

**Summary Report of Washed and Unwashed Mine Tailings (Chat)
from Two Piles at the Tar Creek Superfund Site
Ottawa County, Oklahoma**

Revised June 2003

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Note: This report has been revised from the May 2000 report to include discussions of chat washing operations, wash water analyses, chemical tests for chat, and graphs.

Introduction

The Site Remediation Section of the Land Protection Division of the Department of Environmental Quality sampled two chat piles, the Atlas and Ottawa that are being used for commercial purposes. These piles are located near Picher, Oklahoma, which is in the Tar Creek Superfund Site (Figure 1). The purpose of this sampling was to provide information concerning the chemical and physical characteristics of the chat. Analyses of chat and process water, before and after washing, will provide data on what effect washing has on the lead content (and other contaminants) of chat and process water. This will help define appropriate management of chat.

Two milled asphalt piles, located near Quapaw, Oklahoma, were sampled to determine the metal concentrations of the used asphalt. The used asphalt came from the Will Rogers Turnpike. It was assumed that chat was used as aggregate in the asphalt during the construction of this highway.

Background

The Tar Creek site encompasses approximately 40 square miles in far northeastern Oklahoma and affects the towns of Quapaw, Commerce, Picher, North Miami, and Cardin. The site is part of the former Tri-state Mining Area that extended from northeastern Oklahoma, through southeast Kansas, and into southwest Missouri. Extensive underground mining for lead and zinc during the early 1900's through the 1960's resulted in the formation of acid mine water that has contaminated the shallow ground water and surface water with iron, sulfate, zinc, lead, and cadmium at the Tar Creek site. Mine tailing piles (chat) and tailings impoundments (mill ponds) now cover the landscape. These artifacts of mining were created as waste material during the processing (milling) of the ore to produce concentrates of lead and zinc for smelting. Only one smelter, located three miles east of Picher, operated in the Oklahoma portion of the mine field from 1918 to 1930. Originally each 40-acre mine lease had its own mill, which explains the numerous piles and millponds. In the early 1930s centralized mills came into being. The increased production due to economies of scale enabled the continued operation of mines with low-grade ore and the rerun of old mill tailings piles (McKnight and Fisher, 1970). A few very large piles (with presumably lower metals concentrations) were produced as a result. Much of the original estimated volume of 160 million cubic yards has been removed for various purposes, mostly commercial, but approximately 50 million cubic yards (70 million tons) of chat still remain.

The early milling process consisted mostly of mechanical and gravity separation procedures (tabling and jigging) to concentrate lead and zinc. Later, the floatation process was added and increased recovery efficiency for lead and zinc ores from 58-70 percent to 80-85 percent.

However, when chat piles were rerun using the floatation process mostly zinc, with lesser amounts of lead, was recovered (McKnight and Fisher, 1970). Much of the lead, being more dense, had been removed by gravity separation.

Mine tailing piles (chat) are comprised of mostly angular chert fragments and contain residual amounts of lead sulfide (galena), zinc sulfide (sphalerite) minerals, and their weathering products. Chat (aka – raw chat, bulk chat, whole chat, pile run, gravel, etc.) consists of material typically ranging in diameter from 15.875 mm (5/8 inch) to less than 0.075 mm (the size fraction that passes the No. 200 sieve). Tailings impoundment sediments (aka - mill pond sediment, tailings, fines, etc.) consist of much smaller material of similar composition. Chat has been used for many purposes including: railroad ballast; aggregate in asphalt roads; aggregate in concrete; gravel for county roads and driveways; and fill material. The use of mine tailings in driveways and as fill material around houses has caused lead contamination of soils in residential areas; that, in turn, contributes to elevated blood lead concentrations in area children. The U.S. Environmental Protection Agency (USEPA) is conducting soil remediation work in the cities in this area to reduce lead exposure and consequently reduction in the number of children with elevated blood lead concentrations. The remedial action value set for lead in the residential soil cleanup at the Tar Creek Superfund Site is 500 mg/kg (EPA, 1997).

Chat is a byproduct of mining and milling operations. As such it has been exempted from regulation as a ‘hazardous waste’ under RCRA (Resource Conservation and Recovery Act, 1976) by the Beville amendment. This exclusion is cited in 40 CFR 261.4 (b) (7). Since chat is a “solid waste from the extraction, beneficiation, and processing of ores and minerals...”, it is an excluded solid waste which is not a hazardous waste. However, because chat contains varying concentrations of lead, which is a ‘hazardous substance’, it falls under the jurisdiction of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).

Several chat washing operations are currently active in the area, including the Ottawa and Atlas chat piles, and their general processes are described below. Chat washing is used to separate the large particles of chat and remove the finer sized particles. Two products are created of different sizes (gravel sized and sand sized). Dry screening (sieving) is also used in the chat processing operations. Dry screening uses a vibrating harp screen with approximately 5 mm openings to separate out the fine sized particles of chat into a stockpile for usage. The large particles from dry screening and raw chat are used as the input materials for the washing (wet sieving) operation. At the washing plant, these materials are combined with water on top of a shaking and inclined sieve with 3/16 inch openings. The larger particles fall off the screen and are collected into a stockpile for sale. The finer particles and water that pass through the screen collect for a short retention time in a small container (water trap) where sand sized particles that settle to the bottom are removed with a rotating screw lift pump. The water and very small particles in suspension that overflow the sand screw container are piped to a settling pond where the fine solids accumulate as sediment. The process water cycles through a series of settling ponds for treatment by sedimentation before being pumped back to the wash plant wet screen as input water. The chat washing operations do not have pipe discharges of wastewater to surface water and the settling ponds represent total retention basins. The settling ponds are old mine tailings impoundments (mill ponds) that were located on top of, or excavated into, clay and shale beds at the surface over much of the area. These are areas of preexisting contamination with

high metals concentrations. Periodically, a small amount of additional makeup water may be needed to replace that process water lost to evaporation. The source of the makeup water at the Ottawa site is from a mine shaft and a small pond, and at the Atlas site the makeup water comes from a small stream.

In general, the chemical tests used to assess the leachable and total metals content in chat are: Toxicity Characteristic Leaching Procedure (TCLP) – SW846 Method 1311; Synthetic Precipitation Leaching Procedure (SPLP) – SW846 Method 1312; Total Metals analyses from acid digestions (Methods 3050, 3051 & 3052) coupled with analysis using Atomic Absorption (AA) or Inductively Coupled Plasma (ICP) instruments; and X-Ray Fluorescence (XRF) spectrometry – Method 6200. These methods define the sample preparation procedures and various analytical instruments used to determine metals concentrations in soils and sediment.

Method 3050 “Acid Digestion of Sediments, Sludges and Soils” uses nitric acid (HNO_3) digestions for graphite furnace atomic adsorption spectrometry (GRFAA) or inductively coupled plasma mass spectrometry (ICP-MS) analyses; and hydrochloric acid (HCL) digestions for inductively coupled plasma atomic emission spectrometry (ICP-AES) or flame atomic adsorption spectrometry (FLAA) analyses. Method 3051 “Microwave Assisted Acid Digestion of Sediments, Sludges, Soil, and Oils” uses HNO_3 or HCL, similar to Method 3050, and microwave energy to extract the acid soluble minerals from the samples prior to analysis. Method 3052 “Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices” uses hydrofluoric acid (HF) and microwave heating to dissolve the entire sample prior to analysis on a specified instrument. Methods 3050 and 3051 are partial digestion procedures while method 3052 is a total digestion procedure. These differences can affect sample quantification. Once prepared for analysis, the ICP (inductively coupled plasma) instrument may be used to quantify the metals concentrations and is specified as Method 6010. The entire analytical procedure is specified, for example as Method 1311/6010 to indicate a TCLP leaching procedure followed by analysis on the ICP-MS.

The TCLP and SPLP tests use 100 grams of sample and 2,000 milliliters (ml) of extraction fluid while the analyses for Total Metals concentrations use 0.5 to 1 gram of sample and repeated digestions with 10 ml nitric (HNO_3), hydrochloric (HCl) and / or hydrofluoric (HF) acid and hydrogen peroxide, with and without microwave assistance. The SPLP and TCLP procedures are similar except the extraction fluid used for the SPLP test is a mixture of mineral acids (sulfuric and nitric) while that of TCLP uses an organic acid (Glacial acetic acid). Both methods utilize one of two extraction solutions of different acidity, determined by the sample characteristics. The TCLP was developed to evaluate metal mobility in sanitary landfills and is the only leaching procedure approved for characterizing hazardous waste under RCRA. The SPLP was developed to evaluate metals mobility in the natural environment by exposing the test material to an extraction fluid that simulates slightly acidic rainwater.

XRF measures the total heavy metals content in soil samples directly. XRF measurements may be affected by particle size, uniformity, homogeneity, surface condition, moisture content, and particle size distribution of a soil sample. Therefore, XRF readings may be corrected by using site-specific calibration standards that have been analyzed by ICP or AA to achieve the most

accurate results. Field instruments are used directly on the ground for screening to give metals concentrations of soils or where low detection limits are not required.

Previous Studies

USACE

The use of chat in highway construction has been occurring for many years. A study of 1,000 foot stretches along three different roads was done in 2000 by the U.S. Army Corps of Engineers (USACE) to determine the lead, cadmium, zinc and arsenic concentrations of roadside soils. Two roads contained chat in the asphalt matrix or other components of the roads while a third road, the control, contained no chat. This study showed that the sampled roadside soils along the roads that used chat had higher concentrations of lead, cadmium and zinc than the soils along the control road. The average soil lead levels for the two roads with chat were 266 mg/kg and 233 mg/kg, while the average for the control road (without chat) was 54 mg/kg. The maximum values were 886 mg/kg and 1,090 mg/kg for the soils near roads containing chat and 189 mg/kg for the control road soils.

Cores of asphalt, base coarse, and sub-base, one from each of the roads, were also evaluated for leachable and total metals content. The lead concentration for the 2 surface (asphalt) samples from the road cores with chat were 402 mg/kg and 328 mg/kg, while that for the control road was 26 mg/kg. Leachable lead for the chat containing asphalt cores was 0.198 mg/l for TCLP (average of 2 samples: 0.037 & 0.359 mg/l) and 0.001 mg/l for the SPLP (from two samples: 0.002 and <0.001 mg/l). The SPLP results are at least one tenth the concentration of TCLP lead and are even less, by another order of magnitude, for cadmium and zinc.

Average lead concentrations in soils were consistently higher near the shoulder of the roads than in the ditch. The control road exhibited an average soil lead level of 112 mg/kg near the shoulder while the average soil lead level in the ditch was 20 mg/kg. USACE concluded the presence of lead in soils along the control road indicates contamination resulting from the historical use of leaded gasoline. Further, the USACE stated that although past use of leaded gasoline has contributed to soil lead levels, chat usage might have produced the higher concentrations of lead in roadside soils.

USACE concluded that the use of chat in asphalt for roads did not pose a problem for commercial / industrial settings (an EPA Action Level of 1,000 mg/kg was assumed). Only nine samples (15%) were above the residential soil lead cleanup value of 500 mg/kg with none above a potential commercial cleanup level of 2,000 mg/kg. However, since roadside soils exhibited elevated lead concentrations above those of the control road, the USACE raised questions about the degree of binding of lead from chat in the asphalt matrix and the fate and transport of lead from chat that may be released to roadside soils over time. The USACE noted that past practice in the area of using fine sized chat material with salt to "sand" the roads during icy weather conditions might have contributed to soil lead levels.

CERCLA Investigations

Remedial Investigations were done for both the Kansas and Missouri portion of the Tri-state mining area. The total lead concentration in Kansas (Dames & Moore, 1993) of bulk (raw) chat

ranged from 100 to 1,660 mg/kg with an average of 750 mg/kg for 16 samples. The total lead concentration in Kansas (Dames & Moore, 1993) for tailings (very fine sized mill pond sediment) ranged from 56 to 13,000 mg/kg with an average of 3,790 mg/kg for 12 samples. The total lead concentration in Missouri (Dames & Moore, 1995) for bulk chat ranged from 22 to 6,000 mg/kg with a mean value of 608 mg/kg for 97 samples. The mean total lead concentration in Missouri (Dames & Moore, 1995) for 156 samples of tailings was 3,963 mg/kg. These data show that the metal concentrations are greater for the smaller sized particles (from sieved chat and tailings) and that lead concentration increases as the particle size of the material decreases.

In the RI/FS for Cherokee County, Kansas, (Baxter Springs and Treece Subsites) a laboratory XRF was used for routine metals analyses of chat. The metals concentrations of some chat samples were determined using both nitric acid (HNO₃) and hydrofluoric acid (HF) digestions with subsequent analysis on ICP. Good correlation was established comparing the analytical results from the HF and HNO₃ methods and the sample results from the HF method were used to calibrate the XRF.

DOI

The United States Department of Interior (DOI) took 30 samples from 16 chat piles located on Indian Lands (CCJM, 1999). Each sample was sieved into five particle size divisions and chemical tests were run on each size fraction. The total lead concentration for each sample was calculated from the measured lead concentration and mass fraction of the sieved particles. The results for “calculated total lead” for whole (raw) chat ranged from 78 mg/kg to 2,289 mg/kg, with a mean value of 830 mg/kg (CCJM, 1999). The lead concentrations for the various particle size divisions of sieved chat ranged from 11,700 mg/kg for the smallest size fraction (< 250 microns) that pass the #60 sieve to 566 mg/kg for the largest particles sizes (> 4.75 mm) that were retained on the #4 sieve. The average total lead concentration for smallest particles was 2,794 mg/kg, and 160 mg/kg for the largest particles. Leachable lead (Method 1312) was also run. These values ranged from 0.659 mg/l to nondetect, with many samples in all of the particle size fractions testing below the nondetect value of 0.018 mg/l (C. C. Johnson et al, 1996).

