

## Reactor Neutron Activation Analysis of Lichens by $k_0$ -NAA Standardization method

B.J.B. Nyarko<sup>1,\*</sup>, Y. Serfor-Armah<sup>1</sup>, E.H.K. Akaho<sup>1</sup>, A.W.K. Kyere<sup>1</sup>,  
G. T. Odamtten<sup>2</sup>

<sup>1</sup>National Nuclear Research Institute, Ghana Atomic Energy Commission,  
P.O. Box LG 80, Legon-Accra, Ghana

<sup>2</sup>Botany Department, University of Ghana, Legon-Accra, Ghana

\*Correspondence Author E-mail: [bjbnyarko@yahoo.co.uk](mailto:bjbnyarko@yahoo.co.uk)

### Abstract

Mining activities contribute immensely to trace element atmospheric pollution. In Ghana, air pollution due to gold mining is the least investigated. In order to obtain preliminary information on air quality in the mining areas, the  $k_0$ -standardization neutron activation analysis (NAA) method was used to analyse lichen samples from Prestea and its surrounding villages. The computed EPI values of both the Hogdahl convention and the modified Westcott-formalism with gold as comparator standards, was used to analyse IAEA lichen 336 certified reference material (CRM) and the lichen samples. The samples were irradiated in one of the inner irradiation site of Ghana Research Reactor-1 (GHARR-1) operating at a thermal neutron flux of  $5 \times 10^{11} \text{ n cm}^{-2} \text{ s}^{-1}$ . Comparison of the results with samples obtained from non-mining area, indicates that values of the suspected heavy metals such as As, Cr, Fe, Sb, and V were far higher than the background values; signifying accumulation of these heavy metals due to gold mining activities. The accuracy and precision of the method has been evaluated.

**Keywords:** Neutron Activation Analysis, Biomonitoring,  $k_0$  Standardization, Heavy metals, Pollution, Gold mining, Lichens.

### Introduction

Heavy metals contribute one of the most dangerous groups of anthropogenic environmental pollutants due to their potential toxicity and their persistence in the environment. Evaluation of the levels of these heavy metal depositions is therefore

very vital. One of the major anthropogenic activities in Ghana with potential environmental hazards is gold mining activity.

In view of the continuing exploitation of the environment by gold mining activities in Ghana, determination of levels of heavy metals in the environment resulting from these mining activities has become critical. Some of the gold bearing ores in Ghana are known to be pyrites (FeS) and arsenopyrites (FeSAs)[1-3]. During the processing of the ores for the gold metal, poisonous substances such as oxides and sulphides of heavy metal pollutants are released into the atmosphere.

Prestea, one of the oldest gold mining centers in Ghana, is situated in the Wassa West district of the western Province of the country. Gold mining activities in and around the township started during the colonial era. Even though, some work has been done in the field of toxic element determination in gold tailing, streams and river bodies, there is no known report of heavy metal pollution in the atmosphere.

Systematic surveys of atmospheric deposition of heavy metals have been performed in several countries using lichen biomonitors [4-8]. Lichens are slow-growing associations of fungi (mycobionts and green algae or cyanobacteria (photobionts)). This symbiotic association forms a common thallus that does not possess roots or waxy cuticles and depends mainly on atmospheric inputs of mineral nutrients. These features of lichens, combined with their extraordinary capability to grow at a large geographical range and to accumulate mineral elements far above their needs, rank them among the best bioindicators of air pollution [9].

In this work, lichen *parmelia in situ* samples collected from Prestea township and surrounding villages have been used to estimate the effect of gold mining activities on the environment using reactor thermal neutrons and the  $k_0$  neutron activation analysis standardization method.

