.755	0.033	-0.003	0.135
23120	0.039	0.036	0.071	0.046	0.107	0.082
23121	0.128	0.106	0.142	0.177	0.298	0.214
23124	0.122	0.407	0.143	0.134	0.338	0.430
23457	2.037	1.548	1.468	1.950	1.643	2.294
23459	0.949	0.893	0.360	0.657	0.421	1.155
23461	1.159	1.065	1.569	1.020	1.523	1.596
23463	1.979	1.680	1.962	1.611	1.652	1.766
23466	0.036	-0.020	0.032	0.014	0.030	0.039
23468	0.246	0.129	0.219	0.198	0.203	0.183
23470	1.507	1.499	1.547	1.604	1.542	1.598
23472	0.127	0.065	0.292	0.043	0.294	0.209
23473	1.609	1.498	1.726	1.464	1.650	1.837
23475	0.846	0.898	1.115	0.757	1.403	1.047
RMSD		0.374	0.466	0.163	0.321	0.170
RMSE	0.127					
J	31					
I	6					
K	3					
95% AL	0.126					

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The following text supersedes that of Section 5.6 found on pages 22-23.

Exhibit 16 summarizes all of the solvent-Freon ratios on one graph. In this graph, the mean solvent-Freon ratio (computed prior to log-transformation of the data) is plotted on a logarithmic scale so that amounts of oil and grease greater or less than the amount extracted by Freon 113 are equidistant from 1.00.

This graph shows that the mean solvent-Freon ratios range from approximately 0.4 to 3.3, depending on the solvent, technique, and type of sample being extracted. Of interest in this graph is the influence of the solvent or technique on the average amount of material extracted. For example, Soxhlet extraction with the alternative solvents seems to extract somewhat more oil and grease than the reference Soxhlet extraction with Freon 113, whereas less oil and grease is extracted with sonication rather than with Soxhlet using Freon 113 unless methylene chloride is used in the sonication extraction. It must be remembered that these ratios represent the *average* amount of material extracted, whereas the RMSD represents the deviation from the amount extracted by Freon 113 on an individual sample basis. Therefore, although these averages are interesting, they cannot be relied upon as the final criterion for equivalence.

The use of Freon 113 as the extracting solvent in the alternative techniques allowed the variables of solvent and technique to be separated. Using cartridges (columns), the amount of oil and grease extracted by Freon 113 and the alternative solvents is less than the amount extracted using the reference separatory funnel method for all solvent-technique combinations except 80/20 with the new cartridges. However, all solvents extract more material than Freon 113 when extracted by the same cartridge technique.

Using 47 mm disks with Freon 113, nearly twice as much oil and grease is extracted than is extracted by Freon 113 in the reference separatory funnel method. The amounts of oil and grease extracted by the alternative solvents using the 47 mm disk are even greater than the amount extracted by Freon 113. For 90 mm disks, the amount of oil and grease extracted by Freon 113 is slightly less than the amount extracted using the reference separatory funnel method, but the alternative solvents extract more than Freon 113 by either the reference separatory funnel extraction or 90 mm disk extraction.

The effects of solvent versus technique cannot be separated for Flon-316 and NDIR because Freon 113 was not tested with the NDIR technique. Therefore, it is not known whether the larger amount of oil and grease indicated by the Flon/NDIR technique results from the use of Flon-316 or results from the way in which the NDIR is calibrated.

It must be emphasized that the solvent to Freon ratios are averaged, and that the differences may not be statistically significant due to the high variability of the data. Although significance tests could be performed, the RMSD is a more reliable indicator of the difference between alternative solvents/techniques and the reference Freon 113 separatory funnel method.

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This exhibit replaces Exhibit 16 shown on page 37 of the original report.

Mean Solvent-Freon Ratios for All Techniques Tested