Thesis

A thesis prepared by K. David Drake (Drake, 1999) studied the leachability of size-fractionated mine tailings in Kansas. Chat samples were sieved into different particle sizes ranging from less than 0.075 mm to greater than 4.76 mm, corresponding to particles passing the #200 sieve (-200) to particles retained on the #4 sieve (+4), respectively. The leachable lead concentrations (TCLP - Method 1311) from one chat pile (TC-3) ranged from 118 mg/l for the smallest size fraction (< 0.075 mm or -200) to 0.450 mg/l for the largest size fraction (> 4.76 mm or +4). The TCLP concentrations for lead from another pile (TC-16) ranged from 10.5 mg/l for the -100 sieve size fraction to not detected (above 2.6 ppb) for the +4 and +8 sieve size fractions. These values for leachable lead in chat are at least ten times greater than those reported by CCJM (1999). This difference is attributed to the more aggressive TCLP tests used by Drake compared to SPLP tests used by CCJM (1996) reported in the DOI section above. This difference was also observed in the USACE asphalt road study.

Drake (1999) also tested some tailings impoundments (mill ponds) samples. The leachable lead concentrations (TCLP 1311) from one mill pond site (TT-11) ranged from 5.4 mg/l for the -200

sieve size (<0.075 mm) fraction to 7.1 mg/l for the larger sieve size fractions (including the +16, +8 & +4 fraction). The TCLP concentrations for lead from another mill pond site (TT-22N) ranged from 62.8 mg/l for the -200 (< 0.075 mm) size fraction to 25.8 mg/l for the larger particle sizes (including those from the +4 and +30 sieve size fractions). The lead concentrations in the leachate increased with decreasing particle size. Drake (1999) suggested that the leaching potential of a sample might be misrepresented, with inconsistent metals concentrations, due to the use of the two extraction solutions in the TCLP test (one being more acidic than the other). This may partially explain the wide variation in lead content for like sized particles. However, a wide variance in concentrations from pile to pile was noted.

Drake (1999) characterized the mineral composition of Kansas chat and tailing impoundment material as predominantly quartz with lesser amounts of carbonates (dolomite and limestone). The sulfide minerals of lead and zinc (galena and sphalerite) were not detected but a secondary zinc mineral, hemimorphite, was prevalent in the finer sized fractions of both the chat (10%) and tailings (5%). The carbonate material represented approximately 25% of the chat and approximately 50% of the tailings composition. Quartz was 73% of the coarse (+50) chat, 65% of the fine (-200) chat, 51% of the coarse (+50) tailings, and 45% of the fine (-200) tailings. Drake identified the main factors affecting leachability as: mineral content (hemimorphite is more soluble) and grain size (large surface area of fine sized material is more reactive).

Chat Pile Sampling and Analysis Procedures

Raw chat is accumulated in large piles from the processing of ore at old mill sites during the days of active mining. Chat washing is used to separate the large particles of chat and remove the finer sized particles and produces stockpiles of coarse chat (wet screened coarse) and sand sized chat (wet screened sand screw fines). A very fine sized sediment (wet screen sediment) is produced as a waste material when suspended solids carried in the process water are deposited in settling ponds.

Dry screening (sieving) is also used in the chat processing operations. Dry sieving produces a stockpile of fine sized material for commercial use (dry screened fines for Type 2 Slurry Seal). The coarse particles from dry sieving (dry screen coarse) are used as raw material along with raw chat in the wet screening (sieving) process.

In November, 1999, the DEQ Site Remediation personnel collected samples of raw chat, washed chat stockpiles, dry sieved chat stockpiles, sediment from settling ponds, and process water used at two chat washing operations near Picher, Oklahoma. The chat washing operations are located at the Ottawa and Atlas chat piles (Figure 1). Raw chat samples were composites from many parts of each of the two chat piles. Two “washed chat” stockpiles were sampled at each site, the ‘wet sieve coarse chat’ that falls off the inclined screen of the wash plant, and the ‘wet sieve sand screw fines’ that are extracted from the water trap by a screw pump. The Oklahoma State Environmental Laboratory (SEL) analyzed the washed and unwashed chat samples for total lead, cadmium, arsenic, and zinc using the ICP (Method 3050/6010). The Toxicity Characteristic Leaching Procedure (TCLP – Method 1311/6010) was run for the lead and cadmium and analyzed using the ICP. Standard Engineering and Testing, a geotechnical laboratory, conducted particle size gradation analyses for two composite samples of raw chat. The U.S. Standard

Sieves used were the: 1/2", 3/8", #10, #40, #80, and #200. The sieves were cleaned between each test by hand shaking and brushing; and any particles stuck in the mesh were removed by hand. Samples from each sieve size fraction were collected for analysis of total lead, cadmium, arsenic, and zinc (Method 3050/6010). The material was also analyzed for TCLP lead and cadmium (Method 1311/6010). The water samples were analyzed for lead (Pb), cadmium (Cd), zinc (Zn), arsenic (As), iron (Fe), calcium (Ca), magnesium (Mg), sodium (Na), potassium (K), alkalinity (alk), chloride (Cl), sulfate (SO₄), total dissolved solids (TDS), and total suspended solids (TSS). Specific conductance (SC), pH, temperature (T), and dissolved oxygen (DO) of the water samples were measured in the field.

In February 2000, DEQ Site Remediation personnel also collected samples from a couple of piles of milled asphalt. These were selected for sampling and chemical analyses because chat was incorporated into the asphalt. Six asphalt samples and one duplicate were collected from two piles, named the Battie Asphalt Pile and the DeHorn Asphalt Pile. These piles are located near Quapaw, Oklahoma and came from the Will Rogers Turnpike. The Oklahoma State Environmental Laboratory (SEL) analyzed the samples for total lead, cadmium, and zinc (3050/6010) and for TCLP lead and cadmium (1311/6010). These were selected for sampling to evaluate the effect of encapsulation since chat was incorporated into the asphalt.

Data Summary

Twelve chat samples were taken from the Ottawa and Atlas chat piles along with two duplicates. Total metals and TCLP analyses of the raw chat, wet screen coarse, wet screen sand screw fines, type 2 slurry seal-dry screen fines, type 2 slurry seal-dry screen coarse, and wet screen sediment samples are given in Table 1 for both chat washing sites.

Nine water samples were collected of the process water (wash water) at both chat washing sites along with an equipment blank. Total metals, general chemistry and several field analyses were conducted on the water samples taken at various locations in the process (input, output, and makeup water). Dissolved metals analyses were conducted on the output water (filtered output water) only. The analytical data for the process water is presented in Table 2.

Twelve samples of sieved chat were analyzed from composites of raw chat taken at both washing sites along with one duplicate. These analytical results are presented in Table 3. There was not enough material in the 1/2" and 3/8" fractions to conduct chemical analyses. The Table also shows the percentage mass of material passing and retained on each sieve.

The analytical results of six samples of milled asphalt and one duplicate are presented in Table 4. The samples represent composites from various locations (north, middle and south sides) around the two piles.

Discussion of Results

Washed and Raw Chat

Arsenic results were not detected above 40 mg/kg. Total cadmium concentrations were relatively low, ranging from 18 to 97 mg/kg, and total zinc concentrations were relatively high,

ranging from 3,766 to 19,882 mg/kg. Total lead concentrations are intermediate in relative value, ranging from 116 (for the wet screen coarse at Atlas) to 2,014 mg/kg (for the wet screen sediment at Ottawa).

The wet screen sediment collected near the process water outfall represents the fine material washed from raw chat and deposited in the settling pond. These samples and the fine material from dry sieving have the highest lead concentrations as seen in Figure 2 (Ottawa Site) and Figure 3 (Atlas Site). The washed chat has smaller lead concentrations than raw chat, both having much less lead than in the sediments. With a few exceptions, the washed material (wet screen coarse and sand screw fines) has lower lead concentrations than raw chat and dry sieved chat. In general, the total metals concentrations for lead, cadmium, and zinc of the washed chat, raw chat, dry screened chat, and sediment samples increased as the particle size decreased. The exceptions occur at the Atlas site where the trend in metals concentrations are reversed for the dry sieved material - the higher lead concentrations were found in the larger particles. At the Ottawa pile relatively high total zinc and cadmium concentrations were found in the wet screen coarse and in raw chat compared to the concentrations of the sediment samples.

TCLP lead and cadmium (i.e., leachable lead and cadmium) concentrations generally increase as the particle size decreases. The TCLP samples display similar trends to total metals results: 1) higher metals concentrations are found in the fine sized particles; and 2) the washed material has lower metals concentrations than raw chat or dry sieved chat as shown in Figures 4 and 5. The TCLP data does not have the exceptions to the general trend of increasing metal concentration with decreasing particle size that were noted in the total metals analyses data.

Both total and leachable metals concentrations at the Ottawa Chat Pile were higher than at the Atlas Chat Pile. The total metals concentrations in mg/kg for raw chat at the Ottawa compared to the Atlas chat piles were: 732 versus 358 for lead, 57 versus 41 for cadmium, and 11,086 versus 8,266 for zinc. Comparison of the leachable concentrations in mg/l between Ottawa and Atlas chat piles are: 18.02 versus 2.750 for lead, and 0.792 versus 0.403 for cadmium. This could be due to several reasons such as the number of times the chat pile was processed or the difference in original content (mineralogy) of the ore.

Process Water

The makeup water, input water, and filtered output water were all clear, while the non filtered output water was very turbid. The input water and filtered output water have similar concentrations (of cations) as seen in Figures 6 & 7. However, the filtered output water has slightly higher lead and cadmium concentrations at the Atlas site and slightly lower iron and lead concentrations at the Ottawa site compared to input water. The higher lead and cadmium at the Atlas site may be the result of slightly lower pH of the output water.

The output waters at both sites have much higher metal concentrations for Pb, Cd, Zn, Fe, Ca, Mg, and K than the input water (Figures 6 & 7). The total suspended solids (TSS) concentration for the output water at the Atlas site was 10,364 mg/l and 1,872 mg/l at the Ottawa site. The corresponding TSS values for input waters were 9 mg/l at the Atlas site and 42 mg/l at the Ottawa site. The high metal concentrations observed in the 'unfiltered' output waters at both sites come from the suspended solids carried by these waters. The general chemistry parameters

(alkalinity, total dissolved solids, chloride, and sulfate) were relatively unchanged comparing input to output values. The measured field values for SC, T, and DO were also relatively constant when comparing input and output waters for both sites, except for a slight decrease in pH from 7.34 to 6.75 at the Atlas site.

Figures 6 and 7 display similar trends in the parameter composition when comparing the process waters between both sites; except that an increase in SO₄ and a decrease in Cl are noted in the output water at the Ottawa pile. The process water concentrations are generally slightly higher at the Ottawa site compared to the Atlas site, except for a TSS value of 10,364 mg/l at the Atlas site (this value appears to be anomalous).

At the Ottawa site, the input water and makeup water are compared (Figure 8) and display significant differences. The waters are of poor quality with lead, zinc, and total dissolved solids (TDS) concentrations exceeding maximum concentrations levels (MCLs). High Na, Cl, and SO₄ were noted in the makeup water from the pond. While high Fe and low Cl concentrations were observed in the makeup water from the mineshaft. The input water was intermediate between the Na and Cl concentrations of the makeup waters indicating possible mixing. The high Fe concentration, with correspondingly low dissolved oxygen (DO), of the mineshaft water is characteristic of groundwater in the area. Zinc, Ca, and Mg concentrations for all three waters are similar. Higher Pb and Cd concentrations are noted for the input water compared to the makeup waters. This result may indicate chat as a possible source contributing these elements either as dissolved components or as colloidal particles that have not settled out as process water circulates through the settling ponds. Increases in metals concentrations over time due to evaporation may provide another explanation but chemical reactions may complicate the picture and corresponding increases in the other parameters were not observed.

Sieved Chat

The range of values for total lead for each of the sieved samples from the Ottawa Chat Pile is 70 to 6,668 mg/kg (Figure 9); and for the Atlas Chat Pile the range is 25 to 1,789 mg/kg (Figure 10). An anomalously high zinc value and correspondingly high cadmium value are present in the coarse material (-4, +10) in the Ottawa chat. High zinc and cadmium values were also observed in wet screened coarse material at the Ottawa site and in the sand screw fines at Atlas. These are considered anomalies produced by a few grains of a zinc mineral (containing small amounts of cadmium as a substitution element) that dominate the analytical results due to the small sample size used in total metals analyses. The range of values for the leachable lead for each of the sieved samples for the Ottawa Chat Pile is 2.87 to 116.56 mg/l; and for the Atlas Chat Pile is 1.24 to 15.57 mg/l (Figure 11). The anomalously high cadmium (and zinc) levels in total analyses are not seen in the TCLP results (Figure 12). The larger sample size of the TCLP analyses is thought to be the reason. The TCLP results from the sieved samples, show that as the particle size decreases the lead content increased.

A comparison of total versus leachable lead and cadmium is shown on Figures 13 and Figure 14. Total lead correlates well with leachable lead while cadmium results are scattered.

Particle size distribution curves for raw chat from Ottawa and Atlas piles are presented on Figure 15. The chat from both sites display similar particle size distribution with approximately 50

percent of the material less than #10 sieve (<2 mm) and approximately 20 percent is less than #40 sieve (<420 um). Stated another way, more than approximately 80 percent of the chat volume by weight is greater than the #40 sieve size (> 420 mm).

Figures 16 and 17 show the distribution of lead and zinc mass with respect to sieved chat particle size. About 75 percent of the lead occurs in the fine sized chat (less than the #40 sieve). This represents less than about 20% of the total chat material. However, this portion of the chat material contains less than 52% of the zinc mass. The percentages for zinc are slightly questionable due to the anomaly noted above.

Asphalt Samples

The total lead, cadmium, and zinc concentrations are generally less than the values for the raw and washed chat except for one anomaly, where a lead value of 2,228 mg/kg was measured. The data is given in Table 4. The values for total lead concentrations ranged from 141 to 252 mg/kg, excluding the anomalously high value, with a censored mean of 176 mg/kg. The total cadmium concentrations ranged from 12 to 35 mg/kg, with a mean value of 19.1 mg/kg. Zinc concentrations ranged from 1,761 to 5,104 mg/kg with a mean of 2,804 mg/kg. The leachable lead and cadmium concentrations from the milled asphalt are much lower than the values for the raw chat and washed chat (Figure 18). The TCLP values ranged from < 0.050 to 0.221 mg/l with a mean of 0.132 mg/l for lead; and from 0.005 to 0.014 mg/l with a mean of 0.01 mg/l for cadmium.

