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DETERMINATION OF THE TOXICITY CHARACTERISTIC FOR METALS IN SOIL -
A COMPARISON OF THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE
AND TOTAL METAL DETERMINATION

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TABLE OF CONTENTS

ABSTRACT	1
I. INTRODUCTION	2
II. EXPERIMENTAL	4
A. Samples	4
B. Total Determination Method	4
C. Toxicity Characteristic Leaching Procedure	5
D. Comparison of Total Determination Method and Toxicity Characteristic Leaching Procedure	6
E. Quality Control	7
III. RESULTS AND DISCUSSION	8
A. Metal to Metal Variations	8
B. Duplicates	11
IV. CONCLUSIONS	15
ACKNOWLEDGMENTS	16
REFERENCES	16
APPENDIX A	30

LIST OF TABLES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Detection Limits in Soil (mg/kg)	17
2.	Percent Extracted by TCLP	18
3.	Duplicates for Total Determination Method	18
4.	Duplicates Extracted by TCLP	19
5.	Duplicates with Bullets Extracted by TCLP	19
A1	TCLP Extraction Efficiency for Sb, As, and Ba	31
A2	TCLP Extraction Efficiency for Cd, Cr, and Cu	33
A3	TCLP Extraction Efficiency for Pb, Mg, and Se	35
A4	TCLP Extraction Efficiency for Ag, Sn, and Zn	37
A5	Duplicate Results for Total Determination Method	39
A6	Duplicate Results for Total Determination Method	40
A7	Duplicate Results for TCLP	41
A8	Duplicate Results for TCLP	43
A9	Duplicate Results with Bullets	45

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Extraction of Metals by TCLP	20
2.	TCLP vs. Total Determination Method for As	20
3.	TCLP vs. Total Determination Method for Ba	21
4.	TCLP vs. Total Determination Method for Cd	21
5.	TCLP vs. Total Determination Method for Cd	22
6.	TCLP vs. Total Determination Method for Cr	22
7.	TCLP vs. Total Determination Method for Cu	23
8.	TCLP vs. Total Determination Method for Cu	23
9.	TCLP vs. Total Determination Method for Cu	24
10.	TCLP vs. Total Determination Method for Pb	24
11.	TCLP vs. Total Determination Method for Pb	25
12.	TCLP vs. Total Determination Method for Pb	25
13.	TCLP vs. Total Determination Method for Mg	26
14.	TCLP vs. Total Determination Method for Mg	26
15.	TCLP vs. Total Determination Method for Ag	27
16.	TCLP vs. Total Determination Method for Zn	27
17.	Duplicate Results for Total Determination Method	28
18.	Duplicate Results for TCLP	28
19.	Duplicate Results for TCLP	29
20.	Duplicate Results for TCLP with Visible Bullet	29

DETERMINATION OF THE TOXICITY CHARACTERISTIC FOR METALS IN SOIL - A COMPARISON OF THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE AND TOTAL METAL DETERMINATION

by

Dean A. Bass and John D. Taylor

ABSTRACT

A comparison is made of the concentrations of metals extracted from soils using the Toxicity Characteristic Leaching Procedure (TCLP) and a total determination method. This information is of interest in two ways. First, it is hoped that a relationship might be established between the amount of each metal determined after extraction by the TCLP and the amount determined using a total determination method. And second, data are also presented which indicate the general extractability of various metals in soil samples using the TCLP. This study looks specifically at inorganic elements (Sb, As, Ba, Cd, Cu, Cr, Pb, Mg, Hg, Se, Ag, Sn, and Zn) in soils from a firing range.

The results of this study show that total determination methods for metals can not generally be used for heterogeneous samples, such as soil samples from a firing range. Some correlation between a total determination method and TCLP was observed when Ba and Cd were present in the samples at lower concentrations (less than 80 mg/kg for Ba and less than 25 mg/kg for Cd); however, additional data are necessary to verify this correlation.

The results of this study show that total determination methods for metals can not generally be used for heterogeneous samples, such as soil samples from a firing range. Some correlation between a total determination method and TCLP was observed when Ba and Cd were present in the samples at lower concentrations (less than 80 ppb for Ba and less than 25 ppb for Cd); however, additional data are necessary to verify this correlation.

I. INTRODUCTION

The Resource Conservation and Recovery Act (RCRA) was established in 1976 to protect human health and the environment. This regulation defines RCRA hazardous waste as waste that is ignitable, corrosive, reactive, or that exhibits the toxicity characteristic. The toxicity characteristic is determined using the Toxicity Characteristic Leaching Procedure (TCLP). This procedure is U.S. EPA Method 1311 [1] and is officially described in 40 CFR 261, Appendix II-Method 1311 (Toxicity Characteristic Leaching Procedure). The TCLP is used to simulate, in a laboratory, conditions which could be present in a sanitary landfill, in order to determine whether any inorganic elements, i.e. metals, or organic compounds could be leached from the waste in the landfill.

The TCLP for metals involves an 18 hour agitation period in addition to the steps required in a total determination method, thus, the TCLP is time consuming and relatively more expensive. However, a provision in the procedure allows for the determination of the total amount of each contaminant in order to demonstrate that a waste does not exhibit toxic characteristics. If the total concentration of a contaminant is such that the appropriate regulatory levels for that contaminant could not possibly be exceeded, the TCLP need not be performed [1]. Conrad et al. [2] have described an approach that reduces the number of TCLP analyses by substituting total determination methods and thereby saving analysis costs.

This study looks specifically at determining the concentrations of inorganic elements (Sb, As, Ba, Cd, Cu, Cr, Pb, Mg, Hg, Se, Ag, Sn, and Zn) in soils from a firing range. Antimony, Cu, Mg, Sn, and Zn are not usually included as part of a TCLP extract analysis, however, these metals were measured in these samples for other reasons and the results are presented here for general interest. A comparison is made between measured concentrations of metals extracted from soils using TCLP and a total determination method.

The information collected in this study is of interest for two reasons. First, it was hoped that a relationship might be established between the amount of each metal determined after extraction by the TCLP and the amount determined using a total determination method; second, the general extractability of metals into TCLP extraction fluids can be inferred from this data. Initially, the study examines overall extraction efficiency for all metals of interest in all samples. Then results are analyzed for each metal in order to evaluate differences in extraction on a metal by metal basis. Given this breakdown, further analysis of the results is performed to determine sample variations and explore potential sources of variability.

