# Leaching of Lead and Other Elements from Portable Electronics, Part II

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**Abstract**
Portable electronic device circuit packs were ground up to more than meet EPA sieving criteria and were then subjected to the well known EPA Method 1311 leaching protocol. Based on work from the previous study, mainly Fe, Ni, Cu, Zn, and Pb were investigated. Leachate was also separately treated with egg whites and cardboard and the dissolved metal concentrations were found to be significantly reduced. Individual spikes of 500mg Fe, Ni, Cu, Zn, and Pb (artificial leachate) were run through columns of topsoil, sand, gravel, vermiculite and perlite to compare to the earlier work using topsoil, in a continuing effort to show what might happen if leachate were to escape from a landfill.

**Introduction**
Our previous work[1] dealt with looking at grinding efficiencies for printed circuit packs (PCBs), the analysis of the TCLP leachates, the effect of carbonate and sulfide and the capacity of topsoil to take up such leachates. One portion of the present work looks at two representative materials that would be in municipal solid waste (MSW) and their effect of heavy metal laden leachate. The materials chosen were eggs, specifically the whites, and cardboard. These materials were not chosen at random. Paper products make up 40 - 50% of the waste stream[2] and although the amount of cardboard is being reduced as more and more is being recycled, it nevertheless still makes up a large proportion of MSW, even more so on a volume or surface area basis.

Although the proportion of eggs in a land fill would be vanishingly small on a percentage basis, some will be there as a result of portions sticking to egg shells, eggs thrown out because they were rotten, some accidentally broken and portions scraped from plates into the garbage. And like cardboard eggs also have a reasonably high level of sulfur content[3]. Previously it was shown that sulfide has a significant effect on the solubility of the heavy metals extracted by the EPA leachate.

Sand and gravel are other materials surely to be found around some landfills, so they were chosen as "soil" materials to examine for their ion exchange capacity. Vermiculite and perlite were chosen more for interest. As purchased by the consumer, both of these materials are not in their natural form. Perlite[4] is an artificially created frothy glass, similar to natural pumice. The flash evaporation of the water in the rock known as pearlstone is heated to its softening point (1500 – 200C) results in a light, porous material with a large surface area. Its low density makes it ideal as a filler in plasters and concretes and for use by the home gardener. Vermiculite[5] in its raw form is a hydrated magnesium aluminosilicate which is exfoliated by heating before been sold for retail purchase. Its ion exchange ability is retarded by removing the waters of hydration. This work will show how much ability it still has to hold metal ions from artificial leachate.

This paper again uses the EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP)[6] as the testing basis for the leaching studies. This is the most common form of leaching test currently used in North America. Several individuals have expressed concern about this test[7, 8]. The reason for this lies in the extreme solubility of lead acetate, as opposed to more common salts of lead. See Table 1[9-11]. Note that this table corrects some unusual values listed in the equivalent table of reference one. It is important to keep in mind that this test is to determine the absolute worst case for leachate formation, NOT to determine what actually leaves the landfill as leachate. The concern that has been raised about this test is that this very important distinction is not always made, especially by those that read report data and have no chemical background.

In the present work the protocols called out in the EPA method have been adhered to for the testing. However, the samples for this study were ground to small particles and powders.

The samples in this study were analysed for the following elements: silver, barium, copper, iron, nickel, zinc and lead. The latter five were the individual elements found to have the highest concentrations in the previous study. Lead was an obvious choice, as this element and its compounds are singled out in the RoHS directive[10, 11] to essentially be eliminated from electronics covered by the directive. For classes of electronics and specific cases where their use will at least be allowed for a while, see the directive. The other elements are heavy metals of varying concern.

Silver and copper was chosen for two reasons – they are principle elements in the new Pb-free solders, commonly referred to as SAC alloys and there is also enough economic worth in printed circuit assemblies (PCAs) to consider their recovery. Zinc is included in some specific Pb-free solders. Iron and nickel are common materials in lead frame materials and nickel is of course used in ENIG surface finishes. Some concern has been expressed over nickel and barium.

**Table 1 - Solubility of Some Lead Salts**

(Superscripts are degrees Celsius)

| | g/100 ml | mg/L |
|---|---|---|
| **Lead acetate** | **$221^{50}$** | **2,210,000** |
| **$44.3^{20}$** | **443,000** | |
| **Lead chloride** | **$1.08^{25}$** | **10,800** |
| **Lead sulfate** | **$0.0044^{25}$** | **44** |
| **Lead hydroxide** | **$0.00012^{20}$** | **1.2** |
| **Lead carbonate** | **0.0001** | **1** |
| **Lead phosphate** | **$0.000014^{25}$** | **0.14** |
| **Lead sulfide** | **"insoluble"** | |

**Experimental**

Samples used for this study were taken from whole portable electronic units, different models from the previous study, but of the same general type and concentration of metals. They were soldered with Sn36Pb2Ag solder. The plastic casings, the LCD modules and RF shields were not subjected to testing.

As already mentioned in the Introduction, the analytical protocol used is EPA method 1311, TCLP. Samples were cut to EPA specified size using a cross-sectioning cut-off saw. As per 1311 instructions, initial work showed that extraction fluid # 1 was necessary (acetic acid/NaOH/ASTM Type II water).

The grinding in this study was performed with an IKA M20 universal grinder with a tungsten carbide blade. The grinding time was 20 minutes. The ground boards were then sieved through a 9.5 mm (3/8 in.) mesh sieve (as per 1311) and then were also sieved using 500 µm (#35), and 180 µm (#80) sieves. The portions that fell through the #35 mesh were combined and used for this study.

Each sample plus the requisite amount of EPA leaching solution were put in unused, clean plastic bottles. These were carefully closed with Teflon tape being used on the bottle threads. The bottles were then clamped and taped into a tumbler[12] capable of holding up to 12 bottles simultaneously. The tumbler mixed the samples end-over-end at 30 rpm for 18 hours as per the EPA method.

Each of the treated leachate portions were then filtered using the EPA required filter matt and then each portion was divided into 4 parts.

The first part of the leachate for each board was analyzed using ICP.

Twenty grams of egg white was added to an additional 40 ml part of the leachate for each board and was mixed for 5 min in a blade mixer. This mixture was then divided into two parts. The first part was filtered through a TCLP glass fiber filter (0.7 um) and the extract was analyzed by ICP. The second part of the mixture was centrifuged for 15 min using an International Equipment Company; Model CL centrifuge run at a setting of 5000 rpm and the extract was analyzed by ICP.

Twenty grams of cardboard was added to another 40 ml part of the leachate for each board and was mixed for 5 min in a blade mixer. This mixture was then divided into two parts. The first part was, as above, filtered through a TCLP glass fiber filter (0.7 um) and the extract was analyzed by ICP. The second part of the mixture was centrifuged for 15 min and the extract was analyzed by ICP.

The fourth part of the leachate for each board was itself used like a leaching solution for leaching soil. The soil: leachate proportion of 1:20 was used for each board and was tumbler mixed for 18 hours (according to EPA method). The "Method blank" was EPA leaching solution #1 mixed with soil in the same proportion 1:20. Then, each leachate was filtered using the EPA required filter matt and analyzed by ICP.

A separate set of experiments was also conducted. Twenty chromatographic columns were prepared using topsoil, gravel, sand, vermiculite and perlite four for each soil type. All of the twenty columns were then each spiked with 500ug of the elements Fe, Ag, Cu, Ba, Zn, Ni and Pb in 5 ml of 2% $HNO_3$.

Seven separate portions of 20 ml of Milli-Q water were percolated through each of the first set of ten columns, two columns for each soil type. Each portion of the eluant was collected and analyzed separately by ICP. For the second set of ten columns, seven 20ml portions of EPA leaching solution #1 were used for each column instead. Every 20 ml portion of the eluants collected was also analyzed separately by ICP.