The censored mean concentration for total lead of 176 mg/kg, is less than half the value that the USACE measured for asphalt cores (365 mg/kg). The mean concentration for total cadmium (19.1 mg/kg) is also less than the USACE asphalt data (30.2 mg/kg). The leachable lead and cadmium for the milled asphalt (0.132 & 0.01 mg/l) are slightly less than for the cored asphalt values (0.198 & 0.032 mg/l).

Conclusions

All but one of the samples of the washed and unwashed chat at the Atlas Chat Pile had lead concentrations below the 500 mg/kg action level established for the residential remediation at the Tar Creek Superfund site. All but one of the samples of the Ottawa Chat Pile had lead concentrations above the 500 mg/kg value. One possible explanation for different concentrations of metals between piles is that some piles were run through the milling process one, two, or three times to recover additional lead and zinc. Another explanation is the composition of ore varied from place to place. Lower residual metals concentrations could be expected in chat from a centralized mill since it could process lower grade ore economically.

The sieved data at both sites show that metals concentrations increase with decreasing particle size of the chat. In general, the concentration of metals in washed chat products is less than raw chat, dry sieved chat and sediments. However, several exceptions were observed.

Even though the total lead concentrations at the Atlas Pile were low, the sediment from the washing operations tested in excess of 1,500 mg/kg for lead and the fines of the sieved raw chat contained 1,789 mg/kg lead compared to an average of 364 mg/kg for the two raw chat samples.

This means that "clean" chat (Pb below 500 mg/kg), contains some size fractions with elevated metal concentrations. The elevated metals occur in the fine sized fractions. These smaller size fractions represent a possible threat or hazard, having greater potential for leaching, transport and uptake by human or environmental receptors compared to washed coarse chat.

Sample preparation techniques can drastically affect the analytical results in chat. The large range in size fractions for chat, unlike soil samples, may cause inconsistent, non-representative analytical results depending on the sample preparation procedure. These procedures may vary from: a sample being ground to a uniform size and passed through a #10 (<2 mm) sieve prior to analysis; or analyzing only the -10 portion without prior grinding; or analyzing a representative sample without sieving or grinding. It is problematic to grind the larger chat particles into the (-10) size since they are composed mostly of very hard abrasive silica (chert). The sieving only procedure mentioned above might produce uncharacteristically high analytical results since sieving alone would bias the sample toward the finer sized material where the high lead concentrations are known to exist. To evaluate the degree of variation that may be attributed to undefined sample preparation protocols for chat, aliquots of the same sieved chat samples were sent to two independent laboratories for total metals analyses of lead, cadmium, and zinc. The analytical results were comparable between the SEL and the EPA Region VI laboratory with only relatively small differences in lead concentrations for the individual particle sizes of the sieved chat (DEQ, 2002).

The results from the asphalt samples indicate that chat coated with asphalt partially ties up the metals. The total concentrations of lead, cadmium, and zinc are less than the values for the raw chat and washed chat except for one anomaly. Only the one sample with a lead concentration of 2,228 mg/kg was at a level of concern; the next highest value was 252 mg/kg. Since the leachable lead value of 0.155 mg/l for that sample is within the range of the other samples (< 0.050 to 0.221 mg/l), the anomalously high total lead value may be an artifact of the small sample size used for total metals analyses. Since chat encapsulated in asphalt displays very low leachable lead and cadmium concentrations compared to raw and washed chat, the use of chat in asphalt is considered safe for the environment, having relatively low potential for release of metals.

Material from dry sieving has high metals concentrations that severely restrict its potential uses, unless encapsulated. Washing removes the fine material with high metals concentrations, so use of washed chat is considered acceptable for many purposes. Fine particles of chat and sediment deposited in the old mill ponds have high metals concentrations.

The high suspended solids loads carried by chat wash waters are effectively removed in settling ponds. Removal of the fine sized material with high metals concentrations from chat improves its quality for commercial use. The deposition of a large amount of the fine chat material into the sedimentation basins, that are old mill ponds and represent areas of preexisting contamination, does not result in increased environmental degradation.

Although the makeup water and process waters are of poor quality, they are contained in and recycled through these total retention basins that overlying clay soils or shale bedrock. As a consequence, no additional environmental degradation is realized.

There is a good correlation between total lead and leachable lead concentrations. This is not the case for cadmium due to anomalously high total cadmium values. Compared to total metals analysis, the TCLP analysis is thought to be more representative of metal content in chat samples because of the larger sample size used in the analysis (100 grams for TCLP compared to 0.5 to 1 grams for Total metals). Leachable metals concentrations from SPLP tests are much lower than values obtained from TCLP tests on the same sample.

Recommendations

Because of the limited data obtained in this study, additional testing of chat washing operations should be conducted to substantiate the conclusions made, (e.g., that lower metals concentrations occur in washed chat versus raw chat). Also, process water should be evaluated over time to document possible increases in contaminant concentrations with repeated cycling of wastewater through the system. Sieve analysis should be conducted on the sediments from the settling ponds along with chemical analysis of each size fraction to determine the amount of fine material and mass of metals removed from chat piles and deposited in the ponds through washing.

Analyses of washed coarse chat particles should be compared to crushed (fine sized) particles of the same sample to evaluate the effect of increased surface area on metals concentration.

Vegetative caps, covers or possibly liners over the settling ponds after chat washing operations are completed is needed to limit exposure to high lead concentrations in the sediment. Seepage from impoundment berms or bottom sediment may be minimized through engineered controls (using soil amendments or liners for reduction of soil permeability) to prevent possible future environmental contamination (through releases and seepage to shallow ground water or off site surface water). There may be seepage from the impoundment berms or bottom sediment, but under the current existing site conditions, bottom liners are not considered necessary prior to initiating a washing operation. More importantly, adequate berm free board is necessary to prevent releases during flood conditions. Engineered controls should also be employed to eliminate dust emissions during chat hauling, loading, and dry sieving.

Regulation of chat washing operations falls within the jurisdiction of DEQ for Storm Water and Process Water controls (Water Quality Division) and for dust emission controls (Air Quality division). Also, water withdrawal permits may be required from the OWRB if surface water or ground water is to be used as process water for commercial purposes.

Additional testing and sampling needs to be done to determine the long-term effects of using chat-containing asphalt.

The extraction solution used in the TCLP tests should be reported with the analyses. Sample preparation protocol for chat may need to be standardized to make data comparisons.

List of Acronyms

| | |
|--------|---|
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act of 1980, also known as Superfund: Amended in 1986 by the Superfund Amendments and Reauthorization Act (SARA). |
| DEQ | Oklahoma Department of Environmental Quality |
| DOI | The United States Department of Interior |
| EPA | United States Environmental Protection Agency |
| ROD | Record of Decision: Documents selection of cost-effective Superfund Financed remedy. |
| SEL | The Oklahoma State Environmental Laboratory |
| TCLP | Toxicity Characteristic Leaching Procedure |
| USACE | United States Army Corps of Engineer |
| SPLP | Synthetic Precipitation Leaching Procedure |
| mg/kg | milligram per kilogram |
| mg/l | milligram per liter |
| RCRA | Resource Conservation and Recovery Act |
| RI/FS | Remedial Investigation / Feasibility Study |
| OWRB | Oklahoma Water Resources Board |

