



Project Summary

Removal of PCBs from Contaminated Soil Using the CF Systems[®] Solvent Extraction Process: A Treatability Study

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The United States Environmental Protection Agency (EPA), through its Superfund Technical Assistance Response Team (START) and Superfund Innovative Technology Evaluation (SITE) Programs, completed a treatability study to determine the effectiveness of solvent extraction in separating polychlorinated biphenyls (PCBs) from soil collected at the Springfield Township Dump (STD) Superfund site near Davisburg, MI.

The study consisted of a total of six runs, in which 100 lb of dried soil was processed for each run. Based on preliminary analytical results, three 20-min extraction cycles were chosen as the most economical way to achieve the project objectives. Therefore, this three-extraction cycle condition was repeated twice to acquire data for three runs operated at the same condition. The other three runs consisted of two, four, and five 20-min extraction cycles.

The results of the study indicated that on average approximately 98 percent removal of PCBs was achieved for the test runs using three extraction cycles. The four- and five-extraction cycle runs were the most effective in reducing the concentration of PCBs in soil, to 1.8 and 2.2 mg/kg, respectively. This indicated that the number of extraction cycles required for attaining the lowest concentrations of PCBs in product solids was greater than three but less than or equal to five since there was no discernible improvement in PCB removal from four to five extraction cycles. However, results from

oil and grease analysis suggest that higher removal efficiencies may be possible with additional extraction cycles.

Analysis of the filtered process water collected from all six runs indicated that PCBs were detected only in the filtrate from the two-cycle run [1.9 micrograms per liter ($\mu\text{g/L}$)]. PCBs were not detected ($<1.0 \mu\text{g/L}$) in the filtrate collected from the other five runs.

This Project Summary was developed by EPA's START and SITE programs in Cincinnati, OH to announce key findings of a solvent extraction treatability study that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Project Background

EPA conducted a treatability study on soil collected from the STD Superfund site. The approximately 4-acre site is located near the town of Davisburg, MI (Figure 1). Between 1966 and 1968 the STD was used for the disposal of drummed and liquid industrial waste. Primary contaminants in the soil (a fine-to-coarse-grained sand) include: arsenic, lead, and barium; volatile organic compounds (VOCs); and semivolatile organic compounds (SVOCs), which include PCBs and the pesticide dieldrin.

Onsite incineration had been specified in the Record of Decision for remediating the soil at the site, but negative public opinion toward incineration has led to the consideration of treatment alternatives. Based upon preliminary bench-scale testing on soil samples taken from the site,



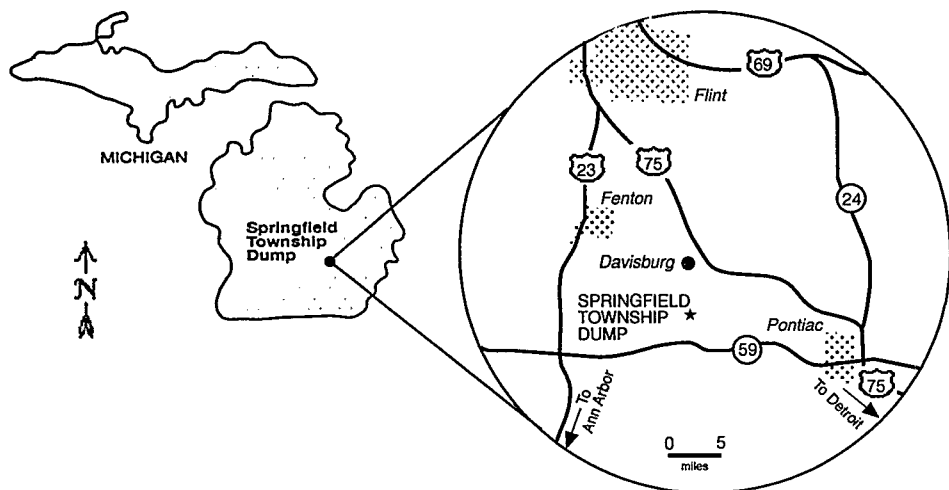


Figure 1. Location of the STD Superfund site.

the CF Systems® (CFS) solvent extraction process was believed to be such an alternative. Therefore, a treatability study was conducted to determine whether the technology would be effective in treating soils at the STD to the desired cleanup standard.