II. EXPERIMENTAL

A. Samples

Soil samples used in this study were collected from firing ranges on a U.S. Army installation in Grafenwöhr, Germany. The samples came from pistol, rifle and hand grenade ranges. Samples were also collected from a variety of soil depths. The purpose of the original project was to evaluate the threat of metal contamination from these ranges to local groundwaters. For this purpose both total determination methods and TCLP extractions were performed on collected soil samples.

The soil samples from all ranges were classified as a sandy-loam soil. Potential sources of contamination included bullets, bullet fragments, metal jackets and casings, and gunpowder residues. Variations based on the type of range were to be expected and are only discussed to the extent to which they could result in sample to sample variations. This paper is only concerned with the comparison between the total determination method and the TCLP extraction. Conclusions regarding the extent of the metal contamination in these samples are presented elsewhere [3].

B. Total Determination Method

Soil samples were digested for total metal determinations using U.S. EPA Method 3050A (Acid Digestion of Sediments, Sludges, and Soils) [1]. Metal concentrations in the digestates were determined using U.S. EPA Method 6010 (Inductively Coupled Plasma Atomic Emission Spectroscopy, ICP-AES) [1]. The digestion method involved taking 1-2 g representative samples and heating them (about 95°C) in nitric acid and hydrogen peroxide. The samples were then refluxed in hydrochloric acid, filtered and diluted to 200 mL. The final acid concentration was about 2.5% (v/v) hydrochloric acid and 2.5% (v/v) nitric acid. The ICP-AES procedure used a two point calibration with standards prepared by dilution of certified solutions procured

from NIST or suppliers that provided NIST traceability. Measurements were made with a JY-86 ICP-AES from Instruments, S.A., Inc. This instrument consists of a 1-m JY-48 polychromator simultaneous, and a JY-38 scanning-monochromator sequential analysis system. The metals measured by ICP-AES included the TCLP metals (As, Ba, Cd, Cr, Pb, Ag, and Se), as well as other metals of general interest (Sb, Cu, Mg, Sn, and Zn).

Mercury was measured using U.S. EPA Method 7470 (Mercury in Liquid Waste) which employs cold vapor atomic absorption spectrophotometry and includes its own total digestion method. Method 3050A (total acid digestion) is not used in the total mercury determination. The digestion method used in Method 7470 involved taking 0.2 g representative samples and heating them (about 95°C) in nitric and sulfuric acid. The samples were treated with potassium permanganate, then with potassium persulfate, and finally, with sodium chloride-hydroxylamine sulfate. They were then analyzed using a Leeman PS200 Automated Mercury Analyzer System. The cold vapor procedure used a five-point calibration with standards prepared by dilution of certified solutions procured from NIST or suppliers that provided NIST traceability.

C. Toxicity Characteristic Leaching Procedure

For TCLP, U.S. EPA Method 1311 (Toxicity Characteristic Leaching Procedure) published in 40 CFR 261, Appendix II, was used. Due to the limited quantities of material, 50 g samples were used, instead of the normal 100 g. Following the TCLP extraction, the filtered extracts were digested using U.S. EPA Method 3010A. Analysis of the digested extract was by ICP-AES and flame atomic absorption (AA). Flame AA measurements were performed on a Buck 200A Atomic Absorption Spectrophotometer with a 250 mm Ebert- mount diffraction grating monochromator. An air/acetylene flame was used for all elements except As which was determined using a nitrous oxide/acetylene flame. Hollow cathode lamps were used as light sources for all elements. All instrument parameters were according to Perkin-Elmer Standard

Conditions Procedures. The flame AA procedure used a five-point calibration with standards prepared by dilution of certified solutions procured from NIST or suppliers that provided NIST traceability.

D. Comparison of Total Determination Method and Toxicity Characteristic Leaching Procedure

Two sample aliquots were taken, one for analysis using the TCLP, and the other using the total determination method. The concentration of metal in the soil was determined for both procedures (TCLP and total determination method) and the percent extracted by the TCLP was determined as follows:

$$\% \text{ Metal Extracted by TCLP} = \frac{[\text{Metal}]_{\text{TCLP}}}{[\text{Metal}]_{\text{Total}}} \times 100 \quad (1)$$

where $[\text{Metal}]_{\text{TCLP}}$ is the concentration of metal in the soil determined by the TCLP and $[\text{Metal}]_{\text{Total}}$ is the concentration of metal in the soil determined by the total determination method.

NOTE: The percent metal extracted by TCLP was not calculated for any given sample if either metal concentration was below the detection limit. That is, if the concentration extracted by the TCLP was below the detection limit but the concentration determined by the total determination method was 50 ppm, then the percent metal extracted by TCLP would not be calculated for this sample. The detection limits for each metal are given in Table 1. These detection limits are approximate and may vary based on sample size and daily fluctuations in instrument conditions.

E. Quality Control

Quality control samples were run in accordance with the U.S. EPA Methods. These included sample duplicates, laboratory control samples (LCSs), spikes, spike duplicates, post digestion spikes, serial dilutions and calibration verifications.

Laboratory duplicates are two separate portions of sample that are taken through the entire extraction and analysis procedures. This QC helps evaluate sample homogeneity. Typically, one laboratory duplicate is processed for each batch of soils digested together (usually consisting of about ten samples). For this study, additional duplicates were analyzed to better understand the source of the variations and these results are also presented.

III. RESULTS AND DISCUSSION

This study examined the overall extraction efficiency for all metals in numerous soil samples collected from a firing range. Results were examined for each metal to evaluate differences in extraction on a metal by metal basis. Duplicates were also examined in detail to better understand sources of variations.