Analysis for the metals was conducted with a Perkin Elmer Optima 3000 DV ICP-OES unit (Inductively Coupled Plasma – Optical Emission Spectrometer). The ICP-OES was calibrated for each element and calibrations were checked by verification standards for quality control purposes. Recovery of the verification standard for each element was between 90% and 110%. Method Detection Limits are shown in Table 2.

**Table 2- Metal Analysis Method Detection Limits for Optima 3000 DV ICP**

| Element | MDL (ppm) |
|---|---|
| Silver (Ag) | 0.003 |
| Copper (Cu) | 0.004 |
| Iron (Fe) | 0.002 |
| Nickel (Ni) | 0.003 |
| Lead (Pb) | 0.007 |
| Zinc (Zn) | 0.002 |
| Barium (Ba) | NA |

**Results and Discussion**

***First part of the circuit board leachate***

Two PCBAs from different but identical units were ground up as described in the experimental section. The results are shown in Figure 1 and Table 3. As can be seen, the agreement in the results between the two PCAs is quite good. Only for the iron results and for the copper results for material through the #80 mesh screen are the results different by more than 2 ppm.

When comparing the results for the material captured by the screens and what went through, only nickel has values that are even of the same order of magnitude. Iron, the hardest of the elements measured, was the only one retained more on the screens than passed through.

A comparison of the materials that went through the #80 mesh from the last study and from this study is shown in Table 4 and Figure 2. The data from the previous study is the average for three samples, including one where there was a different color to the solution due to a higher concentration of copper from the other two. The average values from this study are for the results tabulated in Figure 3 for the two boards used. The agreement is actually again quite good; when one considers that different models of devices were used in the two studies.

**Table 3 - ICP Analysis Results for Materials from PCBAs Leached by EPA Method 1311**

| (all values in mg/L) | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|
| Board #1 On #35 + on #80 (mg/L) | 7.74 | 0.3 | 592 | 17.6 | 0.66 | 2.64 |
| Board #2 On #35 + on #80 (mg/L) | 7.19 | 0.3 | 525 | 17.9 | 0.47 | 3.06 |
| Board #1 Through #80 mesh (mg/L) | 31.2 | 470 | 7 | 39.2 | 34.4 | 490 |
| Board #2 Through #80 mesh (mg/L) | 32.5 | 452 | 1.86 | 39.6 | 34.4 | 492 |

**Table 4 - Comparison of Previous and Current Results for Leaching of Pulverized Handsets**

| | Previous study | Current study |
|---|---|---|
| **Cu** | **360** | **461** |
| **Fe** | **51** | **4.43** |
| **Ni** | **22** | **39.4** |
| **Pb** | **258** | **491** |
| **Zn** | **23** | **34.4** |

**Second and third parts of the circuit board leachate**

Separate portions of 40 ml of the leachate of material through the #80 mesh were treated with either 20 grams of egg white or 20 grams of cardboard, mixed and then either filtered or centrifuged. The resulting solutions were analyzed by ICP. The results are shown in Figure 2 and Table 4.

The egg whites had essentially no effect on barium and nickel concentrations, only a moderate effect on zinc, but rather dramatic results on the concentrations of iron, copper and lead. Except in the case of iron, the results were even more

dramatic with cardboard. Whether this is due to poorer affinity for iron or additional iron coming from the cardboard is not known, as the cardboard was not tested itself for leachate potential.

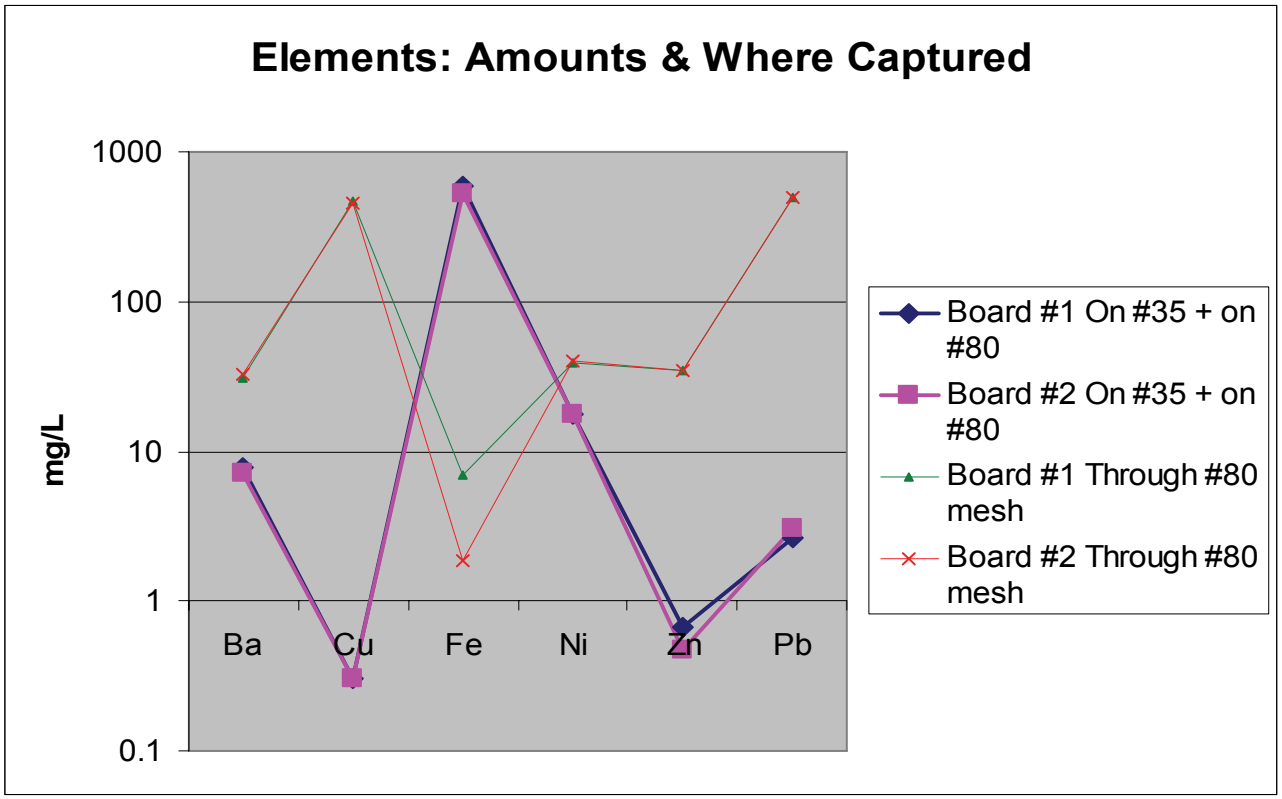



**Figure 1 - EPA Method 1311 Leaching Results for Two Ground Printed Circuit Packs**

**Table 4 - Effect of Egg Whites or Cardboard on Concentrations of Heavy Metals**

| | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|
| Average Through #80 Mesh (mg/L) | 31.85 | 461 | 4.43 | 39.4 | 34.4 | 491 |
| Average Through #80 Mesh + Egg Whites (mg/L) | 32.25 | 60.6 | 0.034 | 37 | 23 | 17.6 |
| Average Through #80 Mesh + Cardboard (mg/L) | 12.3 | 28.75 | 0.145 | 16.05 | 10.06 | 2.365 |

