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Cooperative Testing Of Municipal Sewage Sludges By The Toxicity Characteristic Leaching Procedure And Compositional Analysis

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COOPERATIVE TESTING OF MUNICIPAL SEWAGE SLUDGES BY THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE AND COMPOSITIONAL ANALYSIS

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TABLE OF CONTENTS

ABSTRACT

The Toxicity Characteristic Leaching Procedure (TCLP) is a testing procedure that has been developed by the Office of Solid Waste (OSW) for determining whether or not solid wastes, including municipal sewage sludges, are hazardous based upon toxicity. This procedure was a proposed replacement for the Extraction Procedure (EP), used for this purpose since 1980. In the TCLP, the concentrations of analytes in the extracts are compared to Toxicity Characteristic (TC) regulatory levels. If concentrations of analytes in the TCLP extract meet or exceed these regulatory levels, the wastes are classified as hazardous.

In 1985-86, when these studies were conducted, it was felt that the proposed TCLP and TC regulatory levels might cause a number of municipal sewage sludges from Publicly Owned Treatment Works (POTWs) to be classified as hazardous. Hence, the Office of Water (OW), in cooperation with OSW, began testing municipal sewage sludges. Both total and TCLP fractions of the 18 sewage sludges were analyzed for selected analytes. The Association of Metropolitan Sewerage Agencies (AMSA) cooperated with EPA's OW and OSW in this study, analyzing split samples of sludges from 12 of the POTWs using identical analytical instructions sent by the EPA laboratory. Time and budget did not permit rigid policing of the AMSA laboratories to assure that they actually did use identical procedures.

None of the 18 sludges tested by any of the laboratories had TCLP extract concentrations that exceeded the proposed TC regulatory levels.

In the sludges studied, the volatile analytes were found to be the most likely class of contaminants that might cause them to be classified as hazardous, (i.e., three of 18 sludges had volatile TCLP analyte contents within less than an order of magnitude [one of the three was within a factor of three] of the proposed TC regulatory levels). However, because the final promulgated TC regulatory levels are, on average, two to three times higher than the proposed TC regulatory levels for the volatile toxic organic TCLP compounds, it would seem unlikely that the volatile compounds would result in any POTW sludges being classified as hazardous. Because the concentrations of the metal, semivolatile, pesticide, and herbicide constituents in analytes in TCLP extracts of the tested sewage sludges were lower than the respective TC regulatory levels by about one to two orders of magnitude, it would seem even less likely for these classes of contaminants to result in sludges being classified as hazardous.

For most contaminants except metals, there were non-detects in the TCLP extracts, and there were very few contaminants detected by both laboratories on the same sludge sample. Only for barium, p-cresol, and xylene did split sample analyses on the same sludge by the EPA and AMSA laboratories show detected measurements. There was substantial variation in the split sample results for barium with the level of barium detected by the EPA laboratory always being higher than detected by the AMSA laboratories. On the other hand, the variation in the split sample detects were less for p-cresol and xylene with no laboratory's results being consistently higher. The variation may have resulted because of subsample differences, sludge matrix interferences when using the SW-846 analytical protocol, or differences in the actual procedures used by the laboratories. The split sample results for barium would have to be viewed as questionable because of the large degree of consistently skewed variation.

When the concentrations of metals in TCLP and EP extracts were compared, there were no consistent differences in the amounts of a metal extracted. In general, the AMSA laboratories had lower reporting limits than did the EPA laboratory.

The 18 sludges came from POTWs that ranged in flow from less than 10 to over 600 million gallons per day (MGD) with less than one to over 90 percent of the flow being of industrial origin. The total compositional and TCLP extract contents of the proposed 52 TCLP analytes were not particularly high in these sludges. Some limited information is presented in the report about the various industrial pretreatment programs at the tested facilities. It is not known whether these industrial pretreatment programs had any bearing on the relatively low contents of analytes detected in the tested sludges.

The volatile contaminants benzene and chloroform that came closest to exceeding the respective TC regulatory levels were in a TCLP extract of a sludge from a smaller POTW. This POTW had a flow of about one million gallons per day (MGD) and less than one percent industrial flow.

One possible reason for the higher level of volatile analytes observed in the tested smaller POTW is that an insufficient volume of sludge was generated to dilute out occasional discharges of TC contaminants that might have occurred. Unfortunately our study did not include information for assessing how the TCLP analyte contents in the sludges were impacted by the type, size, and nature of the industries discharging to each POTW or by the type of wastewater and sludge treatment employed at each facility.

One important limitation of these studies is that only 18 of the more than 15,000 POTWs in the United States (US) were included in the study. Only one of the 18 tested POTW sludges came from a POTW that was close to one MGD in size. POTWs of less than one MGD in size constitute nearly 90% of all POTWs in the US. Another limitation is that the 18 POTWs were not selected in a manner that would allow statistically valid extrapolation of the results to the POTWs nationwide. However, the POTWs were selected on a basis of high to low hydraulic and industrial flow with the expectation that these parameters would be somewhat inclusive of wastewater inputs and resultant sludges that might cause the sludge to be classified as hazardous.

The analytical data were used to obtain a very rough estimate of the total content of contaminants in sludges that would result in TC regulatory level exceedance. These rough estimates can be calculated from the following formula: [(TC times 100)/(divided by the median percentage)] where the median percentage is derived from the fraction of the total analytes extracted by the TCLP within a class of compounds. While these estimating percentages are different within a class of extracted TCLP contaminants, the median percentages of the volatiles extracted were generally greatest at 30%, followed by metals at 0.03%, and semivolatiles, pesticides and herbicides at 0.01%. Because of the considerable variability in percentages of the different analytes extracted, additional TCLP testing would be needed where these estimating percentages, applied to the total compositional analyte contents of the TC contaminants in sludge, would predict a TCLP analyte content that was at all close (perhaps within an order of magnitude) to the TC regulatory level.

The cost impact upon small POTWs for testing could be substantial. The cost was about $1,200.00 to $1,500.00 (1988 dollars) for the complete analysis of a single sample without replication. Increased replication might be necessary and increase the cost for facilities if the TCLP extract contaminant levels were closer to the TC regulatory levels.

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INTRODUCTION

The Toxicity Characteristic Leaching Procedure (TCLP) is a testing procedure that has been developed by the Office of Solid Waste (OSW) for determining whether or not solid wastes, including municipal sewage sludges, are hazardous based upon toxicity. This procedure was a proposed replacement for the Extraction Procedure (EP), used for this purpose since 1980. Both procedures were designed to simulate leaching from a landfill under a mismanagement scenario (codisposal of wastes with municipal wastes in an unlined landfill).

The TCLP testing procedure was proposed as a method to extract and test wastes for hazardousness. The test compares the concentration of analytes in the extracts to Toxicity Characteristic (TC) regulatory levels. If concentrations of analytes in the TCLP extract meet or exceed these regulatory levels, the wastes are classified as hazardous. The TC regulatory levels had been proposed by the Environmental Protection Agency (EPA) to identify those wastes that contain certain toxic constituents at levels that can leach to groundwater and thereby pose a threat (hazard) to human health and the environment. The TC regulatory levels for toxic organic compounds were determined based upon chronic toxicity reference levels and compound specific dilution/attenuation factors, generated from a groundwater transport model.

Both the TC regulatory levels and the TCLP were proposed in the Federal Register on June 13, 1986, (51 FR 21648). This new proposed TC added 38 more toxic organic compounds than the 14 compounds included in the EP test. While this study evaluated the 52 elements for which TCs had been proposed, the rule was promulgated in final form on March 29, 1990, (55 FR 11798) with TC regulatory levels for only 25 additional toxic organic compounds rather than the 38 originally proposed. On June 29, 1990, (55 FR 26986) the TCLP was reformated to conform to the SW-846 method's format, including quality assurance and quality control (QA/QC) requirements. OSW plans to finalize the SW-846 QA/QC requirements in April 1991 across all methods, including the TCLP.

At the time the studies were conducted, it was felt that the proposed TCLP and TC regulatory levels could have a substantial impact on municipal sewage sludges from Publicly Owned Treatment Works (POTWs). Hence, the Office of Water (OW), in cooperation with OSW, began testing municipal sewage sludges. Six POTW sludges were tested in November 1985, followed by the testing of 12 additional sludges in May and June of 1986. The expanded testing program was undertaken because of the very limited number of POTWs sampled and the tentative nature of the initial test. The Association of Metropolitan Sewerage Agencies (AMSA) cooperated with EPA's OW and OSW in this expanded study, using identical analytical instructions sent by the EPA laboratory. Time and budget did not permit rigid policing of the AMSA laboratories to assure that they actually did use identical procedures.

This report describes the results of the testing of 12 sludges that occurred in 1986. It also summarizes the testing and discusses the results of the six-POTW sludge test conducted in 1985 and is updated by a summary to indicate the potential impacts on sludge management of the changes in the TCLP and TC rule that occurred from its proposed to final form.

METHODS AND MATERIALS

Sludge and POTW Characteristics

Samples of sewage sludge were collected for the expanded study by each of the 12 cooperating AMSA members using the procedure given in Appendix A. Sludges from each of these POTWs had the properties shown in Table 1. Sludge samples were split, with one split being sent to EPA's contract laboratory (S-CUBED*) and the second split being retained for analysis by the AMSA cooperator for 11 of the 12 POTWs who could arrange to either test the sludge themselves or have a contract laboratory do it.

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*Vendor and trade names are included solely for the benefit of the reader and do not imply endorsement by the U.S. Environmental Protection Agency.

TABLE 1. CHARATERISTICS OF THE TWELVE POTW'S AND THEIR SLUDGES IN THE 1986 EPA - AMSA TCLP AND COMPOSITIONAL TEST SERIES

POTW	Daily Flow Range, MGD	Percent Industrial Flow	Type of Wastewater Treatment	Sludge Parameters			
				Type of Treatment	Sampled from the	pH	% Water
G	30-60	50	Primary & waste activated	No digestion, lime & ferric vacuum filters	Filters, composite cake	12.2	79
H	100-150	35	Waste activated	No digestion ferric vacuum filters	Conveyor from filter discharge	5.8	83
I	Over 600	30	Primary & Waste activated	Anaerobically digested	Draw off from digestor	7.4	95
J	4-10	90	Waste activated extended air	Aerobically digested (60 days), belt filter press	Conveyor from the filter discharge	6.3	81
K	65-100	30	Waste activated	Anaerobically digested, lime, vacuum filters	Vacuum Filter cake before lime	4.6	82
L	70-100	40	Primary & pure oxygen	Anaerobically digested, polymer, centrifuged	Conveyor from the filter discharge	7.8	82

TABLE 1, Cont. CHARATERISTICS OF THE TWELVE POTW'S AND THEIR SLUDGES IN THE 1986 EPA-AMSA TCLP AND COMPOSITIONAL TEST SERIES

POTW	Daily Flow Range, MGD	Percent Industrial Flow	Type of Wastewater Treatment	Sludge Parameters			
				Type of Treatment	Sampled from the	pH	% Water
M	Over 600	30-40	Primary & waste activated	No digestion polymer belt filter press	Filter	7.0	77
N	40-60	25	Waste activated	No digestion, Lime & ferric Vacuum filter		10.7	78
O	275-325	5	Waste activated	Anaerobically digested	Storage	6.4	96
P	125-175	40	Primary	Polymer, vacuum filter, incineration	Bottom ash	8.3	0
Q	80-120	55	Primary & waste activated, pure oxygen	Low pressure oxidation-ZIMPRO, vacuum filter	Conveyor from the filter discharge	5.6	66
R	80-100	50	Waste activated	Anaerobically digested, polymer, belt filter press	Storage	6.6	70

For the most part, the attempt was to include a range of sewage sludges in the test program from POTWs that were expected to have higher levels of constituents and therefore might cause failure with respect to the TC. This higher constituent level and possible failure was expected because of the larger size of these POTWs and their type of industrial input. A second criterion for POTW selection was their willingness to cooperate by either testing the sludges themselves or having a contractor do it. Because of this second criterion, not all of the treatment facilities selected were expected to have higher levels of TC contaminants.

Analytical and QA/QC Procedures

The collected samples were analyzed by the EPA contract laboratory for the targeted 42 volatile, 67 semivolatile, 10 metal and 29 pesticide and herbicide compounds shown in Tables 2A through 2D. These compounds were selected for analysis by the EPA contract laboratory based upon (1) a consideration of the list of contaminants of interest in the TCLP, (2) the list of 40 CFR 261, Appendix VIII constituents recommended for analysis in "Guidance on Issuing Permits to Facilities Required to Analyze Groundwater for Appendix VIII Constituents" dated January 31, 1986, (3) "The 1986 Industrial Technology Division List of Analytes", and (4) the Superfund Contract Laboratory Program (CLP) list of analytes. Final analyte selections from these lists were then based upon (a) the likelihood that the compound would be present in a POTW sludge (given the compound's general level of commercial production and use), its water solubility, and detectability in previous studies of

TABLE 2A. VOLATILE ORGANICS GENERAL METHOD MEDIA REPORTING LIMITS* (SW-846 METHOD 8240)

Compound	Compositional Wet, mg/kg	TCLP mg/l
TCLP ANALYTES		
Acrylonitrile	0.20	0.010
Benzene	0.10	0.005
Carbon disulfide	0.10	0.005
Carbon tetrachloride	0.10	0.005
Chlorobenzene	0.10	0.005
Chloroform	0.10	0.005
1,2-Dichloroethane	0.10	0.005
1,1-Dichloroethylene (1,1-Dichloroethene)	0.10	0.005
Isobutanol (2-Methyl-1-propanol)	0.20	0.010
Methylene chloride	0.10	0.005
Methyl ethy ketone (2-Butanone)	0.20	0.010
Pyridine	0.20	0.010
1,1,1,2-Tetrachloroethane	0.20	0.010
1,1,2,2-Tetrachloroethane	0.10	0.005
Tetrachloroethylene (Tetrachloroethene)	0.10	0.005
Toluene	0.10	0.005
1,1,1-Trichloroethane	0.10	0.005
1,1,2-Trichloroethane	0.10	0.005
Trichloroethylene (Trichloroethene)	0.10	0.005
Vinyl chloride	0.20	0.010
NON-TCLP ANALYTES		
Bromidichloromethane	0.10	0.005
2-Chloro-1,3-butadiene	0.20	0.010
Chloroethane	0.20	0.010
3-Chloropropene	0.20	0.010
Dibromochloromethane	0.10	0.005
1,2-Dibromoethane	0.20	0.010
trans-1,4-Dichloro-2-butene	0.20	0.010
1,1-Dichloroethane	0.10	0.005
trans-1,2-Dichloroethene	0.10	0.005
1,2,-dichloropropane	0.10	0.005
trans-1,3-Dichloropropene	0.10	0.005
cis-1,3-Dichloropropene	0.10	0.005
Diethylether	0.20	0.010
Ethyl acetate	0.20	0.010
Ethylbenzene	0.10	0.005
2-Hexanone	0.20	0.010
Methacrylonitrile	0.20	0.010
4-Methyl-2-pentanone	0.20	0.010
Styrene	0.10	0.005
1,2,3-Trichloropropane	0.20	0.010
Vinly acetate	0.20	0.010
Total xylenes	0.10	0.005

*S-Cubed Laboratory

TABLE 2B. SEMIVOLATILE ORGANICS GENERAL METHOD MEDIAN REPORTING LIMITS* (SW-846 METHOD 827C)

Compound	Compositional Wet, mg/kg	TCLP mg/l
<u>TCLP ANALYTES</u>		
bis(2-chloroethyl)ether	1.3	0.01
0-Cresol(2-Methylphenol)	1.3	0.01
m-Cresol(3-Methylphenol)	1.3	0.01
p-Cresol(4-Methylphenol)	1.3	0.01
1,2-Dichlorobenzene	1.3	0.01
1,4-Dichlorobenzene	1.3	0.01
2,4-Dinitrotoluene	1.3	0.01
Hexachlorobenzene	1.3	0.01
Hexachlorobutadiene	1.3	0.01
Hexachloroethane	1.3	0.01
Nitrobenzene	1.3	0.01
Pentachlorophenol	6.4	0.05
Phenol	1.3	0.01
2,3,4,6-Tetrachlorophenol	2.6	0.01
2,4,5-Trichlorophenol	6.4	0.05
2,4,6-Trichlorophenol	1.3	0.01
<u>NON-TCLP ANALYTES</u>		
Acenaphthene	1.3	0.01
Acenaphthylene	1.3	0.01
Aniline	6.4	0.02
Anthracene	1.3	0.01
Benzo(a)anthracene	1.3	0.01
Benzo(a)pyrene	1.3	0.01
Benzo(b)fluoranthene	1.3	0.01
Benzo(g,h,i)perylene	1.3	0.01
Benzo(k)fluroanthene	1.3	0.01
bis(2-chloroethoxy)methane	1.3	0.01
bis(2-chloroisopropyl)ether	1.3	0.01
bis(2-ethylhexyl)phthalate	1.3	0.01
4-Bromophenyl phenylether	1.3	0.01
Butylbenzlphthalate	1.3	0.01
4-Chloroaniline	1.3	0.01
4-Cloro-3-methylphenol	1.3	0.01
2-Chloronaphthalene	1.3	0.01
2-Chlorophenol	1.3	0.01
4-chlorophenyl phenylether	1.3	0.01
Chrysene	1.3	0.01
Dibenzacridine	1.3	0.01
Dibenz(a,h)anthracene	1.3	0.01
1,3-Dichlorobenzene	1.3	0.01

*S-Cubed Laboratory

TABLE 2B Cont. SEMIVOLATILE ORGANICS GENERAL METHOD MEDIAN REPORTING LIMITS* (SW-846 METHOD 8270)

Compound	Compositional Wet, mg/kl	TCLP mg/l
<u>NON-TCLP ANALYTES continued</u>		
2,4-Dichlorophenol	1.3	0.01
2,6-Dichlorophenol	6.4	0.01
Diethyl phthalate	1.3	0.01
Dimethyl phthalate	1.3	0.01
2,4-Dimethylphenol	1.3	0.01
Di-n-butylphthalate	1.3	0.01
4,6-Dinitro-2-methylphenol	6.4	0.05
2,4-Dinitrophenol	6.4	0.05
2,6-Dinitrotoluene	1.3	0.01
Di-n-octyl phthalate	1.3	0.01
Dipnenylamine	1.3	0.01
Fluoranthene	1.3	0.01
Fluorene	1.3	0.01
Hexachlorocyclopentadiene	1.3	0.01
Indeno(1,2,3-cd)pyrene	1.3	0.01
Isophorone	1.3	0.01
2-Methylnaphthalene	1.3	0.01
Naphthalene	1.3	0.01
2-Nitrophenol	1.3	0.01
4-Nitrophenol	6.4	0.01
Pentachloroethane	6.4	0.01
Phenanthrene	1.3	0.01
2-Picoline	1.3	0.01
Pyrene	1.3	0.01
1,2,4,5-Tetrachlorobenzene	6.4	0.01
2,3,5,6-Tetrachlorophenol	1.3	0.01
1,2,3-Trichlorobenzene	1.3	0.01
1,2,4-Trichlorobenzene	1.3	0.01

*S-Cubed Laboratory

TABLE 2C. METALS ANALYSIS GENERAL METHOD MEDIAN REPORTING LIMITS*

Compound	SW-846W Method	Compositional Dry, mg/kg	TCLP, mg/l	EP, mg/l
<u>TCLP ANALYTES</u>				
Arsenic	7060	4.3	0.25	0.10
Barium	7080	15	0.90	0.90
Cadmium	7130	5.1	0.10	0.10
Chromium	7190	16	0.33	0.33
Lead	7420	4.0	0.62	0.62
Mercury	7441/7440	1.4	0.01	0.01
Selenium	7740	2.7	0.10	0.10
Silver	7760	2.8	0.09	0.09
<u>NON-TCLP-ANALYTES</u>				
Nickel	7520	16	0.22	0.22
Thallium	7840	20	0.43	0.43

*S-Cubed Laboratory

TABLE 2D. PESTICIDES AND CHLORINATED HERBICIDES GENERAL METHOD MEDIAN REPORTING LIMITS* (SW-846 METHOD 8080 & 8150, RESPECTIVELY)

Compound	Compositional Wet, mg/kg	TCLP, mg/l	EP, mg/l
TCLP ANALYTES			
Chlordane	1.1	0.001	0.001
Endrin	0.21	0.2	0.0002
Heptachlor	0.11	0.0001	0.0001
Lindane (gamma-HC)	0.11	0.0001	0.0001
Methoxychlor	1.1	0.001	0.001
Toxaphene	2.1	0.002	0.002
2,4-D	0.02	0.02	0.02
2,4,5-TP (Silvex)	0.02	0.02	0.02
NON-TCLP ANALYTES			
Aldrin	0.11	0.001	0.0001
Aroclor-1016	1.1	0.001	0.001
Aroclor-1221	1.1	0.001	0.001
Aroclor-1232	1.1	0.001	0.001
Aroclor-1242	1.1	0.001	0.001
Aroclor-1248	1.1	0.001	0.001
Aroclor-1254	2.1	0.002	0.002
Aroclor-1260	2.1	0.002	0.002
alpha-BHC	0.11	0.0001	0.0001
beta-BHC	0.11	0.0001	0.0001
delta-BHC	0.11	0.0001	0.0001
4,4-DDD	0.21	0.2	0.0002
4,4-DDE	0.21	0.2	0.0002
4,4-DDT	0.21	0.2	0.0002
Dieldrin	0.21	0.2	0.0002
Endosulfan I	0.11	0.0001	0.0001
Endosulfan II	0.21	0.2	0.0002
Endosulfan sulfate	0.21	0.2	0.0002
Endrin aldehyde	0.05	0.2	0.0002
Heptachlor epoxide	0.11	0.0001	0.0001
2,4,5-T	0.02	0.02	0.02

*S-Cubed Laboratory

POTW wastewaters and sludges; (b) the compound's general level of toxicity; (c) the capability to effectively and quantitatively analyze for the compound, including availability of standards; and (d) the cost of the analyses and the experience and capability of most contract laboratories to perform the analyses specified for the POTWs. All of the originally proposed 52 TCLP contaminants were included in the target compound lists.

Analyses were run on the TCLP extracts and total digests (total compositional content) of each sewage sludge (on a dry weight basis). The purpose of running a compositional analysis was to determine if there were any direct relationship between total content of the various toxic constituents in the sludge and in the amount of the constituent extracted from the sludge by the TCLP.

The detailed analytical procedures used are contained in Table 3 and in reference (1). The sample analyses were also subjected to the QA and QC procedures contained in reference (2) and summarized later in this report (See also Appendix B).

RESULTS AND DISCUSSION

Volatiles

Results of the analytical determinations of the total compositional and TCLP study are contained in Tables 4A to 8B. Only those non-TCLP

TABLE 3. STANDARD ANALYTICAL PROCEDURES*

LEACHING

LEACHING TECHNIQUE	REFERENCE METHOD
Extraction Procedure (EP Toxicity)	1310 (SW-846)
Toxicity Characteristic Leaching Procedure (including Zero Headspace Extraction)	Federal Register Vol. 51 No. 9, Appendix 1

SAMPLE PREPARATION

ANALYTE	METHOD	REFERENCE METHOD (SW-846)
Metals (compositional)- Flame and furnace AAS Analyses	Acid Digestion of Sludge	3050
Metals (leachate samples)- Flame AAS analyses	Acid Digestion of Leachate	3010
Metals (leachate samples)- Furnace AAS analyses	Acid Digestion of Leachate	3020
Mercury (compositional)	Cold Vapor Analysis Preparation	7471
Mercury (leachate samples)-	Cold Vapor Analysis Preparation	7470
Semivolatile Organic Compounds**(compositional)	Sonication/Solvent Extraction	3550
Semivolatile Organic Compounds**(leachate Samples)	Continuous Liquid/Liquid Extraction	3520
Volatile Organic Compounds- (compositional)	Purge and Trap	5030
Volatile Organic Compounds- (leachate samples)	Purge and Trap	5030

*From Table by S-CUBED, A Division of Maxwell Laboratories, Inc.

**Increases Organocholrine pesticides and herbicides, PCB's and base-neutral/acid extractable compounds.

TABLE 3 Cont. STANDARD ANALYTICAL PROCEDURE

METALS ANALYSES

ANALYTE	METHOD	REFERENCE METHOD (SW-845)
Arsenic	Furance AAS	7060
Barium	Flame AAS	7080
Cadmium	Flame AAS	7130
Chromium (Total)	Flame AAS	7190
Lead	Flame AAS	7420
Mercury (compositional)	Cold Vapor AAS	7441
Mercury (leachate)	Cold Vapor AAS	7440
Nickel	Flame AAS	7520
Selenium	Furnace AAS	7740
Silver	Flame AAS	7760
Thallium	Flame AAS	7840

ORGANIC COMPOUNDS ANALYSES

ANALYTE	METHOD	REFERENCE METHOD (SW-845)
Organochlorine Pesticides and PCB's	Gas Chramatography/ Electron Capture Detection	8080
Chlorinated Phenoxy Acid Herbicides	Derivatization; Gas Chromatography/Electron Capture Detection	8150
Volatile Organic Compounds*	Gas Chromatography/Mass Spectrometry	8240
Semivolatile Organic Compounds* (Base Neutral/Acid Extractables)	Gas Chromatography/Mass Spectrometry	8270

*Analysis conducted on sludge (compositional) and TCLP Leachate only.

analytes that were found to be above the reporting limits* of any of the laboratories are shown in these tables. The results are presented as actual data, unless the constituents present were at levels below the reporting limits. The median reporting limits for the TCLP and compositional analyses are given in Tables 2A through 2D for the EPA contract laboratory and the actual reporting limits for all the AMSA laboratories or their contractors are given in Appendix C.