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FIGURES

TAR CREEK SUPERFUND SITE

Ottawa County, Oklahoma

LEGEND

Hydrography

Roads

CHAT_PILES

Existing_Wells

Mine_Drainage

Monitor_Wells

ROAD_SAMPLES

N

E

S

W

Miles

0

0.5

1

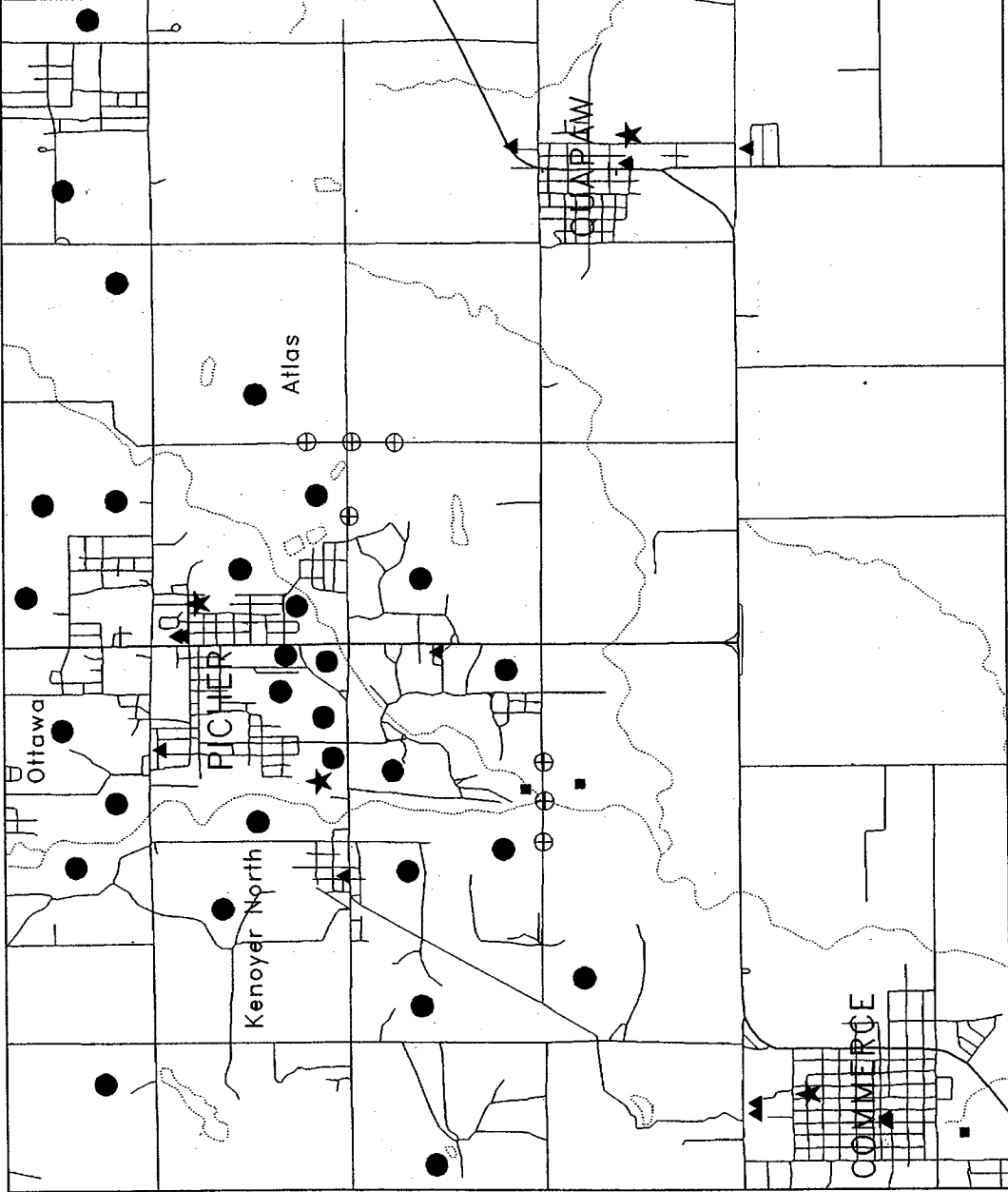


FIGURE 2: OTTAWA CHAT WASHING DATA
Total Concentrations of Lead, Cadmium & Zinc

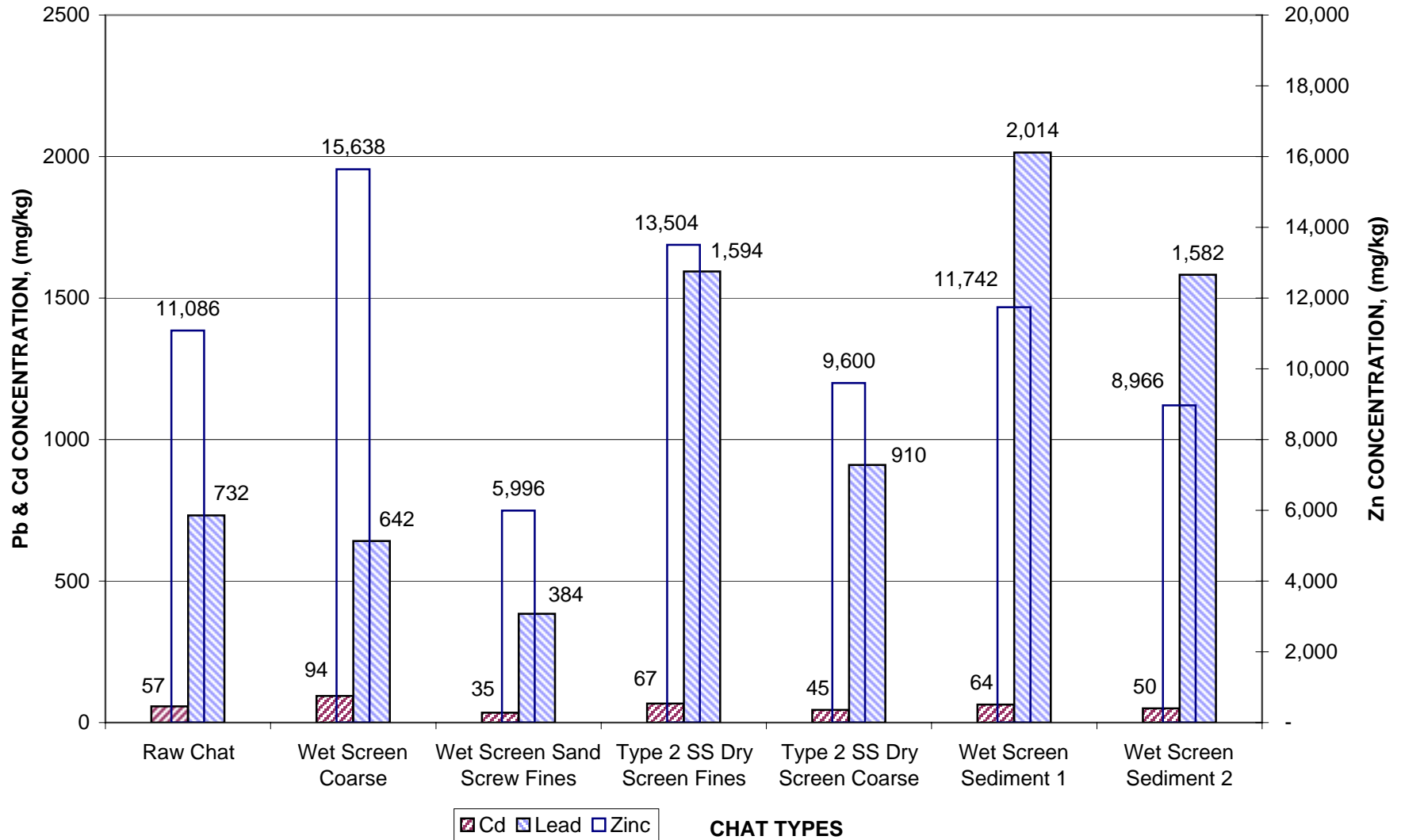
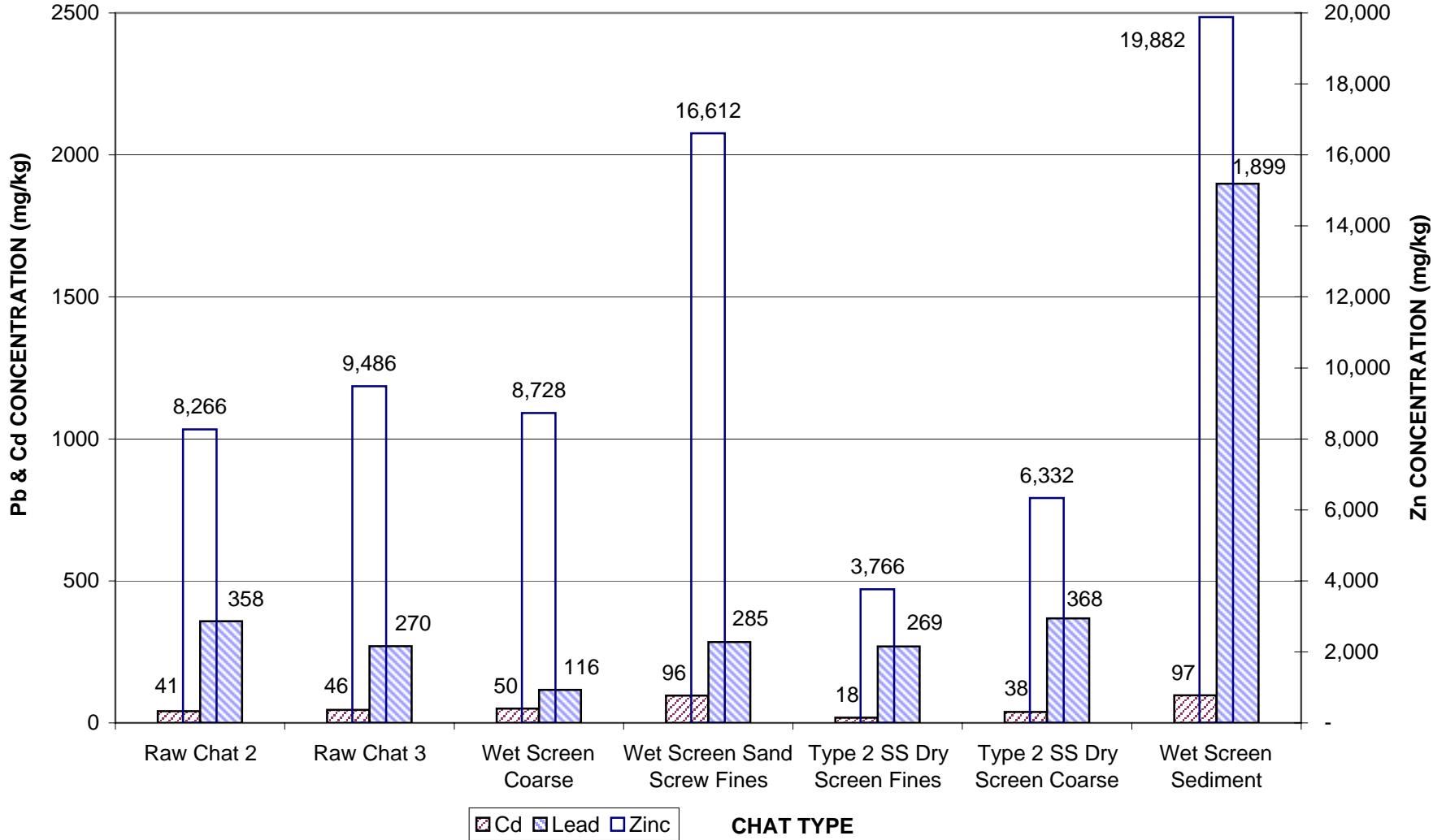
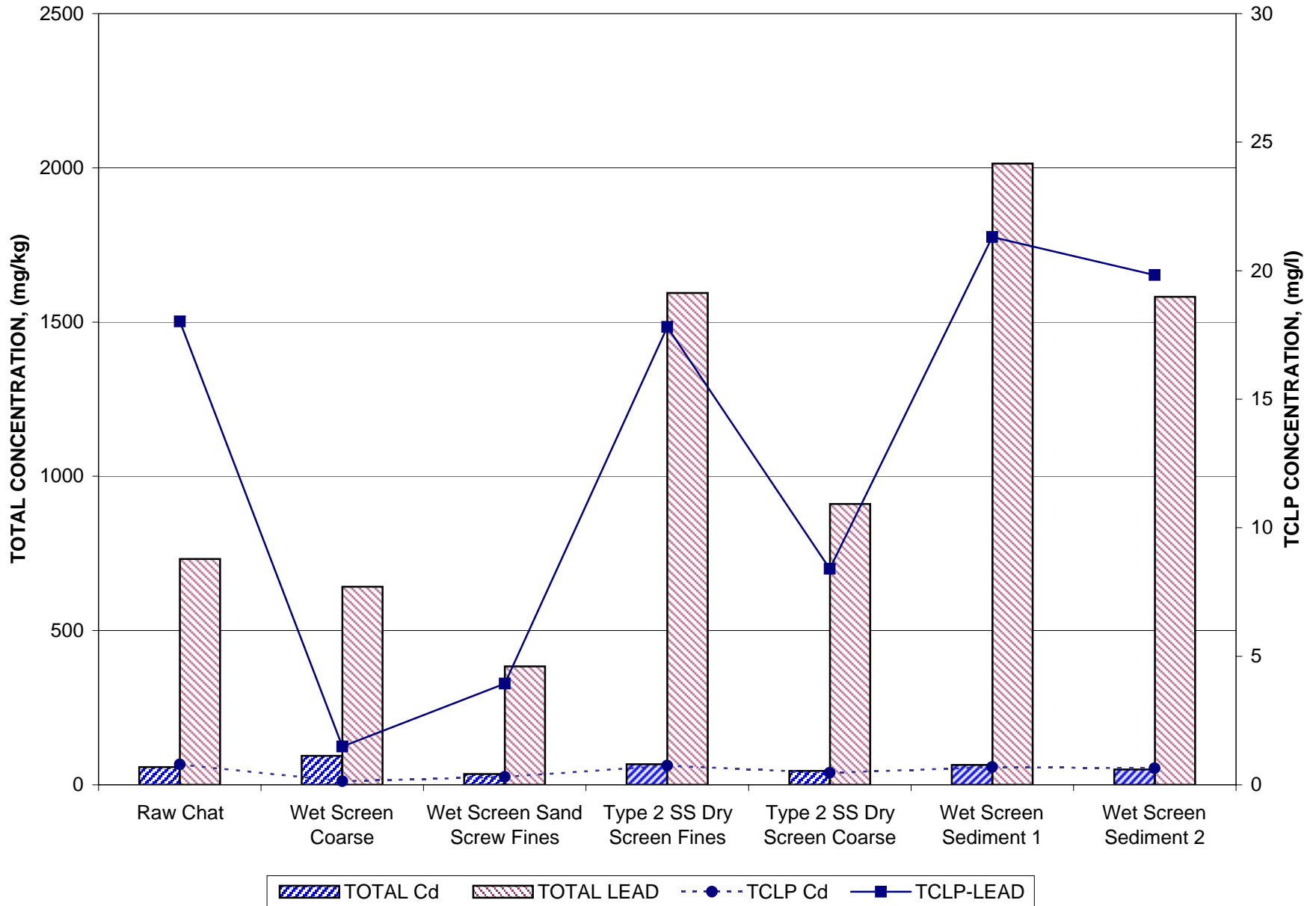


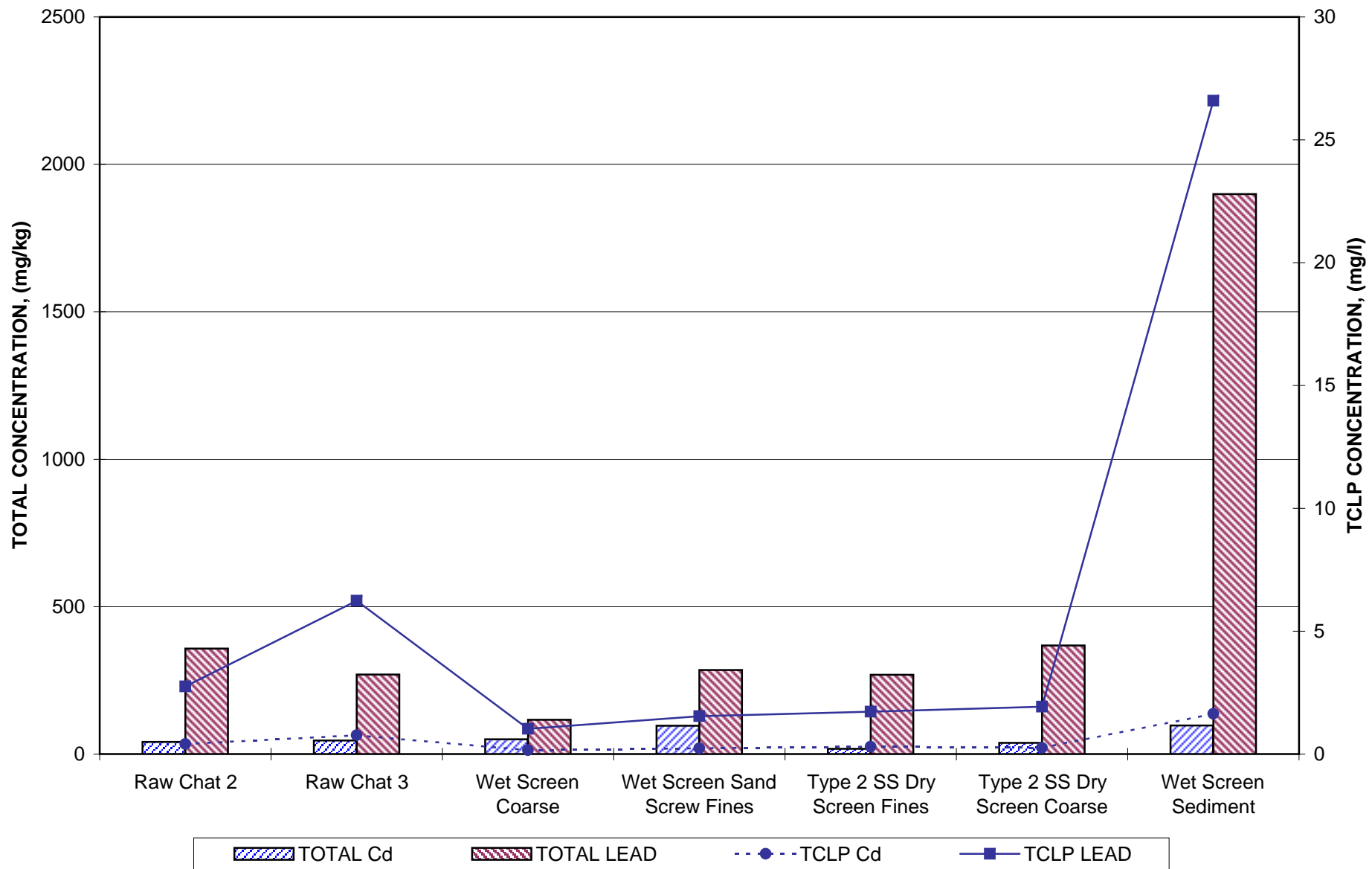
FIGURE 3: ATLAS CHAT WASHING DATA
Total Concentration of Lead, Cadmium & Zinc