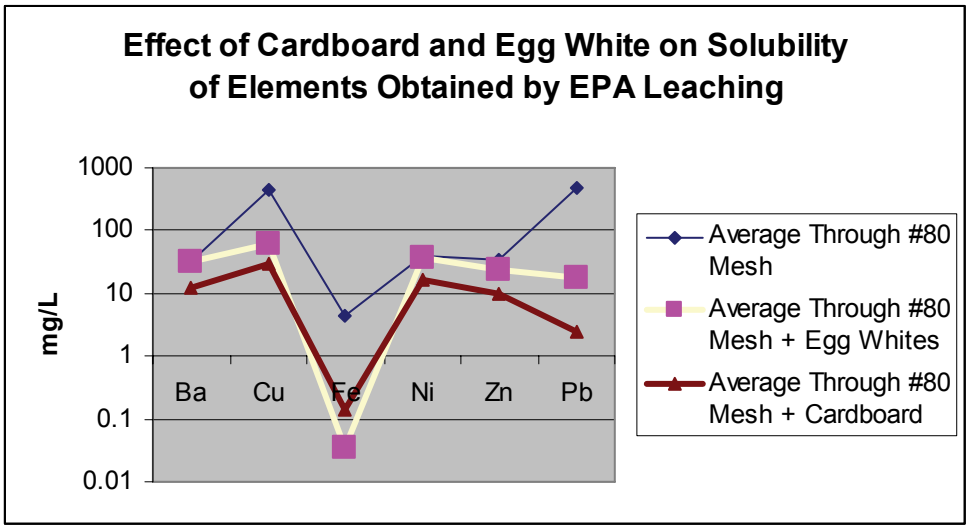



**Figure 2 - Effect of Common Landfill Materials on Heavy Metal Leachate Solubility**

**Fourth part of the circuit board leachate**

This portion of the investigation dealt with the extent of how tightly heavy metals are bound to topsoil. After leaching the two different separated sizes of ground particles from the two circuit boards, collecting the leachates and filtering, they were then mixed in the 1:20 portion with topsoil, tumbled, filtered and then reanalyzed. The results below in Table 5 and Figure 3 show very good agreement between the results for the two boards. The values show that the topsoil has a dramatic effect upon the amount of heavy metals in solution. The following show the approximate values in mg/l for the ionic concentrations before and after mixing with topsoil: (Ba – 32 -> 6), (Cu – 461 -> 12), (Fe – 4.4 -> 0.03), (Ni – 39.4 -> 6.0), (Zn – 34.4 -> 8.7) and (Pb – 491 -> 9). These correspond to the following decreases in concentrations: Ba 6x, Cu 38x, Fe 147x, Ni 7x, Zn 4x and Pb 61x. These are quite large decreases in concentrations.

**Table 5 - Results of a 1:20 Mixture of Topsoil:EPA Leachate**

| | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|
| #1 On #35 + on #80 + Soil (mg/L) | 1.42 | 0.332 | 0.674 | 0.861 | 0.742 | 0.177 |
| #1 Through #80 mesh + Soil (mg/L) | 5.66 | 11.50 | 0.030 | 5.980 | 8.540 | 8.100 |
| #2 On #35 + on #80 + Soil (mg/L) | 1.36 | 0.323 | 0.548 | 0.892 | 0.762 | 0.095 |
| #2 Through #80 mesh + Soil (mg/L) | 6.03 | 11.90 | 0.027 | 6.100 | 8.850 | 9.560 |

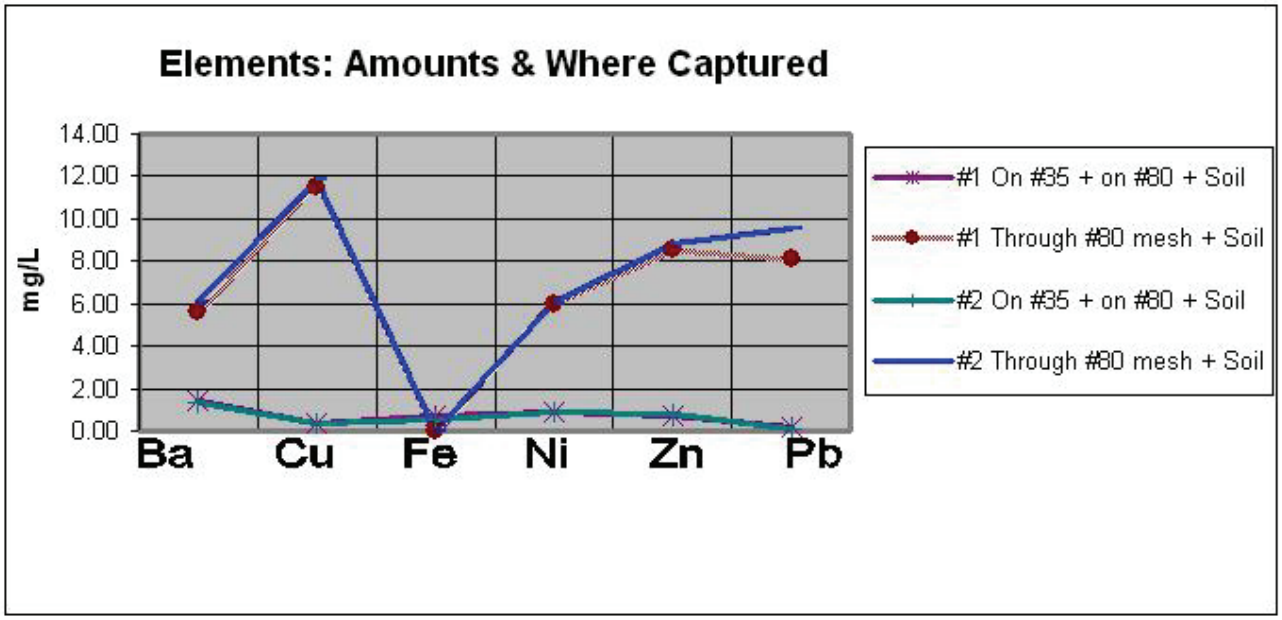



**Figure 3 - Leachate mixed with topsoil in a Leachate:Topsoil ratio of 20:1, mixed, filtered and analyzed**

**Last part of the investigation**

This portion of the study deals with the capability of columns of "soils" to take up elements from artificial leachate and keep it.

A solution of soluble salts of barium, silver, copper, iron, nickel, zinc and lead was made up. Five milliliter spikes containing 500 ug of each metal ion were each put on columns with different "soils". The columns were packed with either: 5 grams of topsoil; 10grams of sand; 10grams of vermiculite; 10grams of perlite or 145grams of gravel. Ideally the same mass or same surface area of material would have been used in each case. However, this was not possible due to not knowing the surface area of the materials and the differing abilities of the materials to hold the salt solutions. The extremes of essentially no porosity and channeling had to be avoided. This could only be done by using less topsoil and a significantly larger amount of gravel.

Seven 20 ml portions of deionized water were then run through two columns of each type of material. The resulting seven twenty milliliter aliquots for each column were collected and analyzed separately to determine the amount of the heavy metals from the artificial leachate not held by the topsoil, sand, gravel, vermiculite and perlite. This was repeated for the other set of columns with the same "soils" and same size of metal spiked solutions added, but the EPA 1311 leaching solution was used instead of deionized water for running through the columns. This was done to determine the differing abilities of these two liquids (DI water and EPA solution) to retrieve metals from soil. The detailed spreadsheets for topsoil and the other "soil" materials are given in the APPENDIX. See Figures 4 – 8 for graphical representations of the results.

The tables of data show that in many cases the blanks gave significant amounts of the ions of interest coming from the columns. The blank values were subtracted from the values obtained for the spiked columns. In the cases where negative values were obtained, zero values were entered in the accompanying graphs.