A. Metal to Metal Variations

The percent metal extracted, as calculated using equation 1, varied for each metal. The source of this variation could be differences in the solubility of the different metals in the extraction fluid or may be related to the form and distribution of the metal in the sample. Table 2 shows the average results on a metal by metal basis. Figure 1 shows a graphical representation of the calculated average extractability for each metal. Also shown in Table 2 is the range of results and the number of measurements used to obtain the results. For samples which had undetectable levels of analytes, fewer values of percent extractability could be calculated (e.g., the value for As is based on one calculated result because all other As results were below the detection limit). For Hg and Se, analyte levels were all below the detection limits and no comparisons could be made. The data used to generate Table 2 and Fig. 1 are given in Appendix Tables A1 to A4. These tables include results that were below the detection limit and therefore not included in Table 2 or Fig. 1. A "u" following a result, indicates that the concentration of an analyte was below the instrument detection limit. These "u" values may vary based on sample size and slight variations in the instrument detection limit. For samples in which data were not collected on a particular metal, the cell has been left blank.

Two metals, Sb and Sn, were extracted in excess of 100%. These two metals are believed to be lost during the routine soil digestion using EPA Method 3050. This digestion uses HCl and the analytes may be lost as their respective chlorides during heating, a commonly observed phenomenon with this method. The digestion of the extraction fluids also uses HCl, however, the presence of the TCLP extraction fluid may reduce the loss of Sn and Sb. Other individual percent extracted values were also observed to exceed 100%. In spite of conscious efforts to take representative samples in each case, a large range of percent extractions for any analyte is most likely due to the heterogeneous nature of the samples, which will be discussed later in this paper.

In this study, extractions greater than 100% occurred for Sb, Cd, Pb, and Sn (see Table 2). Antimony and Sn, as explained earlier, can be lost during the digestion with HCl. Also, these metals are not required as part of the TCLP. Cadmium and Pb, which are required as part of the TCLP, showed one occurrence each (2 out of 50 samples) of percent extraction exceeding 100% (see Tables A2 and A3 respectively). These occurrences were probably caused by the heterogeneous nature of the samples. Both these points would be considered outliers at the 99.5% confidence level, using Grubb's Outlier Test [4].

We observed that the percent extraction by TCLP varied widely for all metals analyzed (Table 2 and Fig. 1). To further assess the relationship between the concentrations of metals extracted by the two methods, concentrations of metals extracted using the total determination method were plotted against the concentrations extracted by the TCLP (Figs. 2-16). Linear regressions were performed on the data to determine whether there was any correlation between the methods. A high value of r^2 (>0.90) indicates a correlation of the data. Graphs were not prepared for Sb and Sn because of the loss of these metals during the total determination method.

Also, graphs were not plotted for Se and Hg, for which all measurements were below the instrument detection limit. (Metal concentrations which fall below the instrument detection limit are reported at the instrument detection limit and therefore appear in these figures on the edge of the method detection limit regions.)

The relationship between the concentration of As extracted by TCLP and the total determination method is shown in Fig. 2. Most values were reported at the detection limit and no correlation can be established.

The relationship between the concentration of Ba extracted by TCLP and the total determination method is shown in Fig. 3. There is no correlation ($r^2 = 0.44$) between methods when the entire concentration range is considered. There does appear to be a correlation ($r^2 = 0.97$) for concentrations of Ba less than 80 mg/kg. However, it is unusual that as the concentration of Ba increases in this concentration range (based on the total determination method), the concentration of Ba extracted by TCLP decreases slightly. This may be due to the form of the Ba in the soil or some other competing chemical process in the TCLP, or may be a coincidence caused by the heterogeneous nature of the samples. More data are necessary to determine whether there is a relationship between the total determination method and the TCLP.

Figures 4 and 5 show the results for Cd. Again, there is no correlation ($r^2 = 0.62$) between methods when the entire concentration range is considered. However, for total Cd concentrations less than 25 mg/kg a correlation ($r^2 = 0.93$) can be made. The r^2 value increases to 0.99 if total Cd concentrations are restricted to those between 10 and 25 mg/kg. For the remaining metals, Cr, Cu, Pb, Mg, Ag, and Zn, no correlations were observed between the total determination method and the TCLP (Figs. 6-16).

Apart from the anomalous high percent extractions for Sb and Sn, Cd was found to be the most "extractable" metal. Although this may be due to the form of the cadmium in the sample, it has been generally observed in this laboratory that cadmium extracts better than the other elements typically determined with the TCLP.

B. Duplicates

Duplicate determinations are made to assess sample homogeneity. Depending on what is known about each sample aliquot and what parameters are compared, other types of information may also be obtained. Duplicate measurements were made and compared for the TCLP, for the total determination method and for samples with and without bullets (TCLP). These provided information on the sample aliquots used in these methods and helped to determine whether a potential source of variations could have been unnoticed bullets or fragments of bullets in the sample. Relative percent difference (RPD) was used to quantify duplicate variations. This was calculated using the following equation:

$$\% \text{ RPD} = \frac{|\text{Sample} - \text{Duplicate}|}{(\text{Sample} + \text{Duplicate}) / 2} \times 100 \quad (2)$$

The RPDs were not calculated if one or both of the measurements were at or below the instrument detection limit. If no RPDs were obtained for a given element, then the element was not included in the tables or figures.

Results of duplicate measurements are presented in three categories. First, the average, minimum, and maximum RPD for samples digested by EPA Method 3050 (total determination method) are shown in Table 3, and the average RPDs are illustrated graphically in Fig. 17 for each set of duplicate digested samples. Second, the average, minimum, and maximum RPD for samples extracted by the TCLP are shown in Table 4, and the average RPDs are illustrated graphically in Figs. 18 and 19 for each set of duplicates. Sample numbers in Figs. 18 and 19 are

for convenience only, and they do not correspond to the same numbers in Figs. 17 and 20.

Finally, the average, minimum, and maximum RPDs for TCLP samples are shown in Table 5, and the average RPDs are illustrated graphically in Fig. 20 for each set of duplicates. For the duplicates with a bullet two 50 g aliquots of the same soil sample were selected. A bullet was included as part of one of the 50 g aliquots, and a typical sampling, which contained no visible bullet, was used for the other. The TCLP did not totally dissolve the bullet.

