

# Validation of Atomic Absorption Spectroscopy (AAS) for Trace Elements Analysis of Environmental Samples

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## Abstract

*The investigation focused on the validation of the methodology used in determining the presence of specific trace elements in environmental soil concern. Atomic Absorption Spectroscopy (AAS) available in Gombe State University, Nigeria was employed in analyzing the elemental concentrations on the certified values in three reference materials obtained from the International Atomic Energy Agency (IAEA) and National Institute of Standards & Technology (NIST). The elements determined were Cd, Co, Cr, Cu, Mg, Mn, Ni, and Pb. The percentage recovery of the measured trace elements concentrations were found within the range of 90–100% of the certified values for most of the elements; the machine was calibrated using standard solution.*

**Keywords:** AAS, Gombe, reference materials, trace elements, percentage recovery

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## INTRODUCTION

Trace elements may be either essential or nonessential, the key elements are those that are required by an organism to maintain its normal physiological function [1]. The significance of trace elements in human health and disease has been reviewed in numerous scientific publications [2, 3]. This definition of essential elements is based mainly on the observation that inadequate intake causes impairment of human health which can be reversed by administering the elements. Nonessential trace elements are considered toxic and are not required for physiological processes, but essential elements can also cause toxicity when concentrated at excessive levels [4]. Without essential elements the organism cannot complete its normal life cycle or achieve normal healthy growth [5].

Presently an increasing interest has been manifested in the determination of trace elements in many fields of science and technology. It is highly demanded to investigate the machine being used in determining the trace elements so that to avoid given wrong data about the concentration of

trace elements. In this context, Atomic Absorption Spectrometry (AAS) technique plays an important role due to its well-established advantages; e.g. significant accuracy and well documented to support field decision making with reduced overall cost, greater sensitivity and detection limits than other method and very small sample is required [6, 7]. In one application of this method, the samples to be analyzed are Certified Reference Material (CRM) and Standard Solution (SS) that is prepared according to the manufacturer's manual. The CRMs used in this work were obtained from the National Institute of Standards and Technology (NIST) and the International Atomic Energy Agency (IAEA). The CRM is selected to have a matrix that is reasonably similar to that of the samples [8].

Validation of analytical methods is a subject of considerable interest and it is the process of determining the suitability of methodology of providing useful analytical data [9]. The data obtained are useful in evaluating the uncertainty associated with measurements [10]. The focus here was to validate the AAS

methodology by determining the concentrations of elements in different Reference Materials (RMs) and the SS. In this paper, the measured concentrations of elements in three Standard Reference Materials (SRMs)—IAEA SOIL-7, NIST 1633b and 1633c [11, 12]—were presented and the recovery percentage were evaluated from certified and experimental values.

## MATERIALS AND METHODS

The above-mentioned reference materials (IAEA SOIL-7, NIST 1633b and 1633c) were utilized as a source of trace element, and SS were also prepared to investigate the validity of AAS in Gombe State University, Gombe, Nigeria. All the reagents used were of the analytical standard.