**Topsoil**

For the DI water leaching of topsoil, only nickel recovery was bigger than the blank values. It would appear that the presence of the materials in the spike was actually retarding the DI water from leaching more material from the column of topsoil. Even ignoring the blank, the total amount of zinc, lead and silver removed from the soil in both cases was less than 0.7 ppm per element. Only very small portions of zinc and lead were leached with the first 20 ml portion of DI water and then no more was detected during analysis. It is quite possible that the lead and zinc detected was a small portion of these ions that had never been taken up by the soil, but had remained in solution and so was easily swept out by the first aliquot. The values for copper and nickel were also small, 4 and 2 ppm, respectively. The most dramatic change in the amount leached over the course adding the seven aliquots of DI water was for barium, where after the first two 20 ml leaches with DI water the amount coming off the soil was reduced to essentially 0.01 mg/L. The results for the two separate columns were in good agreement, within 1 ppm of each other.

As expected, leaching with the EPA solution resulted in more materials being taken out of the topsoil. The blank values are also bigger, except for nickel. It is also noted that the agreement between the two columns run was actually quite poor. One column had most values in the single and double digit range while for the other one the values were double and triple digit. The definitive reason for this is known. Perhaps the topsoil form one column to the other was different or, more likely; the one with the larger values had channels in the material. Nevertheless, average values were plotted in Figure 4 for the EPA solution leach. Only iron again had a huge blank reading, with very significant amounts of barium, copper, nickel and zinc being recovered.

**Other "soils"**

Although all the other graphs exhibit the same general shape for the EPA leaching solution curves as seen in the topsoil graph, the amount of recovered heavy metals is much higher than for topsoil. Not surprisingly the sand holds the least, with the perlite, gravel and vermiculite showing about 80% recovery, except for the iron. Taking into account the blank, generally speaking the iron is the most tightly held by the different soils, with essentially little recovery from topsoil, sand or gravel when the soils are leached with the EPA solution or DI water.

Sand and gravel behave similarly whether the leaching medium is DI water or the EPA solution. This is not too surprising, as both materials are generally not considered very reactive. The reproducibility of results for sand was quite good, with the largest difference between the two columns being 26 ppm for zinc leached with DI. This represents a ~6% difference. Similar agreement was found for the two columns of gravel.

There are a few instances for perlite and gravel where DI water is apparently more aggressive in removing the metal ions. However, whether a 20% difference is real or is just the result of the natural variation of the system due to channeling, etc. is difficult to say. However, in the cases of lead leaching from sand, vermiculite and perlite where the difference is 40% or greater, it is likely that this is a real difference and the EPA solution is more aggressive, as expected.

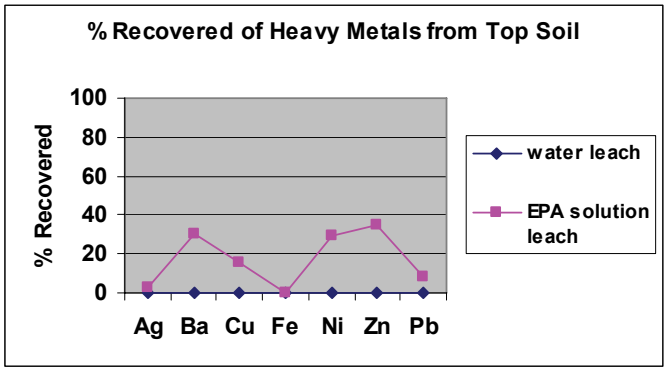
Raw vermiculite that has not been heated is known to have considerable ion exchange ability, especially in the presence of carboxylic acids[13]. However, once the vermiculite has been industrially heated and the waters of hydration are gone, the structure is more "fixed". This would account for the relatively poor ability of the vermiculite to hold the metal ions.

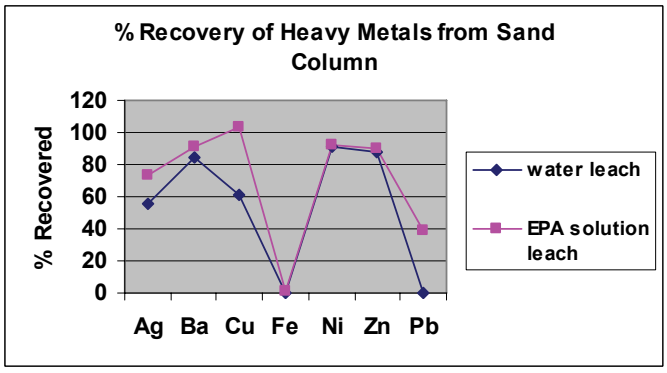
**Conclusions**

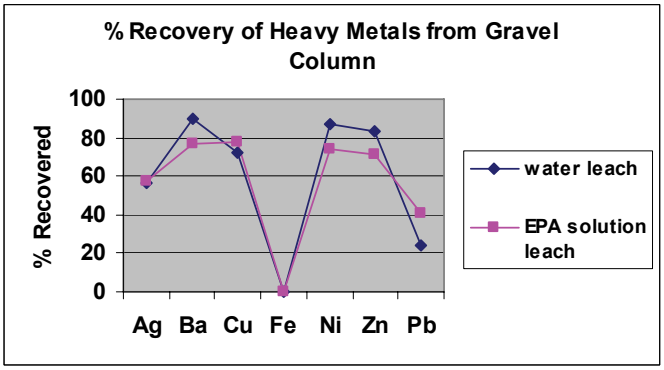
The present work has shown that there are several materials that could slow down if not stop the escape of heavy metals from a landfill. Any sulfur containing material will either have free sulfide or produce it during decomposition, resulting in one of the most potent precipitators of lead. Since landfills contain an abundance of paper, even with recycling, it is not likely lead will be escaping from any landfills soon.

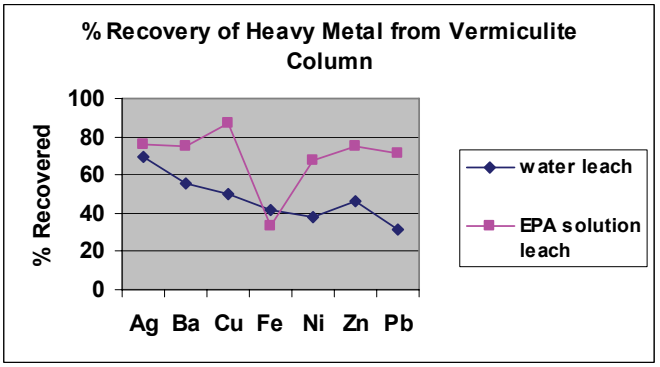
Even if it does escape from a landfill, the analytical work completed shows that any heavy metals that leach into topsoil is not going anywhere, as the topsoil is extremely effective in sequestering lead and other metals. However, sand and gravel, which are more likely to be found lower down in the ground surrounding a landfill, are less able to hold the heavy metals. But looking at lead only, one sees that for a DI water leach, which is reasonably aggressive because of the chemical driving force, that sand will still only release 0.06% of a 500 mg/5ml solution of lead. Gravel will release about 25%. It is highly unlikely that there would be a plume of 100 ppm dissolved lead leaving a landfill. The possibility of any significant amounts of lead leaving a modern landfill in the geologically near future is quite remote.