In general, there were more volatile TCLP analytes reported by the AMSA contract and/or AMSA POTW laboratories compared to the EPA contract laboratory (Table 4A and 4B). This was largely because of the respective higher reporting limits for the EPA contract laboratory discussed elsewhere in this report. There also were fewer volatile compositional analytes reported by the EPA contract laboratory compared with AMSA.

The levels of these volatile constituents were generally quite low, but are the class of compounds that are most likely to cause sludges to exceed the TC. As reported for Cities "A" and "E" in Appendix D, Table D-3), their sludges came close to exceeding the proposed TC because of the volatile constituents chloroform and benzene in the TCLP extract.

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*Reporting limits are defined as concentration levels below which, there is not good confidence in the result. This reporting limit is for the concentration levels determined during the analysis of the given samples under practical and routine laboratory conditions. Reporting limits differ from detection limits. Detection limits are those concentration levels below which the compound can not be detected under ideal laboratory conditions.

TABLE 4A. VOLATILE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGES COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/l	Concentration of Volatiles in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
		G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Acrylonitrile	5.0	*	*	*	*	*	*	*	**	*	*	*	*
Benzene	0.07	*	*	*	*	*	*	*	**	*	*	*	*
Carbon disulfide	14.4	*	*	*	*	*	0.0002	*	**	*	*	0.34	*
Carbon tetrachloride	0.07	*	*	*	*	*	**	*	**	*	*	*	*
Chlorobenzene	1.4	*	*	*	*	*	*	*	**	*	*	0.009	0.01
Chloroform	0.07	*	*	*	*	*	0.0019	*	**	*	*	*	*
1,2,-Dichloroethane	0.40	*	*	*	*	*	*	*	**	*	*	*	*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.1	*	*	*	*	*	**	*	**	*	*	*	*
Isobutanol (aka§ 2-Methyl-1-propanol)	36	*	*	*	*	*	**	*	**	*	*	*	*
Methylene chloride	8.6	*	*	*	*	*	0.0092	*	**	*	*	*	*
Methyl ethyl ketone (aka§ 2-Butanone)	7.2	*	*	0.53	0.85	*	0.0165	*	**	2.2+	1.3+	*	*
Pyridine	5.0	*	*	*	*	*	*	*	**	*	*	*	
1,1,1,2, Tetra-chloroethane	10.0	*	*	*	*	*	*	*	**	*	*	*	*
1,1,2,2, Tetra-chlorethane	1.3	*	*	*	*	*	*	*	**	*	*	*	*
Tetrachloroethylene (aka Tetrachloroethene)	0.1	*	*	*	*	*	**	*	**	*	*	*	*
Toluene	14.4	0.016	0.029	*	*	0.005	0.0008	*	**	0.95	0.16	*	0.003
1,1,1-Trichloro-ethane	30	*	*	*	*	*	*	*	**	*	*	*	*
1,1,2-Trichloro-ethane	1.2	*	*	*	*	*	*	*	**	*	*	*	*
Trichloroethylene (aka Trichloroethene)	0.07	*	*	*	*	*	**	*	**	*	*	*	*
Vinyl Chloride	0.05	*	*	*	*	*	*	*	**	*	*	*	*

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As
+ = Close to the Toxicity Characteristic Regulatory Level

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

TABLE 4A Cont. VOLATILE ANALYTE CONCENTRATION IN TCLP EXTRACTS OF SLUDGE

Constituent	Concentration of Volatiles Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acetone	**	*	**	1.4	**	0.11	**	**	**	1.7	**	**
Dibromomethane	**	*	**	**	**	**	**	**	**	**	**	**
1,1-Dichloroethane	*	**	*	**	*	**	*	**	*	**	*	*
Ethylbenzene	*	0.0085	*	**	*	0.0006	*	**	*	*	*	0.002
2, Hexanone	*	**	*	**	*	**	*	**	*	**	*	**
Methyl isobutyl ketone (aka§ 4-methyl-2-pentanone)	*	*	*	**	*	0.0048	*	**	*	**	*	**
Styrene	*	**	*	**	*	**	*	**	*	**	*	*
Trans 1,2-dichlorethene	*	**	*	*	*	**	*	**	*	**	*	*
Xylene	0.016	0.050	*	*	*	0.0045	*	**	*	**	0.01	0.013

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 4A cont. VOLATILE ANALYTE CONCENTRATION IN TCLP EXTRACTS OF SLUDGES COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory Level, mg/l ++	Concentration of Volatiles in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
		M (35)		N (35)		O (5)		P (40)		Q (55)		R (50)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Acrylonitrile	5.0	*	*	*	*	*	*	*	*	*	*	*	*/*
Benzene	0.07	*	*	*	*	*	*	*	*	*	*	*	*/**
Carbon disulfide	14.4	0.053	*	*	0.0028	*	*	*	*	*	*	0.03	0.011/**
Carbon tetrachloride	0.07	*	*	*	*	*	*	*	*	*	*	*	*/*
Chlorobenzene	1.4	*	*	*	*	*	*	*	*	*	*	*	*/*
Chloroform	0.07	*	*	*	0.0026	*	*	*	0.0053	*	*	*	*/*
1,2,-Dichloroethane	0.40	*	*	*	*	*	*	*	*	*	*	*	*/*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.1	*	*	*	*	*	*	*	*	*	*	*	*/*
Isobutanol (aka§ 2-Methyl-1-proponal)	36	*	*	*	*	*	*	*	*	*	*	*	*/**
Methylene chloride	8.6	*	*	*	0.018	*	*	*	0.026	*	*	*	*/0.004
Methyl ethyl ketone (aka§ 2-Butanone)	7.2	*	0.25	*	*	*	0.4	*	*	*	0.26	*	*/**
Pyridine	5.0	*	*	*	*	*	*	*	*	*	*	*	*/**
1,1,1,2, Tetra-chloroethane	10.0	*	*	*	*	*	*	*	*	*	*	*	*/*
1,1,2,2, Tetra-chlorethane	1.3	*	*	*	*	*	*	*	*	*	*	*	*/*
Tetrachloroethylene (aka Tetrachloroethene)	0.1	*	*	*	0.0075	*	*	*	*	*	*	*	*/*
Toluene	14.4	*	0.032	*	0.004	*	0.027	*	*	0.005	*	0.005	0.016/0.013
1,1,1-Trichloro-ethane	30	0.010	0.027	*	*	*	*	*	*	*	*	*	*/*
1,1,2-Trichloro-ethane	1.2	*	*	*	*	*	*	*	*	*	*	*	*/0.002
Trichloroethylene (aka Trichloroethene)	0.07	*	*	0.009	0.0079	*	*	*	*	*	*	*	*/*
Vinyl Chloride	0.05	*	*	*	*	*	*	*	*	*	*	*	*/**

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

TABLE 4A Cont. VOLATILE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGES

Constituent	Concentration of Volatiles in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acetone	**	1.5	**	0.082	**	*	**	*	**	1.1	**	0.31/**
Dibromomethane	**	*	**	*	**	*	**	**	**	*	**	*/**
1,1-Dichloroethane	*	*	*	*	*	*	*	*	*	*	*	*/**
Ethylbenzene	*	*	*	*	*	*	*	*	*	*	*	*/**
2, Hexanone	*	*	*	*	*	*	*	*	*	*	*	*/**
Methyl isobutyl ketone (aka§ 4-methyl-2-pentanone)	*	*	*	*	*	*	*	*	*	*	*	*/**
Styrene	*	*	*	0.0032	*	*	*	**	*	*	*	*/**
Trans 1,2 dichlorethene	0.034	0.051	*	*	*	*	*	**	*	*	*	*/*
Xylene	0.025	0.041	0.012	0.012	0.006	*	*	*	0.01	*	0.008	*/*

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 4B. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGE

Constituent	Concentration of Volatile Analytes in POTW Sludge (% Industrial Flow), mg/kg G (50) EPA	G (50) AMSA	H (35) EPA	H (35) AMSA	I (30) EPA	I (30) AMSA	J (90) EPA	J (90) AMSA	K (30) EPA	K (30) AMSA	L (40) EPA	L (40) AMSA
TCLP ANALYTES												
Acrylonitrile	*	*	*	*	*	*	*	**	*	*	*	*
Benzene	*	0.056	*	*	*	0.011	*	**	*	*	*	*
Carbon disulfide	*	*	*	*	*	0.035	*	**	*	*	*	*
Carbon tetrachloride	*	*	*	*	*	*	*	**	*	*	*	*
Chlorobenzene	*	*	*	*	*	0.031	*	**	*	*	*	0.92
Chloroform	*	*	*	*	*	*	*	**	*	*	*	*
1,2,-Dichloroethane		*	*	*	*	*	*	**	*	*	*	*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	*	*	*	*	*	**	*	**	*	*	*	*
Isobutanol (aka§ 2-Methyl-1-proponal)	*	*	*	*	*	**	*	**	*	*	*	*
Methylene chloride	*	0.20	*	*	*	0.026	*	**	*	*	*	3.22
Methyl ethyl ketone (aka 2-Butanone)	*	0.70	*	*	*	0.33	*	**	*	*	*	*
Pyridine	*	*	*	*	*	*	*	**	*	*	*	*
1,1,1,2, Tetra-chloroethane	*	*	*	*	*	*	*	**	*	*	*	*
1,1,2,2, Tetra-chlorethane	*	*	*	*	*	*	*	**	*	*	*	*
Tetrachlorethylene (aka Tetrachloroethene)	*	0.39	*	*	*	**	*	**	*	*	*	*
Toluene	0.74	3.82	0.18	*	0.28	0.75	0.23	**	*	100	*	0.39
1,1,1-Trichloro-ethane	*	*	*	*	*	*	*	**	*	*	*	*
1,1,2-Trichloro-ethane	*	*	*	*	*	*	*	**	*	*	*	*
Trichloroethylene (aka Trichloroethene)	*	0.15	*	*	*	**	*	**	*	*	*	*
Vinyl Chloride	*	*	*	*	*	0.022	*	**	*	*	*	*

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 4B Cont. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGES

Constituent	Concentration of Volatiles Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acetone	**	2.05	**	*	**	0.82	**	**	**	*	**	**
Dibromomethane	**	*	**	**	**	0.048	**	**	**	**	**	**
1,1-Dichloroethane	*	*	*	*	*	**	*	**	*	*	*	*
Ethylbenzene	0.31	1.58	*	*	*	0.074	*	**	*	*	*	0.33
2, Hexanone	*	*	*	*	*	*	0.40	**	*	*	*	**
Methyl isobutyl ketone (aka§ 4-methyl-2-pentanone)	*	*	*	*	*	0.91	*	**	*	*	*	**
Styrene	*	*	*	*	*	*	*	**	*	*	*	2.2
Trans 1,2-dichlorethene	*	*	*	**	*	**	*	**	*	**	*	
Xylene	1.78	8.84	*	*	*	0.28	*	**	*	*	*	2.2

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 4B cont. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGE

Constituent	Concentration of Volatile Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Acrylonitrile	*	*	*	*	*	*	*	*	*	*	*	*/**
Benzene	*	*	*	*	*	0.22	*	*	*	*	*	*/*
Carbon disulfide	*	0.043	*	*	*	*	*	0.022	*	*	*	*/*
Carbon tetrachloride	*	*	*	*	*	*	*	*	*	*	*	*/**
Chlorobenzene	*	*	*	*	*	1.0	*	*	*	*	*	*/*
Chloroform	*	*	*	0.10	*	*	*	*	*	*	*	*/0.03
1,2,-Dichloroethane	*	*	*	*	*	*	*	*	*	*	*	*/*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene	*	*	*	*	*	*	*	*	*	*	*	*/*
Isobutanol (aka§ 2-Methyl-1-propanol)	*	*	*	*	*	*	*	*	*	*	*	*/*
Methylene chloride	*	0.14	*	0.039	*	*	*	0.25	*	*	*	0.20/*
Methyl ethyl ketone (aka 2-Butanone)	*	0.43	*	0.25	*	3.1	*	*	*	*	*	*/**
Pyridine	*	*	*	*	*	*	*	*	*	*	*	*/**
1,1,1,2, Tetra-chloroethane	*	*	*	*	*	*	*	*	*	*	*	*/*
1,1,2,2, Tetra-chlorethane	*	*	*	*	*	*	*	*	*	*	*	*/*
Tetrachlorethylene (aka Tetrachloroethene)	*	0.50	*	0.74	*	*	*	*	*	2.5	*	*/*
Toluene	0.15	3.2	*	0.33	0.19	1.2	0.43	0.018	0.29	2.5	*	0.40/1.9
1,1,1-Trichloro-ethane	*	*	*	0.12	*	*	*	0.060	*		*	*/*
1,1,2-Trichloro-ethane	*	*	*	*	*	*	*	*	*		*	*/*
Trichloroethylene (aka Trichloroethene)	*	0.28	*	0.69	*	*	*	*	*		*	*/*
Vinyl Chloride	*	*	*	*	*	*	*	*	*		*	*/**

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 4B Cont. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGE

Constituent	Volatile Analyte Concentration in POTW Sludge (% Industrial Flow), mg/kg											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acetone	**	*	**	1.0	**	11.0	**	0.23	**	26.4	**	*/**
Dibromomethane	**	**	**	**	**	**	**	*	**	**	**	*/**
1,1-Dichloroethane	*	0.074	*	*	*	*	*	*	*	*	*	*/**
Ethylbenzene	*	1.16	*	0.25	*	0.47	*	*	*	0.89	*	0.14/0.75
2, Hexanone	*	*	*	*	*	*	*	*	*	*	*	*/**
Methyl isobutyl ketone (aka§ 4-methyl-2-pentanone)	*	*	*	*	*	*	*	*	*	*	*	*/**
Styrene	*	*	*	0.26	*	*	*	*	*	1.2	*	*/**
Trans 1,2 dichlorethene	*	3.8	*	0.11	*	1.1	*	*	*	*	*	*/*
Xylene	*	6.6	*	1.2	0.006	3.3	*	*	*	3.9	*	0.47/**

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

Also, City "K" sludge came within a factor of three of exceeding the TC regulatory level because of the volatile constituent methyl ethyl ketone (MEK) (Table 4A). The concentration of MEK in the TCLP extract of sludge from City "K" was 1.3 to 2.2 mg/l, depending upon the analytical laboratory (compared with the proposed regulatory level of 7.2 mg/l). (Note: The final promulgated TCs were higher (Table 16) and the chance for TC exceedance is, therefore, less).

Values reported for volatile TCLP analytes by EPA differed from the values reported by AMSA laboratories by as much as six-fold [e.g., for identical split samples of sludge from Cities "I" and "K" analyzed for toluene (Table 4A)]. The differences might actually be higher, but it is difficult to tell because of all the analytical results that were below the reporting limits. Such variability would make it difficult to accurately determine that any given analysis is accurately indicating that the tested sludge has passed or failed the TC when the resultant concentration of the given analyte in the TCLP extract is close to the regulatory level. It might be that the variability is less when the level of the constituent is present in higher concentration, such as for MEK in City "K" sludge rather than for toluene in Cities "I" and "K" sludges. However, too little data was available to make such an assessment.

The total compositional level of TCLP volatile analytes in sludge are mostly in the 0.02 to 4 mg/kg range on a dry weight basis. There was as much as a 21-fold variation between the results obtained by the two separate laboratories doing the analyses on their separate splits of identical sludge samples (for example, for Cities "M" and "P" for toluene in Table 4B).

Semivolatiles

The results of the semivolatile TCLP and compositional analyses were similar to those of the volatile analyses with respect to uniformity among laboratories (Tables 5A and 5B). However, there were far fewer semivolatile TCLP or compositional analytes detected at reportable levels. Furthermore, the TCLP analyte concentrations were quite low compared to the TC regulatory levels.

There was up to an eight-fold difference in the amount of semivolatile analytes in the TCLP extract reported by the EPA and AMSA laboratories for their respective identical splits of the same sample (for example, for City "L" in Table 5A for phenol). This eight-fold variation for semivolatile TCLP analytes is less important than the six-fold variation for volatile TCLP analytes with respect to exceedance of the TC for sewage sludge, because the overall level of semivolatile constituents in sludges is so far below the proposed and final TC regulatory levels.

The total compositional level of TCLP semivolatile analytes in sludge are mostly in the 0.5 to 15 mg/kg range on a dry weight basis (Table 5B). There were a number of non-TCLP analytes detected where the

TABLE 5A. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TOXICITY THRESHOLDS LEVELS

Constituent	Toxicity Characteristic Regulatory Level, mg/l ++	Concentration of Semivolatile Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Bis(2-chloroethyl) ether	0.05	*	*	*	*	*	*	*	**	*	*	*	*
o-Cresol (aka§ 2-Methyl Phenol)	10	*	*	0.07	*	*	*	*	**	*	*	*	*
m-Cresol (aka 3-Methyl Phenol)	10	*	0.75	*	*	*	*	*	**	*	*	*	*
p-Cresol (aka§ 4-Methyl Phenol)	10	*		0.26	0.23	*	*	*	**	0.17	0.26	*	*
1,2 Dichlorobenzene	4.3	*	*	*	*	*	*	*	**	*	*	*	*
1,4 Dichlorobenzene	10.8	*	*	*	*	*	*	*	**	*	*	*	*
2,4 Dinitrotoluene	0.13	*	*	*	*	*	*	*	**	*	*	*	*
Hexachlorobenzene	0.13	*	*	*	*	*	*	*	**	*	*	*	*
Hexachlorobutadiene	0.72	*	*	*	*	*	*	*	**	*	*	*	*
Hexachloroethane	4.3	*	*	*	*	*	*	*	**	*	*	*	*
Nitrobenzene	0.13	*	*	*	*	*	*	*	**	*	*	*	*
Pentachlorophenol	3.6	*	*	*	*	*	*	*	**	*	*	*	*
Phenol	14.4	*	*	*	*	*	0.0008	*	**	0.02	*	0.13	0.017
2,3,4,6-Tetrachlorophenol	1.5	*	*	*	*	*	*	*	**	*	*	*	*
2,4,5-Trichlorophenol	5.8	*	*	*	*	*	*	*	**	*	*	*	*
2,4,6-Trichlorophenol	0.30	*	*	*	*	*	*	*	**	*	*	*	*

* = Below Reporting Limits

§ = Also Known As

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

TABLE 5A cont. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TOXICITY THRESHOLDS LEVELS

Constituent	Concentration of Semivolatile Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acenaphthene	*	**	*	**	*	**	*	**	*	**	*	*
Acenaphthylene	*	**	*	**	*	**	*	**	*	**	*	*
Anthracene	*	**	*	**	*	**	*	**	*	**	*	*
Benzoic Acid	**	**	**	**	**	**	**	**	**	**	**	**
Benzo (a) -anthracene	*	**	*	**	*	**	*	**	*	**	*	*
Benzo (b) -fluoranthene	*	**	*	**	*	**	*	**	*	**	*	*
Bis (2-Ethylhexal) phthalate	*	**	*	**	*	**	*	**	*	**	*	*
Butyl benzyl phthalate	*	**	*	**	*	**	*	**	*	**	*	0.017
4-Chloroanaline	*	**	*	**	*	**	*	**	*	**	*	**
Chrysene	*	**	*	**	*	**	*	**	*	**	*	*
Diethyl phthlate	*	**	*	**	*	**	*	**	*	**	*	*
Di-n-butyl phthalate	*	**	*	**	*	**	*	**	*	**	*	0.16
Di-n-octyl phthalate	*	**	*	**	*	**	*	**	*	**	*	*
Fluoranthene	*	**	*	**	*	**	*	**	*	**	*	*
Fluorene	*	**	*	**	*	**	*	**	*	**	*	*
Indeno (1,2,3,-cd) pyrene	*	**	*	**	*	**	*	**	*	**	*	*
2-Methylnapthalene	*	**	*	**	*	**	*	**	*	**	*	**
Napthalene	*	**	*	**	*	**	*	**	*	**	*	0.002
Phenanthrene	*	**	*	**	*	**	*	**	*	**	*	**
Pyrene	*	**	*	**	*	**	*	**	*	**	*	*

* = Below Reporting Limits
** = Not Analyzed

TABLE 5A cont. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TOXICITY THRESHOLDS LEVELS

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/l	Concentration of Semivolatile Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
		M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Bis(2-chloroethyl) ether	0.05	*	*	*	*	*	*	*	*	*	*	*	*/*
o-Cresol													
(aka§ 2-Methyl Phenol)	10	*		*	*	*		*	*	0.28		*	*/0.0023
m-Cresol													
(aka§ 3-Methyl Phenol)	10	*	0.68	*	*	*	1.9	*	*	*	0.19	*	*/
p-Cresol													0.0008
(aka§ 4-Methyl Phenol)	10	1.5		*	*	0.39		*	*	1.5		*	*/
1,2 Dichlorobenzene	4.3	*	*	*	*	*	*	*	*	*	*	*	*/0.0003
1,4 Dichlorobenzene	10.8	*	*	*	*	*	*	*	*	*	*	*	*/0.0002
2,4 Dinitrotoluene	0.13	*	*	*	*	*	*	*	*	*	*	*	*/*
Hexachlorobenzene	0.13	*	*	*	*	*	*	*	*	*	*	*	*/*
Hexachlorobutadiene	0.72	*	*	*	*	*	*	*	*	*	*	*	*/*
Hexachloroethane	4.3	*	*	*	*	*	*	*	*	*	*	*	*/0.004
Nitrobenzene	0.13	*	*	*	*	*	*	*	*	*	*	*	*/*
Pentachlorophenol	3.6	*	*	*	*	*	*	*	*	*	*	*	*/*
Phenol	14.4	*	*	*	*	0.39	*	*	*	0.08	*	*	*/0.019
2,3,4,6-Tetrachlorophenol	1.5	*	*	*	*	*	*	*	*	*	*	*	*
2,4,5-Trichlorophenol	5.8	*	*	*	*	*	*	*	*	*	*	*	*
2,4,6-Trichlorophenol	0.30	*	*	*	*	*	*	*	*	*	*	*	*
NON-TCLP ANALYTES													
Bis (2-Ethyl hexal) phthalate		*	*	*	*	0.39	*	*	*	*	*	*	*/0.002
Butyl benzyl phthalate		*	*	*	*	*	*	*	*	*	*	*	*/*
Diethyl phthlate		*	*	*	*	*	*	*	*	*	*	*	*/*
Di-n-butyl phthalate		*	*	*	*	*	*	*	*	*	*	*	*/0.0005
Di-n-octyl phthalate		*	*	*	*	*	*	*	*	*	*	*	*/*
Napthalene		*	*	*	*	*	*	*	*	*	*	*	*/0.0012

* = Below Reporting Limits
§ = Also Known As

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

laboratory reporting limits were lower (e.g., for City "R" in Table 5A for one of the two AMSA laboratories and City "I's" AMSA laboratory in Table 5B).

Metals

The results of the TCLP and total compositional metal analyses, obtained from the laboratories doing the work for AMSA and EPA, are presented in Tables 6A and 6B. The reported metal TCLP analyte concentrations for the EPA contract laboratory were consistently higher than those reported by the AMSA cooperators and POTW laboratories by a factor of up to ten. This amount of variation could be very critical if the levels of TCLP constituents were any closer to the TC regulatory levels.

The total compositional metal concentrations were much closer in value between both laboratories (generally within a factor of three). Also, the EPA laboratory compositional metal analyte concentrations were neither consistently higher nor lower than those reported by the AMSA laboratories. The compositional TCLP metal concentrations were mostly in the 1 to 2000 mg/kg range on a dry weight basis.

