

## Study of Analysis of Land Fill Sludge From Maiduguri Nigeria

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### Abstract

A study of four sample preparation methods for analysis of toxic element zinc and lead from landfill sludge of two localities of Maiduguri Metropolis, in the North East of Nigeria is presented in this work. The results showed that digestion with nitric acid and Hydrogen peroxide, and the use of High-pressure decomposition vessels are considered to be more efficient. Digestions in pressure vessels require less acid than digestions in open containers. The pressure vessels reduce volatilisation losses and can promote mineralisation.

### Introduction

Sludge disposal is one of many problems confronting landfill treatment in Nigeria. The ultimate fate of most landfill sludges is burial in landfills. Such an activity poses the possibility of contaminating the ground water with toxic elements for example arsenic, zinc and lead, and other leached from the buried sludges or sludge ashes. This work was prompted during the evaluation of ground water of North East of Nigeria as a guide to oxide zone mined recovery.

Even though the Nigeria Environmental protection agency recommends the measurement of heavy metals in landfill sludges, no procedures are specified for these determinations. The use of the usual heavy metal determination of atomic absorption spectroscopy for some elements like zinc and lead in landfill sludges requires the destruction of the organic matrix and the dissolution of the metals with neither loss nor contamination of the contents of the analysis.

Several dry and wet ashing techniques have been used to prepare samples for atomic absorption spectrometry (Delfino and Enderson, 1988; Plehn and Rhee, 1990) among the earlier methods.

Feinberg and Ducauze, (1980) reported higher recoveries of zinc and lead when the ashes from sludges were solubilized in a hydrochloric - nitric acid mixture rather than in nitric acid alone. Sulfuric acid has also been used as an ashing aid for the food products to decrease the volatility of zinc and lead compounds.

In the case of landfill sludges, there are conflicting reports on the efficiency of muffle furnace ignition compared to wet digestion (Ritter, Bergman and Cathern, 1990).

This comparison of several sample preparation procedures for the determination of zinc and lead in landfill sludges of landfill sludges from Maiduguri in N.E. of Nigeria has therefore been undertaken to enlighten some procedures for sludge analysis. Another aim of this work is to evaluate the amounts of toxic element contents in the landfill disposal of landfill sludges in North East of Nigeria as this imposes an environment hazards on human health. Some of the methods of Jenius and Katz

(1983) were reviewed and applied for the determination of Landfill sludges in the two major areas of Maiduguri in the North East of Nigeria.

### Experimental

#### Sample Collection:

Samples of landfill sludge were collected from the landfill in two environments of Maiduguri Metropolis namely MM - SEW1 and MM - SEW2. These sites serves the major areas.

#### Analytical Procedures:

Four procedures were used to prepare the sludge samples for the determination of zinc and lead by atomic absorption spectrometry. Muffle furnace ignition and digestion with nitric acid were supplemented with digestion in high-pressure-decomposition vessels and digestion with nitric acid and hydrogen peroxide.

#### Procedure 1: Muffle Furnace Ignition

Five 200mg sample of the dried sludges were weighed into zirconium crucible (B-J enterprises, No. 15LF) and ignited in a Bunsen flame. The charred samples were placed in a 450° muffle furnace for 2 hours to complete the ignition. The ignited residues were cooled and leached three times with 5ml portions of hot, 25% (V/V) redistilled nitric acid in distilled water. The leachates were filtered through No. 42 paper (Whatman) into 50ml volumetric flasks and brought to volume with distilled water. These solutions were used for the determination of zinc and lead by atomic absorption spectrometry.

#### Procedure 2: Digestion with Nitric acid

Five 200mg samples of the dried sludges were weighed into 125ml Erlenmeyer flasks, treated with 5ml redistilled nitric acid, and heated gently until the volume was reduced to approximately 1ml. The flasks containing the samples as well as those including blanks were cooled, treated with 5ml redistilled nitric acid, and again heated gently. This process was repeated 4 times to a point where

additional acid produced no apparent changes in the samples. The contents of the flasks were treated with 5ml of distilled water, through No. 42 paper into 50ml volumetric flask and brought to volume with distilled water. These solutions were used for the determination of zinc and lead by atomic absorption spectrometry.

**Procedure 3: Digestion with Nitric Acid in High-Pressure decomposition vessels**

Five 200mg samples of each of the dried landfill sludges were weighed into the PTFE cups of the digestion vessels (No. 4745) and each, including blanks were treated with 2.5ml of redistilled nitric acid. The vessels were closed and placed in a 100° oven overnight. The digested contents were treated with 5ml of distilled water filtered through No. 42 paper into 50ml volumetric flasks and brought to volume with distilled water. The zinc and lead levels of these solutions were determined by atomic absorption spectrometry.