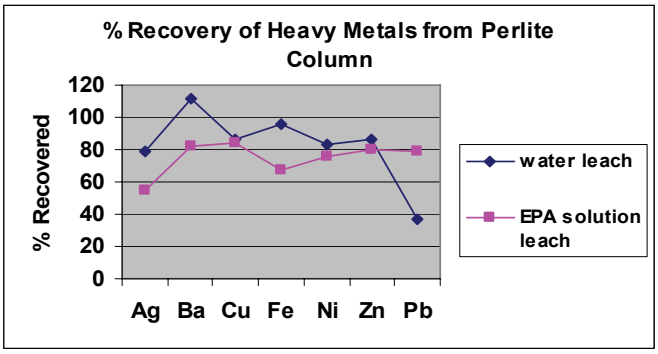
**Figures 4 – 8% Recovery of Heavy Metals from Different Soil**



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**Referemces**

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15. Personal experience of the author

**APPENDIX**

## Soil 5g

Extraction with water

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 1.32 | 0.032 | 0.57 | | 0.021 | 0.08 |
| Spike1.1 | | 0.973 | 0.018 | 0.595 | | 0.016 | 0.034 |
| Spike2.1 | | 0.957 | 0.02 | 0.51 | | 0.017 | 0.025 |
| Blank2 | | 0.374 | 0.035 | 0.73 | | | 0.014 |
| Spike1.2 | | 0.13 | 0.046 | 0.842 | 0.027 | | |
| Spike2.2 | | 0.188 | 0.048 | 0.842 | 0.032 | | |
| Blank3 | | 0.028 | 0.047 | 1.2 | 0.011 | | |
| Spike1.3 | | 0.016 | 0.04 | 0.764 | 0.02 | | |
| Spike2.3 | | 0.02 | 0.043 | 0.798 | 0.023 | | |
| Blank4 | | 0.021 | 0.043 | 1.05 | 0.01 | | |
| Spike1.4 | | 0.014 | 0.034 | 0.643 | 0.017 | | |
| Spike2.4 | | 0.015 | 0.034 | 0.633 | 0.018 | | |
| Blank5 | | 0.016 | 0.036 | 0.851 | 0.009 | | |
| Spike1.5 | | 0.011 | 0.027 | 0.515 | 0.012 | | |
| Spike2.5 | | 0.012 | 0.032 | 0.522 | 0.014 | | |
| Blank6 | | 0.012 | 0.026 | 0.625 | 0.006 | | |
| Spike1.6 | 0.002 | 0.008 | 0.019 | 0.359 | 0.01 | | |
| Spike2.6 | 0.02 | 0.008 | 0.02 | 0.367 | 0.011 | | |
| Blank7 | | 0.013 | 0.027 | 0.675 | 0.009 | | |
| Spike1.7 | 0.004 | 0.01 | 0.02 | 0.434 | 0.015 | | |
| Spike2.7 | 0.003 | 0.011 | 0.023 | 0.432 | 0.014 | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | | 35.68 | 4.92 | 114.02 | 0.9 | 0.42 | 1.88 |
| Spike1(ug) | 0.12 | 23.24 | 4.08 | 83.04 | 2.02 | 0.32 | 0.68 |
| Spike2(ug) | 0.46 | 24.22 | 4.4 | 82.08 | 2.24 | 0.34 | 0.5 |
| Theoreticaly % | 0.024 | -2.488 | -0.168 | -6.196 | 0.224 | -0.02 | -0.24 |
| Recovery of Spike | 0.092 | -2.292 | -0.104 | -6.388 | 0.268 | -0.016 | -0.276 |

Extract with leaching solution

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 1.89 | 0.028 | 0.451 | 0.011 | 0.028 | 0.037 |
| Spike1.1 | | 1.66 | 0.03 | 0.324 | 0.038 | 0.082 | 0.035 |
| Spike2.1 | 0.46 | 16.4 | 7.47 | 10.6 | 13.5 | 16.3 | 4.15 |
| Blank2 | | 0.718 | 0.049 | 1.31 | 0.016 | 0.047 | 0.031 |
| Spike1.2 | | 0.681 | 0.121 | 2.09 | 0.061 | 0.128 | 0.03 |
| Spike2.2 | 0.158 | 0.166 | 0.359 | 2.21 | 0.168 | 0.142 | 0.151 |
| Blank3 | | 0.198 | 0.123 | 6.75 | | 0.034 | 0.019 |
| Spike1.3 | | 0.229 | 0.14 | 2.78 | 0.057 | 0.042 | 0.016 |
| Spike2.3 | 0.117 | 0.146 | 0.155 | 0.91 | 0.098 | 0.104 | 0.065 |
| Blank4 | | 0.105 | 0.08 | 4.81 | | 0.02 | 0.1 |
| Spike1.4 | | 0.166 | 0.131 | 2.86 | 0.056 | 0.04 | 0.009 |
| Spike2.4 | 0.097 | 0.289 | 0.06 | 0.258 | 0.102 | 0.166 | 0.028 |
| Blank5 | | 0.085 | 0.061 | 3.93 | | 0.017 | 0.009 |
| Spike1.5 | | 0.136 | 0.089 | 1.97 | 0.041 | 0.037 | |
| Spike2.5 | 0.099 | 0.411 | 0.052 | 0.222 | 0.118 | 0.196 | 0.032 |
| Blank6 | | 0.087 | 0.068 | 4.57 | | 0.018 | 0.008 |
| Spike1.6 | | 0.108 | 0.072 | 1.61 | 0.032 | 0.028 | 0.004 |
| Spike2.6 | 0.089 | 0.41 | 0.047 | 0.207 | 0.113 | 0.186 | 0.031 |
| Blank7 | | 0.062 | 0.044 | 2.59 | | 0.012 | |
| Spike1.7 | 0.091 | 0.37 | 0.053 | 0.254 | 0.109 | 0.169 | 0.034 |
| Spike2.7 | 0.092 | 0.371 | 0.052 | 0.254 | 0.108 | 0.169 | 0.034 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | | 62.9 | 9.06 | 488.22 | 0.54 | 3.52 | 4.08 |
| Spike1(ug) | 1.84 | 67 | 12.72 | 237.76 | 7.88 | 9.68 | 2.56 |
| Spike2(ug) | 22.24 | 364.06 | 164.74 | 306.22 | 284.84 | 345.64 | 90.68 |
| Theoreticaly % | 0.368 | 0.82 | 0.732 | -50.092 | 1.468 | 1.232 | -0.304 |
| Recovery of Spike | 4.448 | 60.232 | 31.136 | -36.4 | 56.86 | 68.424 | 17.32 |

# Sand (10g)

Extract with water

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 1.13 | 0.016 | 0.231 | 0.056 | 0.157 | 0.032 |
| Spike1.1 | 12.7 | 21.5 | 15.1 | 0.078 | 22.4 | 21.4 | 0.047 |
| Spike2.1 | 12.7 | 22.1 | 15.4 | 0.174 | 22.9 | 22.7 | 0.049 |
| Blank2 | | 0.021 | 0.016 | 0.107 | 0.011 | 0.016 | |
| Spike1.2 | 0.792 | 0.227 | 0.146 | 0.016 | 0.167 | 0.108 | |
| Spike2.2 | 0.700 | 0.237 | 0.151 | 0.011 | 0.167 | 0.120 | |
| Blank3 | | | | 0.276 | | | |
| Spike1.3 | 0.173 | 0.043 | | 0.072 | | | |
| Spike2.3 | 0.165 | 0.045 | | 0.046 | | | |
| Blank4 | | | | 0.343 | | | |
| Spike1.4 | 0.084 | 0.036 | | 0.04 | | | |
| Spike2.4 | 0.075 | 0.038 | | 0.072 | | | |
| Blank5 | | | | 0.336 | | | |
| Spike1.5 | 0.052 | 0.019 | | 0.144 | | | |
| Spike2.5 | 0.044 | 0.020 | | 0.124 | | | |
| Blank6 | | | | 0.218 | | | |
| Spike1.6 | 0.047 | 0.015 | | 0.108 | | | |
| Spike2.6 | 0.045 | 0.021 | | 0.273 | | | |
| Blank7 | | | | 0.191 | | | |
| Spike1.7 | 0.047 | 0.016 | | 0.081 | | | |
| Spike2.7 | 0.045 | 0.018 | | 0.218 | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | | 23.02 | 0.64 | 34.04 | 1.34 | 3.46 | 0.64 |
| Spike1(ug) | 277.9 | 437.12 | 304.92 | 10.78 | 451.34 | 430.16 | 0.94 |
| Spike2(ug) | 275.48 | 449.58 | 311.02 | 12.9 | 461.34 | 456.4 | 0.98 |
| Theoretical % | 55.58 | 82.82 | 60.856 | -4.652 | 90 | 85.34 | 0.06 |
| Recovery of Spike | 55.096 | 85.312 | 62.076 | -4.228 | 92 | 90.588 | 0.068 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Spike1(ug) | 278 | 437 | 305 | 11 | 451 | 430 | 1 |
| Spike2(ug) | 275 | 450 | 311 | 13 | 461 | 456 | 1 |