Extraction Procedure (EP) and TCLP extractions and analyses were also run on each sludge sample by the EPA contract laboratory. The results of the EP and TCLP metal extract analyses were then compared (Table 7). Often, where there was a reportable determination for the

TABLE 5B. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Semivolatile Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Bis(2-chloroethyl) ether	*	*	*	*	*	*	*	**	*	*	*	*
o-Cresol (aka§ 2-Methyl Phenol)	*	*	0.07	*	*		*	**	*	*	*	*
m-Cresol (aka 3-Methyl Phenol)	9.5		*	*	*	1.31	*	**	*	*	*	*
p-Cresol (aka§ 4-Methyl Phenol)	*	0.021	0.26	11.3	*		*	**	*	*	*	5.6
1,2 Dichlorobenzene	*	*	*	*	*	*	*	**	*	*	*	0.67
1,4 Dichlorobenzene	*	*	*	*	*	0.31	*	**	*	*	*	1.0
2,4 Dinitrotoluene	*	*	*	*	*	*	*	**	*	*	*	*
Hexachlorobenzene	*	*	*	*	*	*	*	**	*	*	*	*
Hexachlorobutadiene	*	*	*	*	*	*	*	**	*	*	*	*
Hexachloroethane	*	*	*	*	*	*	*	**	*	*	*	*
Nitrobenzene	*	*	*	*	*	*	*	**	*	*	*	*
Pentachlorophenol	*	*	*	*	*	*	*	**	*	*	*	*
Phenol	*	*	*	*	*	0.60	*	**	*	*	*	14.4
2,3,4,6-Tetrachlorophenol	*	*	*	*	*	*	*	**	*	*	*	*
2,4,5-Trichlorophenol	*	*	*	*	*	*	*	**	*	*	*	*
2,4,6-Trichlorophenol	*	*	*	*	*	*	*	**	*	*	*	*

* = Below Reporting Limits
** = Not Analyzed
§ = Also Known As

TABLE 5B cont. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Semivolatile Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
NON-TCLP ANALYTES												
Acenaphthene	*	*	*	*	*	0.49	*	**	*	*	*	*
Acenaphthylene	*	*	*	*	*	0.48	*	**	*	*	*	*
Anthracene	*	*	*	*	*	0.46	*	**	*	*	*	*
Benzoic Acid	**	*	**	*	**	0.66	**	**	**	*	**	**
Benzo(a)-anthracene	*	*	*	*	*	1.43	*	**	*	*	*	*
Benzo(b)-fluoranthene	*	*	*	*	*	2.64	*	**	*	*	*	*
Bis (2-Ethyl hexal) phthalate	*	65	*	17.0	*	94.3	*	**	*	*	*	*
Butyl benzyl phthalate	*	*	*	*	*	0.91	*	**	*	*	*	*
4-Chloroanaline	*	*	*	*	*	0.70	*	**	*	*	*	**
Chrysene	*	*	*	*	*	2.44	*	**	*	*	*	*
Diethyl phthlate	*	*	*	*	*	**	*	**	*	*	*	0.56
Di-n-butyl phthalate	*	*	*	*	*	**	*	**	*	*	*	1.9
Di-n-octyl phthalate	2.7	8	*	*	*	0.98	*	**	*	*	*	13.0
Fluoranthene	*	*	*	*	*	3.47	*	**	*	*	*	*
Fluorene	*	*	*	*	*	0.43	*	**	*	*	*	*
Indeno(1,2,3,-cd)pyrene	*	*	*	*	*	1.35	*	**	*	*	*	*
2-Methylnapthalene	*	*	*	*	*	0.55	*	**	*	*	*	**
Napthalene	*	*	*	*	*	1.01	*	**	*	*	*	13.0
Phenanthrene	*	*	*	*	*	2.56	*	**	*	*	*	**
Pyrene	*	*	*	*	*	4.37	*	**	*	*	*	*

* = Below Reporting Limits
** = Not Analyzed

TABLE 5B cont. SEMIVOLATILE ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Semivolatile Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Bis(2-chloroethyl) ether	*	*	*	*	*	*	*	*	*	*	*	*/*
o-Cresol (aka§ 2-Methyl Phenol)	12		*	*	*		*	*	*		*	*/*
m-Cresol (aka§ 3-Methyl Phenol)	*	*	*	*	*	1500	*	*	*	*	*	*/*
p-Cresol (aka§ 4-Methyl Phenol)	58		*	*	*		*	*	12		*	*/
1,2 Dichlorobenzene	*	*	*	*	*	*	*	*	*	*	*	*/*
2,4 Dichlorobenzene	*	*	*	*	*	*	*	*	*	*	*	*/*
2,4 Dinitrotoluene	*	*	*	*	*	*	*	*	*	*	*	*/*
Hexachlorobenzene	*	*	*	*	*	*	*	*	*	*	*	*/0.28
Hexachlorobutadiene	*	*	*	*	*	*	*	*	*	*	*	*/0.18
Hexachloroethane	*	*	*	*	*	*	*	*	*	*	*	*/0.8
Nitrobenzene	*	*	*	*	*	*	*	*	*	*	*	*/*
Pentachlorophenol	*	*	*	*	*	*	*	*	*	*	*	*/*
Phenol	2.6	*	*	*	*	*	*	*	*	*	*	*/*
2,3,4,6-Tetrachlorophenol	*	*	*	*	*	*	*	*	*	*	*	*/*
2,4,5-Trichlorophenol	*	*	*	*	*	*	*	*	*	*	*	*/*
2,4,6-Trichlorophenol	*	*	*	*	*	*	*	*	*	*	*	*/*
NON-TCLP ANALYTES												
Bis (2-Ethyl hexal) phthalate	*	*	*	47	*	340	*	*	*	680	*	*/12.29
Butyl benzyl phthalate	*	*	*	*	*	*	1.3	*	*	*	*	*/*
Diethyl phthlate	*	*	*	*	*	*	*	*	*	*	*	*/*
Di-n-butyl phthalate	*	*	*	*	*	*	*	*	*	*	*	*/*
Di-n-octyl phthalate	*	*	*	*	*	*	*	*	*	*	*	*/*
Napthalene	1.9	*	*	*	*	*	*	*	*	*	*	*/0.2

* = Below Reporting Limits
§ = Also Known As

TABLE 6A. METAL ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory Level, mg/1 ++	Concentration of Metals in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/1											
		G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Arsenic	5.0	*	0.005	0.12	*	*	*	*	**	*	*	*	*/*
Barium	100	2.6	0.2	*	0.6	3.3	0.3	1.6	**	3.9	0.8	2.5	0,04/0.07
Cadmium	1.0	*	0.2	*	0.04	*	0.17	*	**	*	*	*	*/*
Chromium	5.0	*	0.2	*	0.3	*	0.08	*	**	*	*	*	0.02/0.02
Lead	5.0	*	0.21	*	*	*	*	*	**	*	*	*	*/*
Mercury	0.2	*	0.0032	*	*	*	*	*	**	*	*	*	0.0002/0.0008
Selenium	1.0	*	0.001	*	*	*	*	0.23	**	0.15	*	0.23	**/**
Silver	5.0	*	1.0	*	*	*	0.01	*	**	*	*	*	0.02/*
NON-TCLP ANALYTES													
Nickel		*	0.6	0.24	0.22	*	0.5	*	**	*	*	*	0.04/0.04
Thallium		*	**	*	*	*	**	*	**	*	*	*	*/0.007

* = Below Reporting Limits
** = Not Analyzed
++ = Proposed and Final Regulatory Levels are the Same for Metals

TABLE 6A cont. METAL ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/l	Concentration of metals in TCLP Extracts POTW Sludge (% Industrial Flow), mg/l											
		M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Arsenic	5.0	*	*	*	*	*	*	*	0.31	*	*	*	**/0.0016
Barium	100	1.6	0.57	1.6	0.65	2.0	0.70	0.98	0.10	0.99	0.24	2.3	**/*
Cadmium	1.0	0.12	0.10	*	0.043	*	0.025	*	0.041	0.14	0.21	*	**/*
Chromium	5.0	*	*	*	*	*	*	*	*	*	*	*	**/*
Lead	5.0	*	*	*	*	*	*	*	*	*	*	*	**/0.002
Mercury	0.2	*	*	*	*	*	*	*	*	*	*	*	**/*
Selenium	1.0	*	*	*	*	*	*	*	0.047	*	*	*	**/*
Silver	5.0	*	*	*	*	*	*	*	*	*	*	*	**/0.0003
NON-TCLP ANALYTES													
Nickel		0.81	0.68	2.9	3.0	0.28	0.39	*	**	*	0.27	0.76	**/0.096
Thallium		*	*	*	*	*	*	*	**	*	*	*	**/**

* = Below Reporting Limits
** = Not Analyzed
++ = Proposed and Final Regulatory Levels are the Same for Metals

TABLE 6B. METAL ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Metals in POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Arsenic	4.5	2.3	4.9	6.0	15	*	44	**	*	5	16	13/12
Barium	548	229	334	180	537	600	390	**	458	340	480	**/**
Cadmium	24	46	32	20	95	160	*	**	21	17	26	26/26
Chromium	340	368	2520	4700	1415	1300	300	**	196	180	229	270/260
Lead	99	115	319	180	283	344	*	**	92	100	211	260/290
Mercury	*	1.9	*	1.0	*	0.02	*	**	*	0.6	*	6,2/5.8
Selenium	*	1.4	*	*	*	*	*	**	*	*	*	2,6/3,6
Silver	*	46	*	8.0	4.8	0.22	*	**	*	12	13	57/62
NON-TCLP ANALYTES												
Nickel	60	92	100	60	162	110	119	**	*	12	56	75/73
Thallium	*	**	*	*	*	**	*	**	*	*	*	*/*

* = Below Reporting Limits
** = Not Analyzed

TABLE 6B cont. METAL ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Metals in POTW Sludge (% Industrial Flow), mg/kg											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Arsenic	21	7.4	*	*	12	*	52	25	16	*	21	**/ 4.0
Barium	514	370	424	170	334	520	751	337	627	360	570	**/2240
Cadmium	131	82	6.6	10	54	61	15	16	305	360	*	**/ 2.3
Chromium	384	400	161	240	6200	2870	109	104	574	600	178	**/ 165
Lead	556	550	326	310	2010	2250	257	255	519	500	131	**/ 155
Mercury	*	1.1	*	1.5	*	4.6	*	*	*	4.6	*	**/ 3.7
Selenium	8.6	13	*	*	3.5	*	*	*	9.4	9.8	*	**/ 0.37
Silver	13	29	12	29	*	230	8.9	16	11	27	4.7	**/ 24.5
NON-TCLP ANALYTES												
Nickel	194	190	423	500	388	470	59	70	863	580	337	**/382
Thallium	*	*	*	*	*	*	*	**	*	*	*	**/**

* = Below Reporting Limits
** = Not Analyzed

TABLE 7. COMPARISON OF TCLP AND EP ANALYTE CONCENTRATIONS IN POTW SEWAGE SLUDGE FROM TWELVE CITIES AS DETERMINED BY THE DIFFERENT LABORATORIES

Constituent	City TCLP and EP Analyte Extract Concentration, mg/l																			
	G				H			I			J			K			L			
	AMSA		EPA		AMSA	EPA		AMSA	EPA		AMSA	EPA		AMSA	EPA		AMSA		EPA	
	TCLP	EP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	TCLP	EP
Arsenic	0.005	0.005	*	0.2	*	*	0.12	*	*	*	**	*	*	*	*	*	*	*	*	*
Barium	0.20	0.20	2.6	2.6	0.60	2.0	*	0.30	3.3	*	**	1.6	*	0.8	3.9	*	0.04	0.07	2.5	1.4
Cadmium	0.20	0.20	*	0.11	0.04	*	*	0.17	*	*	**	*		*	*	*	*	*	*	*
Chromium	0.20	0.20	*	*	0.30	*	*	0.08	*	*	**	*	*	*	*	*	0.02	0.02	*	*
Lead	0.21	0.16	*	*	*	*	*	*	*	*	**	*	*	*	*	*	*	*	*	*
Mercury	0.0032	0.001	*	*	*	*	*	*	*	*	**	*	*	*	*	*	0.0002	0.0008	*	*
Selenium	0.001	*	*	*	*	*	*	*	*	*	**	0.23	*	*	0.15	*	**	**	0.23	*
Silver	1.0	1.0	*	*	*	*	*	*	*	*	**	*	*	*	*	*	0.02	*	*	*
NON-TCLP ANALYTES																				
Nickel	0.60	*	*	0.30	0.22	0.24	*	0.05	*	*	**	*	*	*	*	*	0.04	0.04	*	*
Thallium	**	**	*	*	*	*	*	**	*	*	**	*	*	*	*	*	*	0.007	*	*

* = Below Reporting Limits
** = Not Analyzed

TABLE 7 cont. COMPARISON OF TCLP AND EP ANALYTE CONCENTRATIONS IN POTW SEWAGE SLUDGE FROM TWELVE CITIES AS DETERMINED BY THE DIFFERENT LABORATORIES

Constituent	City TCLP and EP Analyte Extract Concentrations, mg/l																	
	M			N			O			P			Q			R		
	AMSA	EPA		AMSA	EPA		AMSA	EPA		AMSA	EPA		AMSA	EPA		AMSA	EPA	
	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP	TCLP	TCLP	EP
TCLP ANALYTES																		
Arsenic	*	*	*	*	*	*	*	*	*	0.31	*	0.43	*	*	*	0.0016	*	*
Barium	0.57	1.6	*	0.65	1.6	*	0.70	2.0	*	0.10	0.98	*	0.24	0.99	*	0.24	2.3	*
Cadmium	0.10	0.12	*	0.043	*	*	0.025	*	*	0.041	*	*	0.21	0.14	*	*	*	*
Chromium	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
Lead	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	0.002	*	*
Mercury	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
Selenium	*	*	*	*	*	*	*	*	*	0.047	*	*	*	*	*	*	*	*
Silver	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	0.0003	*	*
NON-TCLP ANALYTES																		
Nickel	0.68	0.81	0.36	3.0	2.9	3.0	0.39	0.28	*	**	*	*	0.27	*	*	0.096	0.76	*
Thallium	*	*	*	*	*	*	*	*	*	**	*	*	*	*	*	*	*	*

* = Below Reporting Limits
** = Not Analyzed

TCLP, the EP was below reporting limits. However, there were no consistent differences in amounts of metals extracted by the TCLP or the EP.

Pesticides and Herbicides

The results of the TCLP and total compositional pesticide and herbicide analyses were similar in most ways to those of the semivolatile analyses (Tables 8A and 8B). The total compositional concentration of TCLP pesticide and herbicide analytes in this study are in the 0.1 to 10 mg/kg range on a dry weight basis. Also, it can be concluded similarly for sludge pesticide, herbicide, semivolatile, and metal analytes that their TCLP extract concentrations are one to two orders of magnitude below the TC regulatory levels. It was not possible to determine the variation in the amount of a specific pesticide or herbicide constituent detected in a split sample of a given sludge analyzed by the two laboratories, since essentially all measurements by the EPA contract laboratory were below the reporting limits.

Pretreatment Status of the POTWs

The POTWs selected for this study mostly served larger communities. The industrial contributors to many of these facilities were thought to be of a nature that might cause the resultant sludges to have higher levels of TCLP analytes. This was especially true in the EPA-AMSA cooperative 12 POTW study. The overall study, however, also included a

TABLE 8A. PESTICIDE AND HERBICIDE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TOXICITY THRESHOLDS LEVELS

Constituent	Toxicity Characteristic Regulatory Level, mg/1 ++	Concentration of Pesticide and Herbicide Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l — G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Chlordane	0.03	*	*	*	*	*	0.00047	*	**	*	*	*	*
Endrin	0.003	*	*	*	*	*	0.00002	*	**	*	*	*	*
Heptachlor	0.001	*	*	*	*	*	*	*	**	*	*	*	*
Lindane (gamma-BHC)	0.06	*	*	*	*	*	*	*	**	*	*	*	*
Methoxychlor	1.4	*	*	*	*	*	*	*	**	*	*	*	*
Toxaphene	0.07	*	*	*	*	*	*	*	**	*	*	*	*
2,4-D	1.4	*	*	*	*	*	*	*	**	*	*	*	*
2,4,5,TP (Silvex)	0.14	*	*	*	*	*	*	*	**	*	*	*	*
NON-TCLP ANALYTES													
Aldrin		*	*	*	*	*	*	*	**	*	*	*	*
alpha-BHC		*	*	*	*	*	*	*	**	*	*	*	*
beta-BHC		*	*	*	*	*	*	*	**	*	*	0.0001	*
delta-BHC		*	*	*	*	*	*	*	**	*	*	*	*
Chlordene "237"		**	**	**	**	**	**	**	**	**	**	**	**
2,4-DB		**	*	**	*	**	**	**	**	**	**	**	**
4,4'-DDD		*	*	*	*	*	*	*	**	*	*	*	*
4,4'-DDT		*	*	*	*	*	*	*	**	*	*	*	*
Dinoseb		**	*	**	*	**	*	**	**	**	**	**	**
Endosulfan II		*	*	*	*	*	*	*	**	*	*	*	*
Endrin ketone		**	**	**	**	**	**	**	**	**	**	**	**
Hexachloronobornadiene		**	**	**	**	**	**	**	**	**	**	**	**
Heptachlornobornene		**	**	**	**	**	**	**	**	**	**	**	**
MCPP		**	*	**	*	**	**	**	**	**	**	**	**
Octachlorocyclopentene		**	**	**	**	**	**	**	**	**	**	**	**
PCB-1248		*	*	*	*	*	**	*	**	*	*	*	*
PCB-1254		*	*	*	*	*	**	*	**	*	*	*	*

* = Below Reporting Limit
** = Not Analyzed

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

TABLE 8A cont. PESTICIDE AND HERBICIDE ANALYTE CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TOXICITY THRESHOLDS LEVELS

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/1	Concentration of Pesticide and Herbicide Analytes in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/l											
		M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
		EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES													
Chlordane	0.03	*	*	*	*	*	*	*	*	*	*	*	*/*
Endrin	0.003	*	*	*	*	*	*	*	*	*	*	*	*/0.0002
Heptachlor	0.001	*	*	*	*	*	*	*	*	*	*	*	*/*
Lindane (gamma-BHC)	0.06	*	*	*	*	*	*	*	*	*	*	*	*/*
Methoxychlor	1.4	*	*	*	*	*	*	*	*	*	*	*	*/*
Toxaphene	0.07	*	*	*	*	*	*	*	*	*	*	*	*/**
2,4-D	1.4	*	*	*	*	*	*	*	*	*	*	*	*/**
2,4,5,TP (Silvex)	0.14	*	*	*	*	*	*	*	*	*	*	*	*/**
NON-TCLP ANALYTES													
Aldrin		*	*	*	*	*	*	*	*	*	*	*	*/*
alpha-BHC		*	*	*	*	*	*	*	*	*	*	*	*/0.0001
beta-BHC		*	*	*	*	0.0001	*	*	*	*	*	*	*/0.0001
delta-BHC		*	*	*	*	*	*	*	*	*	*	*	*/*
Chlordene "237"		**	**	**	**	**	**	**	**	**	**	**	**/0.0003
2,4-DB		**	**	**	**	**	**	**	*	**	**	**	**/**
4,4'-DDD		*	*	*	*	*	*	0.12	*	*	*	*	*/0.0001
4,4'-DDT		*	*	*	*	*	*	*	*	*	*	*	*/*
Dinoseb		**	**	**	**	**	**	**	*	**	**	*	**/**
Endosulfan II		*	*	*	*	*	*	*	*	*	*	*	*/*
Endrin ketone		**	**	**	**	**	**	**	**	**	**	**	**/0.0004
Hexachloronobornadiene		**	**	**	**	**	**	**	**	**	**	**	**/0.0001
Heptachlornobornene		**	**	**	**	**	**	**	**	**	**	**	**/0.0002
MCPP		**	**	**	**	**	**	**	*	**	**	**	**/**
Octachlorocyclopentene		**	**	**	**	**	**	**	**	**	**	**	**/*
PCB-1248		*	*	*	*	*	*	*	*	*	*	*	*/**
PCB-1254		*	*	*	*	*	*	*	*	*	*	*	*/**

* = Below Reporting Limits
** = Not Analyzed

++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

TABLE 8B. PESTICIDE AND HERBICIDE ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Pesticide and Herbicide Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	G (50)		H (35)		I (30)		J (90)		K (30)		L (40)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Chlordane	*	*	*	*	*	0.65	*	**	*	0.43	*	*
Endrin	*	*	*	*	*	0.057	*	**	*	*	*	*
Heptachlor	*	*	*	*	*	*	*	**	*	*	*	*
Lindane (gamma-BHC)	*	*	*	*	*	0.013	*	**	*	*	*	*
Methoxychlor	*	*	*	*	*	*	*	**	*	*	*	*
Toxaphene	*	*	*	*	*	*	*	**	*	*	*	*
2,4-D	*	*	*	1.6	*	*	*	**	*	8.9	*	*
2,4,5,TP (Silvex)	*	*	*	0.46	*	*	*	**	*	5.8	*	*
NON-TCLP ANALYTES												
Aldrin	*	*	*	*	*	*	*	**	*	*	*	*
alpha-BHC	*	*	*	*	*	*	*	**	*	*	*	*
beta-BHC	*	*	*	*	*	*	*	**	*	*	*	*
delta-BHC	*	*	*	*	*	*	*	**	*	*	*	*
Chlordene "237"	**	**	**	**	**	**	**	**	**	**	**	**
2,4-DB	**	*	**	*	**	**	**	**	**	6.8	**	**
4,4'-DDD	*	*	*	*	*	*	*	**	*	*	*	*
4,4'-DDT	*	*	*	*	*	0.19	*	**	*	*	*	*
Dinoseb	**	*	**	*	**	*	**	**	**	3.8	**	**
Endosulfan II	*	*	*	*	*	*	*	**	*	*	*	*
Endrin ketone	**	**	**	**	**	**	**	**	**	**	**	**
Hexachloronobornadiene	**	**	**	**	**	**	**	**	**	**	**	**
Heptachlornobornene	**	**	**	**	**	**	**	**	**	**	**	**
MCPP	**	*	**	*	**	**	**	**	**	28.9	**	**
Octachlorocyclopentene	**	**	**	**	**	**	**	**	**	**	**	**
PCB-1248	*	*	*	*	*	1.36	*	**	*	*	*	*
PCB-1254	*	*	*	*	*	0.53	*	**	*	*	*	*

* = Below Reporting Limits

TABLE 8B cont. PESTICIDE AND HERBICIDE ANALYTE CONCENTRATIONS IN TOTAL SLUDGE

Constituent	Concentration of Pesticide and Herbicide Analytes in POTW Sludge (% Industrial Flow), mg/kg											
	M (35)		N (25)		O (5)		P (40)		Q (55)		R (50)	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Chlordane	*	*	*	*	*	*	*	*	*	*	*	*/*
Endrin	*	*	*	*	*	*	*	*	*	*	*	*/*
Heptachlor	*	*	*	*	*	*	*	*	*	*	*	*/*
Lindane (gamma-BHC)	*	*	*	*	*	*	*	*	*	*	*	*/0.36
Methoxychlor	*	*	*	*	*	*	*	*	*	*	*	*/*
Toxaphene	*	*	*	*	*	*	*	*	*	*	*	*/**
2,4-D	*	*	*	*	*	*	*	*	*	*	*	*/**
2,4,5,TP (Silvex)	*	*	*	*	*	*	*	*	*	*	*	*/**
NON-TCLP ANALYTES												
Aldrin	*	*	*	*	*	*	*	*	*	*	*	*/1.65
alpha-BHC	*	*	*	*	*	*	*	*	*	*	*	*/*
beta-BHC	*	*	*	*	*	*	*	*	*	*	*	*/0.34
delta-BHC	*	*	*	*	*	*	*	*	*	*	*	*/*
Chlordene "237"	**	**	**	**	**	**	**	**	**	**	**	**/3.94
2,4-DB	**	**	**	**	**	**	**	*	**	**	**	**/**
4,4'-DDD	*	*	*	*	*	*	0.12	*	*	*	*	*/0.68
4,4'-DDT	*	*	*	*	*	*	*	*	*	*	*	*/*
Dinoseb	**	**	**	**	**	**	**	*	**	**	*	**/**
Endosulfan II	*	*	*	*	*	*	*	*	*	*	*	*/0.72
Endrin ketone	**	**	**	**	**	**	**	**	**	**	**	**/**
Hexachloronobornadiene	**	**	**	**	**	**	**	**	**	**	**	**/*
Heptachlornobornene	**	**	**	**	**	**	**	**	**	**	**	**/2.49
MCPP	**	**	**	**	**	**	**	*	**	**	**	**/**
Octachlorocyclopentene	**	**	**	**	**	**	**	**	**	**	**	**/2.07
PCB-1248	*	*	*	*	*	*	*	*	*	*	*	*/**
PCB-1254	*	*	*	*	*	*	*	*	*	*	*	*/**

* = Below Reporting Limits
** = Not Analyzed

few facilities (especially in the six POTW study) with less than 1% industrial input. These facilities were thought to produce "domestic" sludges with low levels of TCLP analytes.

The results, however showed that "domestic" sludge from the smallest facility (City "A" with less than 1% industrial input and 10 MGD flow, Table D-2 in Appendix D) came closest of all the POTWs studied to exceeding the proposed TC regulatory levels. The concentrations of both benzene and chloroform in the TCLP extract of City "A" sludge were within a factor of three or less of the respective proposed TC regulatory levels (Table D-3 in Appendix D). This result was in sharp contrast to the very low level of TCLP analytes found in the "domestic" sludge from City "B", which also had less than 1% industrial input, but a flow of over 300 MGD.

Postulated reasons for the striking differences in City "A" and "B" sludge TCLP analyte concentration were (a) differences in pretreatment programs, (b) differences in the type of industrial input, (c) differences in the type of treatment, and/or (d) the fact that the smaller facility lacked sufficient flow to dilute occasional discharges of TCLP contaminants.