**FIGURE 4: LEACHABLE (TCLP) VS TOTAL LEAD & CADMIUM
at the Ottawa Chat Pile**



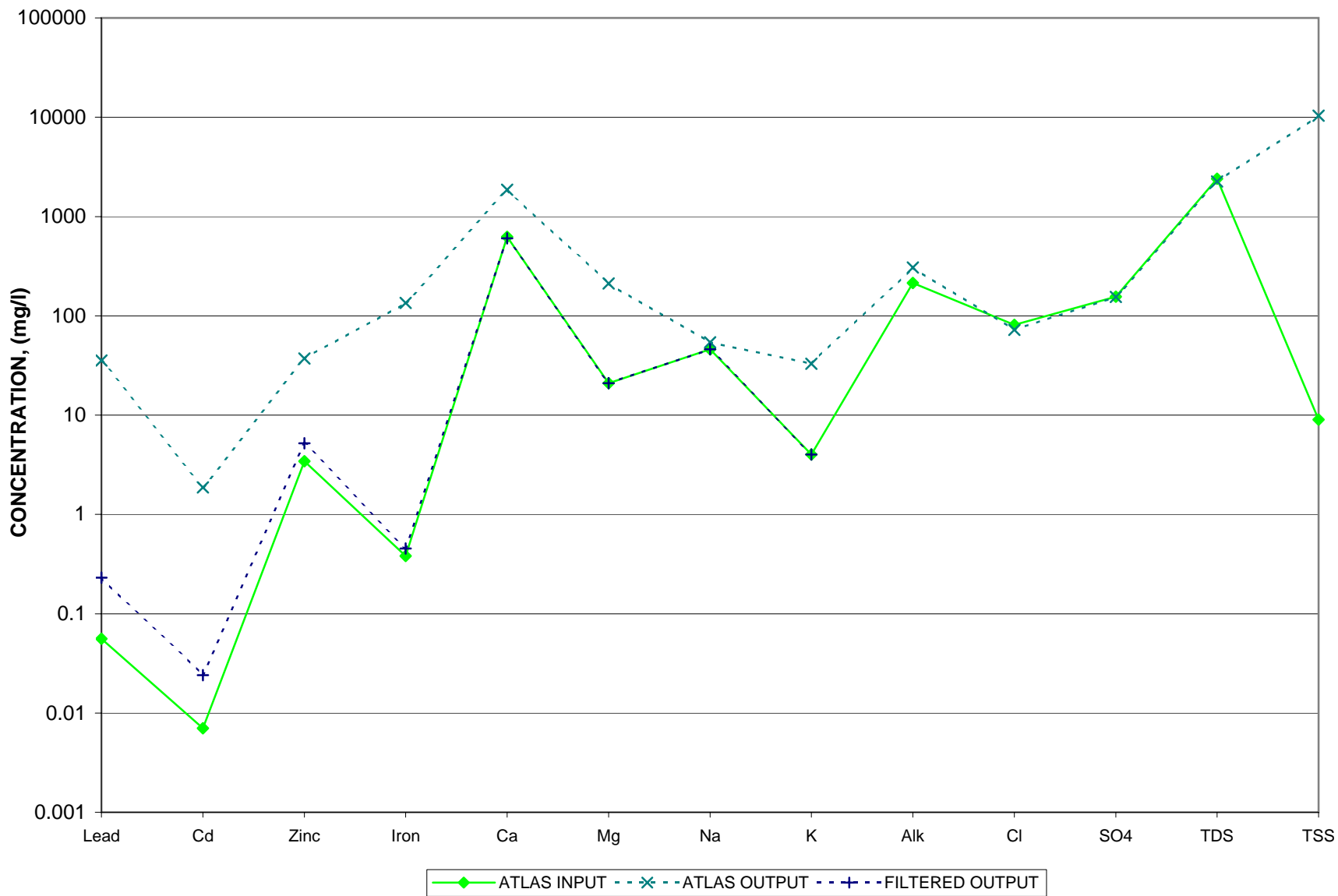
**FIGURE 5: LEACHABLE (TCLP) VS TOTAL LEAD & CADMIUM
at the Atlas Chat Pile**



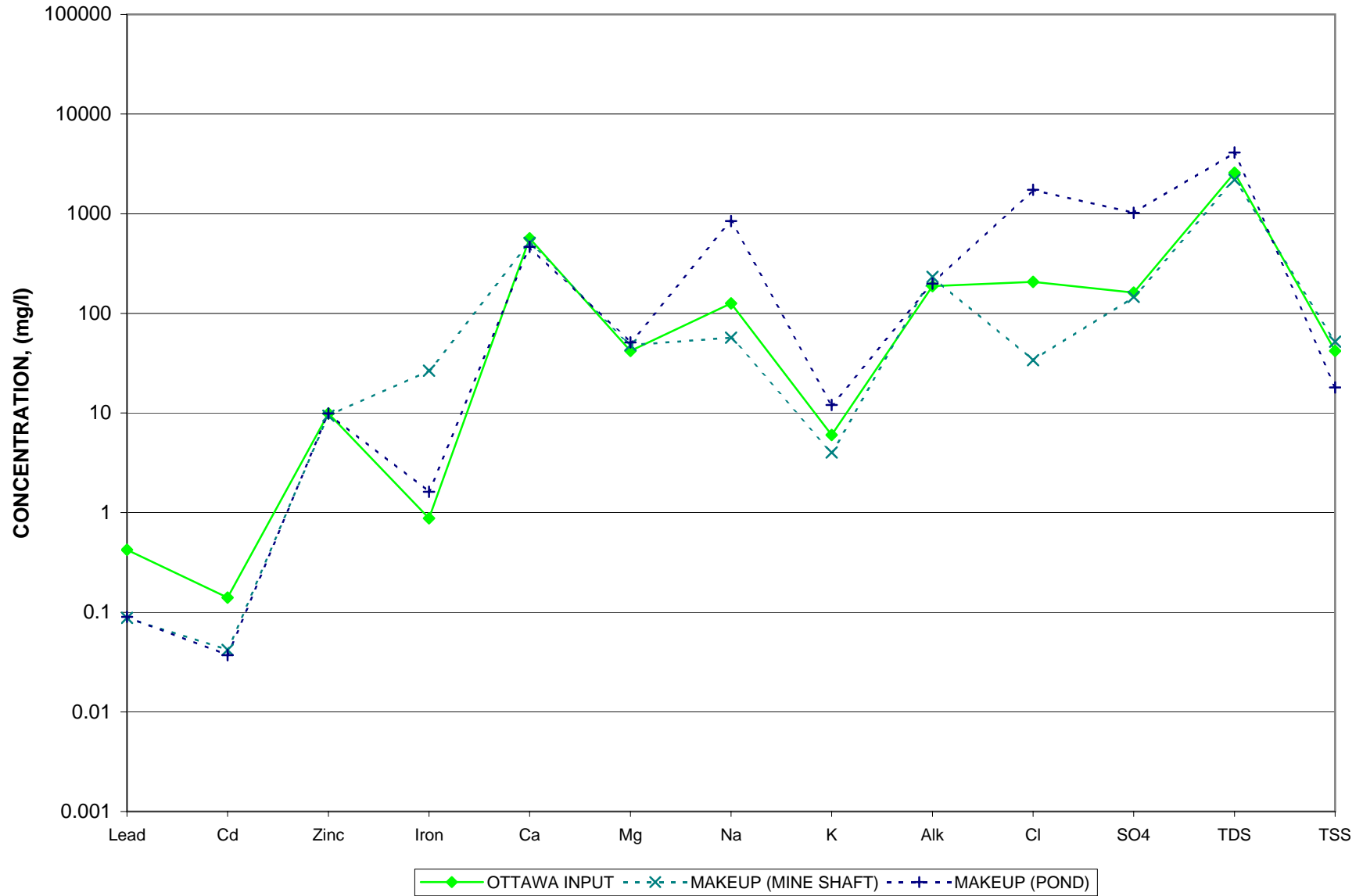
**FIGURE 6: CHAT WASHING PROCESS WATER
at the Ottawa Chat Pile**



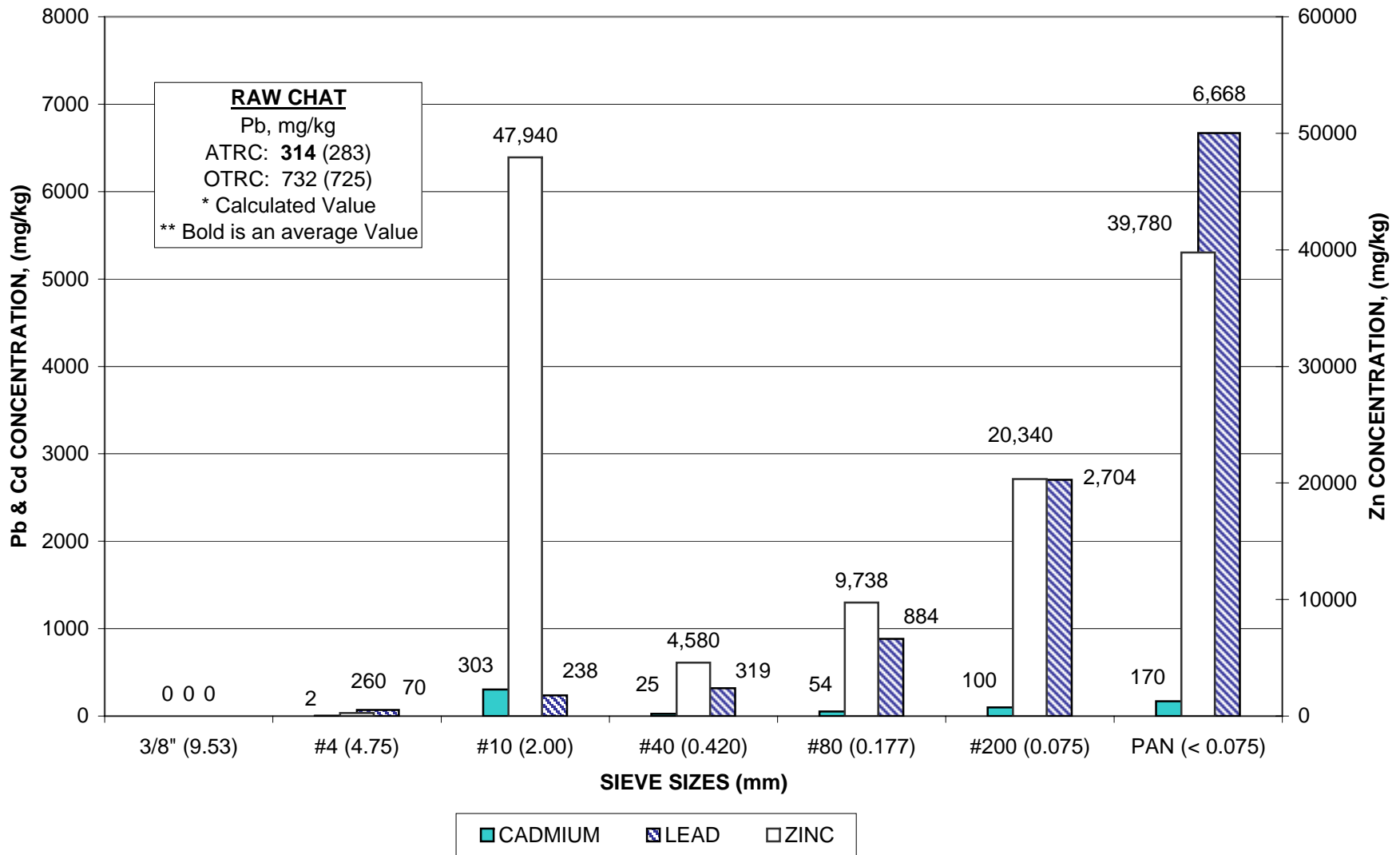
**FIGURE 7: CHAT WASHING PROCESS WATER
at the Atlas Chat Pile**



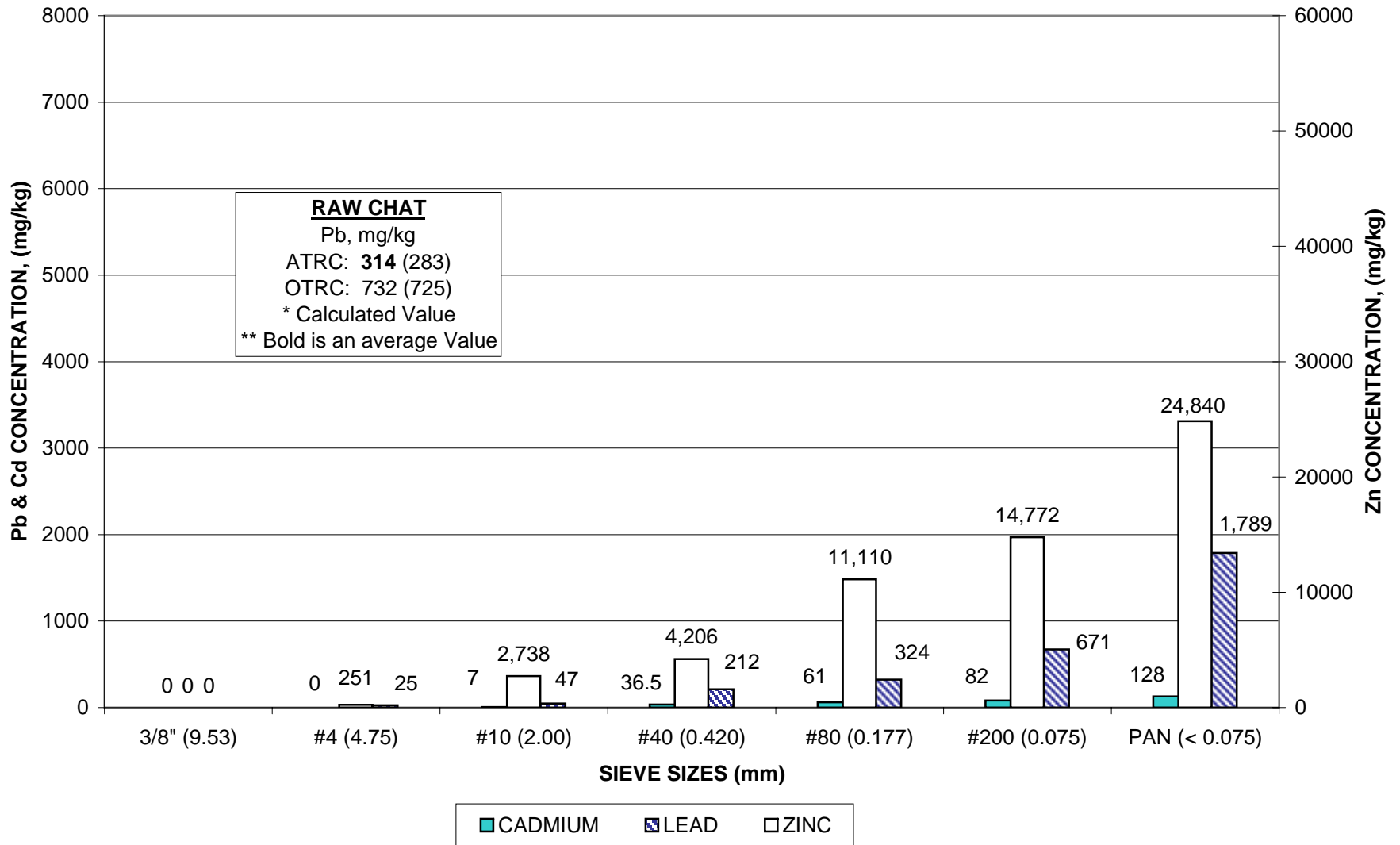
**FIGURE 8: CHAT WASHING MAKEUP WATER
at the Ottawa Chat Pile**