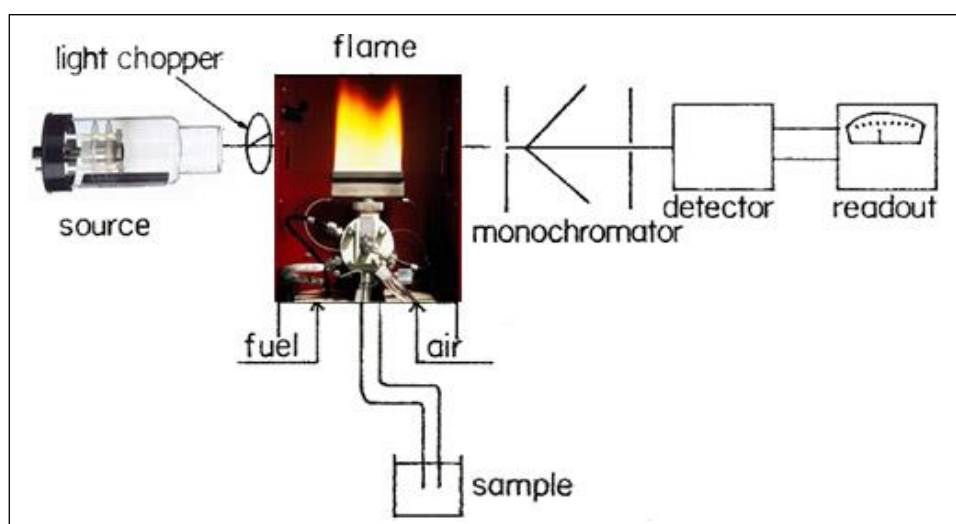
SS of eight metals (Cd, Co, Cr, Cu, Mg, Mn, Ni, and Pb) were prepared by diluting AAS standard solutions (1000 ppm) of 0.1 ml of each SS; the solution were taken into 100 ml flask and made up to the mark with 2% HNO<sub>3</sub>. The concentrations of trace elements in the soil of the sample were determined using the AAS method. The samples were prepared in the same way as the readiness indicates for the AAS. Care was taken during the sampling preparation to minimize the contamination of the SS and SRMs due to excessive direct contact [13], which could give rise to errors in the measured concentration of the trace elements.

The standards were prepared in an identical manner with the samples to reduce matrix

effect, as well as uniform absorption [14]. Three standard reference materials—IAEA SOIL-7, NIST 1633b and NIST 1633C—were analyzed using the AAS facility of Gombe State University, Gombe, Nigeria.

In order to make measurements using AAS it is necessary to produce a population of ground state atoms as efficiently as possible and pass resonance radiation of the element to be measured through the population of atoms [15]. In an ideal system the light-measuring end of the system should only measure at the wavelength being absorbed, as any other radiation sensed decreases the sensitivity of measurement [16]. The layout of a basic flame AAS is shown in Figure 1.

Light from a line source of characteristic wavelength for the element being determined passes through the flame (which is typically wide [4–6 inches], giving a reasonably long pathlength for detecting small concentrations of atoms) into which has been sprayed on the sample solution. The region of the spectrum to be measured is selected by a monochromator. The isolated spectral line falls on the photomultiplier, the detector and the output is amplified and sent to a readout device meter, analogue, or digital, through a computer data processing system, to a chart recorder, digital display unit or printer. The intensity of the resonance line is measured twice, once with the sample in the flame and once without. The ratio of the two readings is a measure of the amount of absorption, hence the amount of element in the sample [17].



*Fig. 1: Basic Flame Spectrometer.*

### Calibration of Standard Solution

SSs of eight samples (Cu, Cd, Co, Pb, Ni, Mn, Cr and Mg) were prepared according to the manufacturer procedure for AAS to be used [18]. Known 1000 mg/l concentration of the metal solution was prepared from their salts. The reagents include nitric acid (HNO<sub>3</sub>) and hydrochloric acid (HCl). The calibration was performed using five or six aqueous SSs in 2% of HNO<sub>3</sub> or HCl depending on the specification of the element. The calibration SSs had the folding concentrations: 0.0, 1.0, 2.0, 3.0, 4.0 and 5.0 mg/l (Cu, Co, Cr, and Pb, respectively) and 0.0, 1.0, 2.0, 3.0, and 4.0 mg/l (Ni) and 0.0, 0.4, 0.8, 1.2, 1.6 and 2.0

mg/l (Cd and Mn) 0.0, 0.4, 0.8, 1.2, and 1.5 mg/l (Mg) for AAS calibration.

### RESULTS AND DISCUSSION

The results of the absorbance from the SSs with their corresponding concentrations are shown in Table 1. Linear graph relationships for the results were plotted and the R<sup>2</sup>-values obtained for most of the results was 99%. The concentration of the following elements: Cd, Co, Cr, Cu, Mg, Mn, Ni, and Pb, were reinvestigated (IAEA SOIL-7, NIST 1633b and NIST 1633c) and the values were compared with literature values of RMs (Table 2).