Approximately 1,158 lb of soil were obtained directly from PCB "hot zones" at the STD site and then screened onsite to remove oversize material (>1/2-in. diameter), which was approximately 168 lb (14.5 percent). Of the approximately 1,000 lb of material screened to ≤1/2-in. diameter, CFS used approximately 150 pounds to conduct a series of bench-scale tests in order to establish basic operating conditions for the treatability study. The remaining volume was shipped to Hazen Research, Inc. in Golden, CO, which is the home base for CFS's Mobile Demonstration Unit (MDU).

The treatability study was conducted using CFS's pilot-scale MDU on a batch-mode. Liquified propane was the solvent chosen to extract the organic compounds from the STD soil. Figure 2 illustrates the basic CFS process and the sample locations.

At Hazen Research Inc., the feed material was air-dried, further screened to remove oversize material (>1/4-in. diameter), and mixed to produce a homogenous test feed. Table 1 summarizes the results of the test soil screening for removal of oversize material.

The contaminated soil was fed in 100-lb batches into the extractor and thoroughly mixed with approximately 150 lb of sol-

vent for each cycle. Following phase separation of the solvent and contaminants from solids, the solvent/contaminant mixture passes from the extraction system to the solvent recovery system. Once in the solvent recovery system, the solvent is vaporized from the contaminant, condensed, and recycled back to the extraction system as fresh solvent. After all extraction cycles are completed, water is added to the extractor and mixed with the solids to aid in removing any residual propane. Product solids are discharged as a slurry in water and then filtered to form a filter cake.

Test Objectives and Conditions

The primary objectives for the treatability study were:

- Determine the effectiveness of removing PCBs from STD soil to the remedial action standard (RAS) of ≤1 mg/kg.
- Determine PCB concentrations in the filtrate water to ensure proper disposal.

Secondary objectives of the treatability study included but were not limited to verifying the absence of PCBs in the pilot-scale unit prior to testing, determining residual concentrations of dieldrin in the product solids, and determining mass balance for total materials.

The CFS pilot-scale treatability study was conducted in two phases, which included a total of five main process runs. Phase I consisted of three test runs, each consisting of a different number of extraction cycles. The first run consisted of three 20-min extraction cycles, the second run consisted of four 20-min extraction cycles, and the third run consisted of five 20-min extraction cycles.

Preliminary analytical results, using hexane as the extracting agent, indicated that the primary objective of producing solids having ≤1.0 mg/kg PCB concentration was met for the three-cycle run. Therefore, Phase II consisted of two additional test runs using three 20-min extraction cycles each, since this process condition was believed to be the most economically feasible in achieving the objective. It was later determined that these preliminary re-

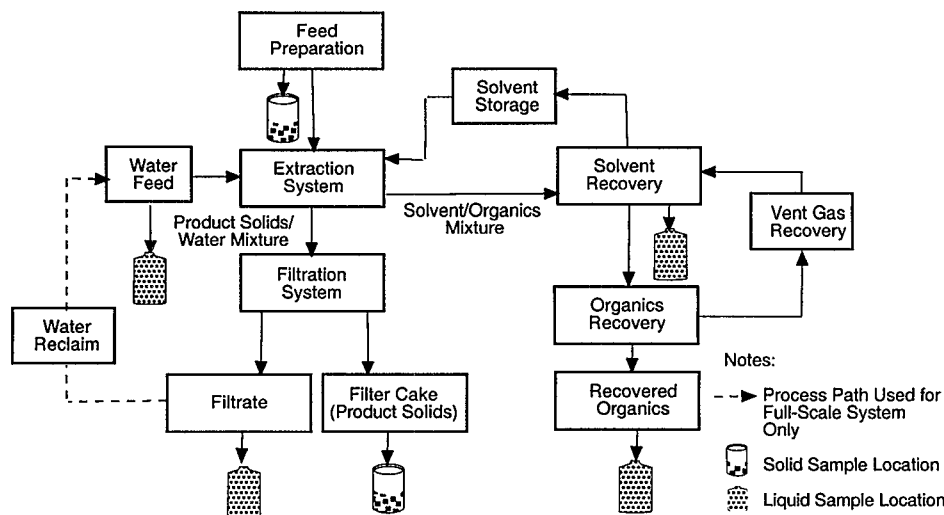


Figure 2. CFS® Process Diagram.