Investigation revealed that pretreatment differences were apparently not the reason. Table 9 shows the pretreatment status of all 18 POTWs. Both Cities "A" and "B" have only begun to implement pretreatment programs, while most of the other facilities have had

TABLE 9. PRETREATMENT STATUS OF COOPERATING POTWs

Local Program	new 1986	new 1986	survey 1981, state appr 1984	began 1950, num limits 1975		Since 1977, Fed appr 1980	Since 1973, Most recent 1983	Formal since 1983	Since 1970, Fed appr 1985	Late 1970s, Fed appr 1985	Since 1981, Fed appr 1983	Since 1973, Cat std 1983	Since 1977, most recent 1986	Fed 1984, new 1987	Inv 1982, Fed appr 1984	Since 1965, appr 1981	Survey 1980	Since 1978, most recent 1984
City	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
Constituent								--- mg/mL ---										
Arsenic	a	a	a	3.0	-	1.0	7.0	-	-	b	0.3	2.0	1.0	-	-	-	0.25	-
Cadmium	a	a	a	15.0	-	0.1	0.9	0.8	2.0	b	0.2	1.0	2.0	0.11	2.0	0.1	0.25	1.0
Chromium-Total	a	a	a	10.0	-	3.0	25.0	-	25.0	b	5.0	2.0	25.0	2.77	5.0	5.0	7.5	5.0
Copper	a	a	a	15.0	-	3.0	15.0	6.0	3.0	b	2.0	5.0	4.5	1.20	5.0	1.5	7.5	5.0
Lead	a	a	a	40.0	-	1.0	5.0	2.0	0.5	b	1.5	2.0	1.0	0.60	2.0	0.2	0.5	1.0
Mercury	a	a	a	5.0	-	0.005	0.0005	-	0.0005	b	0.01	0.05	0.005	0.005	0.05	-	0.02	-
Nickel	a	a	a	12.0	-	3.0	10.0	4.0	10.0	b	3.0	5.0	5.0	1.62	3.0	2.0	13.0	5.0
Silver	a	a	a	2.0	-	3.0	12.0	-	-	b	0.2	1.0	2.0	0.43	-	-	0.25	-
Zinc	a	a	a	10.0	-	5.0	20.0	8.0	15.0	b	6.0	5.0	15.0	2.61	5.0	3.0	25.0	5.0
Cyanide	a	a	a	10.0	-	-	1.7	-	10.0	b	-	5.0	2.0	0.58	0.2	-	2.5	-
Total Tox Org	a	a	a	-	-	4.57	-	-	-	b	-	-	-	2.13	-	-	-	-
Fat/Oil/Grease	a	a	a	-	-	100	-	-	250	b	-	-	-	-	-	-	100	-

a = No local limits yet
b = Industrial plant specific limits

pretreatment programs for longer periods. This left differences in flow, treatment or type of industrial input as the most probable reason.

It was noted that several printing facilities discharged into the City "A" POTW. It is not known if this would have caused an elevated level of volatiles in the sludge. In any event several printing facilities also discharge into City "B's" POTW, but on a relative basis their discharge make up much less of City "B's" 1% industrial flow. Cities "A" and "B" both had primary plus waste actived treatment, with City "B" also having nitrification and ferric chloride treatment for phosphorus removal. City "A" stablized its sludge by lime addition, while City "B" anaerobically digested its sludge. It is not known if the differences in treatment had any influence on the levels of volatile constituents in the sludges. One possible reason for the higher TCLP analytes in the smaller facility's sludge (City "A") appeared to be its lack of ability to dilute out discharged contaminants with large volumes of flow.

Smaller facilities, however, do not necessarily have increased levels of TCLP analytes in their sludge. For example, City "J" had a POTW that treated less than 10 MGD flow, but that had a very high industrial component. City "J" also has an intensive industry-specific pretreatment program that seems to be very effective in controlling levels of TCLP analytes in their sludge (Table 4A, 5A, 6A, and 8A). This is inspite of major petrochemical industries discharging into their facility. Therefore, treatment differences may also be important.

Two other facilities had a volatile TCLP analyte whose concentration in sludge was relatively close to the proposed TC. The POTW serving City "E" had about a 30 MGD average flow and 60% industrial input (Table D-2 in Appendix D). City "K's" POTW had a flow of over 65 MGD with about a 30% industrial input (Table 1). Chloroform, extracted from City "E's" sludge, came within a factor of seven of the proposed TC (Table D-3 in Appendix D), while methyl ethyl ketone, extracted from City "K's" sludge, came within a factor of about four of the proposed TC (Table 4A).

Pretreatment efforts were probably more intensive in City "E" than "K", but neither of the efforts were probably as intensive in City "J". An index of the effectiveness of pretreatment is the change in concentration of contaminants in the influent wastewater and residual sludge with time after the initiation of pretreatment. Such changes in metal concentrations can be seen in Table F-1 of Appendix F. For the most part these metal concentrations have decreased with time as the pretreatment programs have become established. Approximately 75% of the influent metal levels (i.e., 35 of 46 influent metal concentrations for which there was data) decreased from 1980 to 1986. Likewise, about 65% of the sludge metal levels (42 of 63 sludge metal levels) decreased during that period. There was very little comparable historical data on the levels of toxic organic chemicals from the studied facilities.

Taking into consideration the trend toward reduced metal contaminant content in sludges since 1980 as pretreatment programs have been instituted, one can predict more improvement in sludge quality as more attention is placed on pretreatment and management to control toxic organic as well as inorganic constituents. As sludge quality increases, there will be less likelihood of the concentration of contaminants in the TCLP extracts of sludges exceeding the TC regulatory levels. In addition, from the discussion within this section, one can predict that potential problems due to elevated levels of TCLP analytes in sludge will likely be greatest where pretreatment is not practiced and where flow, and hence potential for the dilution of discharged contaminants, is small.

Reporting Limit Impacts on Data

A comparison of the analytical determinations as well as the reporting limits for City "N's" sludge are given for selected volatile TCLP and compositional analytes in Table 10. Note for the volatile analyte carbon disulfide that there is a reported value for its presence in the TCLP extract by the AMSA contract laboratory but not by the EPA contract laboratory. Note further that the AMSA Laboratory's TCLP reporting limit is lower than EPA's. For this same compound there were no reported values for the sludge's compositional content, even though (as just mentioned) an actual value was reported in the TCLP extract. While this result could be because of laboratory contamination, it is more likely a result of the considerably higher reporting limits for the compositional determinations as compared to the TCLP leachate analysis.

TABLE 10. THE RELATIONSHIP BETWEEN REPORTING LIMITS AND THE ABILITY TO DETECT SEWAGE SLUDGE COMPOSITIONAL AND TCLP EXTRACTED ANALYTES

Constituent	Analytical Data for City N+				Reporting Limits+			
	AMSA		EPA		AMSA		EPA	
	TCLP mg/l	Comp. mg/kg	TCLP mg/l	Comp. mg/kg	TCLP mg/l	Comp. mg/kg	TCLP mg/l	Comp. mg/kg
Carbon disulfide	0.0028	*	*	*	0.002	0.0033	0.005	0.10
Chloroform	0.0026	0.024	*	*	0.002	0.0033	0.005	0.10
Methyl ethyl keytone (aka§ 2-Butanone)	*	0.057	*	*	0.010	0.016	0.01	0.20
Trichloroethylene (aka Trichloroethene)	0.0079	0.160	0.009	*	0.002	0.0033	0.005	0.10

+ = Wet Weight Basis
* = Below Reporting Limits
§ = Also Known As

Also, please note in Table 10 that there are reported values for chloroform both for TCLP and compositional determinations by the AMSA contract laboratory. These numbers are both above the AMSA contract laboratory reporting limits, but are below the EPA contract laboratory reporting limits. Similar observations can be made for the other two analytes methyl ethyl ketone (MEK) and trichloroethylene in Table 10 and also for the metal analytes given for City "L" in Table 11. An additional possible reason for MEK being detected in the TCLP extract, but not in the total sludge compositional analysis (based upon the compound's properties and the testing procedure) may be gained from a discussion by D. F. Bishop in Appendix E.

An important conclusion here is that this inability to detect a specific constituent by the EPA contract laboratory compared with the AMSA laboratory was common for all classes of constituents because of the higher EPA laboratory reporting limits. This finding indicates the often overlooked necessity to specifically request in the sampling and analytical plan that an adequate level of sensitivity be obtained to meet the needs of the study, (i.e., so that reporting limits are at a level consistent with meeting the study's objectives).

Quality Assurance and Quality Control

We have included a section in this report on Quality Assurance and Quality Control (QA/QC). This section reports findings by the EPA contract laboratory almost verbatim as follows:

TABLE 11. THE ABILITY TO DETECT METAL ANALYTES IN TCLP AND EP EXTRACTS OF SEWAGE SLUDGE AS INFLUENCED BY REPORTING LIMITS

Constituent	Analytical Data for City L+					Reporting Limits+			
	TCLP, mg/l			EP, mg/l		TCLP, mg/l		EP, mg/l	
	AMSA(1)	AMSA(2)	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA
TCLP ANALYTES									
Arsenic	*	*	*	0.0023	*	0.003	0.25	0.0023	0.10
Barium	0.04	0.07	2.5	*	1.4	0.03	0.90	0.1	0.90
Cadmium	*	*	*	*	*	0.01	0.10	0.01	0.10
Chromium	0.02	0.02	*	*	*	0.10	0.33	0.01	0.33
Lead	*	*	*	*	*	0.10	0.62	0.01	0.62
Mercury	0.0002	0.0008	*	*	*	0.0002	0.01	0.0002	0.01
Selenium	**	**	0.23	*	*	**	0.10	0.0005	0.10
Silver	0.02	*	*	*	*	0.02	0.09	0.01	0.09
NON-TCLP ANALYTES									
Nickel	0.04	0.04	*	*	*	0.01	0.22	*	0.22
Thallium	*	0.007	*	*	*	0.006	0.43	*	0.43

\+ = Wet Weight Basis
* = Below Reporting Limits
** = Not Analyzed

QA Objectives

Quality assurance objectives for precision, accuracy and completeness were established in the QA Project Plan (2). These objectives were expressed in terms of the relative percent deviation (RPD) for duplicate analyses, percent recovery of matrix spike compounds, and percent of samples for which all analyses were completed, respectively. These objectives were as follows:

- Metals (Ag, AS, Cd, Cr, Pb, Hg, Ni, Se, Tl)

Matrix	Precision (RPD)	Accuracy (% Recovery)	Percent Completeness
Sludge	30	70 - 130	95
Leachate	20	75 - 125	95

- Organic Compounds

Table B-1 in Appendix B details the accuracy and precision objectives for the compounds used in spiked sample analyses.

QC Sample Results

Two of the 12 POTW samples (one out of each set of six) were subjected to a specific QC analysis, incorporating the analysis of a matrix-spiked sample (in duplicate) with respect to all analytical procedures employed for both organics and metals. Originally, a duplicate analysis was also incorporated in this scheme. However, because very few, if any, organic analytes were detected within the POTW sludge samples, the performance of a duplicate analysis was not judged to be worthwhile. Rather, the results of the matrix spike duplicate analysis were utilized to address analytical precision.

Results of the matrix spike/matrix spike duplicate analyses are provided in Appendix B Tables B-2 to B-7. Because sample volume requirements for the various analyses frequently approached the volume received by S-Cubed, it was necessary to use different samples for the matrix spike/matrix spike duplicate analyses for some of the various analytical methods employed.

For the volatile and semivolatile organic analyses, recoveries of spiked compounds and the reproducibility of those recoveries were consistently within the QA objectives with respect to two of the three matrices tested (the TCLP and EP extracts, but not the sludge matrix). Problems were encountered with both matrix spike recoveries and precision of the compositional analysis of sludge samples for volatile organic compounds. The initial QC analysis of these samples indicated erratic recovering of spiked compounds, thus initiating corrective action. Other sample aliquots were spiked and analyzed; however, similarly erratic results were produced. Analytical and instrumental conditions were checked to ensure compliance with SW-846 protocols. It can only be assumed, after implementation and completion of corrective action, that

SW-846 protocols have major limitations in producing acceptable data for the matrices of interest to this study. The problems encountered are probably the result of two major areas of difficulty:

1. An extremely complex matrix containing many interfering compounds.

2. Possible irreversible and variable adsorption of analytes within the highly organic POTW sludge matrix.

Results of the matrix spike analyses for pesticides indicated recoveries that were consistently within the established QA objectives with respect to all three matrices. The reproducibility (precision) of these recovery measurements was well within the objectives, with the exception of the EP extraction of the sample from City "P", where the second matrix spike achieved consistently lower recoveries than the first (Table B-6 in Appendix B).

With respect to the herbicide QC sample analysis, the recovery of 2,4-D was consistently below the minimum established QA objectives. For the compositional matrix, the analysis was also poorly reproducible. It is believed that this results from the method employed, with specific reasons as follows:

1. Ether is not the optimum solvent for extraction of phenoxyacid herbicides from complex organic matrices such as POTW sludges.

2. Complex organic matrices require substantial dilution to reduce matrix interferences, and may interact with spiked phenoxyacid herbicides.

Results of the metals QC sample analyses (Tables B-8 to B-10 in Appendix B) revealed the significant difficulties associated with the measurement of metal spike recoveries from a complex organic matrix containing variable but substantial native concentrations of the various metals.

First, because the measured concentration in the unspiked sample must be subtracted from the measured concentration in the spiked sample prior to recovery calculations, potential errors associated with the first measurements add to the potential errors associated with the second. Where native metal concentrations are similar to, or greater than, the spike concentration, this leads to a large potential error in the measured recovery and an inapplicability of the QA objectives. This occurred in many of the cases where the measured recoveries were outside the QA objectives.

Second, the complex matrix of a POTW sludge precludes the level of analytical accuracy expected from cleaner environmental samples.

In particular, the objective of 70 to 130 percent for recovery measurements set in the QA Project Plan (2) are probably unrealistic. A goal of 50 to 150 percent is probably more reasonable and has been used in Tables B-8 through B-10 to mark measured recoveries as outside QA objectives. However, a recovery goal of 70 to 130 percent has been applied to the leachates.

As a routine check on recovery of various types of organic analytes in the GC/MS analysis, surrogate compounds were spiked into each sample processed. Surrogates were spiked at the 50 to 200 ug/l (0.05 to 0.20 mg/L) level in all leachates and at the 1 to 10 mg/L level in the sludges for composite analysis. The recoveries measured are listed in Appendix B Tables B-11 and B-12.

All planned analyses were successfully completed, thereby meeting the completeness goal. (End of S-Cubed discussion.)

It is of interest to compare the variability of results of the QA/QC study by the EPA contract laboratory with the variability between the EPA and AMSA analytical results for identical splits of a sludge sample. For metals, the variability of results of the TCLP extract analysis was far greater for the split samples analyzed by two laboratories than for the analysis of the duplicate matrix-spiked samples by the EPA contract laboratory. On the other hand, the compositional determinations by the two laboratories were relatively close for metals results in the split samples compared with the compositional analysis of the duplicate matrix-spiked samples by the EPA laboratory.

Meaningful comparisons between laboratories were not possible for most contaminants other than metals because the reporting limits of the two laboratories were relatively high, especially for the EPA contract laboratory. Furthermore, sensitivity was lost due to complex sludge matrix interferences and low contaminant concentrations.

There were varying degrees of QA/QC efforts used by the different laboratories. Using a standard set of QA/AC procedures was not an absolute requirement of this cooperative study. Rather, the various laboratories could choose whether or not to follow the recommended QA/QC procedures already discussed.

Costs

The reported costs of analyses by the various contract laboratories were about $2,400 per one replicate of a sludge for TCLP and compositional analyses with the actual cost depending upon the differing amounts of other services being performed. The cost for one TCLP analysis with limited QA/QC was about $1,200 to $1,500 in 1988.

Relationship Between TCLP and Compositional Content in Sludges

The ratios of the TCLP analyte concentration (wet weight basis) to the compositional analyte concentration (dry weight basis) within each

TABLE 12A. RATIO OF THE TCLP EXTRACT METAL CONCENTRATION TO THE COMPOSITIONAL CONCENTRATION IN POTW SEWAGE SLUDGES

Constituent	City G EPA	City G AMSA	City H EPA	City H AMSA	City I EPA	City I AMSA	City J EPA	City J AMSA	City K EPA	City K AMSA	City L EPA	City L AMSA
TCLP ANALYTES												
Arsenic	*	0.002	*	*	*	*	*		*	*	*	*/*
Barium	0.005	0.0009	0.006	0.003	0.006	0.0005	0.004		0.009	0.002	0.005	*/*
Cadmium	*	0.004	*	0.002	*	0.001	*		*	*	*	*/*
Chromium	*	0.0005	*	0.00006	*	0.00006	*		*	*	*	0.00007/0.00008
Lead	*	0.002	*	*	*	*	*		*	*	*	*/*
Mercury	*	0.002	*	*	*	*	*		*	*	*	0.00003/0.0001
Selenium	*	0.0007	*	*	*	*	*		*	*	*	*/*
Silver	*	0.02	*	*	*	0.05	*		*	*	*	0.0004/*
NON-TCLP ANALYTES												
Nickel	*	0.007	0.002	0.004	*	0.004	*		*	*	*	*/0.0001
Thallium	*	*	*	*	*	*	*		*	*	*	*/*

* = Data Not Available to Calculate

TABLE 12A Cont. RATIO OF THE TCLP EXTRACT METAL CONCENTRATION TO THE COMPOSITIONAL CONCENTRATION IN POTW SEWAGE SLUDGES

Constituent	City											
	M		N		O		P		Q		R	
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA
TCLP ANALYTES												
Arsenic	*	*	*	*	*	*	*	0.01	*	*	*	*/0.0004
Barium	0.003	0.002	0.004	0.004	0.006	0.001	0.001	0.0003	0.002	0.0007	0.004	*/0.0001
Cadmium	0.0009	0.001	*	0.004	*	0.0004	*	0.003	0.0005	0.0006	*	*/*
Chromium	*	*	*	*	*	*	*	*	*	*	*	*/*
Lead	*	*	*	*	*	*	*	*	*	*	*	*/*0.00001
Mercury	*	*	*	*	*	*	*	*	*	*	*	*/*
Selenium	*	*	*	*	*	*	*	*	*	*	*	*/*
Silver	*	*	*	*	*	*	*	*	*	*	*	*/*0.00001
NON-TCLP ANALYTES												
Nickel	0.004	0.004	0.007	0.006	0.0007	0.0008	*	*	*	0.0005	0.002	*/0.0003
Thallium	*	*	*	*	*	*	*	*	*	*	*	*/*

* = Data Not Available to Calculate

sludge were calculated. These calculations were used to examine whether the compositional content of TCLP analytes could be used as a rough estimator of the respective TCLP extract analyte contents. The ratios for metals are presented in Table 12A for the AMSA-EPA 12 sludge study and in Table D-9 in Appendix D for the earlier six sludge study. The mean and median ratios for the 18 sludges are presented in Table 12B. As can be seen in Table 12B, the mean and median ratios were different for the different metals. In general the ratio was of greater magnitude for metals which were more readily extractable during the TCLP. For example, the metals chromium and selenium (median ratio of 0.0007) are not as easily extracted by the TCLP as are the metals barium and silver (median ratio of 0.003).

Calculations were made using these ratios to estimate total compositional metal levels in sludges at which the metal TC regulatory levels might be exceeded (Table 12C). The large variance (by more than two orders of magnitude) in the ratio for a given metal in different and even in identical splits of the same sludge, depending upon the specific laboratory and analytical run, indicate their value only as very rough estimators of metal levels that might cause the TCs to be exceeded. Hence, TCLP testing could be necessary if the determined compositional value for a given metal in sludge was at all close to the corresponding estimated range of compositional metal levels for failure of the TC.

Similarly, ratios of the TCLP analyte extract concentration (wet weight basis) to the compositional concentration (dry weight basis) were

TABLE 12B. SUMMARY OF RATIOS OF THE TCLP EXTRACT METAL CONCENTRATION TO THE COMPOSITIONAL CONCENTRATION IN 18 POTW SEWAGE SLUDGES

Constituent	Range	High ratio / Low Ratio	No. of Means	Mean	Median
TCLP ANALYTES					
Arsenic	0.0004 to 0.01	25	6	0.003	0.002
Barium	0.0001 to 0.009	90	28	0.003	0.003
Cadmium	0.00002 to 0.004	200	14	0.002	0.0002
Chromium	0.000005 to 0.0005	100	9	0.0002	0.0007
Lead	0.00001 to 0.002	200	2	0.001	0.001
Mercury	0.00003 to 0.002	67	3	0.0007	0.0001
Selenium	0.0007 to 0.0007	0	1	0.0007	0.0007
Silver	0.00001 to 0.05	5000	4	0.003	0.003
NON-TCLP ANALYTES					
Nickel	0.0001 to 0.007	70	20	0.003	0.004
Thallium	* to *	*	*	*	*
OVERALL MEAN			87	0.002	0.003

* = Data Not Available to Calculate

TABLE 12C. ROUGH ESTIMATION OF THE METAL THRESHOLD COMPOSITIONAL CONCENTRATIONS FOR FAILING THE TCLP

Constituent	TCLP Toxicity Threshold, mg/l	Estimated Compositional Threshold, mg/kg (basis median ratios)	Estimated Compositional Threshold Range, mg/kg (based on ratio range)
TCLP ANALYTES			
Arsenic	5.0	2,500	500 to 12,500
Barium	100	33,000	10,000 to 1,000,000
Cadmium	1.0	5,000	250 to 5,000
Chromium	5.0	7,000	10,000 to 1,000,000
Lead	5.0	5,000	2,500 to 100,000
Mercury	0.2	2,000	100 to 6,600
Selenium	1.0	1,500	
Silver	5.0	1,500	100 to 50,000

TABLE 13A. RATIO OF THE TCLP EXTRACT VOLATILE CONCENTRATION TO THEIR COMPOSITIONAL CONCENTRATION IN POTW SLUDGES

Constituent	City									
	G		I	L	M	N	O	P	Q	R
	EPA	AMSA	EPA	AMSA	AMSA	AMSA	AMSA	AMSA	EPA	AMSA
Chlorobenzene	*	*	*	0.01	*	*	*	*	*	*
Chloroform	*	*	*	*	*	0.03	*	*	*	*
Methylene chloride	*	*	*	*	*	0.5	*	0.1	*	*
Methyl ethyl ketone (aka§ 2-Butanone)	*	*	*	*	0.6	*	0.1	*	*	*
Tetrachloroethylene (aka Tetrachloroethene)	*	*	*	*	*	0.01	*	*	*	*
Toluene	0.02	0.008	0.02	0.008	0.01	0.01	0.02	*	0.02	0.4/0.007
Trichloroethylene (aka Trichloroethene)	*	*	*	*	*	*	*	*	*	*

* = Data Not Available to Calculate Ratio
§ = Also Known As

TABLE 13B. SUMMARY OF RATIOS OF THE TCLP EXTRACT VOLATILE CONCENTRATION TO THEIR COMPOSITIONAL CONCENTRATION IN 18 POTW SLUDGES

Constituent	Range	High ratio / Low ratio	No. of Means	Mean Ratio	Median Ratio
Chlorobenzene	0.01	*	1	0.01	0.01
Chloroform	0.03	*	1	0.03	0.03
Methylene chloride	0.1 to 0.5	5	2	0.3	0.3
Methyl ethyl ketone (aka§ 2-Butanone)	0.1 to 0.6	6	2	0.4	0.4
Tetrachloroethylene (aka Tetrachloroethene)	0.01	*	1	0.01	0.01
Toluene	0.007 to 0.4	57	10	0.05	0.02
Trichloroethylene (aka§ Trichloroethene)	0.01	*	1	0.01	0.01
OVERALL MEAN	0.01 to 0.4		18	0.1	0.05

* = Data Not Available to Calculate Ratio
§ = Also Known As

TABLE 13C. ROUGH ESTIMATION OF THE VOLATILE THRESHOLD CONCENTRATIONS FOR FAILING THE TCLP

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/l	Estimated Compositional Thresholds, mg/kg +++ (basis median ratios)*	Based on the Overall Mean Ratio (0.1)**	Median Ratio (0.05)**	Mean Range of Ratios (0.01 to 0.4)**
Acrylonitrile	5.0		50	25	12.5 to 500
Benzene	0.07		0.7	0.35	0.2 to 7
Carbon disulfide	14.4		144	72	36 to 1440
Carbon tetrachloride	0.07		0.7	0.4	0.2 to 7
Chlorobenzene	1.4	140	14	7	3.5 to 140
Chloroform	0.07	2.3	0.7	0.4	1.8 to 7
1,2,-Dichloroethane	0.40		4	2	1 to 40
1,1,-Dichloroethylene (aka 1,1,-Dichloroethene)	0.1		1	0.5	0.3 to 10
Isobutanol (aka§ 2-Methyl-1-propanol)	36		360	180	90 to 3600
Methylene chloride	8.6	29	86	43	22 to 860
Methyl ethyl ketone (aka§ 2-Butanone)	7.2	144	72	36	18 to 720
Pyridine	5.0		50	25	12 to 500
1,1,1,2, Tetra-chloroethane	10.0		100	50	12 to 1000
1,1,2,2, Tetra-chlorethane	1.3		13	6.5	3.2 to 130
Tetrachloroethylene (aka Tetrachloroethene)	0.1	10	1	0.5	0.2 to 10
Toluene	14.4	288	144	72	36 to 1440
1,1,1-Trichloro-ethane	30		300	150	75 to 3000
1,1,2-Trichloro-ethane	1.2		12	6	3 120
Trichloroethylene (aka Trichloroethene)	0.07	5	0.7	0.4	2 to 7
Vinyl Chloride	0.05		0.5	0.2	0.1 to 5

* = Calculated individually from the TCLP analytes shown in Table 10B

** = From Table 10B

§ = Also Known As

++ = Proposed Regulatory Levels

+++ = Estimated Values would mostly be Greater when Compared with Final Regulatory Levels given in Table 16

TABLE 14A. RATIO OF THE TCLP EXTRACT SEMIVOLATILE, HERBICIDE AND PESTICIDE CONCENTRATION TO THEIR COMPOSITIONAL CONCENTRATION IN POTW SLUDGES

Constituent	City										Mean
	H		I		L		M		R		
	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	EPA	AMSA	
SEMIVOLATILES											
p-Cresol (aka§ 4-Methyl Phenol)	*	0.02	*	*	*	*	0.03	*		*	0.02
Hexachloroethane	*	*	*	*	*	*	*	*	*	0.005	0.005
Phenol	*	*	*	0.001	*	0.001	*	*	*		0.001
PESTICIDES & HERBICIDES											
Chlordane	*	*	*	0.0007	*	*	*	*	*	*	0.0007
Endrin	*	*	*	0.0004	*	*	*	*	*		0.0004

* = Data Not Available to Calculate Ratio
§ = Also Known As

calculated for specific analytes in the volatile, semivolatile, and pesticide and herbicide organic compound classes (Tables 13A, 13B and 14A). These ratios were then used to estimate compositional analyte concentrations of compounds (in these three classes of TCLP analytes) which might exceed the respective TCs (Tables 13C and 14B). Because of the very limited presence of TCLP analytes, especially from the semivolatile and pesticide and herbicide classes, there were few ratios and compositional concentrations that could be calculated and estimated.