## Theory

De Corte et al, [10] investigated the possibility of transition from Hogdahl convention to the Westcott-formalism by introducing a factor EPI for calculating the concentration of elements, which is defined as:

$$EPI = \frac{\sigma_{\text{eff}}^*/\sigma_0^*}{\sigma_{\text{eff}}^x/\sigma_0^x} \dots \dots \dots (1)$$

Where the superscripts \* and x denote the values for the monitor  $^{197}\text{Au}$  and the nuclide of interest respectively.

The definition of  $\sigma_{\text{eff}}$  according to Hogdahl-convention is  $\sigma_{\text{eff}} = [f+Q_0(\alpha)]$

Where  $f = \Phi_{\text{th}}/\Phi_{\text{epi}}$  is the ratio of sub-cadmium ( $\Phi_{\text{th}}$ ) to epi-cadmium flux ( $\Phi_{\text{epi}}$ )

$\alpha$  = epithermal flux distribution parameter

$\sigma_0$  = thermal neutron activation cross section for the energy 0.0253 eV and

$Q_0 = I_0(\alpha)/\sigma_0$  with  $I_0(\alpha)$  as modified resonance integral.

According to modified Westcott-formalism,

$$\sigma_{\text{eff}} = \sigma_0(g(T_n)) + r(\alpha)\sqrt{T_n/T_0}S_0(\alpha) \dots \dots \dots (2)$$

Where

$r(\alpha)\sqrt{T_n/T_0}$  = Modified spectra index (MSI)

$g(T_n)$  = Westcott g-factor

$S_0(\alpha) = s_0 E_r^{-\alpha}$  ( with  $s_0 = 2\pi^{-1/2} Q_0$  ) = modified reduced resonance integral

$T_n$  is the neutron temperature

The flux parameter  $r(\alpha)\sqrt{T_n/T_0}$  for any irradiation site is obtained experimentally using either the “cadmium ratio” or the “bare bi-isotopic monitor” techniques [11]

The expression for calculating the concentration of elements in any sample using the  $k_0$ -NAA method is written in the form [10]

$$\rho(\text{g/g}) = \left[ \frac{\left( \frac{N_p/t_m}{\text{SCDW}} \right)^x}{\left( \frac{N_p/t_m}{\text{SCDW}} \right)^*} \right] \cdot \frac{1}{k_0} \cdot \text{EPI} \cdot \frac{\varepsilon_p^*}{\varepsilon_p^x} \quad (3)$$

Where

$\rho$  is the concentration

$N_p$  is the measured peak area corrected for pulse losses

$t_m$  is the measuring time

$\varepsilon_p$  is the peak efficiency

$S = 1 - \exp(-\lambda t_i)$  is the saturation factor with  $\lambda$  as the decay constant and  $t_i$  as the irradiation time

$D = \exp(-\lambda t_d)$  is the decay factor with  $t_d$  as the decay time

$C = [1 - \exp(-\lambda t_m)] / \lambda$  is the counting factor

$W$  is the sample mass of  $x$

$w$  is the weight of the monitor\*. The definition of the  $k_0$ -factor, which is nuclear constant, is

$$k_0 = \frac{M^* \theta^x I_\gamma^x \sigma_0^x}{M^x \theta^* I_\gamma^x \sigma_0^*} \quad (4)$$

where

$M$  is the molar mass

$\theta$  is the isotopic abundance

$I_\gamma$  is the absolute gamma-ray intensity and

$\sigma_0$  is the  $2200\text{ms}^{-1}$  ( $n, \gamma$ ) cross section

The definition of the EPI factor in accordance with equation 1 is dependent on the flux parameters of the formalism used for the prediction of the reaction rate. For the Hogdahl convention, the factor EPI is written in terms of the flux parameters  $f$  and  $\alpha$ .