Table 1. Percentage Of Screened Oversized Material

Location	Starting Material (lbs)	Material Screened (lbs)	% Oversize
Springfield Township Dump	1,158	168 ^a	~ 14.5
Hazen Research Inc.	~626	26 ^b	~ 4.0
Total % oversize $\geq 1/4$ inch			~18.5 ^c

^a Using plastic crating having approximately 1/2-inch openings.

^b Using an ASTM sieve having 1/4-inch openings.

^c Oversize material could be treated following size reduction (i.e., pulverizing) during a full-scale remediation.

sults underestimated the concentration of PCBs in the treated soil. A sixth run consisting of two 20-min extraction cycles was added to test the limits of the pilot unit in treating soil to the desired levels. Table 2 summarizes the process conditions for all six runs.

Analytical Results

Sampling was performed in accordance with an EPA-approved Quality Assurance Project Plan. The critical process streams sampled for each of the six runs included: feed soil; product solids (filter cake); and filtrate water. Samples of the organic extract were taken at the end of Run 6, in order to perform a mass balance on PCBs.

Table 3 summarizes the MDU's PCB percent removal efficiencies for each run and as averages of all six runs and the three triple extraction cycle runs. Oil and

grease (O&G) analysis was also conducted on feed and product solids for each test run to determine propane's capability in extracting semi- and nonvolatile organic compounds in addition to PCBs. Results of the O&G analyses are summarized in Table 4.

PCBs were not detected in the filtrate samples collected for the five main test runs (<1.0 $\mu\text{g/L}$). However, for Run 6, which involved only two extraction cycles, PCBs were detected at 1.9 $\mu\text{g/L}$. The product oil collected at the end of the entire study (3700 g) contained a PCB concentration in excess of 11,000 mg/kg. This shows that the process was effective in concentrating the PCBs within the product oil fraction.

Dieldrin was not detected in the feed soil nor product solids; therefore removal could not be evaluated. More detail of the

analyses and measurements are discussed in the Project Report.

Mass Balance

A total materials balance was conducted to account for all material loaded into the unit for each run and to ensure that the majority of the material was recovered and did not simply remain in one or more of the process components. Any material loaded into the unit or exiting the unit was examined in the total materials mass balance.

Table 5 summarizes the results of the total materials balance for each run and over the entire treatability study. Results indicate that approximately 98% of input material was accounted for in the product streams. The mass balance closure for PCBs was approximately 62%. The low recovery is believed to be at least partially attributable to the inability to drain all of the oil from the extract product tank and associated piping.

Quality of the Data

The data quality objectives established for this pilot-scale treatability study were based on project requirements and thus designed to ensure that the data generated during the study would be of known and acceptable quality to achieve the project's technical objectives.

There were several quality control analyses conducted to evaluate the laboratory performance. These results are discussed in detail in the full report. The critical

Table 2. Process Conditions For All Test Runs

Test Phase	Run No.	Feed Loaded (lbs)	No. of Extraction Cycles	Mixing Time Each Cycle (min)	Mixing Speed	Solvent/Feed Ratio (by weight each cycle)	Extraction Pressure (psi) Avg/Range	Extraction Temp ($^{\circ}\text{F}$) Avg/Range
I	1	100	3	20	Full	1.5/1	315/250-409	133/125-138
	2	100	4	20	0/Full ^a	1.5/1	261/223-308	122/106-133
	3	100	5	20	Full	1.5/1	238/182-294	117/93-150
II	4	100	3	20	Full	1.5/1	266/202-309	124/98-140
	5	100	3	20	Full	1.5/1	243/194-299	119/98-137
Added Run	6	100	2	20	Full	1.5/1	277/231-319	125/110-138

^a During one of the four extraction cycles, the mixer was inoperable; however, a solvent flow was established by recirculating propane from the top of the extractor into the bottom.

Table 3. PCB Removal Efficiencies

Run Number	No. of Extraction Cycles	Soil Feed Concentration ¹ (mg/kg)	Product Solids Concentration ¹ (mg/kg)	Percent Removal
1	3	210	4.9	97.7
2	4	240	1.8	99.3
3	5	340	2.2	99.4
4	3	310 ²	4.0 ²	98.7
5	3	220	5.8	97.4
6	2	220	19.0	91.4
Average³		260/250	6.3/4.9	97.6/98.0

¹ The test method used was SW-846 3540/8080; Aroclor 1254 was the only PCB identified.