The analytical data were used to obtain a very rough estimate of the total content of contaminants in sludges that would result in TC regulatory level exceedance (Table 15). These rough estimates can be calculated from the formula (TC)/(divided by the median ratio) where the median ratio is derived from the fraction of the total analytes extracted by the TCLP within a class of compounds.

While different for the various compounds within a class, the fraction of the various compounds extracted by the TCLP was generally greatest for volatiles and least for semivolatiles, metals, pesticides, and herbicides (Table 15).

TCLP AND TC UPDATE

EPA proposed the TCLP and coupled with TCs in 1986 to replace the Extraction Procedure (EP) for classifying wastes as hazardous based upon toxicity. The proposed TCLP added 38 additional toxic organic

TABLE 14B. ROUGH ESTIMATION OF THE THRESHOLD SEMIVOLATILE, HERBICIDE AND PESTICIDE CONCENTRATIONS FOR FAILING THE TCLP

Constituent	TCLP Toxicity Threshold,++ mg/l	Estimated Compositional Threshold, +++ mg/kg
SEMIVOLATILES		
p-Cresol (aka§ 4-Methyl Phenol)	10	500
Hexachloroethane	4.3	860
Phenol	14.4	14,400
PESTICIDES & HERBICIDES		
Chlordane	0.03	43
Endrin	0.003	6

§ = Also Known As

++ = Proposed Regulatory Levels

+++ = The Estimated Compostional Thresholds would mostly be Greater when Compared with the Final Toxicity Characteristic Regulatory Levels Given in Table 16 in the Update Section of the Report

TABLE 15. Factors for Roughly Estimating Toxicity Characteristic Regulatory Level Exceedance from the Total Content of Contaminants in Sludge.

TCLP Analyte Class	Tables	Range of Mean Ratios for Compounds within a Class*	Median of Mean Ratios*
Volatiles (limited data)		0.2 to 0.4	0.3
Metals		0.0002 to 0.003	0.003
Semivolatiles (very limited data)		0.001 to 0.02	0.001
Pesticides & Herbicides (very limited data)		0.0008 to 0.002	0.001

*These ratios were derived from the fraction of the total analytes extracted by the TCLP. A very rough estimate of the total content of contaminants in sludges that would result in TC regulatory level exceedance can be calculated from the formula (TC)/(divided by the median ratio).

compounds. EPA received many comments on the proposed TCLP and its 52 TC regulatory levels. The comments received and the changes ultimately made to both the TCLP and the TCs are described in detail in the final rule (March 29, 1990, in 55 FR 11798).

Of particular importance to this sewage sludge study, there TCs for only 25 additional toxic organic compounds in the final rule. The promulgated and proposed TCs are compared in Table 16. The final promulgated TC regulatory levels remained unchanged from the proposal for the eight metals and some of the other contaminants. Most of the other contaminants had a less stringent TC, except for several semivolatile toxic organic compounds where the TCs were slightly decreased. Since all of the TCs for volatile toxic organic contaminants have been made less stringent in the promulgated final rule, sewage sludges are even less likely to exceed the TC and be considered hazardous.

TABLE 16. COMPARISON OF PROPOSED AND FINAL TOXICITY CHARACTERISTICS

Constituent	Toxicity Characteristic Proposed, mg/l	Toxicity Characteristic Promulgated Final, mg/l
VOLATILES		
Acrylonitrile	5.0	not promulgated
Benzene	0.07	0.5
Carbon disulfide	14.4	not promulgated
Carbon tetrachloride	0.07	0.5
Chlorobenzene	1.4	100
Chloroform	0.07	6.0
1,2,-Dichloroethane	0.40	0.5
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.1	0.7
Isobutanol (aka§ 2-Methyl-1-propanol)	36	not promulgated
Methylene chloride	8.6	not promulgated
Methyl ethyl ketone (aka§ 2-Butanone)	7.2	200
Pyridine	5.0	5.0+
1,1,1,2, Tetra-chloroethane	10.0	not promulgated
1,1,2,2, Tetra-chlorethane	1.3	not promulgated
Tetrachloroethylene (aka Tetrachloroethene)	0.1	0.7
Toluene	14.4	not promulgated
1,1,1-Trichloro-ethane	30	not promulgated
1,1,2-Trichloro-ethane	1.2	not promulgated
Trichloroethylene (aka Trichloroethene)	0.07	0.5
Vinyl Chloride	0.05	0.2

+ = Reporting limit is greater than the calculated regulatory level, hence reporting limit is used.

§ = Also Known As (aka)

TABLE 16 cont. COMPARISON OF PROPOSED AND FINAL TOXICITY CHARACTERISTICS

Constituent	Toxicity Characteristic Proposed, mg/l	Toxicity Characteristic Promulgated Final, mg/l
SEMIVOLATILES		
Bis(2-chloroethyl) ether	0.05	not promulgated
o-Cresol (aka§ 2-Methyl Phenol)	10	200*
m-Cresol (aka 3-Methyl_ Phenol)	10	200*
p-Cresol (aka§ 4-Methyl Phenol)	10	200*
Cresol		200*
1,2 Dichlorobenzene	4.3	not promulgated
1,4 Dichlorobenzene	10.8	7.5
2,4 Dinitrotoluene	0.13	0.13+
Hexachlorobenzene	0.13	0.13+
Hexachlorobutadiene	0.72	0.5
Hexachloroethane	4.3	3.0
Nitrobenzene	0.13	2.0
Pentachlorophenol	3.6	100
Phenol	14.4	not promulgated
2,3,4,6-Tetrachlorophenol	1.5	not promulgated
2,4,5-Trichlorophenol	5.8	400
2,4,6-Trichlorophenol	0.30	2.0
METALS		
Arsenic	5.0	5.0
Barium	100	100
Cadmium	1.0	1.0
Chromium	5.0	5.0
Lead	5.0	5.0
Mercury	0.2	0.2
Selenium	1.0	1.0
Silver	5.0	5.0
PESTICIDES AND HERBICIDES		
Chlordane	0.03	0.03
Endrin	0.003	0.02
Heptachlor	0.001	0.008
Lindane (gamma-BHC)	0.06	0.4
Methoxychlor	1.4	10.0
Toxaphene	0.07	0.5
2,4-D	1.4	10.0
2,4,5,TP (Silvex)	0.14	1.0

* = If o-, m-, & p-cresol cannot be differentiated, the total cresol regulatory level of 200 is used

+ = Reporting limit is greater than calculated regulatory level, hence reporting limit is used.

§ = Also Known As (aka)

SUMMARY AND CONCLUSIONS

The Toxicity Characteristic Leaching Procedure (TCLP) is a testing procedure that has been developed by the Office of Solid Waste (OSW) for determining whether or not solid wastes, including municipal sewage sludges, are hazardous based upon toxicity. This procedure was a proposed replacement for the Extraction Procedure (EP), used for this purpose since 1980. In the TCLP, the concentrations of analytes in the extracts are compared to Toxicity Characteristic (TC) regulatory levels. If concentrations of analytes in the TCLP extract meet or exceed these regulatory levels, the wastes are classified as hazardous.

In 1985-86 when the studies were conducted, it was felt that the proposed TCLP and TC regulatory levels might cause a number of municipal sewage sludges from Publicly Owned Treatment Works (POTWs) to be classified as hazardous. Hence, the Office of Water (OW), in cooperation with OSW, began testing municipal sewage sludges. Both total and TCLP fractions of the 18 sewage sludges were analyzed for selected analytes. The Association of Metropolitan Sewerage Agencies (AMSA) cooperated with EPA's OW and OSW in this study, analyzing split samples of sludges from 12 of the POTWs using identical analytical instructions sent by the EPA laboratory. Time and budget did not permit rigid policing of the AMSA laboratories to assure that they actually did use identical procedures.

These 18 analyzed sludges, included in two separate tests, were obtained from POTWs that ranged in flow from less than 10 to over 600 million gallons per day (MGD) with less than one to over 90 percent of the flow being of industrial origin.

Any change of TC regulatory levels from proposal to final promulgation have been accounted for in the following important conclusions:

1) No POTW sewage sludge will likely exceed the TC regulatory levels and be considered hazardous.

   - None of the 18 sludges tested by any of the laboratories had TCLP extract concentrations that exceeded the proposed TC regulatory levels.

   - In these studied sludges the volatile analytes were found to be the most likely class of contaminants that might cause a sewage sludge to be classified as hazardous, (i.e., three of 18 sludges had volatile TCLP analyte contents within less than an order of magnitude [one of the three was within a factor of three] of the proposed TC regulatory levels).

     -- Sludge from one POTW (City "K", Table 4A), came close to exceeding the proposed TC regulatory level because of the volatile constituent methyl ethyl ketone. This result was similar to the results of our earlier six sewage sludge TCLP study. In the six POTW study two of the six sludges also approached exceedance of the respective TC regulatory levels because of their content of the volatile components benzene and chloroform (Table D-3 in Appendix D).

- However, because the final promulgated TCs are on average, two to three times higher than the proposed TCs for the volatile toxic organic TCLP compounds, it would seem unlikely that the volatile compounds will result in any POTW sludges being classified as hazardous.

- Because the concentrations of the metal, semivolatile, pesticide, and herbicide constituents in analytes in TCLP extracts of the tested sewage sludges were lower than the respective TC regulatory levels by about one to two orders of magnitude, it would seem even less likely for these classes of contaminants to result in sludges being classified as hazardous.

2) To summarize the results in a different way, TCLP analyte concentrations in 15 of the 18 analyzed POTW sludges were one to two orders of magnitude below the TC regulatory levels. These 15 POTWs were larger in size and most contained an industrial flow component of 30% or more. Two smaller POTWs (less than 10 MGD in size) and one moderately-sized POTW had sludges with TCLP volatile analyte contents that were from 3 to 7 times below the proposed TC regulatory levels. It may be that sludges from smaller facilities are more likely to be considered hazardous than from larger facilities.

- The TCLP contaminants benzene and chloroform that came closest to exceeding the proposed TC regulatory levels were in a TCLP extract of a sludge from a smaller POTWs' sludge. This POTW had a flow that was a little over one million gallons per day (MGD) and less than one percent industrial flow.

- One possible reason for the higher level of volatile analytes observed in the tested smaller POTW is that an insufficient volume of sludge was generated to dilute out occasional discharges of TC contaminants that have occurred. Unfortunately, this study did not include information for assessing how the TCLP analyte contents in the sludges were impacted by the type, size, and nature of the industries discharging to each POTW or by the type of wastewater and sludge treatment employed at each facility.

- The total compositional and TCLP extract contents of the proposed 52 TCLP analytes were not particularly high in the tested sludges. Some limited information is presented in the report about the various industrial pretreatment programs at the tested facilities. It is not known whether these industrial pretreatment programs had any bearing on the relatively low contents of analytes detected in the tested sludges.

- These findings for the tested sludges are contrary to the common assumption that sewage sludges from larger more industrial communities are likely to contain higher levels of volatile, semivolatile, metal, and herbicide and pesticides.

3) For most contaminants except metals, there were non-detects in the TCLP extracts, and there were very few contaminants detected by both laboratories on the same sludge sample. Only for barium, p-cresol, and xylene did split sample analyses on the same sludge by the EPA and AMSA laboratories show detected measurements. There was substantial variation in the split sample results for barium with the level of barium detected by the EPA laboratory always being higher than detected by the AMSA laboratories. On the other hand, the variation in the split sample detects were less for p-cresol and xylene with no laboratory's results being consistently higher. The split sample results for barium would have to be viewed as questionable because of the large degree of consistently skewed variation.

- The EPA contract laboratory concluded in their QA/QC analysis that such analytical variability may have resulted because of compounds within the complex sludge matrices that interfered when using the SW-846 protocols. Further, they concluded that there was possible irreversible and variable adsorption of analytes within the highly organic POTW sludge matrix. A third factor might be differences in subsample contaminant content. In general, the AMSA laboratories had lower reporting limits than did the EPA laboratory.

- This considerable degree of analytical variability could increase the amount of duplication and cost to obtain adequate confidence in the results, especially where the analyte concentrations in the TCLP extracts are close to the TC regulatory levels.

- The cost impact upon small POTWs could be substantial. The cost was about $1,200.00 to $1,500.00 (1988 dollars) for the complete analysis of a single sample without duplication.

4) The analytical data were used to obtain a very rough estimate of the total content of contaminants in sludges that would result in TC regulatory level exceedance.

- These rough estimates can be calculated from the following formula:

(TC times 100)/(divided by the median percentage)

where the median percentage is derived from the fraction of the total analytes extracted by the TCLP within a class of compounds (Table 15).

- While these estimating percentages are different within a class of extracted TCLP contaminants, the median percentages of the volatiles extracted were generally greatest at 30%, followed by metals at 0.03%, and semivolatiles, pesticides and herbicides at 0.01%.

- Because of the considerable variability in percentages of the different analytes extracted (see Tables 12B, 13B, 14A), additional TCLP testing would be needed where these estimating percentages, applied to the total compositional analyte contents of the TC contaminants in sludge, would predict a TCLP analyte content that was at all close (perhaps within an order of magnitude) to the TC regulatory level.

5) When the concentrations of metals in TCLP and EP extracts were compared, there were no consistent differences in the amounts of a metal extracted.

6) One important limitation of these studies is that only 18 of the more than 15,000 POTWs in the United States (US) were included in the study. Only one of the 18 tested POTW sludges came from a POTW that was close to one MGD in size. POTWs of less than one MGD in size constitute nearly 90% of all POTWs in the US. Another limitation is that the 18 POTWs were not selected in a manner that would allow statistically valid extrapolation of the results to the POTWs nationwide. However, the POTWs were selected on a basis of

high to low hydraulic and industrial flow with the expectation that these parameters would be somewhat inclusive of wastewater inputs and resultant sludges that might cause the sludge to be classified as hazardous.

7) The applicability of the test from the viewpoint of reflecting a potentially toxic and hazardous condition for sewage sludges, whether used or disposed in air, on land or into water and at what rate, was not evaluated in this report. We also did not compare TCLP results of sludge and other waste materials.

REFERENCES

(1) Proposed analytical techniques - POTW sludge testing, S-Cubed Laboratories, La Jolla, CA for USEPA, phone 619-453-0060.

(2) Quality assurance project plan for POTW sludge testing, S-Cubed Laboratories, La Jolla, Ca for Dynamac Corporation for USEPA, phone 619-453-0060, May 1986.

APPENDIX A

POTW Sludge Sampling Procedures

APPENDIX A: POTW Sludge Sampling Procedures

The sampling of sludge at your wastewater treatment facility should be performed at the location previously specified.

It is important that four basic objectives be kept in mind regardless of where the sludge samples are actually collected:

(1) Samples should be representative of the bulk material from which they are collected;

(2) The sample should be identical in each of the six glass mason jars (about one quart in volume) and six 40 ml glass vials (VOA vials) having teflon septums at the top;

(3) Sludge character or quality should not be altered as a result of sampling; and

(4) Proper QA procedures such as sample icing for refrigeration, fully filling all containers, and labelling of containers. Also, all procedures employed relative to sample collection are properly documented.

Factors such as accessibility and physical characteristics of the sludge (i.e., solids content, viscosity, etc.) should be considered when selecting a sampling device and/or procedure. To the extent possible, the sampling device should be clean and constructed of an inert or unreactive substance such as glass, stainless steel of teflon. The sampling method will vary depending upon the type of sample requested. Dried sludge in either a "cake" form or within a drying bed should be easily accessible and can be sampled using either a trowel, scoop, shovel, or auger.

Availability and ease of use will probably be the determining factor. A shovel or an auger are better suited for sampling from a deeper bed of material (integrated sample). A sample of a thin layer of sludge cake such as that produced by a centrifuge, belt filter press, vacuum filter, etc. would be more easily collected by means of a trowel or scoop. Sampling the bottom sludge from either a lagoon or settling tank can be accomplished using a small, light weight mechanical grab or dredge sampler. Examples of this type of sampler are an Eckman grab or box dredge, ponar grab or Peterson grab. Mechanical grab samplers generally have closeable jaws, some of which are messenger activated. If the sludge layer is extremely thick, (i.e., several feet or more) a teflon or glass lined coring device can be used. These latter samplers have the added advantage of creating a lesser degree of disturbance but may require more drops. Again, it should be emphasized that whichever sampler is used, proper cleaning procedures should be followed. Moreover, it should be dropped at a location within the lagoon or tank where sludge deposits are most likely to accumulate.

When multiple drops with a sampling device are required or multiple scoops are taken of drier material, it is essential to manually mix these individual samples prior to filling the sample containers. The final composite of these multiple samples should be thoroughly but carefully mixed and then distributed among the six glass jars and six vials. (NOTE: If the conditions of sampling require time compositing or handling which would allow significant loss of volatiles, the taking of separate grab samples in each 40 ml VOA vial is appropriate. Although some sample representativeness may be compromised, the loss of volatile organics through volatilization is extremely rapid and preservation of this fraction through zero headspace storage is simply a more important consideration.)

For purposes of this sampling program, it will be necessary to fill three glass mason jars (about 1 quart volume) and three 40 ml glass vials having teflon septums in the top for each of the two ice chests. One ice chest (with three quart jars and three VOA vials) should be sent to the EPA lab and one ice chest (with the other three quart jars and three VOA vials) is for your lab. Each glass jar and vial should be filled as completely full as possible in order to avoid the loss of volatile compounds. Preservatives must not be added to any of the samples. Samples should be refrigerated and shipped as soon as possible. (See the enclosed May 17th memo for timing.) WE MUST EMPHASIZE AGAIN THAT THE SLUDGE IN EACH OF THE SIX QUART JARS AND SIX VOA VIALS BE AS NEARLY IDENTICAL AS POSSIBLE.

Lastly, it is important that all samples are properly labelled with your identification number and packaged prior to shipment. The samples should be packaged on water ice (not "Freeze Paks") and every attempt should be made to ensure that the sample bottles will not be broken during transit. The mason jars should be wrapped in the provided packing material to prevent their coming into contact with one another. The three 40 ml VOA containers can be wrapped and sealed in the collapsed plastic container being sent to you. The ice chest should also be taped, labelled with the label provided and shipped by overnight shipment.

APPENDIX B

QUALITY ASSURANCE/QUALITY CONTROL DATA

TABLE B-1. QA OBJECTIVE (ORGANIC COMPOUNDS)*

Matrix Spike Compound	Accuracy % Recovery Leachate	Accuracy % Recovery Sludge	Precision RPD Leachate	Precision RPD Sludge	Completeness %
VOLATILE COMPOUNDS					
Benzene	76-127	66-142	11	21	95
Chlorobenzene	75-130	60-133	13	21	95
1,1-Dichloroethene	61-145	59-172	14	22	95
Toluene	76-125	59-139	13	21	95
Trichloroethene	71-120	62-137	14	24	95
BASE-NEUTRAL EXTRACTABLE COMPOUNDS					
Acenaphthene	46-118	31-137	31	19	95
1,3,4-Trichlorobenzene	39-98	38-107	28	23	95
2,4-Dinitrotoluene	24-96	28-89	38	47	95
Di-n-butyl Phthalate**	11-117	29-135	40	47	95
Pyrene	26-127	25-142	31	36	95
1,2,4-Trichlorobenzene	39-98	38-107	28	23	95
ACID EXTRACTABLE COMPOUNDS					
4-Chloro-3-Methylphenol	23-97	26-103	42	33	95
2-Chlorophenol	27-123	25-102	40	50	95
4-Nitrophenol	10-80	11-114	50	50	95
Pentachlorophenol	9-103	17-109	50	47	95
Phenol	12-89	26-90	42	35	95
PESTICIDES					
Aldrin	40-120	34-132	20	31	95
4,4'-DDT	38-127	23-134	27	50	95
Dieldrin	52-126	31-134	18	38	95
Endrin	56-121	42-139	21	45	95
Heptachlor	40-131	35-130	20	31	95
Lindane	56-123	46-127	15	50	95
HERBICIDES					
2,4-D	40-130	25-130	25	45	95

*S-Cubed Laboratory
**Deleted from matrix spike list prior to implementation of analysis.

TABLE B-2. COMPOSITIONAL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 1*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
VOLATILES (METHOD 8240)					
Benzene	K	0.0004	109	107	1
Chlorobenzene		0.0003	114	120	5
1,1-Dichloroethene		0.0003	167**	183**,+	9
Toluene		0.0004	531**,+	505**,+	5
Trichloroethene		0.0003	99.7	109	9
SEMIVOLATILES (METHOD 8270) B/N					
Acenaphthene	N	5.0	102	90	13
1,4-Dichlorobenzene		5.0	60	58	3
2,4-Dinitrotoluene		5.0	94	80	16
Pyrene		5.0	94	84	11
1,2,4-Trichlorobenzene		5.0	78	70	11
SEMIVOLATILES (METHOD 8270) ACID					
4-Chloro-3-methylphenol	N	10.0	72	65	10
2-Chlorophenol		10.0	63	56	12
4-Nitrophenol		10.0	123	103	15
Pentachlorophenol		10.0	21	16+	27
Phenol		10.0	63	55	12
PESTICIDES (METHOD 8080)					
Aldrin	N	0.36	64	52	21
4-4'-DDT		0.90	77	66	15
Dieldrin		0.90	84	95	12
Endrin		0.90	85	74	14
Heptachlor		0.36	93	85	9
Lindane		0.36	99	87	13
HERBICIDES (METHOD 8150)					
2,4-D	M	0.7	0+	6+	200+

* S-Cubed Laboratory
** Interference
\+ Outside QA objectives
%Rec_1 Percent Recovery for Matrix Spike.
%Rec_2 Percent Recovery for Matrix Spike Duplicate.
RPD Relative Percent Difference = $(\%Rec_2 - \%Rec_1) - (\%Rec_1 + \%Rec_2)/2$

TABLE B-3. COMPOSITIONAL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 2*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
VOLATILES (METHOD 8240)					
Benzene	L	0.0004	92.8	95.4	2
Chlorobenzene		0.0003	127	132	3
1,1-Dichloroethene		0.0003	0**,+	205**,+	200**,+
Toluene		0.0004	119	131	8
Trichloroethene		0.0003	126	121	4
SEMIVOLATILES (METHOD 8270) B/N					
Acenaphthene	G	5.0	70	68	3
1,4-Dichlorobenzene		5.0	62	58	7
2,4-Dinitrotoluene		5.0	66	66	0
Pyrene		5.0	55	53	3
1,2,4-Trichlorobenzene		5.0	66	64	3
SEMIVOLATILES (METHOD 8270) ACID					
4-Chloro-3-methylphenol	G	10.0	61	58	5
2-Chlorophenol		10.0	34	33	3
4-Nitrophenol		10.0	23	18	24
Pentachlorophenol		10.0	54	43	23
Phenol		10.0	56	56	0
PESTICIDES (METHOD 8080)					
Aldrin	G	0.40	56	57	2
4-4'-DDT		1.0	112	119	6
Dieldrin		1.0	62	69	11
Endrin		1.0	93	103	10
Heptachlor		0.40	69	80	15
Lindane		0.40	70	78	11
HERBICIDES (METHOD 8150)					
2,4-D	H	0.94	4+	0+	200+

* S-Cubed Laboratory
** Interference
\+ Outside QA objectives
$\%Rec_1$ Percent Recovery for Matrix Spike.
$\%Rec_2$ Percent Recovery for Matrix Spike Duplicate.
RPD Relative Percent Difference = $(\%Rec_2 - \%Rec_1) - (\%Rec_1 + \%Rec_2)/2$.