The data used to generate the tables and figures on the duplicate results are given in Appendix Tables A5 to A9. In these tables the sample results are presented in one row with the duplicate results presented in the following row. The "D" following a sample number indicates that the sample is a duplicate. An "x" following a sample number indicates that a second analysis of that sample and duplicate was performed. This second measurement was treated as a completely separate sample. A "B" signifies that a visible bullet or bullet fragment was present in either the sample or the duplicate. A "u" following a result indicates that the concentration of analyte for that sample was below the instrument detection limit. These "u" values may vary based on sample size and slight variations in the instrument detection limit. The RPD for each element and each pair of samples is given in the column entitled "RPD". Results from these three sets of duplicates (for total determination method, TCLP extracts and TCLP extracts with a bullet) provide different types of information on sample homogeneity.

Several important points should be made with respect to the homogeneity of the sample and the differences between the total determination method and the TCLP extraction. The total determination method uses 1-2 g of sample and the TCLP extraction uses 50 g. Therefore, it is more difficult to get a representative sample for the total determination method. The TCLP extraction uses a weak acid which may cause variations in the amount extracted based on physical size of particles, degree of occlusion of metals or the potential for other competing

chemical reactions during the extraction process. The total determination method uses a strong acid which is less affected by particle size, degree of occlusion of metals or the potential for other competing chemical reactions. All of these factors can impact the reproducibility of the TCLP extraction and the comparison with the total determination method.

For the duplicates that were digested with EPA Method 3050, the average sample RPD was about 17%, and the standard deviation of the RPDs was 20%. None of the elements had consistently high RPDs. Copper had the largest RPD and Ba had the lowest (see Table 3). The RPDs for TCLP measurements averaged 20% and the standard deviation was 28%. It is not clear why duplicate RPDs for TCLP are larger and more variable than those for the total determination method. The 50 g sample aliquot used for the TCLP provides a more representative sample, thus RPDs should be lower. However, several differences in the procedures could effect duplicate RPDs. A possible explanation for the higher RPDs seen with TCLP could be bias in selecting the sample aliquot. In collecting a 50 g sample aliquot, the analyst is able to include larger items (such as bullet fragments) that might not be included in a smaller sample. Another possible reason for the higher RPDs is that the TCLP is more affected by both the chemical and physical form of the metals to be extracted. A given sample may contain metals in various chemical and physical forms, which is generally not a problem with the aggressive total determination method, but will effect the TCLP. This latter theory might be consistent with a mixture of metal compounds and various sized metal particles (e.g., bullet fragments) in the soil.

Assuming that the main source of the lack of reproducibility is bullet fragments, one might expect to find larger RPDs for the metals contained in the bullet (e.g., Pb and Cu). The largest difference was seen for Pb, which is consistent with the expected bullet composition. Other elements which were extracted from the sample with the bullet include Cu, Mg, Sb, and Zn.

Variations in the RPDs might be explained by the way the bullet fragments, or by the types of bullets used in a given area. For example if certain types of bullets tended to fragment (e.g. bullets with more copper) and others did not, then we would be likely to find more bullet duplicates with less copper and more copper fragments in the duplicate total determination and TCLP tests. Copper in particular showed larger variations in RPDs. That is, in some instances the RPDs were low for copper duplicates and sometimes they were high (see Appendix Tables A5 to A9). This was true for all duplicates (total metals, TCLP, and TCLP with bullets). The reason for this is believed to be the scattered use of copper coated bullets on various firing ranges.

IV. CONCLUSIONS

This study compares the amount of metal measured in a soil sample using a total determination method with the amount extracted by the TCLP. Because the TCLP is more time consuming and therefore more expensive than a total determination method, it was hoped that a correlation could be established between the two procedures. Some correlation was observed for Ba and Cd at lower concentrations. This could mean samples are more homogenous with respect to these metals at lower concentrations. However, in general, good correlations between the total metal concentration and the amount extracted in the TCLP were not observed. The lack of correlation may be due to the heterogeneous nature of the samples (mostly because of bullet fragments). This was suggested by high RPDs in the analysis of duplicates for total digestates, TCLP extracts and TCLP extracts with bullets. The samples showed the largest RPDs on duplicate results for lead and copper, which are the major metals in bullets. Ultimately, the lack of correlation means that the extraction of metals by the TCLP cannot be estimated from the total determination method when results are above regulatory levels.

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Table 1. Detection Limits in Soil (mg/kg)

Element	Total Metals (ICP)	TCLP (ICP)	TCLP (AA) ^a
Sb	20	2	0.5
As	20	2	1
Ba	0.6	0.06	0.5
Cd	1	0.1	0.01
Cr	0.4	0.04	0.2
Cu	1	0.1	0.1
Pb	10	1	0.5
Mg	6	0.6	0.05
Hg	0.04 ^b	^b	1.4 ^b
Se	30	3	0.5
Ag	0.6	0.06	0.05
Sn	20	2	1
Zn	0.4	0.04	0.1

^a Flame AA was performed using an air/acetylene flame except for As which used a nitrous oxide/acetylene flame.

^b Hg was determined by cold vapor atomic absorption spectrophotometry.

Table 2. Percent Extracted by TCLP

		Range		N
		Average	Min	
Sb	205%	2%	904%	10
As	12%	12%	12%	1
Ba	25%	12%	51%	9
Cd	46%	1%	156%	17
Cr	4%	1%	15%	16
Cu	6%	0%	29%	29
Pb	26%	4%	150%	33
Mg	6%	0%	26%	32
Ag	8%	3%	15%	6
Sn	1538%	293%	2831%	3
Zn	16%	1%	96%	32

N = the number of comparisons

Table 3. Duplicates for Total Determination Method

		Range		N
		Average	Min	
Sb	16%	4%	36%	4
As	18%	6%	30%	2
Ba	5%	0%	17%	7
Cr	9%	2%	20%	7
Cu	32%	2%	95%	7
Pb	20%	1%	54%	7
Mg	14%	0%	56%	7
Zn	14%	0%	39%	7

N = the number of comparisons

Table 4. Duplicates Extracted by TCLP

		Range		N
		Average	Min	
Sb		41%	0%	13
Cd		14%	0%	6
Cu		16%	0%	13
Pb		26%	1%	10
Mg		10%	0%	14
Sn		5%	0%	12
Zn		26%	0%	14

N = the number of comparisons

Table 5. Duplicates with Bullets Extracted by TCLP

		Range		N
		Average	Min	
Sb		116%	0%	5
Cu		84%	2%	5
Pb		143%	40%	5
Mg		45%	3%	5
Sn		2%	0%	5
Zn		83%	9%	5