² Average concentration of analyses of field duplicate samples (see Table 7).

³ Two values are given; the first is the average of all six runs and the second is the average of the three extraction cycle runs (Runs 1, 4, and 5).

Table 4. O&G Removal Efficiencies

Run Number	No. of Extraction Cycles	Soil Feed Concentration ¹ (mg/kg)	Product Solids Concentration ¹ (mg/kg)	Percent Removal
1	3	4480	112	97.5
2	4	4560	73	98.4
3	5	5870	< 20	> 99.6
4	3	5460	133	97.6
5	3	5140	93	98.2
6	2	7060	279	96.0
Average²		5430/5030	<118/113	>97.8/>97.8

¹ The test method used was SW-846 9071.

² Two values are given; the first is the average of all six runs and the second is the average of the three extraction cycle runs (Runs 1, 4, and 5).

target analyte (PCB Aroclor 1254) was spiked into both a sample of the product solids and into a sample of the product water to determine the accuracy and precision for these matrices. The results of these matrix spike/matrix spike duplicate (MS/MSD) samples (Table 6) show that accuracy and precision were obtained for both matrix types and that the project quality assurance (QA) goals were met.

Field duplicate samples of raw feed, product solids, product oil, and product water were collected and analyzed for PCBs. Field duplicates provide a measure of precision for the combined sampling and analytical processes. Table 7 presents these results, which show that the project relative percent difference (RPD) goal of 40 was easily met.

It should also be mentioned that PCBs were not detected in any of the laboratory method blanks, nor in a rinsate sample collected from the pilot plant prior to the study, indicating that contamination was not a problem.

Conclusions

The analytical test data indicate that the primary goal of producing solids having a ≤ 1.0 mg/kg PCB concentration was not attained by the designated test method used. Of the five main test runs, the closest PCB concentration to the RAS was 1.8 mg/kg in Run 2 product solids, which was approximately a 99.3 percent removal efficiency. The average PCB removal efficiency attained for the three-extraction cycle test runs was 98%.

There is not enough PCB data to indicate whether the additional fifth extraction cycle conducted during Run 3 benefited

Table 5. Total Materials Balance

Run Number	Input (grams)			Output (grams)				(%) Recovery
	Feed Soil ¹	Water	TOTAL	Oil Extract	Slurry	F-1 Filter Solids	Total ³	Material
1	45,400	52,600	98,000	—	71,600	485	72,090	73.6
2	45,800	80,800	126,600	—	147,400	485	147,900	117 ²
3	45,800	93,800	139,600	—	116,200	485	116,700	83.6
4	45,800	88,500	134,300	—	134,800	640	135,400	101
5	45,800	99,500	145,300	—	141,200	640	141,800	97.6
6	45,800	103,000	148,800	3,700	158,800	640	163,100	110
Total	274,400	518,200	792,600	3,700	770,000	3,380	777,000	98

¹ Runs 2-6 include the addition of 454g of sand to fill void space in the extractor.

² Solids not flushed out in Run 1 exited at the end of Run 2.

³ Totals rounded to four significant digits.

Table 6. PCB Aroclor 1254 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Results

Sample	Spike	Sample Conc.	MS	MS % R	MSD	MDS % R ¹	RPD ²
Product Solids	4.9 mg/kg	4.1 mg/kg	8.2 mg/kg	84	9.1 mg/kg	102	10
Filtrate	10.0 µg/L	< 1.0 µg/L	9.0 µg/L	90	7.8 µg/L	78	14

¹ The QA objective for accuracy was a recovery of 50-150 percent.

$$\% \text{ Recovery} = \frac{C_1 - C_o}{C_i} \times 100$$

where,

*C*₁ is the measured concentration in the spiked sample
*C*_o is the measured concentration in the unspiked sample
*C*_i is the known concentration of analyte added to the sample

² The QA objective for precision was an RPD of ≤40.

$$RPD = \frac{(\text{Maximum Value} - \text{Minimum Value})}{(\text{Maximum Value} + \text{Minimum Value})/2} \times 100$$

Table 7. PCB Aroclor 1254 Field Duplicate Results

Sample Matrix	Sample Result	Result 2	RPD*
Feed	350 mg/kg	260 mg/kg	30
Product Solids	4.0 mg/kg	3.9 mg/kg	2.5
Product Oil	11,200 mg/kg	11,300 mg/kg	0.9
Filtrate	< 1 µg/L	< 1 µg/L	NC