TABLE B-4. COMPOSITIONAL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 2*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
VOLATILES (METHOD 8240)					
Benzene	K	0.04	102	104	2
Chlorobenzene		0.04	102	102	0
1,1-Dichloroethene		0.04	100	102	2
Toluene		0.04	105	105	0
Trichloroethene		0.04	102	102	0
SEMIVOLATILES (METHOD 8270) B/N					
Acenaphthene	J	0.20	115	105	9
1,4-Dichlorobenzene		0.20	80	75	6
2,4-Dinitrotoluene		0.20	85	70	19
Pyrene		0.20	95	90	5
1,2,4-Trichlorobenzene		0.20	90	80	12
SEMIVOLATILES (METHOD 8270) ACID					
4-Chloro-3-methylphenol	J	0.40	53	43	21
2-Chlorophenol		0.40	68	65	4
4-Nitrophenol		0.40	45	30	40
Pentachlorophenol		0.40	0	0	0
Phenol		0.40	45	60	29
PESTICIDES (METHOD 8080)					
Aldrin	J	0.0004	72	75	4
4-4'-DDT		0.001	61	71	6
Dieldrin		0.001	113	116	3
Endrin		0.001	101	107	6
Heptachlor		0.0004	95	93	2
Lindane		0.0004	92	94	2
HERBICIDES (METHOD 8150)					
2,4-D	J	0.84	31+	30+	3

* S-Cubed Laboratory
\+ Outside QA objectives
$\%Rec_1$ Percent Recovery for Matrix Spike.
$\%Rec_2$ Percent Recovery for Matrix Spike Duplicate.
RPD Relative Percent Difference = $(\%Rec_2 - \%Rec_1) - (\%Rec_1 + \%Rec_2)/2$.

TABLE B-5. TCLP MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 2*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
VOLATILES (METHOD 8240)					
Benzene	P	0.05	94	96	2
Chlorobenzene		0.05	82	84	2
1,1-Dichloroethene		0.05	88	94	7
Toluene		0.05	84	88	4
Trichloroethene		0.05	104	86	19
SEMIVOLATILES (METHOD 8270) B/N					
Acenaphthene	P	0.20	85	95	11
1,4-Dichlorobenzene		0.20	60	55	9
2,4-Dinitrotoluene		0.20	70	80	13
Pyrene		0.20	90	90	0
1,2,4-Trichlorobenzene		0.20	64	60	8
SEMIVOLATILES (METHOD 8270) ACID					
4-Chloro-3-methylphenol	P	0.40	58	68	16
2-Chlorophenol		0.40	60	68	12
4-Nitrophenol		0.40	28	22	22
Pentachlorophenol		0.40	43	73	52
Phenol		0.40	38	45	18
PESTICIDES (METHOD 8080)					
Aldrin	P	0.0002	77	80	4
4-4'-DDT		0.0005	106	98	8
Dieldrin		0.0005	84	85	1
Endrin		0.0005	90	88	2
Heptachlor		0.0002	80	78	3
Lindane		0.0002	80	80	0
HERBICIDES (METHOD 8150)					
2,4-D	P	0.16	30+	28+	7

* S-Cubed Laboratory
\+ Outside QA objectives
$\%Rec_1$ Percent Recovery for Matrix Spike.
$\%Rec_2$ Percent Recovery for Matrix Spike Duplicate.
RPD Relative Percent Difference = $(\%Rec_2 - \%Rec_1) - (\%Rec_1 + \%Rec_2)/2$.

TABLE B-6. EP MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 1*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
<u>PESTICIDES (METHOD 8080)</u>		0.0003	55	44	22
Aldrin	G	0.0008	74	57	26
4-4'-DDT		0.0008	68	55	21
Dieldrin		0.0008	102	94	8
Endrin		0.0003	56	43	26
Heptachlor		0.0003	98	106	8
Lindane					
<u>HERBICIDES (METHOD 8150)</u>					
2,4-D	G	0.76	26+	26+	0

TABLE B-7. EP MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY ORGANIC ANALYSES, NO. 2*

Compound	City No.	Spike Added mg/kg	% Rec_1	% Rec_2	RPD
<u>Pesticides (Method 8080)</u>					
Aldrin	P	0.003	49	35	33
4-4'-DDT		0.0007	102	52	65
Dieldrin		0.0007	86	56	42
Endrin		0.0007	110	73	40
Heptachlor		0.0003	75	51	38
Lindane		0.0003	91	85	7
<u>HERBICIDES (METHOD 8150)</u>					
2,4-D	P	0.16	7.5+	21	90

* S-Cubed Laboratory
+ Outside QA objectives
$\%Rec_1$ Percent Recovery for Matrix Spike.
$\%Rec_2$ Percent Recovery for Matrix Spike Duplicate.
RPD Relative Percent Difference = $(\%Rec_2 - \%Rec_1) + (\%Rec_1 + \%Rec_2)/2$.

TABLE B-8. METALS SPIKE/SPIKE DUPLICATE RECOVERY COMPOSITIONAL MATRIX*

SET No. 1

Compound	Method	Conc. Unspiked Sample mg/kg	Conc. Spike Added mg/kg	% Rec_1	% Rec_2	RPD
Arsenic	7060	4.5	27	97	91	6.4
Barium	7080	548	540	145	37+	119+
Cadmium	7130	24	5.4	141	131	7.4
Chromium	7190	340	27	134	120	11
Lead	7420	99	27	127	47+	92+
Nickel	7520	60	27	131	131	0
Selenium	7740	ND	5.4	143	106	30
Silver	7760	ND	263	86	77	11
Thallium	7840	ND	27	137	137	0

Set No. 2

Compound	Method	Conc. Unspiked Sample (mg/kg)	Conc. Spike Added (mg/kg)	% Rec_1	% Rec_2	RPD
Arsenic	7060	52	27	32+	0+	200+
Barium	7080	751	540	91	91	0
Cadmium	7130	15	5.4	100	109	8.6
Chromium	7190	109	27	157+	90	27
Lead	7420	257	27	136	128	6.1
Mercury	7440	ND	2.0	72	78	8.0
Nickel	7520	59	27	129	105	20
Selenium	7740	ND	5.4	59	84	35+
Silver	7760	8.9	27	55	9+	144+
Thallium	7840	ND	27	70	54	26

* S-Cubed Laboratory
ND Not detected
\+ Outside QA objectives
RPD Relative Percent Difference = $(\%Rec_1 - \%Rec_2) + (\%Rec_1 + \%Rec_2)/2 \times 100$
$\%Rec_1$ First Sample Recovery
$\%Rec_2$ Duplicate Sample Recovery

TABLE B-9. METALS SPIKE/SPIKE DUPLICATE RECOVERY TCLP MATRIX*

Set No. 1

Compound	Method	Conc. Unspiked Sample mg/L	Conc. Spike Added mg/L	% Rec_1	% Rec_2	RPD
Arsenic	7060	ND	5.4	111	88	23+
Barium	7080	1.6	108	64+	65+	1.6
Cadmium	7130	ND	1.08	97	100	3.0
Chromium	7190	ND	5.4	122	117	4.2
Lead	7420	ND	5.4	99.4+	99.4	0
Mercury	7440	ND	0.216	111	117	5.3
Nickel	7520	ND	5.4	94	99	5.2
Selenium	7740	0.23	1.08	30+	45+	40+
Silver	7760	ND	5.4	81	73	10.4
Thallium	7840	ND	5.4	95	107	12

Set No. 2

Compound	Method	Conc. Unspiked Sample mg/L	Conc. Spike Added mg/L	% Rec_1	% Rec_2	RPD
Arsenic	7060	ND	5.4	110	189+	53+
Barium	7080	0.98	108	5.2+	5.2+	0
Cadmium	7130	ND	1.08	107	106	0.94
Chromium	7190	ND	5.4	93	101	8.2
Lead	7420	ND	5.4	4+	59+	175+
Mercury	7440	ND	0.216	97	91	6.4
Nickel	7520	ND	5.4	97	100	3.0
Selenium	7740	ND	1.08	75	82	8.9
Silver	7760	ND	5.4	6.2+	2.4+	88+
Thallium	7840	ND	5.4	106	107	0.94

* S-Cubed Laboratory
ND Not detected
\+ Outside QA objectives
RPD Relative Percent Difference = $(\%Rec_1 - \%Rec_2) \div (\% Rec_1 + \% Rec_2)/2 \times 100$
$\%Rec_1$ First Sample Recovery
$\%Rec_2$ Duplicate Sample Recovery

TABLE B-10. METALS SPIKE/SPIKE DUPLICATE RECOVERY
EP LEACHATE MATRIX*

Set No. 1

Compound	Method	Conc. Unspiked Sample mg/L	Conc. Spike Added mg/L	% Rec_1	% Rec_2	RPD
Arsenic	7060	0.20	5.4	102	115	12
Barium	7080	2.6	108	95	76	18
Cadmium	7130	0.11	1.08	102	95	7.1
Chromium	7190	ND	5.4	123	118	4.1
Lead	7420	ND	5.4	109	111	1.8
Mercury	7440	ND	0.216	-	80.5	-
Nickel	7520	0.30	5.4	98	94	4.2
Selenium	7740	ND	1.08	86	66+	26+
Silver	7760	ND	5.4	92	93	1.1
Thallium	7840	ND	5.4	108	105	2.8

Set No. 2

Compound	Method	Conc. Unspiked Sample mg/L	Conc. Spike Added mg/L	% Rec_1	% Rec_2	RPD
Arsenic	7060	0.43	5.4	99	95	4.1
Barium	7080	ND	108	95	76	22+
Cadmium	7130	ND	1.08	101	103	2.0
Chromium	7190	ND	5.4	102	105	2.9
Lead	7420	ND	5.4	11+	9+	20
Mercury	7440	ND	0.216	66+	65+	1.5
Nickel	7520	ND	5.4	102	105	2.9
Selenium	7740	ND	1.08	71	92	26+
Silver	7760	ND	5.4	97	99	2.0
Thallium	7840	ND	5.4	110	109	0.9

* S-Cubed Laboratory
ND Not detected
\+ Outside QA objectives
RPD Relative Percent Difference = (%Rec_1 - %Rec_2/) + (% Rec_1 + % Rec_2)/2 x 100
%Rec_1 First Sample Recovery
%Rec_2 Duplicate Sample Recovery

TABLE B-11. COMPOSITIONAL MATRIX SURROGATE PERCENT RECOVERY SUMMARY - ORGANIC ANALYSIS*

Compound	Percent Recovery-City No.											
	G	H	I	J	K	L	M	N	O	P	Q	R
VOLATILE ORGANICS (METHOD 8240)												
4-Bromofluorobenzene	117	103	150	NA	78	86	74	89	90	82	100	99
1,2-Dichloroethane-D_4	79	84	83	NA	69	44	82	118	70	105	88	87
Toluene-D_8	103	100	104	NA	93	97	98	95	118	103	100	96
SEMIVOLATILE ORGANICS (METHOD 8270)												
2-Fluorobiphenyl	69	34	59	52	71	59	75	73	73	22	82	79
2-Fluorophenol	33	26	57	49	80	62	61	63	60	1	59	65
Nitrobenzene-D_6	79	29	57	47	75	60	73	71	67	0	107	65
Phenol-D_5	56	19	21	26	65	49	66	58	59	5	72	58
Terphenyl-D_{14}	95	31	77	58	61	54	66	73	81	27	62	73
2,4,6-Tribromophenol	43	24	62	43	78	58	91	57	66	45	102	66

* S-Cubed Laboratory
ND Not analyzed

TBLE B-12. TCLP SURROGATE PERCENT RECOVERY SUMMARY - ORGANIC ANALYSIS*

Compound	Percent Recovery-City No.											
	G	H	I	J	K	L	M	N	O	P	Q	R
VOLATILE ORGANICS (METHOD 8240)												
4-Bromofluorobenzene	94	95	96	94	96	96	94	93	96	97	96	98
1,2-Dichloroethane-D_4	86	77	84	86	84	86	84	89	85	80	88	89
Toluene-D_8	92	94	95	93	95	95	92	84	93	101	99	100
SEMIVOLATILE ORGANICS (METHOD 8270)												
2-Fluorobiphenyl	83	92	89	75	72	76	67	111	69	88	81	66
2-Fluorophenol	82	91	79	60	63	48	57	42	45	37	56	23
Nitrobenzene-D_6	96	96	91	65	68	70	68	98	55	98	68	71
Phenol-D_5	75	82	57	46	52	33	41	16	77	22	46	16
Terphenyl-D_{14}	94	100	100	106	72	137	81	113	68	100	102	94
2,4,6-Tribromophenol	40	60	124	72	105	45	85	73	31	38	103	56

* S-Cubed Laboratory
ND Not analyzed

APPENDIX C

AMSA LABORATORY REPORTING LIMITS FOR TCLP AND COMPOSITIONAL ANALYSES OF SEWAGE SLUDGE

APPENDIX C. TABLE C-1A. TCLP VOLATILE REPORTING LIMITS FOR POTW SEWAGE SLUDGE ANALYSES

Constituent	EPA Contract Laboratories (median, mg/l)	TCLP Volatile Reporting Limits of AMSA Contract Laboratories+ for Analyses of POTW Sludges, mg/l											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Acrylonitrile	0.01	0.025	0.5	0.006	0.5	0.05	1.2	0.23	1.2	0.025	1.15	*	1.1
Benzene	0.005	0.005	0.025	0.0001	0.025	0.005	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Carbon disulfide	0.005	0.005	0.025	*	0.025	0.02	0.01	0.002	0.002	0.005	0.002	*	0.002
Carbon tetrachloride	0.005	0.005	0.025	0.0003	0.025	0.002	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Chlorobenzene	0.005	0.005	0.025	0.0004	0.025	0.005	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Chloroform	0.005	0.005	0.025	0.0002	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
1,2,-Dichloroethane	0.005	0.005	0.025	0.002	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.005	0.005	0.025	*	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Isobutanol (aka§ 2-Methyl-1-propanol)	*	0.05	*	*	*	0.02	1.2	0.23	1.2	0.050	1.15	*	1.1
Methylene chloride	0.005	0.005	0.025	*	0.025	0.01	0.025	0.005	0.005	*	0.005	0.001	0.005
Methyl ethyl ketone (aka 2-Butanone)	0.01	0,025	0.025	*	0.025	0.01	0.05	0.01	0.01	0.025	0.01	*	0.010
Pyridine	0.01	0.01	*	*	*	0.02	1.2	0.23	1.2	0.10	1.15	*	1.1
1,1,1,2, Tetra-chloroethane	0.01	0.005	*	*	*	0.003	1.2	0.23	1.2	0.005	1.15	0.0005	1.1
1,1,2,2, Tetra-chlorethane	0.005	0.005	0.025	0.0008	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Tetrachlorethylene (aka§ Tetrachloroethene)	0.005	0.005	0.025	*	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Toluene	0.005	*	0.025	0.002	0.025	0.005	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
1,1,1-Trichloro-ethane	0.005	0.005	0.025	0.0003	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
1,1,2-Trichloro-ethane	0.005	0.005	0.025	0.0004	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0.0005	0.002
Trichloroethylene (aka§ Trichloroethene)	0.005	0.005	0.025	*	0.025	0.003	0.01	0.002	0.002	0.005	0.002	0,0005	0.002
Vinyl Chloride	0.01	0.005	0.05	0.0006	0.050	0.002	0.025	0.005	0.005	0.005	0.002	*	0.005

+ = AMSA Contract Laboratory Unless Indicated by "(POTW)" Meaning AMSA POTW Laboratory
++ = Letter Denotes Municipal POTW, (Number) Denotes the Specific AMSA Contract Laboratory
* = No Data Available
§ = Also Known As

APPENDIX C. TABLE 1A Cont. TCLP VOLATILE REPORTING LIMITS FOR POTW SEWAGE SLUDGE ANALYSES

Constituent	EPA Contract Laboratories Median, mg/l)	TCLP Volatile Reporting Limits of AMSA Contract Laboratories+ for Analyses of POTW Sludges, mg/l											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
NON-TCLP ANALYTES													
Acetone	*	0.05	0.025	*	0.025	0.02	0.25	0.5	0.05	0.05	0.05	*	0.05
Dibromomethane	*	*	*	*	*	*	*	*	*	*	*	*	*
1,1-Dichloroethene	0.005	*	*	0.0003	*	0.003	0.01	0.002	0.002	*	0.002	0.0005	0.002
Ethylbenzene	0.005	*	0.025	*	0.025	0.005	0.01	0.002	0.002	0.05	0.002	0.0005	0.002
2, Hexanone	0.01	*	*	*	*	*	0.05	0.01	0.01	*	0.01	*	0.01
Isopropanol	*	*	*	*	*	*	*	*	*	*	*	*	*
Methyl isobutyl ketone (aka§ 4-methyl-2-pentanone)	0.01	0.01	0.025	*	0.025	0.02	0.05	0.01	0.01	*	0.01	*	0.01
Styrene	0.005	*	*	*	*	0.005	0.01	0.002	0.002	*	0.002	*	0.002
Trans 1,2 dichlorethene	0.005	*	*	0.0002	*	0.005	0.01	0.002	0.002	*	0.002	0.0005	0.002
Xylene	0.005	*	0.025	*	*	0.005	0.01	0.002	0.002	*	0.002	*	0.002

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* = No Data Available

APPENDIX C. TABLE C-1B. COMPOSITIONAL VOLATILE REPORTING LIMITS FOR POTW SEWAGE SLUDGE ANALYSES

Constituent	EPA Contract Laboratories (median)	Dry Weight Basis, mg/kg — Compositional Volatile Reporting Limits of AMSA Contract Laboratories+ for Analyses of POTW Sludges											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Acrylonitrile	1.0	0.12	353	2.4	631	5.0	3.9	1.65	17.2	0.025	68	*	1.3
Benzene	0.5	*	18	0.044	*	0.5	0.034	0.014	0.15	*	0.59	0.5	0.011
Carbon disulfide	0.5	0.023	18	*	33	2.0	0.034	0.014	0.15	0.005	0.59	*	0.011
Carbon tetrachloride	0.5	0.023	18	0.13	33	0.2	0.034	0.014	0.15	0.005	0.59	0.2	0.011
Chlorobenzene	0.5	0.023	18	0.14	33	0.5	0.034	0.014	0.15	0.005	0.59	0.03	0.011
Chloroform	0.5	0.023	18	0.064	33	0.3	0.034	0.014	0.15	0.005	0.59	0.1	0.011
1,2,-Dichloroethane	0.5	0.023	18	0.069	33	0.3	0.034	0.014	0.15	0.005	0.59	1.0	0.011
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.5	0.023	18	*	33	0.3	0.034	0.014	0.15	0.005	0.59	1.0	0.011
Isobutanol (aka§ 2-Methyl-1-propanol	*	0.23	*	*	*	2.0	3.9	1.65	17.2	*	68	*	1.3
Methylene chloride	0.5	0.023	18	*	33	1.0	0.085	0.035	0.38	*	1.5	0.5	0.028
Methyl ethyl ketone (aka 2-Butanone)	0.5	*	*	*	*	1.0	0.17	0.069	0.74	0.025	3.0	*	0.057
Pyridine	1.0	0.47	18	*	*	2.0	3.9	1.65	17.2	0.10	68	*	1.3
1,1,1,2, Tetra-chloroethane	1.0	0.023	*	*	*	0.3	3.9	1.65	17.2	0.005	68	0.5	1.3
1,1,2,2, Tetra-chlorethane	0.5	0.023	18	0.032	33	0.3	0.034	0.014	0.15	0.005	0.59	1.0	0.011
Tetrachlorethylene (aka Tetrachloroethene)	0.5	*	18	*	33	0.3	0.034	0.014	0.15	0.005	0.59	0.5	0.011
Toluene	0.5	*	18	0.84	*	0.5	0.034	0.014	0.15	*	0.59	0.5	0.011
1,1,1-Trichloro-ethane	0.5	0.023	18	0.11	33	0.3	0.034	0.014	0.15	*	0.59	0.2	0.011
1,1,2-Trichloro-ethane	0.5	0.023	18	0.18	33	0.3	0.034	0.014	0.15	0.005	0.59	0.5	0.011
Trichloroethylene (aka Trichloroethene)	0.5	*	18	*	33	0.3	0.034	0.014	0.15	0.005	0.59	0.2	0.011
Vinyl Chloride	1.0	0.023	35	0.22	63	0.2	0.085	0.035	0.38	0.005	1.5	*	0.028

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APPENDIX C. TABLE C-1B Cont. COMPOSITIONAL VOLATILE REPORTING LIMITS FOR POTW SEWAGE SLUDGE ANALYSES

Constituent	EPA Contract Laboratories (median)	Dry Weight Basis, mg/kg — Compositional Volatile Reporting Limits of AMSA Contract Laboratories+ for Analyses of POTW Sludges											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
NON-TCLP ANALYTES													
Acetone	*	*	18	*	*	2.0	0.85	0.35	3.7	*	14.8	*	0.28
Dibromomethane	*	0.047	*	*	*	*	*	*	*	0.005	*	*	*
1,1-Dichloroethene	0.5	0.023	18	0.13	32	0.2	0.034	0.014	0.15	0.005	0.59	1.0	0.011
Ethylbenzene	0.5	*	18	*	*	0.5	0.034	0.014	0.15	0.005	0.59	0.5	0.011
2, Hexanone	1.0	0.05	18	*	32	*	0.17	0.069	0.74	0.01	3.0	*	0.057
Isopropanol (aka§ 2-Methyl-1propanol)	*	*	*	*	*	*	*	*	*	*	*	*	*
Methyl isobutyl ketone aka§ 4-methyl-2-pentanone)	1.0	0.05	18	*	32	2.0	0.17	0.069	0.74	0.01	3.0	*	0.057
Styrene	0.5	0.02	18	*	32	0.5	0.034	0.014	0.15	0.005	0.59	*	0.011
Trans 1,2 dichlorethene	0.5	*	18	0.096	32	0.2	0.034	0.014	0.15	0.005	0.59	*	0.011
Xylene	0.5	*	18	*	*	0.5	0.034	0.014	0.15	0.005	0.59	*	0.001

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:PENDIX C. TABLE C-2A. TCLP SEMIVOLATILE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median, mg/1)	TCLP Semivolatile Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges, mg/1											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Bis(2-chloroethyl) ether	0.01	0.01	0.23	0.0006	0.13	0.01	0.02	0.02	0.02	0.01	0.04	0.005	0.02
o-Cresol (aka§ 2-Methyl Phenol)	0.01	0.01	0.23	*	0.13	0.02	0.1	0.02	0.08	0.01	0.2	0.001	0.02
m-Cresol (aka§ 3-Methyl Phenol)	0.01	*	0.23	*	0.13	0.02	0.1	0.02	0.08	0.01	0.2	0.001	0.02
p-Cresol (aka§ 4-Methyl Phenol)	0.01	*	0.23	*	0.13	0.02	0.1	0.02	0.08	0.01	0.2	0.001	0.02
1,2 Dichlorobenzene	0.01	0.01	0.23	0.0007	0.13	0.01	0.02	0.02	0.02	0.01	0.04	0.0005	0.02
1,4 Dichlorobenzene	0.01	0.01	0.23	0.0006	0.13	0.01	0.02	0.02	0.02	0.01	0.04	0.0005	0.02
2,4 Dinitrotoluene	0.01	0.01	0.23	0.0009	0.13	0.02	0.02	0.02	0.02	0.01	0.04	0.0005	0.02
Hexachlorobenzene	0.01	0.01	0.23	0.001	0.13	0.005	0.02	0.02	0.02	0.01	0.04	0.00005	0.02
Hexachlorobutadiene	0.01	0.01	0.23	0.001	0.13	0.01	0.02	0.02	0.02	0.01	0.04	0.0001	0.02
Hexachloroethane	0.01	0.01	0.23	0.003	0.13	0.005	0.02	0.02	0.02	0.01	0.04	0.004	0.02
Nitrobenzene	0.01	0.01	0.23	0.004	0.13	0.01	0.02	0.02	0.02	0.01	0.04	0.001	0.02
Pentachlorophenol	0.05	0.01	1.2	0.007	0.64	*	0.1	0.02	0.08	0.01	0.2	0.005	0.02
Phenol	0.01	0.01	0.012	0.001	0.13	*	0.1	0.02	0.08	0.01	0.2	0.005	0.02
2,3,4,6-Tetrachlorophenol	0.01	0.01	*	*	*	*	0.1	0.02	0.08	0.01	0.2	*	0.02
2,4,5-Trichlorophenol	0.05	0.01	0.23	*	0.13	*	0.1	0.02	0.08	0.01	0.2	*	0.02
2,4,6-Trichlorophenol	0.01	0.01	0.012	*	0.13	*	0.1	0.02	0.08	0.01	0.2	0.01	0.02