N = the number of comparisons

Figure 1. Extraction of Metals by TCLP

Figure 2. TCLP vs. Total Determination Method for As

Figure 3. TCLP vs. Total Determination Method for Ba

Figure 4. TCLP vs. Total Determination Method for Cd

Figure 5. TCLP vs. Total Determination Method for Cd (Insert from Figure 4)

Figure 6. TCLP vs. Total Determination Method for Cr

Figure 7. TCLP vs. Total Determination Method for Cu

Figure 8. TCLP vs. Total Determination Method for Cu (Insert from Figure 7)

Figure 9. TCLP vs. Total Determination Method for Cu (Insert from Figure 8)

Figure 10. TCLP vs. Total Determination Method for Pb

Figure 11. TCLP vs. Total Determination Method for Pb (Insert for Figure 10)

Figure 12. TCLP vs. Total Determination Method for Pb (Insert for Figure 11)

Figure 13. TCLP vs. Total Determination Method for Mg

Figure 14. TCLP vs. Total Determination Method for Mg (Insert for Figure 13)

Figure 15. TCLP vs. Total Determination Method for Ag

Figure 16. TCLP vs. Total Determination Method for Zn

Figure 17. Duplicate Results for Total Determination Method

Figure 18. Duplicate Results for TCLP (Samples 1 through 7)

Figure 19. Duplicate Results for TCLP (Samples 8 through 14)

Figure 20. Duplicate Results for TCLP with Visible Bullet

APPENDIX A
RAW DATA

Table A1. TCLP Extraction Efficiency for Sb, As, and Ba

SAMPLE	Sb (mg/kg)		% Extr.	As (mg/kg)		% Extr.	Ba (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R111-S037	23.2			17.8u	1.96		97.3	23.4	24%
GTA-R111-S039	21.4u			18.3u	4.36		94.8	33.8	36%
GTA-R111-S049	20.5u			17.5u	1.84u		61.0	10.7	18%
GTA-R122-S016	21.6u			18.5u	1.84u		63.2	10.4	16%
GTA-R122-S022	21.2u			18.1u	1.84u		83.4	19.5	23%
GTA-R122-S029	21.2u			18.1u	1.84u		71.6	8.82	12%
GTA-R124-S003	64.6			19.4	2.26	12%	51.8	12.2	24%
GTA-R124-S007	70.4			18.1u	2.4		57.3	10.6	18%
GTA-R124-S014	21.4u			18.2u	1.84u		28.9	14.7	51%
GTA-R111-S037	23.2	26.1	113%	17.8u			97.3		
GTA-R111-S038	36.6	38.0	104%	18.1u			115		
GTA-R111-S039	21.4u	30.1		18.3u			94.8		
GTA-R111-S041	21.8	12.9	59%	22.6			164		
GTA-R111-S044	20.9u	8.82		17.9u			92.0		
GTA-R111-S049	20.5u	0.68		17.5u			61.0		
GTA-R122-S016	21.6u	8.82		18.5u			63.2		
GTA-R122-S017	21.5u	0.68		18.3u			63.3		
GTA-R122-S022	21.2u	8.82		18.1u			83.4		
GTA-R122-S026	21.4u	4.07		17.7u			48.6		
GTA-R122-S029	21.2u	0.5u		18.1u			71.6		
GTA-R122-S030	22.0u	42.0		18.8u			97.9		
GTA-R124-S003	64.6	45.4	70%	19.4			51.8		

Table A1. TCLP Extraction Efficiency for Sb, As, and Ba (cont'd.)

SAMPLE	Sb (mg/kg)		% Extr.	As (mg/kg)		% Extr.	Ba (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R124-S004	1350	21.0	2%	145			37.1		
GTA-R124-S007	70.4	49.5	70%	18.1u			57.3		
GTA-R124-S009	111	61.7	56%	17.1u			53.7		
GTA-R124-S014	21.4u	10.3		18.2u			28.9		
GTA-R124-S015	20.9u	4.75		17.9u			34.4		
R111-01.0.01015	11.2u	33.0		19.1u			73.1		
R111-02.0.010010	11.2u	16.3		18.7u			37.8		
R111-03.0.218	19.9	51.5	259%	23.2			128		
R111-03.0.229	11.2u	12.8		19u			29.3		
R111-03.1.218	11.1u	48.0		18.8u			89.0		
R111-03.1.220	12.3	111	904%	18.6u			64.8		
R111-04.0.201	11u	2.50		18.7u			40.0		
R111-04.0.209	11.2u	44		19u			86.2		
R111-04.1.205	11.5	47	409%	19u			142		
R111-05.0.01012	11.2u	0.5u		19.1u			27.0		
R111-06.0.229	10.9u	11.5		18.6u			19.9		
R111-08.0.232	11.3u	0.5u		19.1u			20.5		
R122-08.2.529	11.3u	16.4		19.1u			70.5		
R122-08.2.529D	11.3u	10.8		19.1u			72.7		

u = below detection limit

* = total determination method

Table A2. TCLP Extraction Efficiency for Cd, Cr, and Cu

SAMPLE	Cd (mg/kg)		% Extr.	Cr (mg/kg)		% Extr.	Cu (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R111-S037	31.1	16.3	52%	34.8	0.66	2%	276		
GTA-R111-S039	94.6	69.8	74%	27.7	1.02	4%	1560		
GTA-R111-S049	0.95u	0.42		16.3	0.14u		11.0		
GTA-R122-S016	0.99 u	0.12		7.90	0.16	2%	146		
GTA-R122-S022	0.97 u	0.1u		3.00	0.14u		57.0		
GTA-R122-S029	0.97 u	0.1u		5.50	0.14u		23.7		
GTA-R124-S003	1.2	0.56	47%	7.50	0.14u		271		
GTA-R124-S007	0.97 u	0.72		7.20	0.16	2%	6420		
GTA-R124-S014	0.98 u	0.3		6.60	0.62	9%	29.9		
GTA-R111-S037	31.1	21.68	70%	34.8	0.43	1%	276	18.9	7%
GTA-R111-S038	32.3	25.72	80%	41.8	0.43	1%	370	25.4	7%
GTA-R111-S039	94.6	79.98	85%	27.7	0.43	2%	1560	73.4	5%
GTA-R111-S041	32.5	50.75	156%	44.4	0.2u		364	14.6	4%
GTA-R111-S044	73.6	17.26	23%	25.8	0.2u		322	7.68	2%
GTA-R111-S049	0.95u	0.38		16.3	0.2u		11.0	0.75	7%
GTA-R122-S016	0.99 u	0.01u		7.90	0.2u		146	8.42	6%
GTA-R122-S017	0.98 u	0.01u		4.60	0.2u		28.6	0.75	3%
GTA-R122-S022	0.97 u	0.01u		3.00	0.2u		57.0	7.68	13%
GTA-R122-S026	0.95 u	0.01u		3.50	0.2u		51.1	4.46	9%
GTA-R122-S029	0.97 u	0.01u		5.50	0.2u		23.7	0.25	1%
GTA-R122-S030	1.00 u	1.61		39.2	0.2u		32.6	1.2	4%
GTA-R124-S003	1.2	0.38	32%	7.50	0.2u		271	35.9	13%