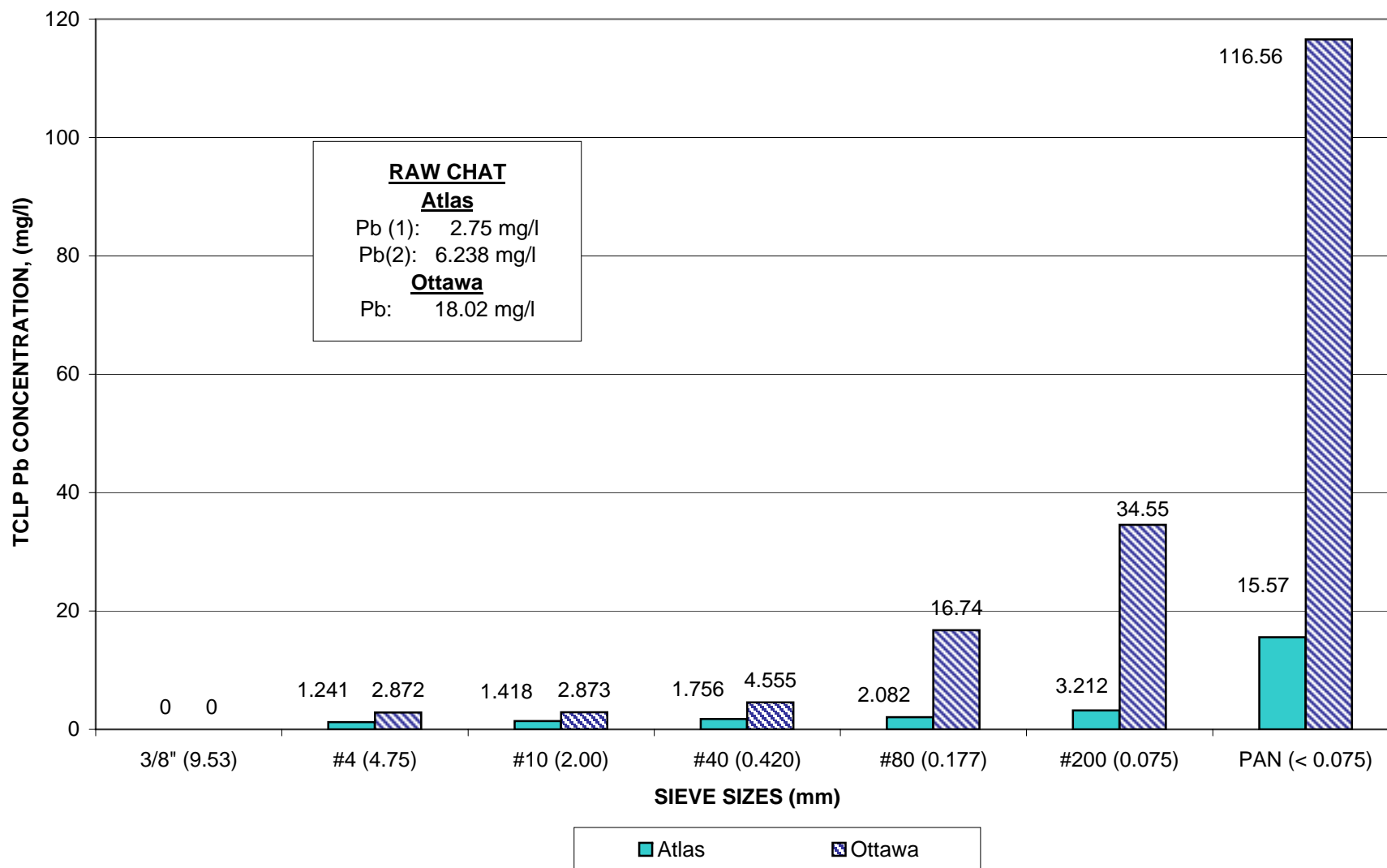
**FIGURE 9: TOTAL LEAD, CADMIUM & ZINC IN SIEVED CHAT
at the Ottawa Chat Pile**