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APPENDIX C. TABLE C-2A Cont. TCLP SEMIVOLATILE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median, mg/l)	TCLP Semivolatile Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges, mg/l											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
NON-TCLP ANALYTES													
Acenaphthene	0.01	*	*	0.0006	*	0.005	0.02	0.02	0.02	*	0.04	*	0.02
Acenaphthylene	0.01	*	*	0.0005	*	0.005	0.02	0.02	0.02	*	0.04	*	0.02
Anthracene	0.01	*	*	0.0009	*	0.01	0.02	0.02	0.02	*	0.04	*	0.02
Benzoic Acid	*	*	*	*	*	0.05	*	*	*	*	*	*	*
Benzo (a) -anthracene	0.01	*	*	0.002	*	0.015	0.02	0.02	0.02	*	0.04	*	0.02
Benzo (b)-fluoranthene	0.01	*	*	0.002	*	0.025	0.02	0.02	0.02	*	0.04	*	0.02
Bis (2-Ethyl hexyl) phthalate	0.01	*	*	0.005	*	0.015	0.02	0.02	0.02	*	0.04	0.001	0.02
Butyl benzyl phthalate	0.01	*	*	0.0008	*	0.01	0.02	0.02	0.02	*	0.04	0.001	0.02
4-Chloroanaline	0.01	*	*	*	*	*	*	*	*	*	*	*	*
Chrysene	0.01	*	*	0.001	*	0.015	0.02	0.02	0.02	*	0.04	*	0.02
Diethyl phthlate	0.01	*	*	0.0007	*	0.01	0.02	0.02	0.02	*	0.04	0.002	0.02
Di-n-butyl phthalate	0.01	*	*	0.0006	*	0.01	0.02	0.02	0.02	*	0.04	0.0005	0.02
Di-n-octyl phthalate	0.01	*	*	0.0009	*	0.015	0.02	0.02	0.02	*	*	*	0.02
Fluoranthene	0.01	*	*	0.0004	*	0.005	0.02	0.02	0.02	*	0.04	*	0.02
Fluorene	0.01	*	*	0.0004	*	0.005	0.02	0.02	0.02	*	0.04	*	0.02
Indeno(1,2,3,-cd)pyrene	0.01	*	*	0.005	*	0.025	0.02	0.02	0.02	*	0.04	*	0.02
2-Methylnapthalene	0.01	*	*	*	*	0.005	*	*	*	*	*	*	*
Napthalene	0.01	*	*	0.0007	*	0.005	0.02	0.02	0.02	*	0.04	0.0005	0.02
Phenanthrene	0.01	*	*	0.0004	*	0.01	0.02	0.02	0.02	*	0.04	*	0.02
Pyrene	0.01	*	*	0.0007	*	0.01	0.02	0.02	0.02	*	0.04	*	0.02

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APPENDIX C. TABLE C-2B. COMPOSITIONAL SEMIVOLATILE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median)	Dry Weight Basis, mg/kg — Compositional Semivolatile Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Bis((2-chloroethylether) ether	6.5	4.7	*	0.024	105	0.4	130	31	120	5.9	260	3	25
o-Cresol (aka§ 2-Methyl Phenol)	6.5	4.7	5.9	*	105	0.8	130	31	120	5.9	260	5	25
m-Cresol (aka 3-Methyl Phenol)	6.5	*	*	*	*	0.8	130	31	120	5.9	260	5	25
p-Cresol (aka§ 4-Methyl Phenol)	6.5	*	*	*	105	0.8	130	31	120	5.9	260	5	25
1,2 Dichlorobenzene	6.5	4.7	*	0.028	105	1.0	130	31	120	5.9	260	3	25
1,4 Dichlorobenzene	6.5	4.7	*	0.022	105	1.0	130	31	120	5.9	260	3	25
2,4 Dinitrotoluene	6.5	4.7	*	0.036	105	0.8	130	31	120	5.9	260	3	25
Hexachlorobenzene	6.5	4.7	*	1.48	105	0.2	130	31	120	5.9	260	0.1	25
Hexachlorobutadiene	6.5	4.7	*	0.44	105	0.8	130	31	120	5.9	260	0.1	25
Hexachloroethane	6.5	4.7	*	0.51	105	0.2	130	31	120	5.9	260	0.8	25
Nitrobenzene	6.5	4.7	*	1.07	105	0.4	130	31	120	5.9	260	3	25
Pentachlorophenol	32	4.7	29	0.26	526	*	130	31	120	5.9	260	6	25
Phenol	6.5	4.7	5.9	0.45	105	*	130	31	120	5.9	260	5	25
2,3,4,6-Tetrachlorophenol	13	4.7	*	*	105	*	130	31	120	5.9	260	*	25
2,4,5-Trichlorophenol	32	4.7	5.9	*	105	*	130	31	120	5.9	260	*	25
2,4,6-Trichlorophenol	6.5	4.7	5.9	*	105	*	130	31	120	5.9	260	5	25

+ = AMSA Contract Laboratory Unless Indicated by "(POTW)" Meaning AMSA POTW Laboratory
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* = No Data Available

APPENDIX C. TABLE C-2B Cont. COMPOSITIONAL SEMIVOLATILE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median)	Dry Weight Basis, mg/kg — Compositional Semivolatile Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
NON-TCLP ANALYTES													
Acenaphthene	6.5	4.7	5.9	0.022	112	0.2	130	31	120	1.0	260	*	25
Acenaphthylene	6.5	4.7	5.9	0.021	112	0.2	130	31	120	1.0	260	*	25
Anthracene	6.5	4.7	5.9	0.035	112	0.4	130	31	120	1.0	260	*	25
Benzoic Acid	*	47	29	*	561	2.0	*	*	*	10	*	*	*
Benzo (a) -anthracene	6.5	4.7	5.9	0.86	112	0.6	130	31	120	1.0	260	*	25
Benzo (b) -fluoranthene	6.5	4.7	5.9	0.74	112	1.0	130	31	120	1.0	260	*	25
Bis (2-Ethyl hexyl) phthalate	6.5	4.7	5.9	0.20	112	0.6	130	31	120	1.0	260	3	25
Butyl benzyl phthalate	6.5	4.7	5.9	0.033	112	0.4	130	31	120	1.0	260	3	25
4-Chloroanaline	6.5	4.7	5.9	*	112	*	*	*	*	1.0	*	*	*
Chrysene	6.5	4.7	5.9	0.043	112	0.6	130	31	120	1.0	260	*	25
Diethyl phthlate	6.5	4.7	5.9	0.029	112	0.4	130	31	120	1.0	260	3	25
Di-n-butyl phthalate	6.5	4.7	5.9	0.022	112	0.4	130	31	120	1.0	260	3	25
Di-n-octyl phthalate	6.5	*	5.9	0.018	112	0.6	130	31	120	1.0	260	*	25
Fluoranthene	6.5	4.7	5.9	0.0084	112	0.2	130	31	120	1.0	260	*	25
Fluorene	6.5	4.7	5.9	0.074	112	0.2	130	31	120	1.0	260	*	25
Indeno(1,2,3,-cd)pyrene	6.5	4.7	5.9	0.094	112	1.0	130	31	120	1.0	260	*	25
2-Methylnapthalene	6.5	4.7	5.9	*	112	0.2	*	*	*	1.0	*	*	*
Napthalene	6.5	4.7	5.9	0.014	112	0.2	130	31	120	1.0	260	0.2	25
Phenanthrene	6.5	4.7	5.9	0.017	112	0.4	130	31	120	1.0	260	*	25
Pyrene	6.5	4.7	5.9	0.030	112	0.4	130	31	120	1.0	260	*	25

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APPENDIX C. TABLE C-3A. TCLP METAL REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median, mg/1)	TCLP Metal Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges, mg/1												
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES														
Arsenic	0.25	*	0.01	0.3	0.01	0.015	0.003	0.01	0.01	0.01	*	0.01	0.0005	*
Barium	0.90	0.2	0.2	0.2	0.2	0.15	0.03	0.1	0.1	0.1	*	0.1	0.1	*
Cadmium	0.10	*	0.005	0.02	0.005	0.05	0.01	0.02	0.02	0.02	*	0.02	0.0002	*
Chromium	0.33	*	0.03	0.03	0.03	0.05	0.01	0.2	0.2	0.2	0.01	0.2	0.001	*
Lead	0.62	*	0.05	0.02	0.05	0.5	0.1	0.2	0.2	0.2	0.2	0.2	0.002	*
Mercury	0.01	*	0.0002	0.0002	0.0002	0.001	0.0002	0.0002	0.0002	0.0002	0.01	0.0002	0.0002	*
Selenium	0.10	0.001	0.01	0.3	0.01	0.04	*	0.02	0.02	0.02	0.047	0.02	0.0002	*
Silver	0.09	1.0	0.02	0.02	0.02	0.1	0.02	0.02	0.02	0.02	0.01	0.02	0.0002	*
NON-TCLP ANALYTES														
Nickel	0.22	*	*	*	0.2	0.05	0.01	0.1	0.1	0.1	*	0.27	0.01	*
Thallium	0.43	*	0.05	*	0.05	0.03	0.006	0.01	0.01	0.01	*	0.01	*	*

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APPENDIX C. TABLE C-3B. COMPOSITIONAL METAL REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median)	Dry Weight Basis, mg/kg — Compositional Metal Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges												
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES														
Arsenic	4.3	*	*	*	*	2	1	3.9	3.4	33	*	6.3	0.5	*
Barium	15	*	*	*	*	20	*	3.8	4.5	40	*	3.1	44	*
Cadmium	5.1	*	*	*	*	8	4	0.77	0.89	7.9	*	0.62	1.0	*
Chromium	16	*	*	*	*	8	4	7.7	8.9	79	*	6.3	50	*
Lead	4.0	*	*	*	*	80	44	7.7	8.9	79	*	6.3	50	*
Mercury	1.4	*	*	*	*	0.2	0.09	0.077	0.089	0.79	0.1	0.063	0.7	*
Selenium	2.7	*	1.0	*	*	5	2.6	3.9	3.4	33	1.0	3.1	0.1	*
Silver	2.8	*	*	*	*	18	4	0.77	0.89	7.9	*	0.62	50	*
NON-TCLP ANALYTES														
Nickel	16	*	12	*	5	8	4	3.9	4.5	40	70	3.1	50	*
Thallium	20	*	29	*	2	1	2.6	3.7	3.4	33	*	3.2	*	*

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APPENDIX C. TABLE C-4A. TCLP PESTICIDE AND HERBICIDE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Laboratories (median, mg/l)	TCLP Pesticide & Herbicide Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges, mg/l											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Chlordane	0.001	0.001	0.02	0.0003	0.003	0.0005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.001
Endrin	0.2	0.0005	0.01	0.00009	0.003	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Heptachlor	0.0001	0.005	0.01	0.00005	0.003	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Lindane	0.0001	0.0005	0.01	0.00003	0.003	0.00005	*	0.0001	0.0001	*	0.002	0.0001	0.0001
Methoxychlor	0.001	0.001	0.01	*	0.045	0.0001	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Toxaphene	0.002	0.01	0.02	0.0003	0.015	0.001	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
2,4-D	0.02	0.005	0.01	*	0.01	0.0005	0.002	0.002	0.002	*	0.002	*	0.0001
2,4,5,TP (Silvex)	0.02	0.005	0.01	*	0.01	0.0002	0.002	0.002	0.002	*	0.002	*	0.002
NON-TCLP ANALYTES													
Aldrin	0.001	*	*	0.00005	*	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	1.0001
alpha-BHC	0.0001	*	*	*	*	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
beta-BHC	0.0001	*	*	*	*	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
delta-BHC	0.0001	*	*	*	*	0.00005	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Chlordene "237"	*	*	*	*	*	*	*	*	*	*	*	0.0001	*
2,4-DB	*	*	*	*	*	*	*	*	*	*	*	*	*
4,4'-DDD	0.2	*	*	*	*	0.0001	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
4,4'-DDT	0.2	*	*	0.0002	*	0.0001	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Dinoseb	*	*	*	*	*	*	*	*	*	*	*	*	*
Endosulfan II	0.2	*	*	0.00014	*	0.00008	0.0001	0.0001	0.0001	*	0.002	0.0001	0.0001
Endrin keytone	*	*	*	*	*	*	*	*	*	*	*	0.0001	*
Hexachloronobornadiene	*	*	*	*	*	*	*	*	*	*	*	0.0001	*
Heptachlornobornene	*	*	*	*	*	*	*	*	*	*	*	0.0001	*
MCPP	*	*	*	*	*	*	*	*	*	*	*	*	*
Octachlorocyclopentene	*	*	*	*	*	*	*	*	*	*	*	0.0001	*
PCB-1248	*	*	*	*	*	0.002	0.0001	0.0001	0.0001	*	0.002	*	0.0001
PCB-1254	*	*	*	*	*	0.002	0.0001	0.0001	0.0001	*	0.002	*	0.0001

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* = No Data Available

APPENDIX C. TABLE C-4B. COMPOSITIONAL PESTICIDE AND HERBICIDE REPORTING LIMITS FOR POTW SLUDGE ANALYSES

Constituent	EPA Contract Lababoratories (median)	Dry Weight Basis, mg/kg Compositional Pesticide & Herbicide Reporting Limits of AMSA Contract Laboratories+ for Analysis of POTW Sludges											
		G(1)++	H(2)	I(POTW)	K(2)	L(POTW)	M(3)	N(3)	O(3)	P(1)	Q(3)	R(POTW)	R(3)
TCLP ANALYTES													
Chlordane	5.5	0.47	0.29	0.10	*	0.01	0.5	1.0	1.0	0.10	0.3	0.1	0.5
Endrin	1.05	0.23	0.15	0.0036	0.13	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Heptachlor	0.55	0.23	0.29	0.002	0.13	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Lindane	0.55	0.23	*	0.012	0.13	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Methoxychlor	5.5	0.47	*	*	0.13	0.002	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Toxaphene	10.5	0.47	5.9	0.10	0.13	0.02	0.5	1.0	1.0	0.10	0.3	*	0.5
2,4-D	0.1	0.047	*	*	*	0.01	0.8	0.8	2.2	0.50	0.3	*	0.5
2,4,5,TP (Silvex)	0.1	0.047	*	*	*	0.004	0.8	0.8	2.2	0.01	0.6	*	0.7
NON-TCLP ANALYTES													
Aldrin	0.55	0.23	0.15	0.002	0.11	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
alpha-BHC	0.55	0.23	0.15	*	0.18	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
beta-BHC	0.55	0.23	0.15	*	0.13	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
delta-BHC	0.55	0.23	0.15	*	0.13	0.001	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Chlordene "237"	*	*	*	*	6.3	*	*	*	*	*	*	0.1	*
2,4-DB	*	0.047	1.2	*	0.13	*	*	*	*	0.01	*	*	*
4,4'-DDD	1.05	0.23	0.16	*	0.13	0.002	0.5	1.0	1.0	0.05	0.3	0.1	0.5
4,4'-DDT	1.05	0.23	0.15	0.088	0.13	0.002	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Dinoseb	*	2.3	0.24	*	0.11	*	*	*	*	0.05	*	*	*
Endosulfan II	1.05	0.23	0.16	0.056	0.39	0.002	0.5	1.0	1.0	0.05	0.3	0.1	0.5
Endrin keytone	*	4.7	5.7	*	0.26	*	*	*	*	5.9	*	0.5	*
Hexachloronobornadiene	*	*	*	*	*	*	*	*	*	*	*	0.1	*
Heptachlornobornene	*	*	*	*	*	*	*	*	*	*	*	0.1	*
MCPP	*	2.3	44	*	19	*	*	*	*	0.05	*	*	*
Octachlorocyclopentene	*	*	*	*	*	*	*	*	*	*	*	0.1	*
PCB-1248	*	0.47	2.9	*	0.53	0.04	0.5	1.0	1.0	0.10	0.3	*	0.5
PCB-1254	*	0.47	2.9	*	0.53	0.04	0.5	1.0	1.0	0.10	0.3	*	0.5

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APPENDIX D

REPORT ON SIX POTW SLUDGE TCLP STUDY

(from a memo by John Walker, dated 7-11-86)

APPENDIX D: Report on Six POTW Sludge TCLP Study

PURPOSE

This report describes the results of Compositional and Toxicity Characteristic Leaching Procedure (TCLP) testing of sewage sludges from six publically owned treatment works (POTWs).

INTRODUCTION

The six POTW sludges were sampled in November 1985 and subsequently analyzed by a laboratory* under contract to the EPA Office of Solid Waste and Emergency Response. Results are incomplete because limited equipment was available at the time of testing and some test procedures have subsequently been revised. More specifically, (1) the zero headspace extractors were unavailable for use on this project until nearly two months beyond the desired maximum two week holding period for sludge samples to be extracted for volatiles, (2) final adjustments were still being made during this period to chemicals being used for the TCLP extraction of samples which are different pH's and (3) necessary equipment and procedures were not available for determining the presence of all 52 compounds in the solutions extracted from the sludges. Because of these difficulties, 15 of the 52 TCLP compounds listed in Table D-1, were not analyzed.

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*ERCO Laboratories in Cambridge, Mass. Mention of tradenames and names of vendors is for the benefit of the reader and does not imply endorsement by the US Environmental Protection Agency.

POTW AND SLUDGE CHARACTERISTICS

Characteristics of the six POTWs involved and their sampled sludges are presented in Table D-2. Average daily flows for the six POTWs ranged from less than 10 to over 500 million gallons per day (MGD) with the industrial contributions varying from less than one percent to about 60 percent. Most of the sludges were anaerobically digested and most were dewatered. One sludge was aerobically digested and not dewatered. The sludge pH's ranged from about 6.4 to 8.0.

RESULTS AND DISCUSSION

The TCLP extract concentrations for those volatile analytes detected (including both the volatiles listed for TCLP analysis or other volatiles found) are given in Table D-3. The data indicated that sludge for City "A" was the worst based upon its volatile contents being closest to the Toxicity Characteristic Regulatory Levels (TCRLs). Sludge "A" came from a POTW with less than 10 MGD flow of which industrial sources contribute less than 1 percent. This sludge approached "failure" of the TCLP test due to the chloroform and benzene concentrations in the TCLP extract. In fact, this sludge would have been considered "hazardous" based upon an earlier proposed TCRL for chloroform that was lower. The presence of several printing and photographic business, discharging wastewater to City "A's" POTW may be part of the explanation of this phenomenon. Sludge "E" also approached "failure". However, it came from a community with about 60 percent of its almost 30 MGD flow from industrial sources. This sludge would also have failed, based upon earlier threshold concentration proposals, again because of its TCLP extract chloroform content.

Still another sludge from City "B" with greater than 300 MGD flow, of which less than one percent was of industrial origin, actually had one value for a volatile compound in the TCLP extract that exceeded the TCRL. The compound was tetrachloroethylene with one measurement indicating a content of 11.0 mg/ml as compared with the TCRL for this compound of 0.1 mg/ml. However, this same volatile compound was not detected in three other TCLP extracts of this same sludge and the high value may likely have been the result of laboratory contamination. Also, the total compositional content of tetrachloroethylene was only 0.16 mg/ml (a mean of two determinations) (Table D-4).

The TCLP extract concentrations for heavy metals are given in Table D-5. None of the sludge TCLP extract metal concentrations were very close to the TCRLs. The TCLP concentrations nearest the TCRLs were for lead and cadmium in POTW Sludge "C". Those TCLP concentrations are about one-tenth the TCRL. The compositional dry weight concentrations of metals (Table D-6) were used along with the TCLP extract concentrations (Table D-5) to calculate the ratios in Table D-9.

Extraction Procedure (EP) metal analyte concentrations were usually lower for the six POTW sludges than were the TCLP metal analyte concentrations except for Sludge from City "C" (Table D-7). While TCLP metal levels were higher than EP metal levels, as might be expected because of a somewhat more vigorous TCLP extractant, the differences were not great. Since metal concentrations in the EP extracts have rarely caused sludges to fail and since the EP and TCLP extract levels are not very different, few POTW sludges are expected to fail because of their metal contents.

Some persons have proposed using wet weight compositional analysis as an index for predicting the TCLP extract concentration. The usefulness of this "index" should depend upon demonstrating that such a relationship exists.

The TCLP extract to wet weight sludge compositional metal content ratios are given in Table D-8. An examination of this data revealed that this ratio is not constant for a given metal. In fact, it varies over 1000-fold for a given metal analyte with the variance apparently being strongly affected by the sludge moisture content. On the other hand, the ratio of the TCLP extract to dry weight sludge compositional metal content ratios (given in Table D-9) varied less (only about 10-fold). Examination of the ratios in Table D-9 for individual metals shows that the ratios are about 100-fold different from one another because of the difference in the TCLP extractabilities (solubilities) of the various metals tested (lead being the most insoluble and barium and especially nickel being the most soluble). Using different ratios each derived from an individual metal or the median ratio derived from all of the individual metal ratios could be used as a multiplier times the dry weight sludge compositional concentration to obtain a rough estimate of the TCLP extract concentration of that metal. While using individual ratios would be more precise, using the median of all ratios could be useful to obtain a very rough estimate.

A similar examination of the ratio of TCLP extract to dry weight compositional concentration for volatiles was attempted (Table D-10). Since volatiles were detected for many fewer compounds, reliable evaluations are not possible for either the constancy of the ratio or its usefulness in predicting the TCLP extract concentrations of volatiles.

The only semivolatiles analytes detected were 1,4- and 1,2-dichlorobenzene (0.31 and 0.35 mg/ml, respectively). Furthermore, no herbicide and pesticide TCLP analytes were detected. Hence, ratios could not be calculated for these three classes of TCLP analytes.

CONCLUSIONS

We reached the following tentative conclusions based upon the incomplete results shown in Tables D-2 through D-10.

1. No POTW sewage sludge failed the test.

2. Two of the six POTW sludges approached failure of the TCLP test for volatile components and would have failed if an earlier set of TCRLs had not been recalculated and changed.

One of the POTW sludges that approached failure was from a smaller community (less than 10 MGD) with 99% of its wastewater input being of domestic origin.

3. Volatile components in POTW sewage sludges are most likely to cause failure of the TCLP test. Failure caused by semivolatile and herbicide and pesticide contents is most unlikely.

4. Since the EP and TCLP analyte concentrations are not too different and since few sludges have had metal analyte concentrations exceeding the EP toxicity thresholds in the past, few sludges are expected to fail the TCLP test and hence, be considered hazardous because of metal content.

5. The ratios of TCLP extract metal concentration to compositional dry weight metal concentration varied only within a factor of about 10 for a given metal in all six sludges. These rations could be used to very roughly predict the TCLP metal extract concentration in sludges.

Use of wet weight sludge compositional metal concentrations to determine the ratio for predicting the TCLP extract metal concentrations was unsuitable because the different sludge moisture levels caused as much as a 1000-fold variation in the ratio of TCLP extract concentration to wet weight sludge concentration.

The use of the TCLP extract concentration to dry weight sludge concentration ratio may be suitable for estimating concentrations of volatiles compounds, but the data were insufficient to support such an hypothesis.

6. Only two semivolatile analytes were detected and these were in only one of the six sludges examined. No pesticides or herbicides were detected in the six sludges.

FUTURE

The above conclusions are clearly tentative, recognizing the uncertainties discussed. To obtain better results and hopefully sounder findings, twelve additional POTW sludges underwent compositional and TCLP testing. Better quality assurance and quality control procedures were used. Samples were collected and split to allow separate testing by each of the 12 POTWs or their contractors and the EPA contract laboratory. Results of both sets of analyses have been assembled and compared. The results of this study constitute the main body of this report to which Appendix D is attached.