* The project objective for precision was an RPD of ≤40.
 NC = Not calculated

PCB removal beyond the four-extraction cycles conducted during Run 2. The two concentration values, for Run 2 (1.8 mg/kg) and Run 3 (2.2 mg/kg), are essentially equal since they are within the range of field sampling and analytical error. However the O&G analytical results can be used to supplement the interpretation of results, with respect to organics removal in general. As Table 4 indicates, when the O&G data is evaluated, the five-extraction cycles used for Run 3 appears to have performed the best for overall organics removal.

The performance of the runs relative to one another is illustrated in Figures 3 and 4. These show the removal of PCBs and O&G, respectively, for each test run as the decline in contaminant concentration from starting feed to product solids as sloped lines. Both figures show the disparity in performance between test runs for the respective parameters, which may not be as apparent when simply looking at percent removal values. Figure 3 clearly shows that Runs 2, 3, and 4 came closer to the test objective, assuming a feed

concentration equal to the average of all runs (250 mg/kg). Their slopes essentially parallel one another. Figure 3 also shows that Runs 1 and 5 had an almost identical performance and that Run 6 had the poorest performance. For O&G removal, Figure 4 indicates that Run 3 produced the "cleanest" solids, while Runs 1, 2, 4, and 5 had similar performance. Again, Run 6 had the poorest performance, indicating that greater than two extraction cycles are required to achieve O&G removal efficiencies > 96%. These results suggest that the extraction process operating conditions could be further optimized to yield higher removal efficiencies than were identified in this study.

Another important conclusion that resulted from the study regarded the volume reduction of hazardous waste. Although the CFS solvent extraction process is not capable of destroying PCBs and other contaminants present in the STD soil (as is the case with solvent extraction technologies in general), it is a means of separating those contaminants from the soil, thereby reducing the volume of haz-

ardous waste that must be treated. This in turn reduces the cleanup costs involved. The cumulative mass of the wet contaminated feed soil for all six runs of the treatability study was approximately 274,000 g. The mass of the oily extract sampled at the completion of Run 6 was approximately 3,700 g. Therefore, the process reduced the overall mass of the contaminated material to 1.35% of its original waste mass. The volume of the feed soil [SG=1.34 g/mL and oil extract (SG=0.87 g/mL) were approximately 204 and 4.3 L, respectively. Therefore, the process reduced the overall volume of the contaminated material to 2.1% of its original waste volume. The highly concentrated oil extracted from the CFS process is either destroyed by incineration or chemical dechlorination.

The full report was submitted in fulfillment of Contract No. 68-C0-0048, Work Assignment No. 0-50, by Science Applications International Corporation under the sponsorship of the U.S. Environmental Protection Agency.