**FIGURE 10: TOTAL LEAD, CADMIUM & ZINC IN SIEVED CHAT
at the Atlas Chat Pile**



**FIGURE 11: LEACHABLE LEAD (TCLP METHOD 1311) IN SIEVED CHAT
at the Atlas & Ottawa Chat Piles**



**FIGURE 12: LEACHABLE CADMIUM (TCLP METHOD 1311) IN SIEVED CHAT
at the Atlas & Ottawa Chat Piles**

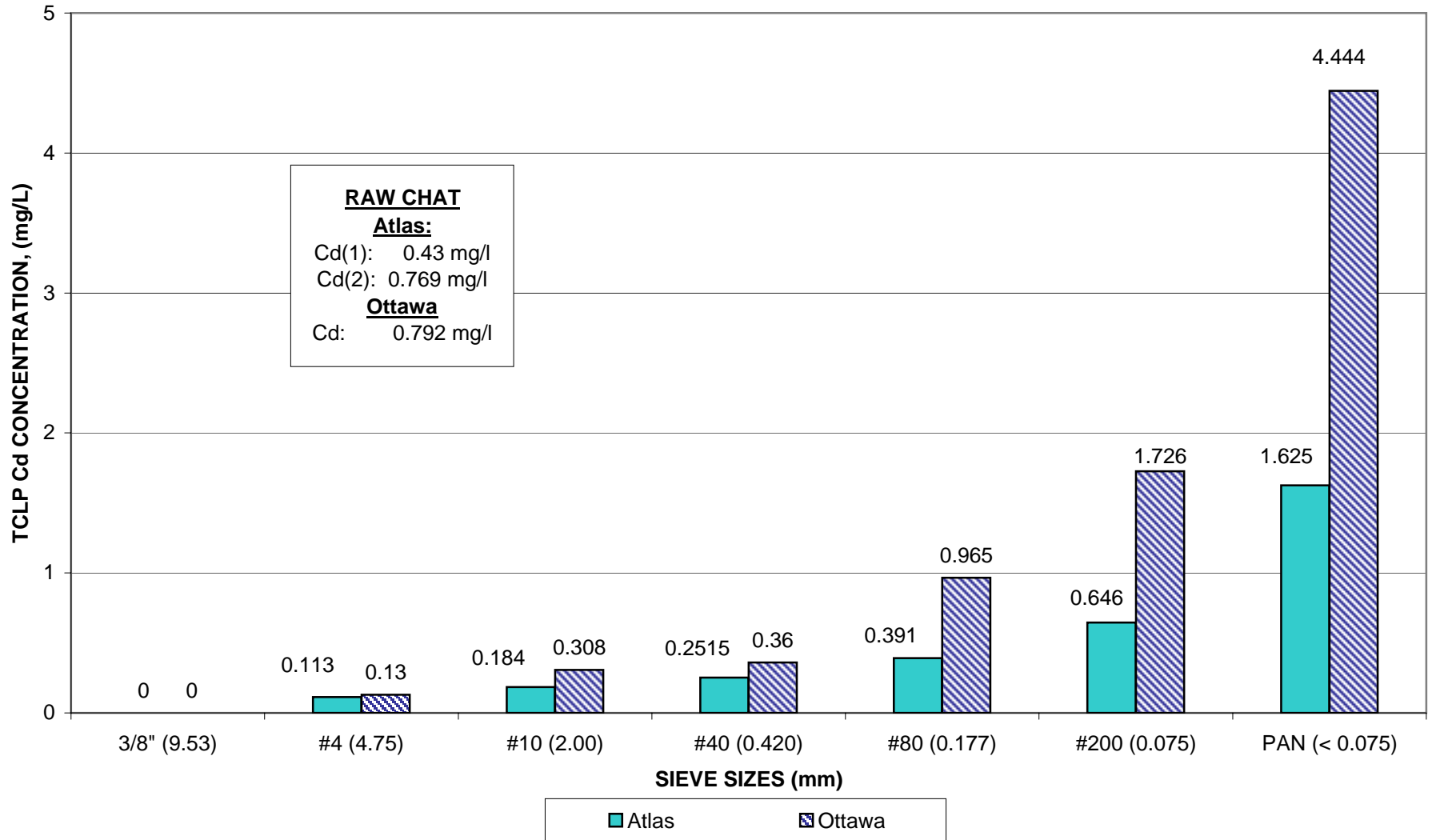


FIGURE 13: TOTAL LEAD VS LEACHABLE (TCLP METHOD 1311) LEAD

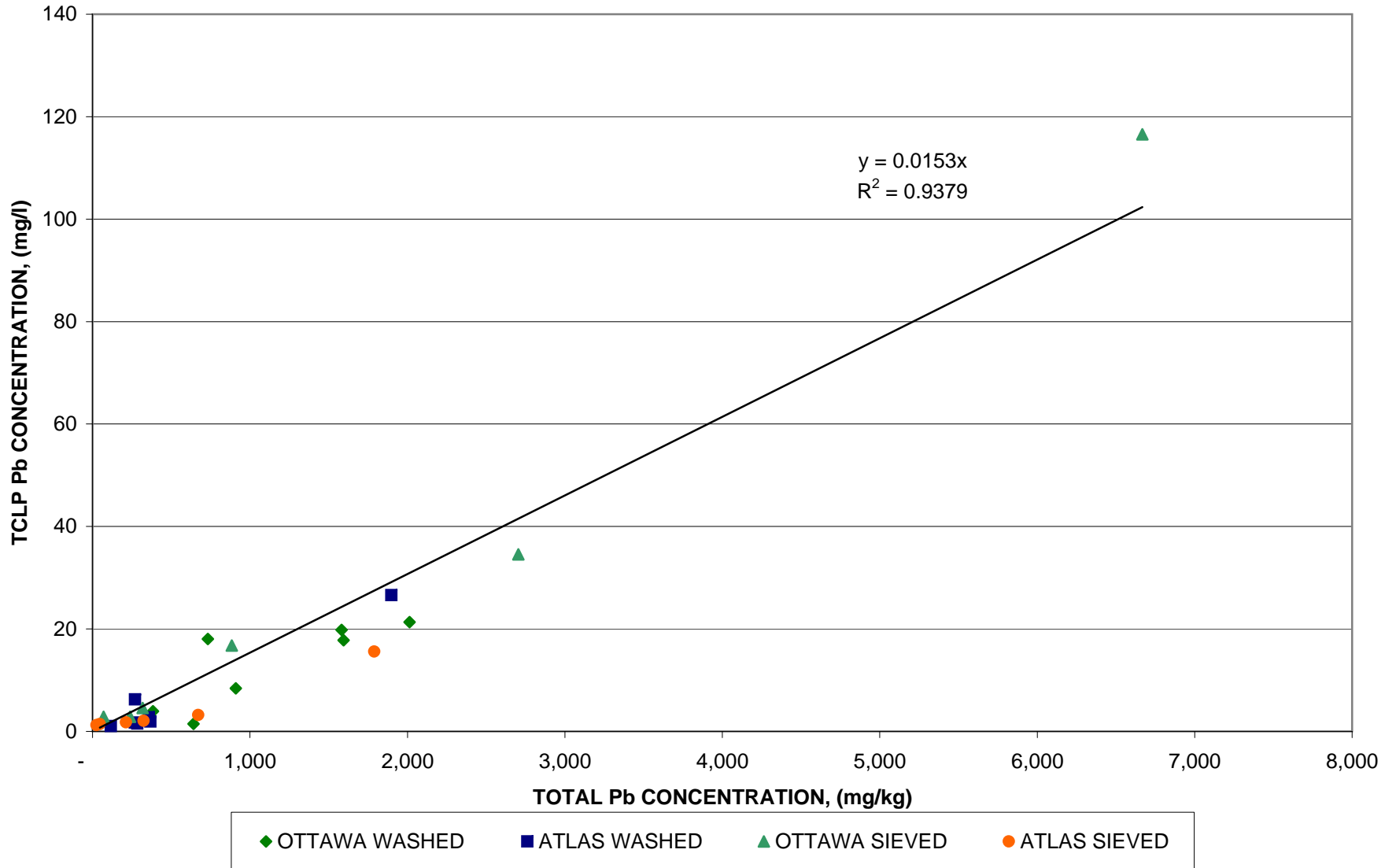


FIGURE 14: TOTAL CADMIUM VS LEACHABLE (TCLP METHOD 1311) CADMIUM

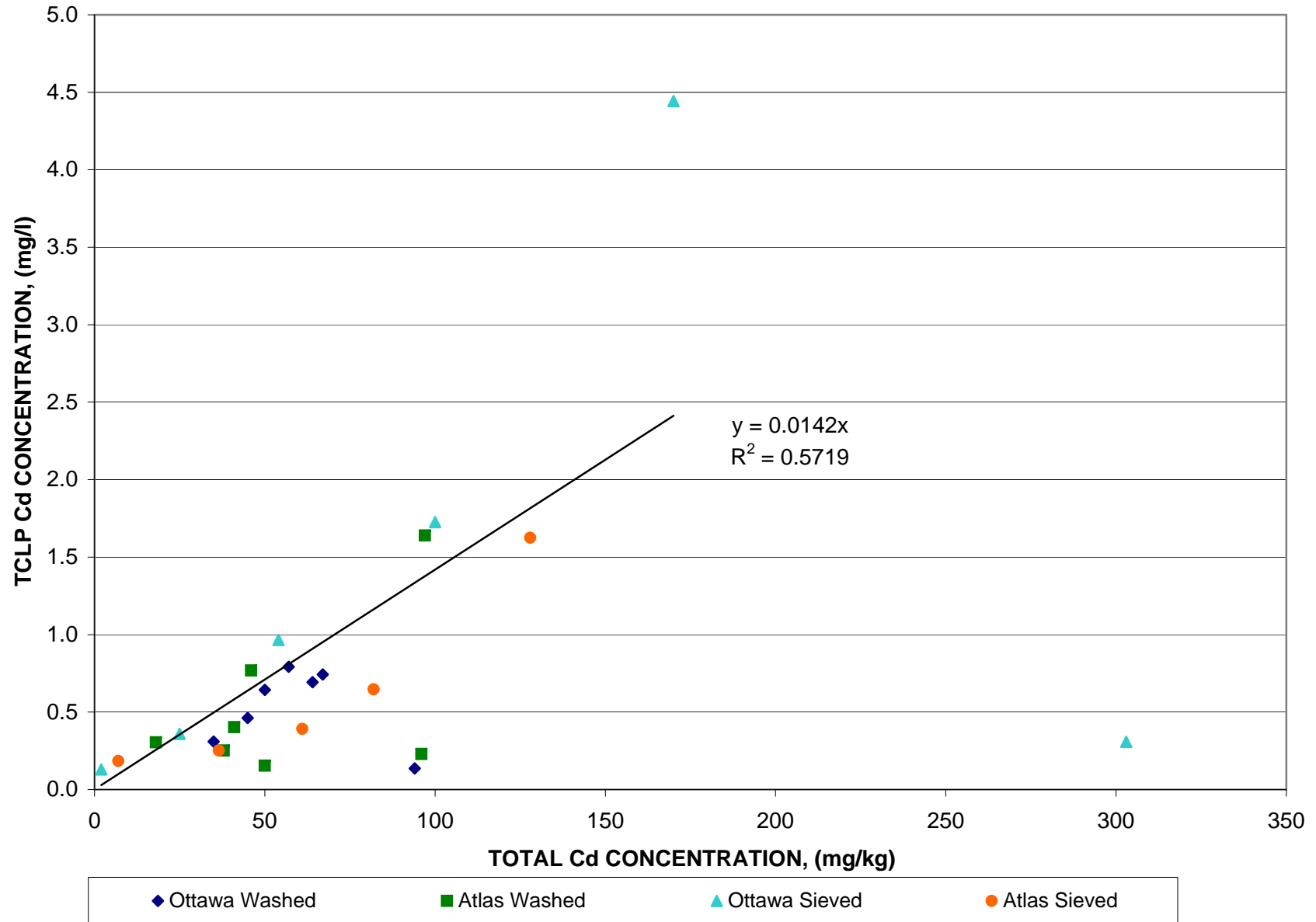
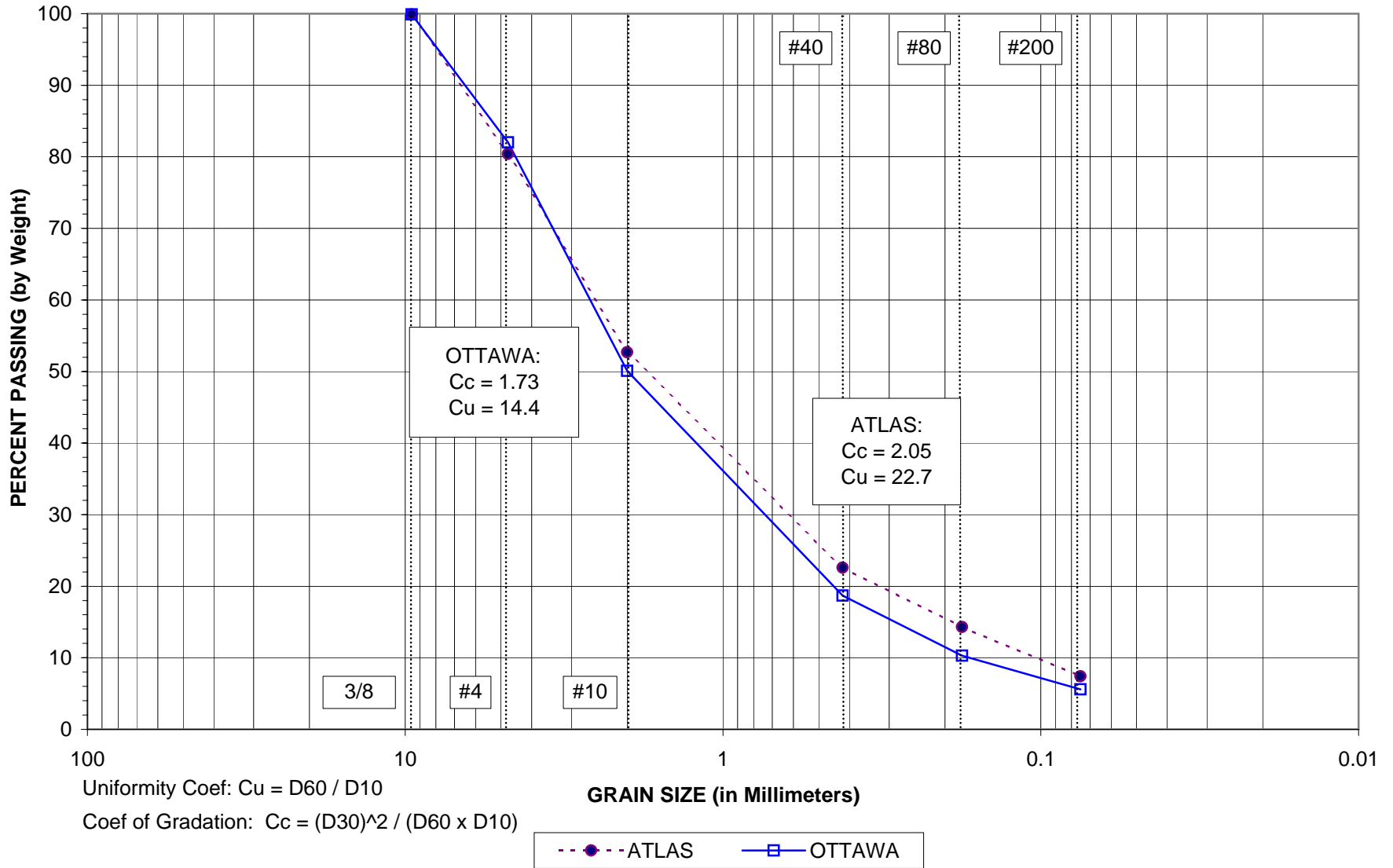


FIGURE 15: SIEVE ANALYSES
Atlas & Ottawa Chat Piles



**FIGURE 16: MASS DISTRIBUTION OF LEAD IN SIEVED CHAT
Atlas & Ottawa Chat Piles**

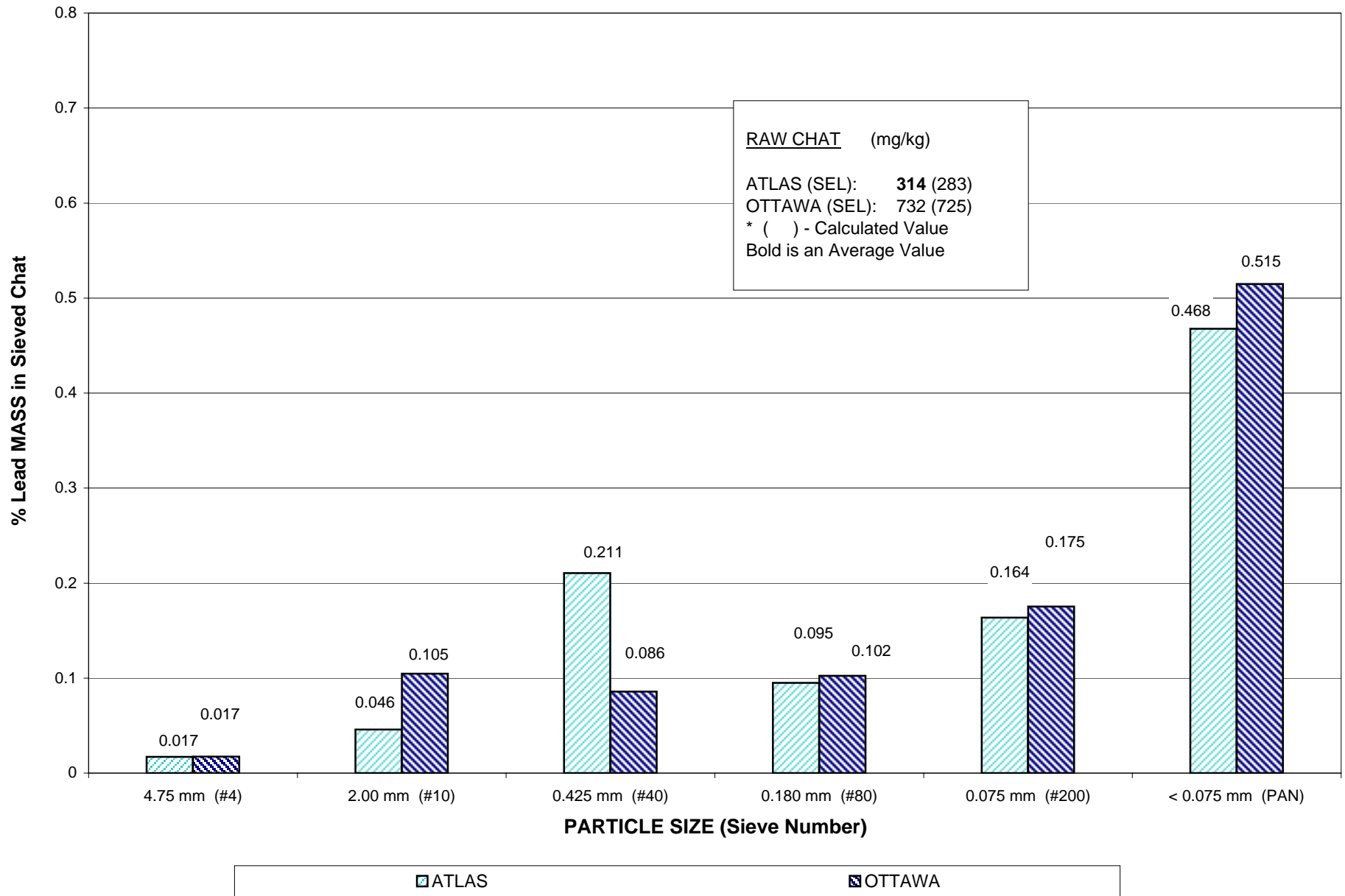
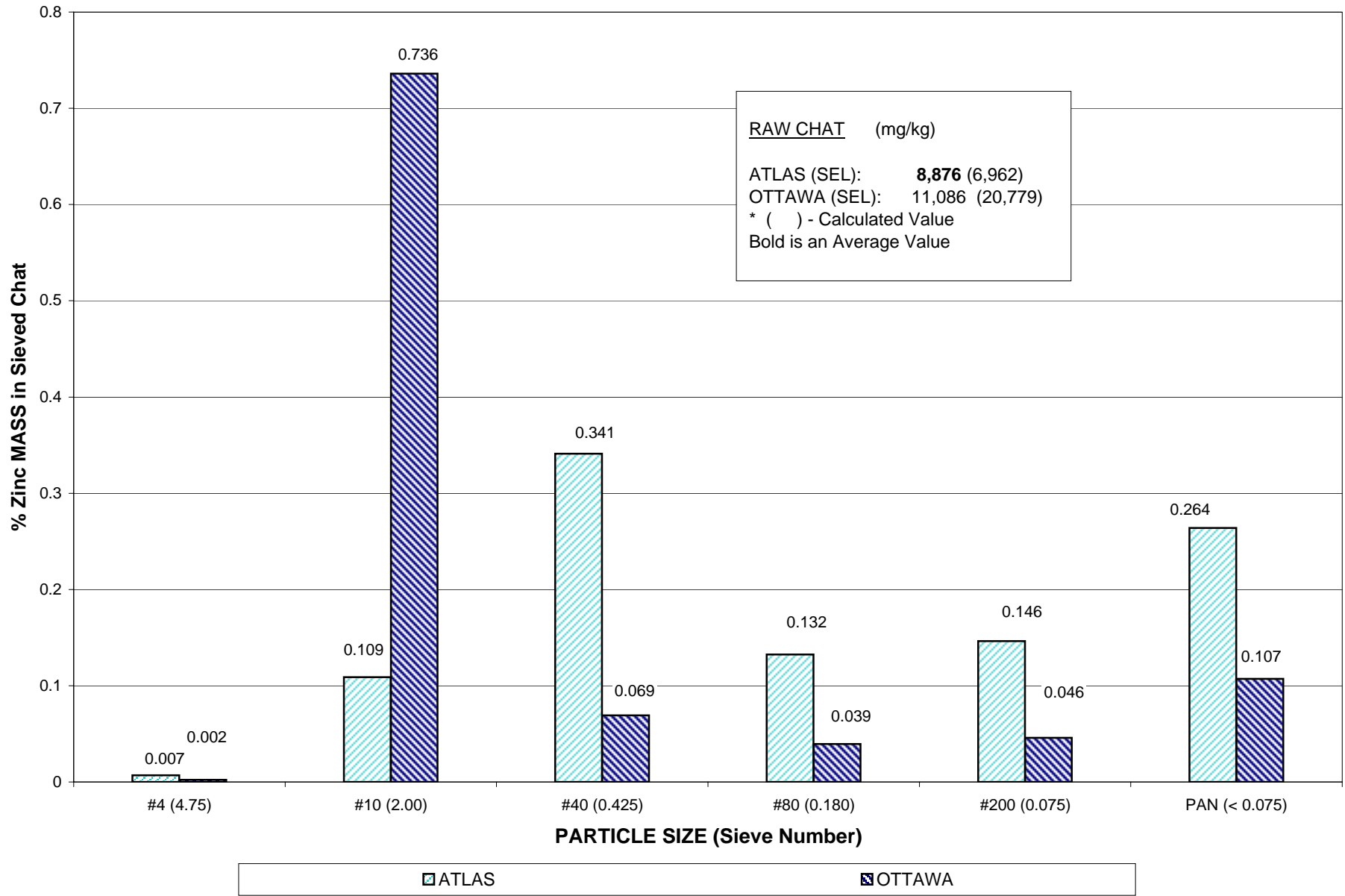
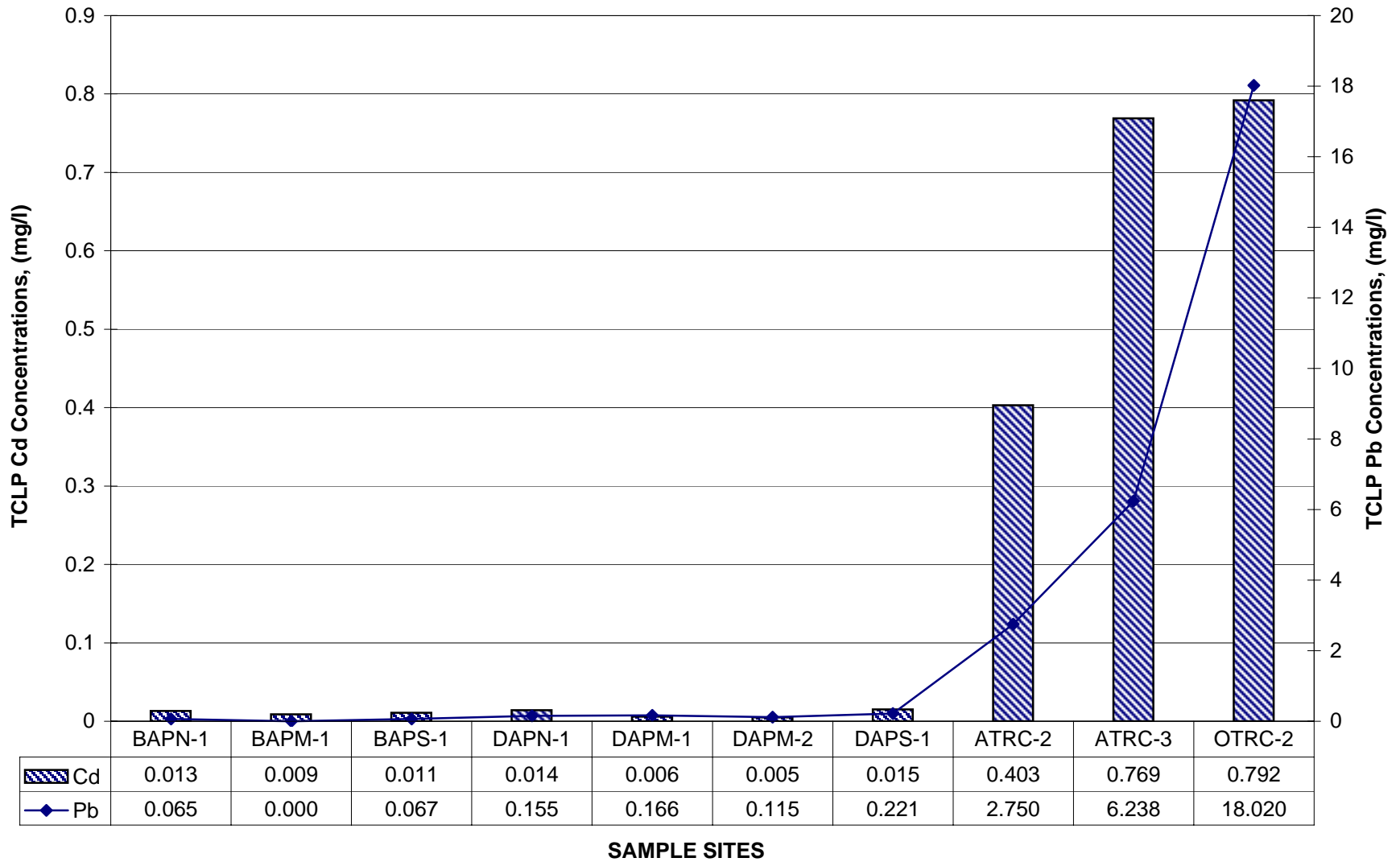