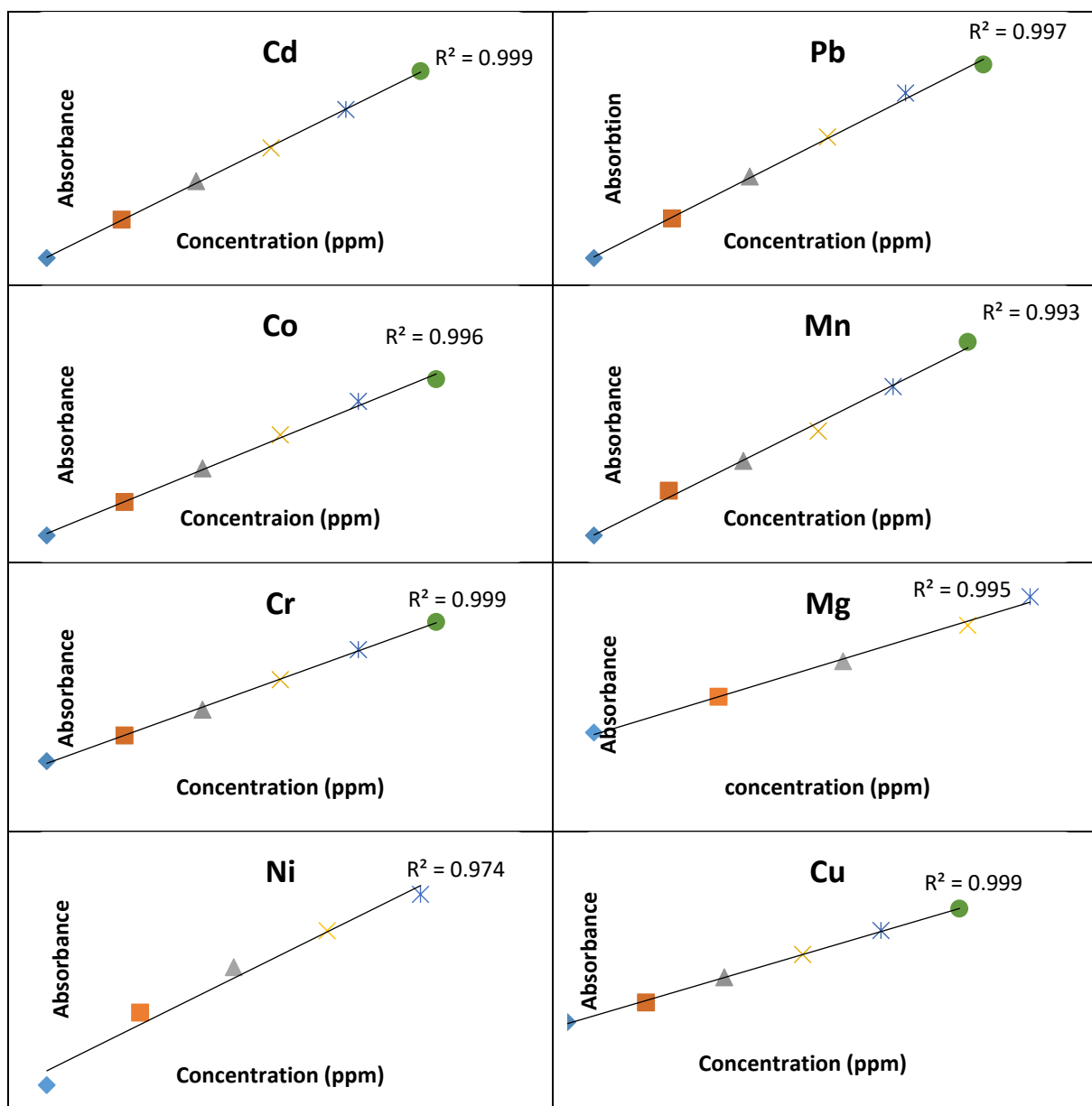


Fig. 2: Relationship Between Absorbance and Concentration.