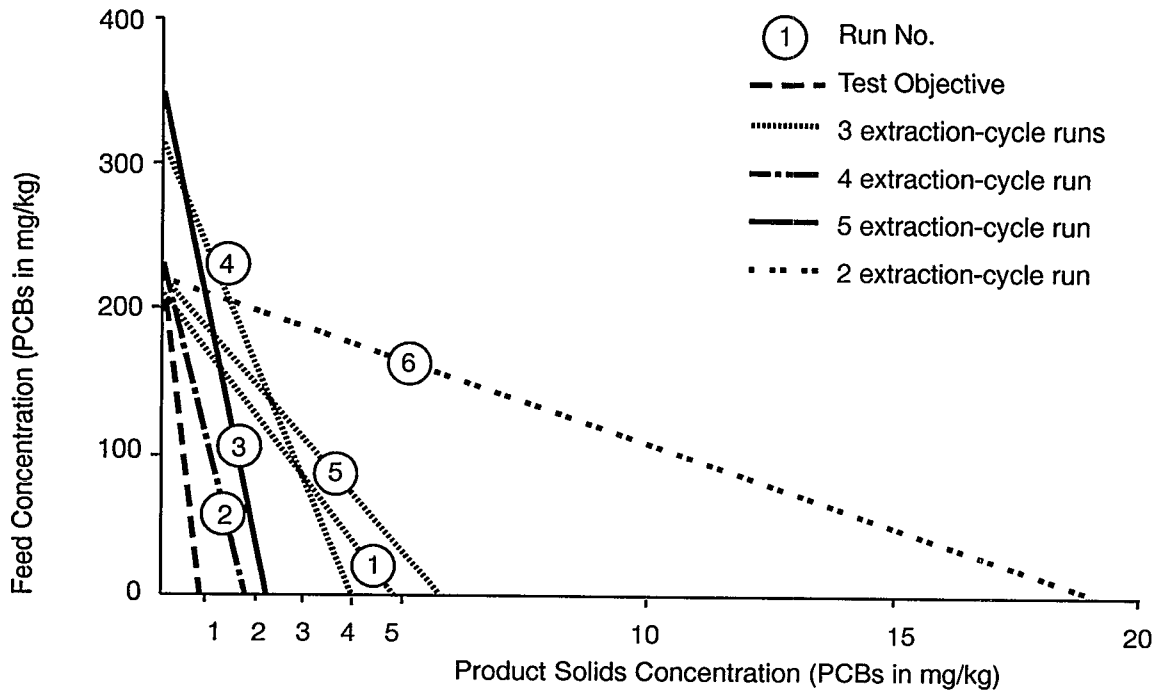


Figure 3. PCB removal trend.