FIGURE 17: MASS DISTRIBUTION OF ZINC IN SIEVED CHAT
Atlas and Ottawa Chat Piles



**FIGURE 18: LEACHABLE (TCLP METHOD 1311) LEAD & CADMIUM
Atlas, Ottawa, & Milled Asphalt Piles**



TABLES

TABLE 1. DATA RESULTS FOR THE CHAT WASHING

Collected by The Oklahoma Department of Environmental Quality on November 23, 1999

| CHAT SAMPLES PROCESS ID | IDENTIFICATION | TOTAL METALS | | | TCLP RESULTS | |
|--|----------------|-----------------|--------------------|-----------------|----------------|-------------------|
| | NUMBER | Lead (mg/kg) | Cadmium (mg/kg) | Zinc (mg/kg) | Lead (mg/l) | Cadmium (mg/l) |
| OTTAWA CHAT PILE | | | | | | |
| Raw Chat | OTRC-2 | 732 | 57 | 11,086 | 18.020 | 0.792 |
| Wet Screen Coarse | OTWSC-1 | 642 | 94 | 15,638 | 1.487 | 0.137 |
| Wet Screen Sand Screw Fines | OTWSSSF-1 | 384 | 35 | 5,996 | 3.938 | 0.310 |
| Type 2 Slurry Seal: Dry Screen Fines | OTT2SS-1 | 1,594 | 67 | 13,504 | 17.810 | 0.742 |
| Type 2 Slurry Seal: Dry Screen Coarse | OTT2SSC-1 | 910 | 45 | 9,600 | 8.406 | 0.462 |
| Wet Screen Sediment 1 | OTWSSSED-1 | 2,014 | 64 | 11,742 | 21.310 | 0.693 |
| Wet Screen Sediment 2 | OTWSSSED-2 | 1,582 | 50 | 8,966 | 19.830 | 0.643 |
| ATLAS CHAT PILE | | | | | | |
| Raw Chat 2 | ATRC-2 | 358 | 41 | 8,266 | 2.750 | 0.403 |
| Raw Chat 3 | ATRC-3 | 270 | 46 | 9,486 | 6.238 | 0.769 |
| Wet Screen Coarse | ATWSC-1 | 116 | 50 | 8,728 | 1.028 | 0.154 |
| Wet Screen Sand Screw Fines | ATWSSF-1 | 285 | 96 | 16,612 | 1.540 | 0.229 |
| Type 2 Slurry Seal: Dry Screen Fines | ATT2SS-2 | 269 | 18 | 3,766 | 1.720 | 0.305 |
| Type 2 Slurry Seal: Dry Screen Coarse | ATT2SSC-1 | 368 | 38 | 6,332 | 1.926 | 0.252 |
| Wet Screen Sediment | ATWSSSED-1 | 1,899 | 97 | 19,882 | 26.590 | 1.640 |
| NOTE: The total for arsenic was also run and was less than the detection limit of 40 mg/kg. | | | | | | |
| TCLP = Toxicity Characteristic Leaching Procedure | | | | | | |

TABLE 3. DATA RESULTS FOR SIEVING THE RAW UNWASHED CHAT

Collected by The Oklahoma Department of Environmental Quality on November 23, 1999 and sieved on December 21, 1999.

ATLAS RAW CHAT PILE (ATRC)

| SIEVE SIZE/NUMBER | % PASSING | % RETAINED | RETENTION SIZE | TOTAL METALS | | | TCLP | |
|--|---|------------|-----------------------|-----------------|--------------------|-----------------|----------------|-------------------|
| | | | | LEAD (mg/kg) | CADMIUM (mg/kg) | ZINC (mg/kg) | LEAD (mg/l) | CADMIUM (mg/l) |
| 1/2 INCH SIEVE (medium size gravel) | 100.0 | 0.0 | 0.500 inch (12.70 mm) | NA | NA | NA | NA | NA |
| 3/8 INCH SIEVE (medium size gravel) | 99.9 | 0.1 | 0.375 inch (9.53 mm) | NA | NA | NA | NA | NA |
| ATRC-4 (fine size gravel) | 80.4 | 19.5 | 0.187 inch (4.75 mm) | 25 | ND | 251 | 1.241 | 0.113 |
| ATRC-10 (very coarse sand or very fine gravel) | 52.7 | 27.7 | 0.079 inch (2.00 mm) | 47 | 7 | 2,738 | 1.418 | 0.184 |
| ATRC-40 (medium size sand) | 22.6 | 30.1 | 0.017 inch (420 um) | 198 [225] | 41 [32] | 7892 [520] | 1.812 [1.700] | 0.251 [0.252] |
| ATRC-80 (fine size sand) | 14.3 | 8.3 | 0.007 inch (177 um) | 324 | 61 | 11,110 | 2.082 | 0.391 |
| ATRC-200 (clay size particles) | 7.4 | 6.9 | 0.003 inch (75 um) | 671 | 82 | 14,772 | 3.212 | 0.646 |
| ATRC-PAN (clay size particles) | | 7.4 | | 1,789 | 128 | 24,840 | 15.57 | 1.625 |
| NOTES: | NA = Data not available to run test | | | | | | | |
| | ND = Non-detect | | | | | | | |
| | Data was collected on November 23, 1999 | | | | | | | |
| | Data was sieved on December 21, 1999 | | | | | | | |
| | Duplicate run on ATRC-40 with result in [] | | | | | | | |
| | ATRC means Atlas Raw Chat | | | | | | | |
| | The total for arsenic was also run and was less than the detection limit of 40 mg/kg. | | | | | | | |
| | TCLP = Toxicity Characteristic Leaching Procedure | | | | | | | |

TABLE 3 CONTINUED. DATA RESULTS FOR SIEVING OF THE RAW UNWASHED CHAT

Collected by The Oklahoma Department of Environmental Quality on November 23, 1999 and sieved on December 21, 1999.

OTTAWA RAW CHAT PILE (OTRC)

| SIEVE SIZE/NUMBER | % PASSING | % RETAINED | RETENTION SIZE | TOTAL METALS | | | TCLP | |
|--|---|------------|-----------------------|-----------------|--------------------|-----------------|----------------|-------------------|
| | | | | LEAD (mg/kg) | CADMIUM (mg/kg) | ZINC (mg/kg) | LEAD (mg/l) | CADMIUM (mg/l) |
| 1/2 INCH SIEVE (medium size gravel) | 100 | 0.0 | 0.500 inch (12.70 mm) | NA | NA | NA | NA | NA |
| 3/8 INCH SIEVE (medium size gravel) | 99.9 | 0.1 | 0.375 inch (9.53 mm) | NA | NA | NA | NA | NA |
| OTRC-4 (fine size gravel) | 82.0 | 17.9 | 0.187 inch (4.75 mm) | 70 | 2 | 260 | 2.872 | 0.13 |
| OTRC-10 (very coarse sand or very fine gravel) | 50.1 | 31.9 | 0.079 inch (2.00 mm) | 238 | 303 | 47,940 | 2.873 | 0.308 |
| OTRC-40 (medium size sand) | 18.7 | 31.4 | 0.017 inch (420 um) | 319 | 25 | 4,580 | 4.555 | 0.36 |
| OTRC-80 (fine size sand) | 10.3 | 8.4 | 0.007 inch (177 um) | 884 | 54 | 9,738 | 16.74 | 0.965 |
| OTRC-200 (clay size particles) | 5.6 | 4.7 | 0.003 inch (75 um) | 2,704 | 100 | 20,340 | 34.55 | 1.726 |
| OTRC-PAN (clay size particles) | | 5.6 | | 6,668 | 170 | 39,780 | 116.56 | 4.444 |
| NOTES: | NA = No data to run test | | | | | | | |
| | Data was collected on November 23, 1999 | | | | | | | |
| | Data was sieved on December 21, 1999 | | | | | | | |
| | OTRC means Ottawa Raw Chat | | | | | | | |
| | The total for arsenic was also run and was less than the detection limit of 40 mg/kg. | | | | | | | |
| | TCLP = Toxicity Characteristic Leaching Procedure | | | | | | | |

**TABLE 4. TESTING OF ASPHALT REMOVED FROM THE WILL ROGERS TURNPIKE
 LOCATED NEAR QUAPAW OKLAHOMA
 Asphalt contained mining waste (chat) of unknown levels for metals.
 Collected on February 25, 2000**

| LOCATION | TOTALS | | | TCLP | |
|------------------------------|---------|---------|---------|---------|---------|
| | LEAD | CADMIUM | ZINC | LEAD | CADMIUM |
| | (mg/kg) | (mg/kg) | (mg/kg) | (mg/l) | (mg/l) |
| BAPNORTH-1 | 158 | 17 | 2,718 | 0.065 | 0.013 |
| BAPMIDDLE-1 | 141 | 15 | 1,761 | < 0.050 | 0.009 |
| BAPSOUTH-1 | 179 | 35 | 5,104 | 0.067 | 0.011 |
| DAPNORTH-1 | 2,228 | 12 | 1,934 | 0.155 | 0.014 |
| DAPMIDDLE-1 | 168 | 18 | 2,594 | 0.166 | 0.006 |
| DAPMIDDLE-2 (DUPLICATE OF 1) | 160 | 17 | 2,708 | 0.115 | 0.005 |
| DAPSOUTH-1 | 252 | 20 | 2,808 | 0.221 | 0.015 |

BAP = BATTIE ASPHALT PILE

DAP = DEHORN ASPHALT PILE

TCLP = Toxicity Characteristic Leaching Procedure