**Table 1: Results of Absorbance and Standard.**

Sample	Ni		Cd		Mn		Mg		Cu		Co		Cr		Pb	
	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.	Con. (ppm)	Abs.
S0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
S1	1	0.08	0.4	0.14	0.4	0.003	0.4	0.25	1	0.18	1	0.03	1	0.12	1	0.18
S2	2	0.13	0.8	0.28	0.8	0.005	0.8	0.5	2	0.41	2	0.06	2	0.24	2	0.37
S3	3	0.17	1.2	0.4	1.2	0.007	1.2	0.75	3	0.62	3	0.09	3	0.38	3	0.55
S4	4	0.21	1.6	0.54	1.6	0.01	1.4	0.95	4	0.84	4	0.12	4	0.52	4	0.75
S5			2	0.68	2	0.013	1.5	0.98	5	1.04	5	0.14	5	0.65	5	0.88

(Note: Con. = concentration, Abs. = absorbance, ppm = part per million)

Table 1 gives the concentration of the elements used in the experiment and the corresponding absorption determined from the AAS. The absorbance was plotted against the concentration of each trace elements and the R-square value for each element was determined as shown in the Figure 2.

Figure 2 indicated that in most of the cases the R-square value for the concentration and the absorbance was about 99% which shows that the correlation between them is very significant [19] and the AAS can be used for any analysis in the determination of trace elements in any sample.

### The Quality Control

The quality control of this experiment was conducted using three different reference material (IAEA SOIL-7, NIST 1633b [Coal Fly Ash] and NIST 1633c [Coal Fly Ash]). Tables 2(a–c) shows the literature values with the corresponding experimental results.

**Table 2a: IAEA SOIL-7.**

Elements	Literature values	This work	% recovery
Cd	1.3	1	76.9
Co	8.9	7.5	84.7
Cr	60	50.55	84.3
Cu	11	8.5	77.3
Mg	1130	ND	0
Mn	631	400	63.4
Ni	ND	ND	100
Pb	60	5.5	91.7

**Table 2b: NIST 1633b (Coal Fly Ash).**

Elements	Literature values	This work	% recovery
Cd	0.784	0.7	89.3
Co	6.3	6.2	98.4
Cr	198.2	196.4	99.1
Cu	112.8	110.2	97.7
Mg	0.482	0.395	82
Mn	131.8	125.1	94.9
Ni	120.6	ND	0
Pb	68.2	66	96.7

**Table 2c: NIST 1633c (Coal Fly Ash).**

Elements	Literature values	This work	% recovery
Cd	0.758	0.75	98.9
Co	42.9	43	100
Cr	258	258	100
Cu	173.7	163	93.8
Mg	0.498	0.3	60.2
Mn	240.2	237.5	98.9
Ni	121	120	99.2
Pb	95.2	90.1	94.6

The quality assurance results (Tables 2a, 2b and 2c) shows that the determinations for most of the elements were in good agreement with the SRMs literature values. The values of the percentage recovery for most of the elements were above 90% in NIST 1633 and NIST 1633c and this is within the permissible limits [20]; but Ni was not detected in NIST 1633b. Most of the elements determined in IAEA SOIL-7 were below 90%. This might be due to the uncertainty in literature values or the concentration was degraded due to long time storage of the soil for over a decade [21].

### CONCLUSIONS

This work investigates the validation of AAS methodology at the Biochemistry Department, Gombe State University, Gombe, Nigeria by determining the elemental concentrations in RMs of different matrices and the SS. Concentrations of Cd, Co, Cr, Cu, Mg, Mn, Ni, and Pb, were determined from SRM and SS, and were found similar to those obtained elsewhere with respect to the IAEA and NIST.

The measured concentrations were in good agreement with the certified reported values in general. Therefore, the results shows that AAS analytical method can be used for determining concentration of trace elements in environmental samples.

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