Table A2. TCLP Extraction Efficiency for Cd, Cr, and Cu (cont'd.)

SAMPLE	Cd (mg/kg)		% Extr.	Cr (mg/kg)		% Extr.	Cu (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R124-S004	7.6	0.38	5%	5.60	0.2u		798	96.93	12%
GTA-R124-S007	.97 u	0.01u		7.20	0.2u		6420	80.22	1%
GTA-R124-S009	2.1	0.03	1%	10.1	0.2u		13200	72.55	1%
GTA-R124-S014	0.98 u	1.25		6.60	0.2u		29.9	8.76	29%
GTA-R124-S015	0.96 u	0.01u		6.00	0.2u		35.0	3.22	9%
R111-01.0.01015	6	1.70	28%	22.3	1	4%	65.8	1	2%
R111-02.0.010010	0.87u	0.28		7.20	0.4	6%	11.0	0.28	3%
R111-03.0.218	18.9	5.51	29%	88.3	0.2u		199	3.88	2%
R111-03.0.229	0.87u	0.36		7.80	0.2u		6.60	0.16	2%
R111-03.1.218	12.2	3.46	28%	32.7	0.4	1%	388	5.62	1%
R111-03.1.220	10.2	2.61	26%	57.8	3	5%	586	1.97	0%
R111-04.0.201	0.86u	0.48		10.5	1	10%	16.4	0.42	3%
R111-04.0.209	96.1	23	24%	27.8	0.6	2%	288	5	2%
R111-04.1.205	22.8	6.38	28%	30.5	0.3	1%	264	7.55	3%
R111-05.0.01012	0.87u	0.12		6.80	1	15%	1.60	0.18	11%
R111-06.0.229	0.85u	0.10		5.30	0.2u		8.20	0.81	10%
R111-08.0.232	0.88u	0.02		8.10	0.2u		4.00	0.1u	
R122-08.2.529	0.88u	0.01u		6.10	0.2u		17.5	0.1u	
R122-08.2.529D	0.88u	0.01u		6.00	0.2u		48.9	0.1u	

u = below detection limit

* = total determination method

Table A3. TCLP Extraction Efficiency for Pb, Mg, and Se

SAMPLE	Pb (mg/kg)		% Extr.	Mg (mg/kg)		% Extr.	Se (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R111-S037	112	5	4%	8070			28.2u	2.9u	
GTA-R111-S039	90.6	10.5	12%	3510			28.9u	2.9u	
GTA-R111-S049	26	1.18u		1640			27.7u	2.9u	
GTA-R122-S016	398	72.4	18%	638			29.2u	2.9u	
GTA-R122-S022	123	38.4	31%	258			28.7u	2.9u	
GTA-R122-S029	31.6	1.18u		416			28.6u	2.9u	
GTA-R124-S003	7870	3880	49%	699			27.6u	2.9u	
GTA-R124-S007	4800	1068	22%	904			28.7u	2.9u	
GTA-R124-S014	1800	754	42%	839			28.9u	2.9u	
GTA-R111-S037	112	12.4	11%	8070	292	4%	28.2u		
GTA-R111-S038	152	15.0	10%	89300	294	0%	44.8		
GTA-R111-S039	90.6	15.0	17%	3510	294	8%	28.9u		
GTA-R111-S041	70.1	2.51	4%	4080	160	4%	28.7u		
GTA-R111-S044	70.7	3.77	5%	3870	133	3%	28.3u		
GTA-R111-S049	26	0.5u		1640	17	1%	27.7u		
GTA-R122-S016	398	37.7	9%	638	5	1%	29.2u		
GTA-R122-S017	41.2	0.5u		356	18	5%	29.0u		
GTA-R122-S022	123	20.2	16%	258	18	7%	28.7u		
GTA-R122-S026	55	0.5u		288	2	1%	28.1u		
GTA-R122-S029	31.6	0.5u		416	22	5%	28.6u		
GTA-R122-S030	314	11.2	4%	6260	387	6%	29.7u		
GTA-R124-S003	7870	4440	56%	699	88	13%	27.6u		

Table A3. TCLP Extraction Efficiency for Pb, Mg, and Se (cont'd.)

SAMPLE	Pb (mg/kg)		% Extr.	Mg (mg/kg)		% Extr.	Se (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R124-S004	126000	13553	11%	512	63	12%	61.9		
GTA-R124-S007	4800	7212	150%	904	45	5%	28.7u		
GTA-R124-S009	13600	9010	66%	724	65	9%	27.1u		
GTA-R124-S014	1800	1023	57%	839	220	26%	28.9u		
GTA-R124-S015	684	254	37%	404	16	4%	28.3u		
R111-01.0.01015	81.2	9.12	11%	11600	137	1%	27.4u		
R111-02.0.010010	22.2	5.32	24%	2140	525	25%	27.5u		
R111-03.0.218	92	11.1	12%	14100	110	1%	27.1u		
R111-03.0.229	15.2u	6.11		776	33	4%	27.5u		
R111-03.1.218	68.1	10.4	15%	4950	147	3%	27.2u		
R111-03.1.220	56.9	17.6	31%	9500	327	3%	26.9u		
R111-04.0.201	40.7	4.07	10%	832	60	7%	27.1u		
R111-04.0.209	104	9.6	9%	3690	136	4%	27.6u		
R111-04.1.205	85.4	11.7	14%	3650	96	3%	27.6u		
R111-05.0.01012	15.2u	1.27		465	2	0%	27.6u		
R111-06.0.229	25	5.20	21%	310	29	9%	26.9u		
R111-08.0.232	29.4	3.23	11%	470	34	7%	27.7u		
R122-08.2.529	35.9	10.7	30%	501	26	5%	27.7u		
R122-08.2.529D	34.7	12.0	35%	473	28	6%	27.7u		