Appendix D. TABLE D-1. TCLP COMPOUNDS

TCLP COMPOUNDS FOR WHICH		
No Analyses Were Run	Analyses Were Run	
Volatiles (No zero head space data)	Volatiles	Semivolatiles
Acrylonitrile	Benzene	Bis(2-chloroethyl) ether
1,1-Dichloroethylene	Carbon disulfide	1,2,-Dichlorobenzene
Isobutanol	Carbon tetrachloride	1,4-Dichloroebenzene
Pyridine	Chlorobenzene	2,4-Dinitrotolulene
1,1,1,2-Tetrachloroethane	Chloroform	Hexchlorbenzene
Tetrachloroethylene	1,2-Dichloroethane	Hexachlorobutadiene
Trichloroethylene	Methylene chloride	Hexachloroethane
	Methyl ethyl keytone	Nitrobenzene
	1,1,2,2-Tetrachloroethane	
	Tolulene	
	1,1,1-Trichloroethane	
	1,1,2-Trichloroethane	
	Vinyl Chloride	
Semivolatiles (No TCLP Data)	Herbicides & Pesticides	Metal
o-Cresol	Chlordane	Arsenic
m-Cresol	Endrin	Barium
p-Cresol	Heptachlor	Cadmium
Pentachlorophenol	Lindane	Chromium
Phenol	Methoxychlor	Lead
2,3,4,6-Tetrachlorophenol	Toxaphene	Mercury
2,4,5-Trichlorophenol	2,4-D	Selenium
2,4,6-Trichlorophenol	2,4,5-TP	Silver

APPENDIX D. TABLE D-2. CHARATERISTICS OF THE SIX POTW'S AND THEIR SLUDGES IN THE 1985 TCLP AND COMPOSITIONAL TEST SERIES

POTW	Daily Flow Range	Percent Industrial Flow	Type of Wastewater Treatment	Sludge Parameters			
				Type of Treatment	Sampled from the	pH	% Water
A	.10	1%	Primary & waste activated	Lime stabilzed (sampled before lime treatment for test)	Belt Filter Press	6.4	80
B	300-500	1%	Primary & waste activated with chemical P removal & nitri-cation	Anaerobically digested and centifuged	Filter	7.4	74
C	50-100	20%	Primary & pure oxygen	Aerobic digestors w/pure oxygen for 5 days	Sludge gravity thickner	6.4	96
D	300-500	20%	Primary & 40% waste activated	Anaerobically digested and centrifuged	Centrifuge	7.9	77
E	10-50	60%	Primary & aeration	Extend aeration	Aeration basin	8.0	79
F	Over 500	8%	Primary & waste activated	Anaerobically digested and centrifuged	Centifuge	7.4	81

APPENDIX D. TABLE D-3. VOLATILE ANALYTES CONCENTRATION IN TCLP EXTRACTS OF SLUDGES COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory +++ Level, mg/L	Concentration of Volatiles in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/L					
		A (1%)	B (1%)	C (20%)	D (20%)	E (60%)	F (8%)
TCLP ANALYTES							
Benzene	0.07	0.026+	*	*	*	*	*
Carbon disulfide	14.4	*	*	*	0.015	*	0.16
Chlorobenzene	1.4	*	*	*	*	*	0.073
Chloroform	0.07	0.023+	*	*	*	0.01+	*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	0.1	0.015	*	*	0.013	0.01	*
Methylene chloride	8.6	0.041	*	0.085	0.063	*	0.1
Methyl ethyl ketone (aka§ 2-Butanone)	7.2	0.51	*	0.34	0.083	0.19	0.59
1,1,2,2, Tetra-chloroethane	1.3	*	*	*	*	0.018	*
Tetrachloroethylene (aka§ Tetrachlorethene)	0.1	0.014	*++	0.042	0.011	0.025	0.069
Toluene	14.4	0.05	*	0.12	0.049	*	*
1,1,1-Trichloroethane	30.0	*	*	0.028	*	*	*
Trichloroethylene (aka§ Trichloroethene)	0.07	*	*	*	*	*	*
NON-TCLP ANALYTES							
Acetone		2.4	*	0.43	*	*	*
Dichlorobenzene		*	*	*	*	*	*
Dimethyl sulfide		*	0.0014	*	*	*	0.14
Dimethyl disulfide		*	*	*	*	*	*
Ethylbenzene		*	*	*	*	*	0.038
Isopropanol		0.036	*	*	*	*	*
Methyl sulfide		*	*	*	*	*	*
Xylene		*	*	*	0.051	*	0.17

§ = Also Known As
* = Below Reporting Limits
+ = Close to the (Toxicity Characteristic Regulatory Thresholds (TCRLs)
++ = There were a number of determinations for this sludge. One TCLP extract determination indicated an 11.0 mg/L concentration for this compound which would have exceeded the TCRL. This number was not thought to be valid; however, because all other determinations indicated that the compound was not detected. Furthermore, the total content (Indicated in Appendix D, Table D-4) was only 0.14--a mean of two replicate determinations.
+++ = Proposed Regulatory Levels; Final Regulatory Levels are Mostly Higher and Are Compared with these Proposed Regulatory Levels in Table 16 in the Update Section of this Report

APPENDIX D. TABLE D-4. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGE (DRY WEIGHT BASIS)

Constituent	Concentration of Volatile Analytes in POTW Sludge (% Industrial Flow), mg/kg					
	A (1%)	B (1%)	C (20%)	D (20%)	E (60%)	F (8%)
TCLP ANALYTES						
Benzene	*	*	*	0.30	*	*
Carbon disulfide	*	*	*	*	*	*
Chlorobenzene	*	0.17	*	*	*	12.1
Chloroform	*	*	0.018	*	*	*
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	*	*	*	*	*	*
Methylene chloride	*	*	15.1	*	*	*
Methyl ethyl ketone (aka§ 2-Butanone)	1.1	9.2	1.9	0.65	*	*
1,1,2,2, Tetra-chloroethane	*	*	*	*	*	*
Tetrachloroethylene (aka§ Tetrachlorethene)	0.75	0.14	5.6	*	*	*
Toluene	*	0.21	51.2	3.4	*	22.1
1,1,1-Trichloroethane	*	*	*	*	*	*
Trichloroethylene (aka§ Trichloroethene)	0.07	*	0.34	0.07	*	*
NON-TCLP ANALYTES						
Acetone	4.7	11.6	*	*	*	*
Dichlorobenzene	*	0.65	*	*	*	*
Dimethylsulfide	8.0	5.4	*	10.4	*	5.8
Dimethyldisulfide	7.0	0.56	*	*	*	1.7
Ethylbenzene	*	0.029	1.3	1.4	0.057	11.6
Isopropanol	*	*	*	*	*	*
Methyl sulfide	*	*	*	*	*	*
Xylene	*	0.38	7.8	7.8	*	52.6

§ = Also Known As
* = Below Reporting Limits

APPENDIX D. TABLE D-5. METAL EXTRACT CONCENTRATIONS IN TCLP EXTRACTS OF SLUDGE COMPARED WITH TCLP TOXICITY THRESHOLD LEVELS

Constituent	Toxicity Characteristic Regulatory ++ Level, mg/L	Concentration of Metals in TCLP Extracts of POTW Sludge (% Industrial Flow), mg/L					
		A (1%)	B (1%)	C (20%)	D (20%)	E (60%)	F (8%)
TCLP ANALYTES							
Arsenic	5.0	*	*	0.012	0.011/0.012	0.018/0.013	0.011
Barium	100	0.63	0.90	0.47	0.46/0.45	0.9/0.76	0.41
Cadmium	1.0	0.001	0.0012	0.10	*/*	0.0021/0.0006	*
Chromium	5.0	0.012	0.011	0.050	*/*	0.013/0.013	*
Lead	5.0	0.002	0.01	0.49	*/*	0.020/0.014	*
Mercury	0.2	*	*	*	*/*	*/*	*
Selenium	1.0	*	*	*	*/*	*/*	*
Silver	5.0	*	*	*	*/*	*/*	*
NON-TCLP ANALYTES							
Nickel		0.97	0.17	0.11	0.77/0.80	1.9/1.7	0.48
Thallium		*	*	*	*/*	*/*	*

* = Below Reporting Limits

++ = Proposed and Final Regulatory Levels are the Same for Metals

APPENDIX D. TABLE D-6. METAL ANALYTE CONCENTRATION IN TOTAL SLUDGE (DRY WEIGHT BASIS)

Constituent	Compositional Metals in POTW Sludge (% Industrial Flow), mg/kg					
	A (1%)	B (1%)	C (20%)	D (20%)	E (60%)	F (8%)
TCLP ANALYTE						
Arsenic	5.7	3.8	41	10	8.0	7.8
Barium	470	330	605	64	275	720
Cadmium	5.3	7.0	160	87	64	14
Chromium	45	280	310	700	2495	260
Lead	97	280	7723	320	315	360
Mercury	2.0	2.0	6.0	4.5	0.87	2.2
Selenium	5.7	2.7	41	12	0.45	5.6
Silver	5.9	14	46	24	5.4	30
NON-TCLP ANALYTE						
Nickel	23	28	68	210	295	140
Thallium	5.7	2.7	41	4.3	4.5	5.6

APPENDIX D. TABLE D-7. COMPARISON OF THE TCLP AND EP ANLAYTE CONCENTRATIONS IN POTW SLUDGE

Constituent	TCLP & EP Toxicity Characteristic Regulatory Levels, mg/L	Sludge Analyte concentration in POTW Sludge (1% Industrial Flow), mg/l A (1%) EP		B (1%) EP		C (20%) EP		D (20%) EP		E (60%) EP		F (8%) EP	
		EP	TCLP	EP	TCLP	EP	TCLP	EP	TCLP	EP	TCLP	EP	TCLP
TCLP/EP ANALYTE													
Arsenic	5.0	*	*	*	*	0.014	0.012	*	0.012	*	0.016	*	0.011
Barium	100	0.23	0.63	0.18	0.90	0.36	0.47	0.33	0.46	0.68	0.84	*	*
Cadmium	1.0	*	0.001	*	0.0012	0.16	0.10	*	*	*	0.0014	*	*
Chromium	5.0	*	0.012	*	0.011	*	0.05	*	*	*	0.013	*	*
Lead	5.0	*	0.002	*	0.01	0.70	0.49	*	*	*	0.017	*	*
Mercury	0.2	*	*	*	*	*	*	*	*	*	*	*	*
Selenium	1.0	*	*	*	*	*	*	*	*	*	*	*	*
Silver	5.0	*	*	*	*	*	*	*	*	*	*	*	*
NON-TCLP/EP ANALYTE													
Nickel		*	0.97	**	0.17	0.24	0.11	1.1	0.78	1.6	1.8	0.30	0.48
Thallium		*	*	**	*	*	*	*	*	*	*	*	*

* = Below Reporting Limits
** = No data

APPENDIX D. TABLE D-8. RATIO OF TCLP METAL ANALYTE CONTENT TO WET WEIGHT COMPOSTIONAL CONTENT AS A MULTIPLIER FOR ESTIMATING THE TCLP ANALYTE CONCENTRATION FROM THE COMPOSTIONAL WET WEIGHT METAL CONTENT

Constituent	TCLP/Wet Weight Content for POTW Sludge, (% Moisture) [pH], ppm					
	A (80%) [6.4]	B (74%) [7.4]	C (96%) [6.4]	D (77%) [7.9]	E (79%) [8.0]	F (81%) [7.4]
TCLP ANALYTE						
Arsenic	-	-	-	-	0.04	0.008
Barium	0.008	0.007	0.03	0.0005	0.13	0.003
Cadmium	0.001	0.0005	0.03	0.03	0.0009	-
Chromium	0.002	0.0001	0.007	-	0.0002	-
Lead	0.0001	0.0001	0.003	-	0.002	-
Mercury	-	-	-	-	-	-
Selenium	-	-	-	-	-	-
Silver	-	-	-	-	-	-
NON-TCLP ANALYTE						
Nickel	0.02	0.02	0.07	0.02	0.23	0.02
Thallium	-	-	-	-	-	-

- = Data not available to calculate the ratio

APPENDIX D. TABLE D-9. RATIO OF TCLP METAL ANALYTE CONTENT TO DRY WEIGHT COMPOSTIONAL CONTENT AS A MULTIPLIER FOR ESTIMATING THE TCLP ANALYTE CONCENTRATION FROM THE COMPOSTIONAL DRY WEIGHT METAL CONTENT

Constituent	TCLP/Dry Weight Content for POTW Sludge, (% Moisture) [pH], mg/kg					
	A (80%) [6.4]	B (74%) [7.4]	C (96%) [6.4]	D (77%) [7.9]	E (79%) [8.0]	F (81%) [7.4]
TCLP ANALYTE						
Arsenic	–	–	–	0.001	0.0009	0.001
Barium	0.001	0.003	0.0008	0.007	0.003	0.003
Cadmium	0.0002	0.0002	0.0007	–	0.00002	–
Chromium	0.0003	0.00004	0.0002	–	0.000005	–
Lead	0.00002	0.00004	0.00006	–	0.00005	–
Mercury	–	–	–	–	–	–
Selenium	–	–	–	–	–	–
Silver	–	–	–	–	–	–
NON-TCLP ANALYTE						
Nickel	0.004	0.006	0.002	0.004	0.006	0.003
Thallium	–	–	–	–	–	–

– = Data not available to calculate the ratio

APPENDIX D. TABLE D-10. VOLATILE ANALYTE CONCENTRATION IN TOTAL SLUDGE

Constituent	Concentration of Volatile Analytes in POTW Sludge (% Industrial Flow), mg/L					
	A (1%)	B (1%)	C (20%)	D (20%)	E (60%)	F (8%)
TCLP ANALYTES						
Benzene	–	–	–	–	–	–
Carbon disulfide	–	–	–	–	–	–
Chlorobenzene	–	0.17	–	–	–	0.007
Chloroform	–	–	–	–	–	–
1,1,-Dichloroethylene (aka§ 1,1,-Dichloroethene)	–	–	–	–	–	–
Methylene chloride	–	–	0.006	–	–	–
Methyl ethyl ketone (aka§ 2-Butanone)	0.46	–	0.18	0.13	–	–
1,1,2,2, Tetra-chloroethane	–	–	–	–	–	–
Tetrachloroethylene (aka§ Tetrachlorethene)	0.019	–	0.008	–	–	–
Toluene	–	–	0.002	0.014	–	–
1,1,1-Trichloroethane	–	–	–	–	–	–
Trichloroethylene (aka§ Trichloroethene)	–	–	–	–	–	–
NON-TCLP ANALYTES						
Acetone	0.51	–	–	–	–	–
Dichlorobenzene	–	–	–	–	–	–
Dimethylsulfide	–	0.0003	–	–	–	–
Dimethyldisulfide	–	–	–	–	–	–
Ethylbenzene	–	–	–	–	–	–
Isopropanol	–	–	–	–	–	–
Methyl sulfide	–	–	–	–	–	–
Xylene	–	–	–	0.007	–	–

– = Data Not Available to Calculate Ratio

APPENDIX E

COMMENTS BY DOLOFF F. BISHOP

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
WATER ENGINEERING RESEARCH LABORATORY
CINCINNATI, OHIO 45268

DATE: June 17, 1987

SUBJECT: Comments on "Cooperative Testing of Municipal Sewage Sludges by the TCLP and by Compositional Analysis"

FROM: Dolloff F. Bishop, Chief
Technology Assessment Branch, WRD

TO: John M. Walker, Physical Scientist, WH-595
Residuals Management Branch

My overall impression of the above report is that a larger data basis is essential to appropriately evaluate the probable impact of toxics on sludge disposal. The TCLP results of the study are, however, consistent with expected partitioning chemistry of the toxics. That is, those organics with a strong tendency to partition to the solids in wastewater (high octanol/water partition coefficient [K_{OW}])will not be efficiently extracted by an acetic acid aqueous extraction (TCLP). Those organics with more affinity for the aqueous phase (lower K_{OW}), such as many of the volatile organics, will be found at higher concentrations in the TCLP extract even though they may be at lower concentrations in the sludge.

Conversely, organics with a high K_{OW} will partition more completely onto the sludge than those with lower K_{OW} and, therefore, for equal influent wastewater concentrations, would appear at higher concentrations in the sludge during compositional analyses. Unfortunately, the analytical measurements by the EPA and AMSA Laboratories in the study are even more variable for the compositional analyses than for the TCLP analyses. Thus I am not sure that the existing data substantiates this high K_{OW} effect of the partitioning chemistry. It is my opinion, however, that the composition analysis will be the more important measurement, especially if the new sludge regulations establish regulatory compositional concentrations for both organics and metals. Thus, the compositional analytical effort needs to be substantially improved.

The lack of comparison data between the EPA and AMSA Laboratories on the same sludge samples is most often related to different detection limits. In the study, the EPA Laboratory used higher detection limits. It appears as if the EPA Laboratory limits have been rather arbitrarily established. I would expect that all laboratories should have used the same procedures and thus have similar detection limits. The use of different detection limits by the various laboratories, however, may indicate the use of different analytical options in the analytical methods. Your report does not address the issue and it needs clarification.

If the analytical methods were the same, then approximately similar detection limits should be agreed to by all laboratories. If, the significantly lower detection limits, as apparently used by the AMSA Laboratories can actually be observed, I would suggest that the EPA Laboratory also apply those limits to its existing data tapes. The use of the lower limits would produce a larger data base for evaluation. In any event, roughly similar detection limits should be applied to all data if identical methods and similar equipment were used.

It may be possible to strengthen the qualitative conclusion of the study that municipal sludges are not likely to fail the TCLP test. Specifically, a statistician with analytical chemistry competency could evaluate the appropriateness of applying a statistical test for significance (students "t" test or other comparison tests) to paired results from the EPA and AMSA Laboratories compared to the TCLP regulatory levels. The comparison using the observed analytical variability would indicate the probability of the sludges in the existing data base for exceeding the TCLP regulatory levels. Paired results (precision) within laboratories could also be used in the statistical evaluation. The availability of more paired results, such as would occur if lower analytical limits were applied to the existing EPA Laboratory measurements, might strengthen the analysis. The statistician, however, needs to assess the effect on the validity of the statistical comparison of having a large number of samples in the study near the methods detection limits.

In the report, the observation of the presence of methyl ethyl ketone (MEK) in the TCLP analyses and its absence (presence below detection limits?) in the compositional analyses can perhaps be explained. Methyl ethyl ketone is a polar solvent with low Henry's Law constant. That is, while the compound has a high vapor pressure, it also is very soluble in water and thus it is not easily stripped from an aqueous medium at ambient temperatures. The purge and trap analytical method at ambient temperature will not efficiently strip the ketone from the samples. However, a known spike of MEK in an aqueous medium can be used to correct for the poor-stripping efficiency and provide some reasonable measure of the ketone concentration in unknowns. The problem is most effectively corrected if a stable labeled MEK is added directly into the matrix with unknown normal MEK. In this case the analytical system can be directed to automatically correct for poor stripping efficiency in the sample matrix.

I am not sure how this problem was addressed by the various laboratories providing the analytical services. They probably used MEK stripped from distilled water as the control approach to correct for low stripping efficiency. Such a correction would be more appropriate for the TCLP test since the organic matrix in the leachate would be relatively low, especially as compared to the organic matrix in the compositional purge and trap tests. While the proper use of stabled labeled MEK in the individual sample matrices should minimize the matrix effects, my guess is that such an approach was not used in the compositional analyses and matrix effects could explain the anomalous results on MEK.

cc: John J. Convery, Director, WRD

APPENDIX F

TRENDS IN AMSA POTW INFLUENT AND SLUDGE METAL CONTENTS AS INFLUENCED BY PRETREATMENT

APPENDIX TABLE F-1. TRENDS IN INFLUENT AND SLUDGE METAL CONTENTS

	Influent, mg/l				Sludge, mg/kg			
Approx. Year	1975	1980	1983	1986	1975	1980	1983	1986
City				CHROMIUM				
A								
C	-	0.104	0.076	0.062	-	241	151	128
D	1.02	0.42	0.29	0.18	2184	1567	937	512
G	1.15+	-	0.23	0.20	420+	-	900	950
H	-	2.44	2.22	1.17	-	6262	4170	5740
L	-	0.135	0.096	0.046	-	-	385	180
N(a)	-	-	-	-	-	574+++	471	506
P(b)	0.022*	0.035	0.030	0.005	392*	596	303	207
R	-	0.139	0.073	0.067	-	207	139	124
City				COPPER				
A	-	-	-	-	-	560	440	359
C	-	1.48	0.89	0.91	-	2375	2270	2498
D	-	-	-	-	1819	957	763	535
G	1.16+	-	0.63	0.22	4700++	-	1700	800
H	-	0.193	0.187	0.094	-	466	393	381
L	-	0.162	0.120	0.113	-	-	613	568
N(a)	-	-	-	-	-	3178+++	2323	3506
P	1.117*	0.233	0.030	0.005	585*	571	371	397
R	-	0.102	0.083	0.095	-	157	214	195
City				NICKEL				
A***	-	-	-	-	-	20	48	22
C	-	0.090	0.075	0.076	-	58	50	59
D	0.31	0.21	0.22	0.10	481	449	362	169
G	0.67+	-	0.14	0.14	-	500	250	260
H	-	0.137	0.119	0.038	-	93	125§	86
L	-	0.105	0.057	0.037	-	-	89	60
N(a)	-	-	-	-	-	521+++	357	543
P	0.503*	0.115	0.165	0.052	193*	132	165	111
R	-	0.207	0.153	0.182	-	204	322	248

(a) = different labs for 1981/83 and 1986
(b) = hexavalent chromium in influent not in sludge
§ = another POTW input
* = 1977 data
** = 1982 data
*** = limited data
+ = 1973-74 data
++ = 1978 data
+++ = 1981 data

APPENDIX TABLE F-1 Cont. TRENDS IN INFLUENT AND SLUDGE METAL CONTENTS

	Influent, mg/l				Sludge, mg/kg			
Approx. Year	1975	1980	1983	1986	1975	1980	1983	1986
City				CADMIUM				
A	-	-	-	-	-	8	9	5
C	0.051	0.026	0.026	0.043	-	81	52	102
D	0.035	0.026	0.034	0.014	93	75	76	44
G	0.24x	0.13	0.03**	0.02	-	900	500	40
H	-	0.052	0.023	0.012	-	113	45	39
L	-	0.018	0.015	0.005	-	-	46	25
N(a)	-	-	-	-	-	23+++	12	15
P	0.027*	0.012	0.013	0.011	54*	21	29	32
R	-	0.002	0.002	0.001	-	7.2	4.7	3.2
City				ZINC				
A	-	-	-	-	-	1240	982	510
C	-	12.6	5.0	3.2	-	8700	8425	6500
D	1.55	0.85	1.00	0.75	4821	3233	3370	1368
G	-	-	-	-	-	-	-	-
H	-	0.555	0.540	0.289	-	1103	1300§	1185
L	-	0.789	0.441	0.239	-	-	2479	1134
N(a)	-	-	-	-	-	3327+++	2376	3802
P	1.188*	0.980	0.685	0.400	3529*	3172	2119	1990
R	-	0.338	0.313	0.242	-	479	572	437
City				LEAD				
A	-	-	-	-	-	220	106	58
C	-	0.58	0.40	0.42	-	1395	1099	1137
D	0.36	0.17	0.15	0.14	1437	663	430	398
G	-	-	-	-	-	-	-	-
H	-	0.152	0.127	0.101	-	394	409§	325
L	-	0.153	0.048	0.038	-	-	307	215
N(a)	-	-	-	-	-	401+++	347	365
P	0.500*	0.210	0.105	0.086	843*	736	608	449
R	-	0.064	0.044	0.024	-	195	404	73

(a) = different labs for 1981/83 and 1986
§ = another POTW input
* = 1977 data
** = 1982 data
*** = limited data
+ = 1973-74 data
++ = 1978 data
+++ = 1981 data
x = 1976 data

APPENDIX TABLE F-1 Cont. TRENDS IN INFLUENT AND SLUDGE METAL CONTENTS

	Influent, mg/l				Sludge, mg/kg			
Approx. Year	1975	1980	1983	1986	1975	1980	1983	1986
City				ARSENIC				
C	-	0.01	0.03	0.008	-	3.4	5.5	2.9
D	0.016	0.006	0.008	0.012	20	10	17	15
City				SELENIUM				
C	-	0.010	0.018	-	-	0.4	2.3	-
D	-	-	-	-	-	8.4	8.6	12.3
City				MERCURY				
A	-	-	-	-	-	2.6	1.1	1.4
C	-	0.0007	0.003	0.006	-	2.4	1.9	1.6
D	0.0013	0.0009	0.0013	0..0011	4.9	4.5	6.2	4.6
N(a)	-	-	-	-	-	0.16+++	1.63	1.1
City				SILVER				
C	-	0.028	0.024	0.022	-	11.6	24.7	2.1
D	-	-	-	-	42	36	46	41
N(a)	-	-	-	-	-	24+++	30	64
City				CHROMIUM^{+6}				
N(a)					-	18	1.4	129

(a) = different labs for 1981/83 and 1986
+++ = 1981 data

APPENDIX TABLE F-1 Cont. TRENDS IN INFLUENT AND SLUDGE CONSTITUENTS

	Influent, mg/l				Sludge, mg/kg			
Approx. Year	1975	1980	1983	1986	1975	1980	1983	1986
City				CYNAIDE				
C	–	0.27	0.27	0.47	–	296	144	239
D	0.28	0.11	0.04	0.02	–	–	–	–
N(a)	–	–	–	–	–	91+++	48	444
City				DDT				
D	–	–	–	–	10.2	2.0	0.5	0.6
City				PCB				
D	–	–	–	–	18.8	1.8	0.5	–
City				TICH				
D	–	–	–	–	31.3	3.8	1.1	0.8
City				OIL & GREASE				
D	90	75	70	65	–	–	–	–
City				PHENOLS				
D	3.7	2.3	2.4	2.4	–	–	–	–

(a) = different labs for 1981/83 and 1986
+++ = 1981 data