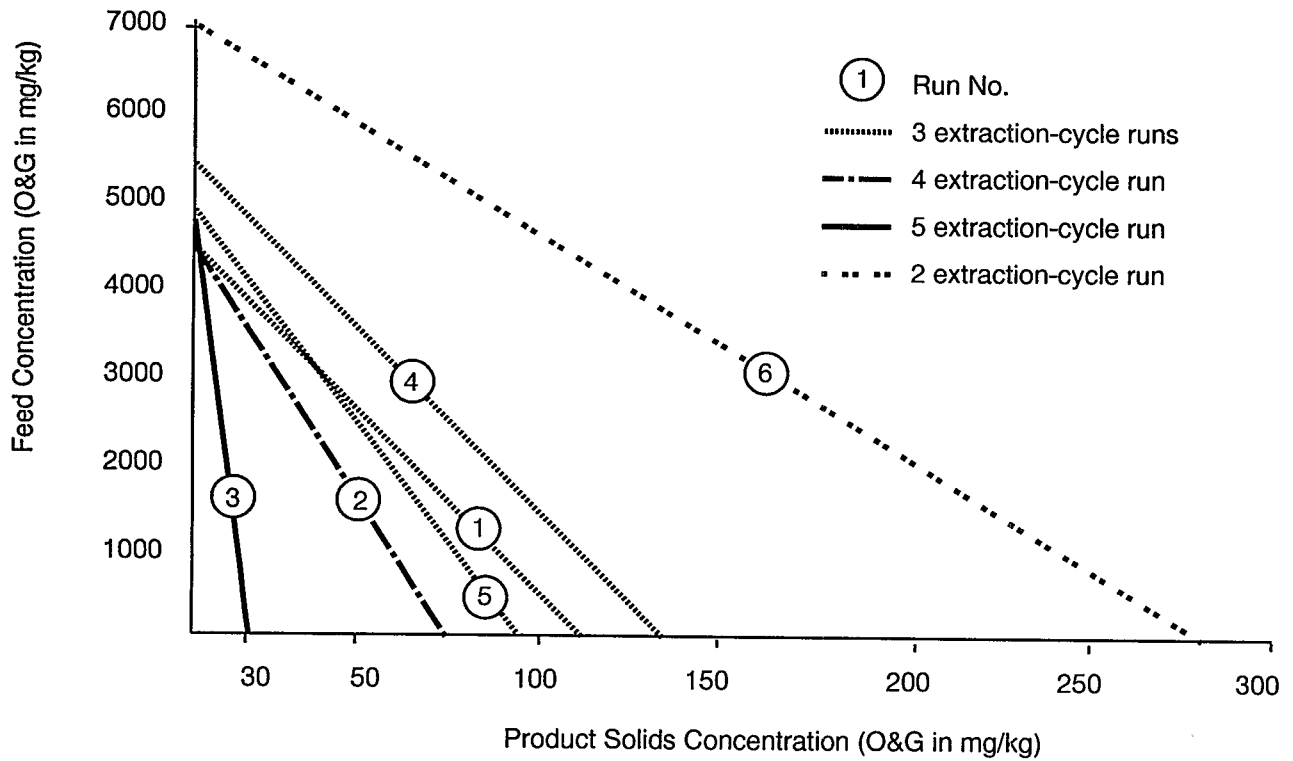
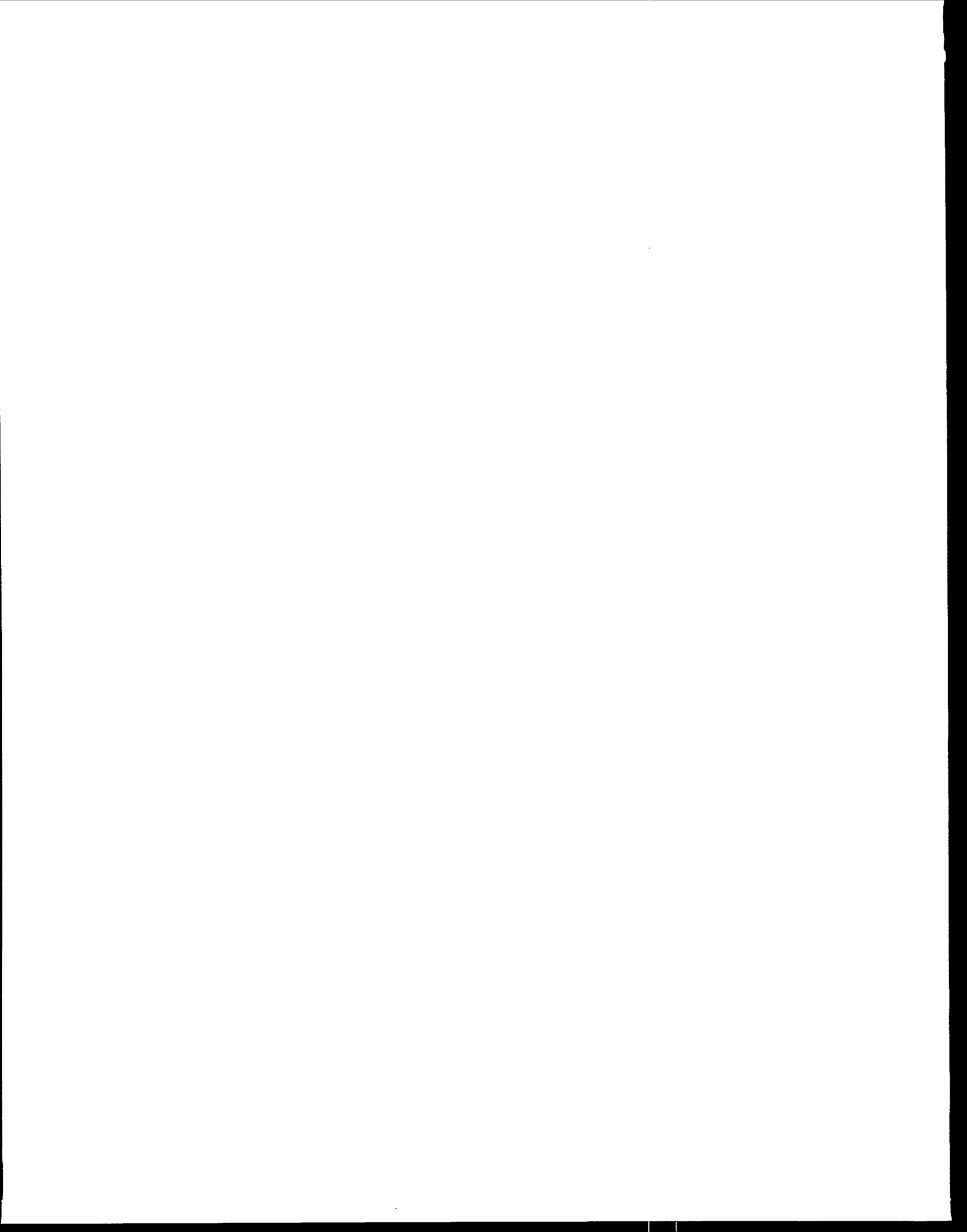


Figure 4. Oil & grease removal trend.



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Mark Meckes is the EPA Project Officer (see below).

The complete report (Order No. PB95-199030; Cost: \$19.50, subject to change) will be available only from:

National Technical Information Service

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The EPA Project Officer can be contacted at:

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