u = below detection limit

* = total determination method

Table A4. TCLP Extraction Efficiency for Ag, Sn, and Zn

SAMPLE	Ag (mg/kg)		% Extr.	Sn (mg/kg)		% Extr.	Zn (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R111-S037	5.9	0.24	4%	21.7u			2120		
GTA-R111-S039	7.6	0.36	5%	23.4			691		
GTA-R111-S049	3.5	.06u		21.3u			48.6		
GTA-R122-S016	2	.06u		22.5u			48.2		
GTA-R122-S022	0.8	0.1	13%	22.1u			23.8		
GTA-R122-S029	1.1	.06u		22.1u			23		
GTA-R124-S003	1.8	0.14	8%	21.2			77.6		
GTA-R124-S007	6.8	0.2	3%	22.1u			700		
GTA-R124-S014	2	0.3	15%	22.2u			105		
GTA-R111-S037	5.9			21.7u	715.77		2120	975	46%
GTA-R111-S038	7.4			22.1u	742.44		5680	1523	27%
GTA-R111-S039	7.6			23.4	662.42	2831%	691	662	96%
GTA-R111-S041	5.6			22.1u	356.4		976	193	20%
GTA-R111-S044	5.8			21.8u	190		703	400	57%
GTA-R111-S049	3.5			21.3u	2.6u		48.6	1.68	3%
GTA-R122-S016	2			22.5u	182		48.2	5.19	11%
GTA-R122-S017	0.83			22.3u	164		26	0.31	1%
GTA-R122-S022	0.8			22.1u	34.8		23.8	2.75	12%
GTA-R122-S026	0.66u			21.6u	15.3		30.2	2.60	9%
GTA-R122-S029	1.1			22.1u	93.3		23	0.15	1%
GTA-R122-S030	7.2			22.9u	671		72.7	1.04	1%
GTA-R124-S003	1.8			21.2	315	1489%	77.6	13.7	18%

Table A4. TCLP Extraction Efficiency for Ag, Sn, and Zn (cont'd.)

SAMPLE	Ag (mg/kg)		% Extr.	Sn (mg/kg)		% Extr.	Zn (mg/kg)		% Extr.
	Total*	TCLP		Total*	TCLP		Total*	TCLP	
GTA-R124-S004	6.1			71.3	208	293%	125	23.6	19%
GTA-R124-S007	6.8			22.1u	155		700	14.7	2%
GTA-R124-S009	14.2			20.9u	262		1350	16.8	1%
GTA-R124-S014	2			22.2u	573		105	8.57	8%
GTA-R124-S015	2.1			21.8u	57.8		39.6	1.07	3%
R111-01.0.01015				17.7u	2.6u		2070	397	19%
R111-02.0.010010				17.8u	19.8		56.2	21.6	39%
R111-03.0.218				17.5u	30.7		1020	219	22%
R111-03.0.229				17.8u	7.67		24.4	2.62	11%
R111-03.1.218				17.6u	30.5		865	139	16%
R111-03.1.220				17.4u	65.7		791	164	21%
R111-04.0.201				17.5u	14.1		46.8	4.37	9%
R111-04.0.209				17.8u	24		558	42.5	8%
R111-04.1.205				17.8u	32.5		1600	299	19%
R111-05.0.01012				17.8u	39.0		24.2	0.56	2%
R111-06.0.229				17.4u	6.88		28.2	1.41	5%
R111-08.0.232				17.9u	3.82		25.1	0.23	1%
R122-08.2.529				17.9u	18.6		21.6	0.38	2%
R122-08.2.529D				17.9u	20.3		21	0.57	3%

u = below detection limit

* = total determination method

Table A5. Duplicate Results for Total Determination Method

SAMPLE	Sb	RPD	As	RPD	Ba	RPD	Cr	RPD	Cu	RPD
GTA-R124-S015	20.9u		17.9u		34.4	1%	6	7%	35	6%
GTA-R124-S015D	20.9u		17.9u		34.6		5.6		33	
R124-04.0.352x	98.0	4%	35.9	30%	839	4%	20.9	6%	1070	2%
R124-04.0.352Dx	102		26.4		808		22.3		1050	
R124-07.0.386x	117	36%	35.6	6%	49.8	7%	6.7	7%	330	21%
R124-07.0.386Dx	169		33.4		46.4		7.2		406	
R122-08.2.529	11.3u		19.1u		70.5	3%	6.1	2%	17.5	95%
R122-08.2.529D	11.3u		19.1u		72.7		6		48.9	
R111-02.0.010010	11.2u		18.7u		37.8	5%	7.2	6%	11	18%
R111-02.0.010010D	11.2u		18.9u		36		6.8		9.2	
R124-04.0.352	67.9	15%	19.3u		848	17%	27	14%	1060	17%
R124-04.0.352D	58.6		19.3u		717		23.4		892	
R124-07.0.386	83.3	10%	19.4u		51.2	0%	9	20%	639	68%
R124-07.0.386D	92.3		19.5u		51		11		313	

u = below detection limit

Table A6. Duplicate Results for Total Determination Method

SAMPLE	Pb	RPD	Mg	RPD	Ag	RPD	Zn	RPD
GTA-R124-S015	684	1%	404	1%	2.1	47%	39.6	0%
GTA-R124-S015D	691		408		1.3		39.6	
R124-04.0.352x	2570	54%	3550	0%			233	0%
R124-04.0.352Dx	1470		3540				233	
R124-07.0.386x	9260	48%	848	4%			81.4	14%
R124-07.0.386Dx	15100		887				94	
R122-08.2.529	35.9	3%	501	6%			21.6	3%
R122-08.2.529D	34.7		473				21	
R111-02.0.010010	22.2	23%	2140	12%			56.2	27%
R111-02.0.010010D	17.7		1900				42.9	
R124-04.0.352	1310	8%	3810	19%			230	16%
R124-04.0.352D	1420		3160				195	
R124-07.0.386	8900	3%	908	56%			112	39%
R124-07.0.386D	8620		1620				75.6	