Extract with leaching solution

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 1.23 | 0.068 | 0.275 | 0.049 | 0.238 | 0.031 |
| Spike1.1 | 16.2 | 22.5 | 22.4 | 0.988 | 22.5 | 21.7 | 3.53 |
| Spike2.1 | 15 | 21.7 | 21.4 | 0.4 | 22.3 | 21 | 2.67 |
| Blank2 | | 0.296 | 0.075 | 0.021 | 0.031 | 0.117 | 0.02 |
| Spike1.2 | 1.74 | 1.53 | 2.08 | 0.03 | 0.425 | 0.792 | 2.47 |
| Spike2.2 | 1.75 | 1.94 | 2.3 | 0.03 | 0.577 | 1.03 | 2.19 |
| Blank3 | | 0.201 | 0.063 | 0.027 | 0.022 | 0.088 | 0.014 |
| Spike1.3 | 0.683 | 0.466 | 0.774 | 0.022 | 0.06 | 0.212 | 1.28 |
| Spike2.3 | 0.756 | 0.493 | 0.788 | 0.034 | 0.054 | 0.23 | 1.32 |
| Blank4 | | 0.154 | 0.066 | 0.047 | 0.021 | 0.086 | 0.014 |
| Spike1.4 | 0.204 | 0.249 | 0.49 | 0.027 | 0.044 | 0.138 | 0.925 |
| Spike2.4 | 0.207 | 0.265 | 0.513 | 0.043 | 0.047 | 0.153 | 0.968 |
| Blank5 | | 0.141 | 0.074 | 0.073 | 0.023 | 0.084 | 0.011 |
| Spike1.5 | 0.066 | 0.207 | 0.389 | 0.06 | 0.04 | 0.116 | 0.733 |
| Spike2.5 | 0.056 | 0.189 | 0.373 | 0.077 | 0.037 | 0.12 | 0.762 |
| Blank6 | | 0.129 | 0.098 | 0.111 | 0.022 | 0.079 | 0.013 |
| Spike1.6 | 0.015 | 0.165 | 0.302 | 0.091 | 0.035 | 0.099 | 0.597 |
| Spike2.6 | 0.017 | 0.173 | 0.311 | 0.089 | 0.034 | 0.113 | 0.609 |
| Blank7 | | 0.097 | 0.068 | 0.3 | 0.018 | 0.067 | 0.016 |
| Spike1.7 | | 0.107 | 0.216 | 0.275 | 0.015 | 0.078 | 0.497 |
| Spike2.7 | | 0.118 | 0.237 | 0.272 | 0.021 | 0.087 | 0.501 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | | 44.96 | 10.24 | 17.08 | 3.72 | 15.18 | 0.119 |
| Spike1(ug) | 378.16 | 504.48 | 533.02 | 29.86 | 462.38 | 462.7 | 200.64 |
| Spike2(ug) | 355.72 | 497.56 | 518.44 | 18.9 | 466.63 | 462.66 | 189.1 |
| Theoretical % | 75.632 | 91.904 | 104.556 | 2.556 | 91.732 | 89.504 | 40.1042 |
| Recovery of Spike | 71.144 | 90.52 | 101.64 | 0.364 | 92.582 | 89.496 | 37.7962 |

# Perlite (10g)

Extract with water

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 0.16 | 0.29 | 0.464 | 0.019 | 0.044 | 0.011 |
| Spike1.1 | 7.85 | 11.3 | 10.8 | 11.1 | 10.3 | 10.7 | 10.8 |
| Spike2.1 | 4.26 | 8.06 | 7.58 | 8.31 | 7.23 | 7.5 | 7.49 |
| Blank2 | | 0.225 | 0.025 | 1.33 | 0.028 | 0.028 | 0.008 |
| Spike1.2 | 8.54 | 11.5 | 11 | 11.1 | 10.4 | 10.8 | 11.1 |
| Spike2.2 | 11.5 | 15.6 | 14.4 | 14.9 | 13.8 | 14.2 | 15 |
| Blank3 | | 0.088 | 0.006 | 0.428 | 0.007 | 0.008 | |
| Spike1.3 | 2.46 | 3.6 | 3.41 | 3.05 | 3.2 | 3.37 | 3.34 |
| Spike2.3 | 2.54 | 3.43 | 3.14 | 2.66 | 3 | 3.15 | 3.21 |
| Blank4 | | 0.03 | | 0.07 | 0.005 | | |
| Spike1.4 | 0.794 | 1.04 | 0.983 | 0.563 | 0.914 | 0.973 | 0.894 |
| Spike2.4 | 0.726 | 0.907 | 0.837 | 0.425 | 0.789 | 0.837 | 0.762 |
| Blank5 | | 0.011 | | 0.017 | 0.006 | | |
| Spike1.5 | 0.261 | 0.361 | 0.344 | 0.081 | 0.326 | 0.345 | 0.251 |
| Spike2.5 | 0.262 | 0.366 | 0.339 | 0.086 | 0.325 | 0.345 | 0.252 |
| Blank6 | | 0.006 | | 0.013 | | | 0.007 |
| Spike1.6 | 0.082 | 0.139 | 0.142 | 0.021 | 0.137 | 0.144 | 0.07 |
| Spike2.6 | 0.101 | 0.158 | 0.151 | 0.025 | 0.149 | 0.158 | 0.085 |
| Blank7 | | 0.003 | | 0.006 | | | |
| Spike1.7 | 0.042 | 0.064 | 0.07 | 0.01 | 0.072 | 0.075 | 0.026 |
| Spike2.7 | 0.058 | 0.092 | 0.093 | 0.015 | 0.094 | 0.099 | 0.032 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | | 10.46 | 6.42 | 46.56 | 1.3 | 1.6 | 0.38 |
| Spike1(ug) | 400.58 | 560.08 | 436 | 518.5 | 414 | 430 | 216 |
| Spike2(ug) | 388.94 | 572.26 | 439.6 | 527.92 | 420.6 | 434 | 149.8 |
| Theoreticaly % | 80.116 | 109.924 | 85.916 | 94.388 | 82.54 | 85.68 | 43.124 |
| Recovery of Spike | 77.788 | 112.36 | 86.636 | 96.272 | 83.86 | 86.48 | 29.884 |