**Procedure 4: Digestion with Nitric Acid in hydrogen Peroxide**

Five 200mg samples of each of the dried sludges were weighed into 125ml Erlenmeyer flasks. The flasks containing the sludge samples and empty flasks serving as blanks were treated with 5ml distilled water. The flasks were heated gently until most of the acid was evaporated. After cooling, 5ml of redistilled nitric acid was added to the flasks and the flasks again were heated gently until the volume was reduced to approximately 1ml. Again, the flasks were allowed to cool. These flasks were treated with 1ml of redistilled nitric acid, 1ml of distilled water and 2ml of 30% Hydrogen peroxide. The flasks were alternately heated, cooled and treated with additional 1ml increments of peroxide until no further changes were apparent in the sample. This was accomplished with 4 additional 1ml increment of peroxide. The contents of the flasks were treated with 5ml distilled water and filtered through No. 42 paper into 50ml volumetric flasks and brought to volume with distilled water. These solutions were used for the determination of zinc and lead by atomic absorption spectrometry.

Table 1: The Amounts of Zinc and Lead in Landfill Sludges of Maiduguri in North-East of Nigeria

Procedure	Amounts of Solids (mg kg)							
	MM-SEW1				MM-SEW2			
	Zn	E	Pb	E	Zn	E	Pb	E
1	61.0	±1.8	2680	±124	58.7	±1.7	2680	±101
2	64.7	±2.2	2970	±66	57.7	±1.8	2760	±51
3	76.7	±1.7	3410	±73	66.3	±5.9	3080	±110
4	74.1	±1.3	3220	±71	61.7	±6.7	2970	±122

Table 2: Relative Recoveries of Zinc and Lead in Sludge of Maiduguri in N.E. of Nigeria

Procedures	MM-SEW1 (in %)		MM-SEW2 (in %)	
	Zn	Pb	Zn	Pb
1	79.5	78.6	88.5	87.0
2	84.4	87.1	87.0	89.6
3	100	100	100	100
4	96.6	94.4	92.1	96.4

An atomic absorption spectrometer model 360, (perkin-Elmer) was used for the determination of zinc and lead in the solutions prepared by the for procedures outlined above. Zinc level were measured in the air-acetylene flame at a wavelength of 283.3nm.

**Results and Discussion**

The results of the zinc and lead determination are presented in Table 1. These results are given on a dry weight basis as the mean and standard deviation for the five samples of each sludge

The mean values were compared on the basis of the sample preparation procedures (see Table 1). Differences were considered to be significant when the calculated mean exceeded those expected as shown by standard deviation values (e) the table.

It appears that the zinc and lead results for the sludge from the MM-SEW1 differ significant

from each other on the basis of sample preparation procedures. The zinc and lead levels for the MM-SEW2 show similar differences between the results obtained from samples that were digested in nitric acid or ignited in the muffle furnace and those obtained from samples that were digested with nitric acid in the high-pressure-decomposition vessels.

In all cases, the zinc and lead levels obtained on samples digested with nitric acid in the high-pressure-decomposition vessels are significantly higher than the corresponding values obtained on samples prepared by muffle furnace ignition or nitric acid digestion. The results obtained from samples digested with nitric acid and hydrogen peroxide (Procedure 4) appear to agree more closely with those from the pressure vessel digestion (Procedure 3) than with those from the other two procedures.

Among the factors responsible for losses during dry ashing are absorption, diffusion and volatilization (Mitchell and Zief, 1980). Significant

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losses of zinc and lead when tissue was dry - ashed in porcelain crucibles have also been reported (Watling and Wardale, 1979). In this work attempt was made to minimize losses due to absorption and/or diffusion by replacing porcelain crucible with zirconium crucibles. However, while volatilization of zinc and lead compounds during ignition is possible, the formation of difficultly soluble residues also may be responsible for the differences in the results.

The results in Table 1 show increasing recoveries of zinc of 61.0, 64.7 and 74.1 mg, and lead of 2680, 2970 and 3220 mg from MM-SEW1 sludge in progressing from samples prepared by muffle furnace ignition to those prepared by nitric acid digestion in high-pressure decomposition vessels. Barnas (1973, 1976) has reported on the use of high-pressure decomposition vessels for the preparation of inorganic and organic materials prior to elemental analysis by atomic absorption spectrometry. The zinc and lead levels determined for the sludge sample from MM-SEW2 shows a similar progression. Using the results obtained with the samples digested nitric acid in the pressure vessel as a reference point, relative recoveries were calculated for the other procedures. These values are presented in Table 2.

Inspection of Table 2 shows that the losses of zinc and lead greater than 10% occur when muffle furnace ignition (Procedure 1) or digestion with nitric acid in open beakers (Procedure 2) is used to prepare sludge samples for atomic absorption spectrometry.

### Conclusion

Consequently, digestion with nitric acid and Hydrogen peroxide (Procedure 4) and the use of high-pressure decomposition vessels (Procedure 3) are

considered more efficient for this purpose. Among the advantages in using these devices are low blanks and high recoveries. Digestions in pressure vessels require less acid than digestions in open containers. Therefore, lesser amounts of impurities from the reagents are added to the samples. The pressure vessels reduce volatilization losses and promote mineralization.

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