u = below detection limit

Table A7. Duplicate Results for TCLP

SAMPLE	Sb	RPD	Cd	RPD	Cu	RPD	Pb	RPD
GTA-R124-S005	49.5	98%	0.03		44.6	7%	7330	70%
GTA-R124-S005D	17.0		0.38		41.4		3532	
GTA-R124-S008	33.2	12%	0.01u		67.8	1%	4985	1%
GTA-R124-S008D	37.3		0.01u		67.4		5024	
GTA-R124-S010	33.2	0%	0.03		66.8	20%	7811	6%
GTA-R124-S010D	33.2		0.01u		81.3		7365	
GTA-R124-S013	25.1	39%	0.38		8.17	64%	1051	13%
GTA-R124-S013D	17.0		0.03		15.8		1193	
GTA-R122-S021	0.68	0%	0.01u		9.66	40%	254	122%
GTA-R122-S021D	0.68		0.01u		6.44		61.7	
GTA-R122-S024	8.82	60%	0.01u		42.1	2%	350	5%
GTA-R122-S024D	4.75		0.01u		43.1		332	
GTA-R122-S027	4.75	0%	0.01u		1.49	0%	0.5u	
GTA-R122-S027D	4.75		0.01u		1.49		0.5u	
GTA-R111-S040	30.1	33%	41.92	2%	37.4	10%	22.8	11%
GTA-R111-S040D	42.0		42.7		33.8		25.5	
GTA-R111-S045	17.0	27%	6.3	6%	7.7	20%	0.5u	
GTA-R111-S045D	12.9		5.92		9.42		0.5u	

Table A7. Duplicate Results for TCLP (cont'd.)

SAMPLE	Sb	RPD	Cd	RPD	Cu	RPD	Pb	RPD
GTA-R111-S046	17.0	112%	14.6	0%	4.21	27%	0.5u	
GTA-R111-S046D	4.75		14.6		3.22		0.5u	
GTA-R111-S048	0.68		2.46	54%	0.99	0%	0.5u	
GTA-R111-S048D	0.5u		1.42		0.99		0.5u	
R111-02.0.01005	46.7	23%	48.2	10%	9.57	11%	11.0	18%
R111-02.0.01005D	37.1		53.5		8.60		9.14	
R111-03.0.222	105	8%	39.16	13%	31.49	0%	19.6	4%
R111-03.0.222D	97.5		44.7		31.4		20.4	
R122-08.2.529	16.4	42%	0.01u		0.1u		10.71	12%
R122-08.2.529D	10.8		0.01u		0.1u		12.03	

Table A8. Duplicate Results for TCLP

SAMPLE	Mg	RPD	Sn	RPD	Zn	RPD
GTA-R124-S005	52.44	1%	200	0%	9.62	7%
GTA-R124-S005D	53.11		200		9.01	
GTA-R124-S008	38.79	17%	155	16%	14.1	0%
GTA-R124-S008D	45.8		182		14.1	
GTA-R124-S010	42.1	12%	173	0%	15.0	10%
GTA-R124-S010D	37.5		173		13.6	
GTA-R124-S013	118	1%	377	2%	5.8	17%
GTA-R124-S013D	117		369		6.87	
GTA-R122-S021	3.44	9%	2.6u		2.6	6%
GTA-R122-S021D	3.77		5.61		2.44	
GTA-R122-S024	6.35	47%	73.8	12%	7.94	2%
GTA-R122-S024D	10.27		83.6		8.09	
GTA-R122-S027	2.6	0%	25.1	0%	1.38	25%
GTA-R122-S027D	2.6		25.1		1.07	
GTA-R111-S040	286	2%	697	7%	220	5%
GTA-R111-S040D	292		751		209	
GTA-R111-S045	144	1%	239	0%	349	25%
GTA-R111-S045D	143		239		449	

Table A8. Duplicate Results for TCLP (cont'd.)

SAMPLE	Mg	RPD	Sn	RPD	Zn	RPD
GTA-R111-S046	945	0%	191	5%	138	53%
GTA-R111-S046D	941		181		79.68	
GTA-R111-S048	107	10%	2.6u		6.41	30%
GTA-R111-S048D	96.43		2.6u		4.73	
R111-02.0.01005	126	26%	32.6	8%	372	142%
R111-02.0.01005D	164		30.1		62.8	
R111-03.0.222	162	5%	52.6	3%	801	7%
R111-03.0.222D	155		54.3		860	
R122-08.2.529	25.7	9%	18.6	9%	0.37	40%
R122-08.2.529D	28.2		20.28		0.57	

Table A9. Duplicate Results with Bullets

SAMPLE	Sn	RPD	Cu	RPD	Mg	RPD	Sb	RPD	Zn	RPD	Pb	RPD
GTA-R124-S012	164	6%	61.9	2%	43.4	6%	41.4	0%	11.9	9%	6353	40%
GTA-R124-S012B	155		63.1		40.8		41.4		13.0		9518	
GTA-R122-S016	182	5%	8.42	166%	5.03	158%	8.82	107%	5.19	125%	37.7	194%
GTA-R122-S016B	191		89.7		43.5		29.2		22.6		2424	
GTA-R122-S020	5.61	0%	116	8%	7.16	27%	4.75	159%	19.4	43%	1778	92%
GTA-R122-S020B	5.61		107		5.46		41.4		30.1		4829	
GTA-R122-S024	73.8	0%	42.1	57%	6.35	32%	8.82	169%	7.94	105%	350	189%
GTA-R122-S024B	73.8		75.8		4.6		106		25.6		12175	
GTA-R122-S026	15.3	0%	4.46	187%	2.07	3%	4.07	144%	2.6	134%	0.5u	200%
GTA-R122-S026B	15.3		135.9		2		25.1		13.1		3054	

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