Extract with leaching solution

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | 0.005 | 0.219 | 0.035 | 0.565 | 0.014 | 0.052 | 0.012 |
| Spike1.1 | 3.3 | 7.01 | 6.98 | 7.56 | 6.61 | 6.91 | 6.78 |
| Spike2.1 | 3.63 | 8.28 | 8.1 | 9.06 | 7.78 | 8.11 | 8.03 |
| Blank2 | 0.004 | 0.191 | 0.016 | 0.684 | 0.015 | 0.014 | 0.005 |
| Spike1.2 | 6.11 | 10.2 | 10.4 | 8.77 | 9.29 | 9.65 | 9.6 |
| Spike2.2 | 7.86 | 12.3 | 12.5 | 10.4 | 11.2 | 11.6 | 11.7 |
| Blank3 | | 0.06 | 0.011 | 0.054 | 0.012 | 0.005 | |
| Spike1.3 | 1.69 | 2.75 | 2.81 | 1.36 | 2.51 | 2.65 | 2.47 |
| Spike2.3 | 1.6 | 2.66 | 2.71 | 1.08 | 2.41 | 2.55 | 2.31 |
| Blank4 | | 0.026 | 0.009 | 0.025 | 0.013 | 0.004 | |
| Spike1.4 | 1.09 | 0.894 | 0.925 | 0.499 | 0.792 | 0.848 | 0.812 |
| Spike2.4 | 0.924 | 0.576 | 0.58 | 0.352 | 0.492 | 0.533 | 0.539 |
| Blank5 | | 0.016 | 0.009 | 0.022 | 0.012 | 0.003 | |
| Spike1.5 | 0.706 | 0.345 | 0.348 | 0.28 | 0.292 | 0.313 | 0.326 |
| Spike2.5 | 0.617 | 0.205 | 0.2 | 0.223 | 0.164 | 0.178 | 0.209 |
| Blank6 | | 0.012 | 0.009 | 0.018 | 0.009 | 0.003 | |
| Spike1.6 | 0.395 | 0.152 | 0.152 | 0.197 | 0.121 | 0.13 | 0.153 |
| Spike2.6 | 0.385 | 0.103 | 0.1 | 0.173 | 0.079 | 0.084 | 0.104 |
| Blank7 | | 0.01 | 0.007 | 0.012 | 0.01 | 0.002 | |
| Spike1.7 | 0.311 | 0.106 | 0.103 | 0.17 | 0.081 | 0.087 | 0.11 |
| Spike2.7 | 0.31 | 0.087 | 0.085 | 0.135 | 0.062 | 0.069 | 0.091 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | 0.155 | 9.585 | 1.745 | 24.775 | 1.63 | 1.4 | 0.28 |
| Spike1(ug) | 255.54 | 394.09 | 399.46 | 338.92 | 360.87 | 377.21 | 371.12 |
| Spike2(ug) | 288.37 | 442.82 | 445 | 383.16 | 404.84 | 421.93 | 419.51 |
| Theoreticaly % | 51.077 | 76.901 | 79.543 | 62.829 | 71.848 | 75.162 | 74.168 |
| Recovery of Spike | 57.643 | 86.647 | 88.651 | 71.677 | 80.642 | 84.106 | 83.846 |

# Vermiculite (10g)

Extract with water

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | 0.021 | 0.994 | 0.09 | 4.78 | 0.039 | 0.078 | 0.012 |
| Spike1.1 | 8.86 | 9.91 | 8.9 | 9.52 | 7.6 | 8.64 | 6.84 |
| Spike2.1 | 5 | 7.77 | 5.84 | 9.38 | 4.24 | 5.3 | 3.29 |
| Blank2 | 0.026 | 0.918 | 0.075 | 7.16 | 0.057 | 0.06 | 0.007 |
| Spike1.2 | 5.25 | 5.16 | 4.12 | 9.82 | 3.05 | 3.8 | 2.47 |
| Spike2.2 | 5.93 | 5.48 | 4 | 10.5 | 2.68 | 3.55 | 2.01 |
| Blank3 | 0.007 | 0.296 | 0.024 | 2.64 | 0.014 | 0.017 | 0.003 |
| Spike1.3 | 1.81 | 1.25 | 0.857 | 5.2 | 0.597 | 0.781 | 0.427 |
| Spike2.3 | 2.36 | 1.19 | 0.838 | 3.15 | 0.555 | 0.772 | 0.392 |
| Blank4 | | 0.119 | 0.009 | 1.32 | 0.008 | 0.005 | 0.004 |
| Spike1.4 | 0.976 | 0.434 | 0.268 | 2.71 | 0.182 | 0.249 | 0.132 |
| Spike2.4 | 1.41 | 0.518 | 0.346 | 1.64 | 0.218 | 0.318 | 0.15 |
| Blank5 | | 0.06 | 0.007 | 0.931 | 0.007 | 0.002 | |
| Spike1.5 | 0.577 | 0.191 | 0.11 | 1.56 | 0.072 | 0.102 | 0.055 |
| Spike2.5 | 0.962 | 0.249 | 0.155 | 0.906 | 0.088 | 0.145 | 0.07 |
| Blank6 | | 0.029 | | 0.649 | 0.005 | | |
| Spike1.6 | 0.319 | 0.085 | 0.045 | 0.981 | 0.029 | 0.041 | 0.024 |
| Spike2.6 | 0.666 | 0.141 | 0.085 | 0.601 | 0.042 | 0.077 | 0.042 |
| Blank7 | | 0.026 | | 0.851 | 0.005 | | 0.005 |
| Spike1.7 | 0.23 | 0.059 | 0.029 | 0.944 | 0.019 | 0.025 | 0.012 |
| Spike2.7 | 0.402 | 0.076 | 0.042 | 0.413 | 0.021 | 0.038 | 0.02 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | 1.08 | 48.84 | 4.1 | 366.62 | 2.7 | 3.24 | 0.62 |
| Spike1(ug) | 360.44 | 341.78 | 286.58 | 614.7 | 230.98 | 271.44 | 199.2 |
| Spike2(ug) | 334.6 | 308.48 | 226.12 | 531.8 | 156.88 | 201.7 | 119.48 |
| Theoreticaly % | 71.872 | 58.588 | 56.496 | 49.616 | 45.656 | 53.64 | 39.716 |
| Recovery of Spike | 66.704 | 51.928 | 44.404 | 33.036 | 30.836 | 39.692 | 23.772 |

Extract with leaching solution

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | 0.002 | 0.779 | 0.078 | 4.06 | 0.016 | 0.098 | 0.014 |
| Spike1.1 | 8.67 | 10.8 | 10.3 | 8.11 | 8.82 | 9.65 | 7.51 |
| Spike2.1 | 8.87 | 11.2 | 10.5 | 8.33 | 8.8 | 9.91 | 7.52 |
| Blank2 | | 0.671 | 0.06 | 3.54 | 0.016 | 0.052 | 0.007 |
| Spike1.2 | 4.62 | 4.62 | 5 | 6.45 | 3.19 | 3.77 | 3.31 |
| Spike2.2 | 4.82 | 4.91 | 5.19 | 6.38 | 3.37 | 4 | 3.51 |
| Blank3 | | 0.48 | 0.049 | 2.02 | 0.015 | 0.033 | 0.003 |
| Spike1.3 | 2.01 | 1.95 | 2.8 | 1.42 | 1.55 | 1.74 | 2.45 |
| Spike2.3 | 2.08 | 2.03 | 2.88 | 1.63 | 1.63 | 1.84 | 2.63 |
| Blank4 | | 0.414 | 0.038 | 0.861 | 0.009 | 0.024 | 0.005 |
| Spike1.4 | 1.32 | 1.42 | 1.63 | 0.956 | 1.13 | 1.22 | 1.67 |
| Spike2.4 | 1.38 | 1.46 | 1.69 | 1.31 | 1.2 | 1.27 | 1.78 |
| Blank5 | | 0.377 | 0.032 | 0.651 | 0.007 | 0.02 | |
| Spike1.5 | 0.904 | 1.17 | 1.04 | 2.15 | 0.901 | 0.917 | 1.21 |
| Spike2.5 | 0.879 | 1.13 | 0.974 | 0.795 | 0.906 | 0.915 | 1.2 |
| Blank6 | | 0.349 | 0.028 | 0.591 | 0.005 | 0.017 | 0.002 |
| Spike1.6 | 0.638 | 0.972 | 0.702 | 0.959 | 0.735 | 0.722 | 0.886 |
| Spike2.6 | 0.638 | 0.945 | 0.696 | 0.834 | 0.743 | 0.719 | 0.889 |
| Blank7 | | 0.321 | 0.025 | 0.479 | 0.004 | 0.014 | 0.005 |
| Spike1.7 | 0.461 | 0.809 | 0.487 | 0.774 | 0.588 | 0.559 | 0.667 |
| Spike2.7 | 0.457 | 0.803 | 0.474 | 0.99 | 0.594 | 0.567 | 0.67 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | 0.04 | 67.82 | 6.2 | 244.04 | 1.44 | 5.16 | 0.72 |
| Spike1(ug) | 372.46 | 434.82 | 439.18 | 416.38 | 338.28 | 371.56 | 354.06 |
| Spike2(ug) | 382.48 | 449.56 | 448.08 | 405.38 | 344.86 | 384.42 | 363.98 |
| Theoreticaly % | 74.484 | 73.4 | 86.596 | 34.468 | 67.368 | 73.28 | 70.668 |
| Recovery of Spike | 76.488 | 76.348 | 88.376 | 32.268 | 68.684 | 75.852 | 72.652 |

# Gravel (145g)

Extract with water

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 0.533 | 0.08 | 5.46 | 0.053 | 0.117 | 0.055 |
| Spike1.1 | 10.6 | 21 | 17.1 | 6.97 | 20.1 | 19.5 | 4.84 |
| Spike2.1 | 9.9 | 21.6 | 16.6 | 6 | 20.3 | 19.9 | 3.63 |
| Blank2 | | 0.099 | 0.056 | 3.66 | 0.033 | 0.04 | 0.035 |
| Spike1.2 | 1.12 | 1.23 | 0.622 | 1.21 | 1.19 | 0.969 | 0.259 |
| Spike2.2 | 1.28 | 1.75 | 0.906 | 1.8 | 1.69 | 1.47 | 0.406 |
| Blank3 | | 0.046 | 0.037 | 2.66 | 0.025 | 0.027 | 0.029 |
| Spike1.3 | 0.75 | 0.251 | 0.28 | 3.49 | 0.129 | 0.124 | 0.495 |
| Spike2.3 | 0.709 | 0.228 | 0.174 | 1.59 | 0.126 | 0.106 | 0.246 |
| Blank4 | | 0.045 | 0.046 | 3.39 | 0.032 | 0.033 | 0.035 |
| Spike1.4 | 0.725 | 0.196 | 0.46 | 7.51 | 0.078 | 0.112 | 0.962 |
| Spike2.4 | 0.567 | 0.115 | 0.114 | 1.74 | 0.04 | 0.035 | 0.246 |
| Blank5 | | 0.05 | 0.056 | 4.09 | 0.03 | 0.041 | 0.034 |
| Spike1.5 | 0.471 | 0.088 | 0.13 | 2.4 | 0.029 | 0.033 | 0.278 |
| Spike2.5 | 0.622 | 0.095 | 0.188 | 3.34 | 0.023 | 0.041 | 0.486 |
| Blank6 | | 0.026 | 0.022 | 1.96 | 0.019 | 0.018 | 0.016 |
| Spike1.6 | 0.353 | 0.056 | 0.093 | 0.096 | 0.016 | 0.022 | 0.204 |
| Spike2.6 | 0.454 | 0.06 | 0.126 | 2.49 | 0.01 | 0.029 | 0.317 |
| Blank7 | | 0.018 | 0.015 | 1.46 | 0.012 | 0.011 | 0.019 |
| Spike1.7 | 0.216 | 0.028 | 0.03 | 1.03 | | | 0.068 |
| Spike2.7 | 0.343 | 0.046 | 0.104 | 2.16 | 0.011 | 0.025 | 0.261 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | 0 | 16.34 | 6.24 | 453.6 | 4.08 | 5.74 | 4.46 |
| Spike1(ug) | 284.7 | 456.98 | 374.3 | 454.12 | 430.84 | 414.76 | 142.12 |
| Spike2(ug) | 277.5 | 477.88 | 364.24 | 382.4 | 444 | 431.04 | 111.84 |
| Theoretic | 56.94 | 88.128 | 73.612 | 0.104 | 85.352 | 81.804 | 27.532 |
| aly % | 55.5 | 92.308 | 71.6 | -14.24 | 87.984 | 85.06 | 21.476 |

Extract with leaching solution

| mg/L | Ag | Ba | Cu | Fe | Ni | Zn | Pb |
|---|---|---|---|---|---|---|---|
| Blank1 | | 0.82 | 0.129 | 5.84 | 0.044 | 0.229 | 0.061 |
| Spike1.1 | 10.9 | 17.3 | 16.9 | 11.4 | 16.8 | 16.1 | 7.23 |
| Spike2.1 | 9.58 | 18.4 | 16.8 | 6.09 | 17.9 | 17 | 5.21 |
| Blank2 | | 0.309 | 0.065 | 4.17 | 0.028 | 0.069 | 0.036 |
| Spike1.2 | 1.56 | 1.87 | 1.67 | 3.55 | 1.32 | 1.24 | 1.5 |
| Spike2.2 | 1.01 | 1.57 | 1.51 | 2.41 | 0.893 | 0.949 | 1.38 |
| Blank3 | | 0.204 | 0.044 | 2.54 | 0.019 | 0.05 | 0.026 |
| Spike1.3 | 0.8 | 0.504 | 0.507 | 1.49 | 0.149 | 0.252 | 0.695 |
| Spike2.3 | 0.672 | 0.533 | 0.563 | 0.858 | 0.137 | 0.267 | 0.768 |
| Blank4 | | 0.158 | 0.036 | 2.39 | 0.019 | 0.045 | 0.022 |
| Spike1.4 | 0.684 | 0.306 | 0.333 | 1.25 | 0.057 | 0.152 | 0.531 |
| Spike2.4 | 0.699 | 0.33 | 0.417 | 2.16 | 0.063 | 0.172 | 0.735 |
| Blank5 | | 0.137 | 0.151 | 2.07 | 0.018 | 0.04 | 0.018 |
| Spike1.5 | 0.559 | 0.204 | 0.226 | 0.936 | 0.03 | 0.106 | 0.414 |
| Spike2.5 | 0.639 | 0.226 | 0.313 | 1.3 | 0.034 | 0.129 | 0.554 |
| Blank6 | | 0.123 | 0.028 | 1.97 | 0.017 | 0.037 | 0.016 |
| Spike1.6 | 0.407 | 0.171 | 0.177 | 1.04 | 0.026 | 0.083 | 0.36 |
| Spike2.6 | 0.567 | 0.196 | 0.253 | 1.03 | 0.029 | 0.11 | 0.492 |
| Blank7 | | 0.096 | 0.022 | 1.47 | 0.017 | 0.029 | 0.019 |
| Spike1.7 | 0.311 | 0.14 | 0.148 | 1.44 | 0.024 | 0.075 | 0.318 |
| Spike2.7 | 0.522 | 0.163 | 0.207 | 0.894 | 0.024 | 0.093 | 0.416 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Blank(ug) | 0 | 36.94 | 9.5 | 409 | 3.24 | 9.98 | 3.96 |
| Spike1(ug) | 304.42 | 409.9 | 399.22 | 422.12 | 368.12 | 360.16 | 220.96 |
| Spike2(ug) | 273.78 | 428.36 | 401.26 | 294.84 | 381.6 | 374.4 | 191.1 |
| Theoretic | 60.884 | 74.592 | 77.944 | 2.624 | 72.976 | 70.036 | 43.4 |
| aly % | 54.756 | 78.284 | 78.352 | -22.832 | 75.672 | 72.884 | 37.428 |