$$\text{EPI}_H = \frac{f + Q_0^*(\alpha)}{f + Q_0^x(\alpha)} \quad (5)$$

where

$Q_0(\alpha)$  is defined as:

$$Q_0(\alpha) = \frac{Q_0 - 0.429}{E_r^\alpha} + \frac{0.429}{(2\alpha + 1)E_{cd}^\alpha} \quad (6)$$

where

$E_r$  is the effective resonance energy (eV) and  $E_{cd} = 0.55\text{eV}$

For the use of the modified Westcott formalism, the EPI factor becomes:

$$EPI_w = \frac{g * (T_n) + r(\alpha)\sqrt{T_n/T_0} \cdot S_0(\alpha)}{g_x(T_n) + r(\alpha)\sqrt{T_n/T_0} \cdot S_0(\alpha)} \quad (7)$$

The computed EPI values for both Hogdahl convention and the Westcott formalism together with the  $k_0$  values and the nuclear data used in this study were taken from ref [14-15] and are shown in table 1. The  $f$ ,  $\alpha$ ,  $T_n$  and MSI values for the irradiation sites were obtained by Akaho and Nyarko [13,14].

**Table 1.** Nuclear Data.

Target Isotope	Formed Isotope	Half-life ( $T_{1/2}$ )	$\gamma$ -ray energy keV	Ko- Values	EPI <sub>w</sub>	EPI <sub>H</sub>
<sup>27</sup> Al	<sup>28</sup> Al	2.24 m	1779	1.75X10 <sup>-2</sup>	1.8462	1.8461
<sup>75</sup> As	<sup>76</sup> As	26.32 h	559	4.97X10 <sup>-2</sup>	0.9266	0.9266
<sup>81</sup> Br	<sup>82</sup> Br	35.3 h	776	2.76X10 <sup>-2</sup>	0.7484	0.7484
<sup>50</sup> Cr	<sup>51</sup> Cr	27.7 d	320	2.62X10 <sup>-3</sup>	1.9179	1.9178
<sup>37</sup> Cl	<sup>38</sup> Cl	37.2 d	1642	1.97X10 <sup>-3</sup>	1.8699	1.8699
<sup>58</sup> Fe	<sup>59</sup> Fe	44.5 d	1099	7.77X10 <sup>-5</sup>	1.8471	1.8470
<sup>41</sup> K	<sup>42</sup> K	12.36 h	1525	9.46X10 <sup>-4</sup>	1.7940	1.7940
<sup>55</sup> Mn	<sup>56</sup> Mn	2.58 h	847	4.96X10 <sup>-1</sup>	1.8140	1.8140
<sup>23</sup> Na	<sup>24</sup> Na	14.66 h	1369	4.68X10 <sup>-2</sup>	1.8847	1.8846
<sup>121</sup> Sb	<sup>122</sup> Sb	2.72 d	564	4.38X10 <sup>-2</sup>	0.6136	0.6138
<sup>50</sup> Ti	<sup>51</sup> Ti	5.76 m	320	3.74X10 <sup>-4</sup>	1.8536	1.8535
<sup>51</sup> V	<sup>52</sup> V	3.75 m	1434	1.96X10 <sup>-1</sup>	1.8915	1.9150
<sup>64</sup> Zn	<sup>65</sup> Zn	244 d	1116	5.72X10 <sup>-3</sup>	1.6758	1.6758
<sup>164</sup> Dy	<sup>165</sup> Dy	2.33 h	94.7	3.57X10 <sup>-1</sup>	1.9614	1.9613
<sup>138</sup> Ba	<sup>139</sup> Ba	83.1 m	165	1.05X10 <sup>-3</sup>	1.8210	1.8208
<sup>151</sup> Eu	<sup>152</sup> Eu	13.5 y	344	10.28	1.8753	1.8750
<sup>176</sup> Lu	<sup>177</sup> Lu	6.71 d	208	1.65X10 <sup>-2</sup>	1.8592	1.8592

## Experimental

### Sampling

Lichen samples were obtained from Prestea and its surrounding villages and from Akim Tafo (control area) from May 2002 to February 2003. Figure 1 shows the

sampling points in and around Prestea. The samples were taken from bark of trees about 1.0 m above ground level.

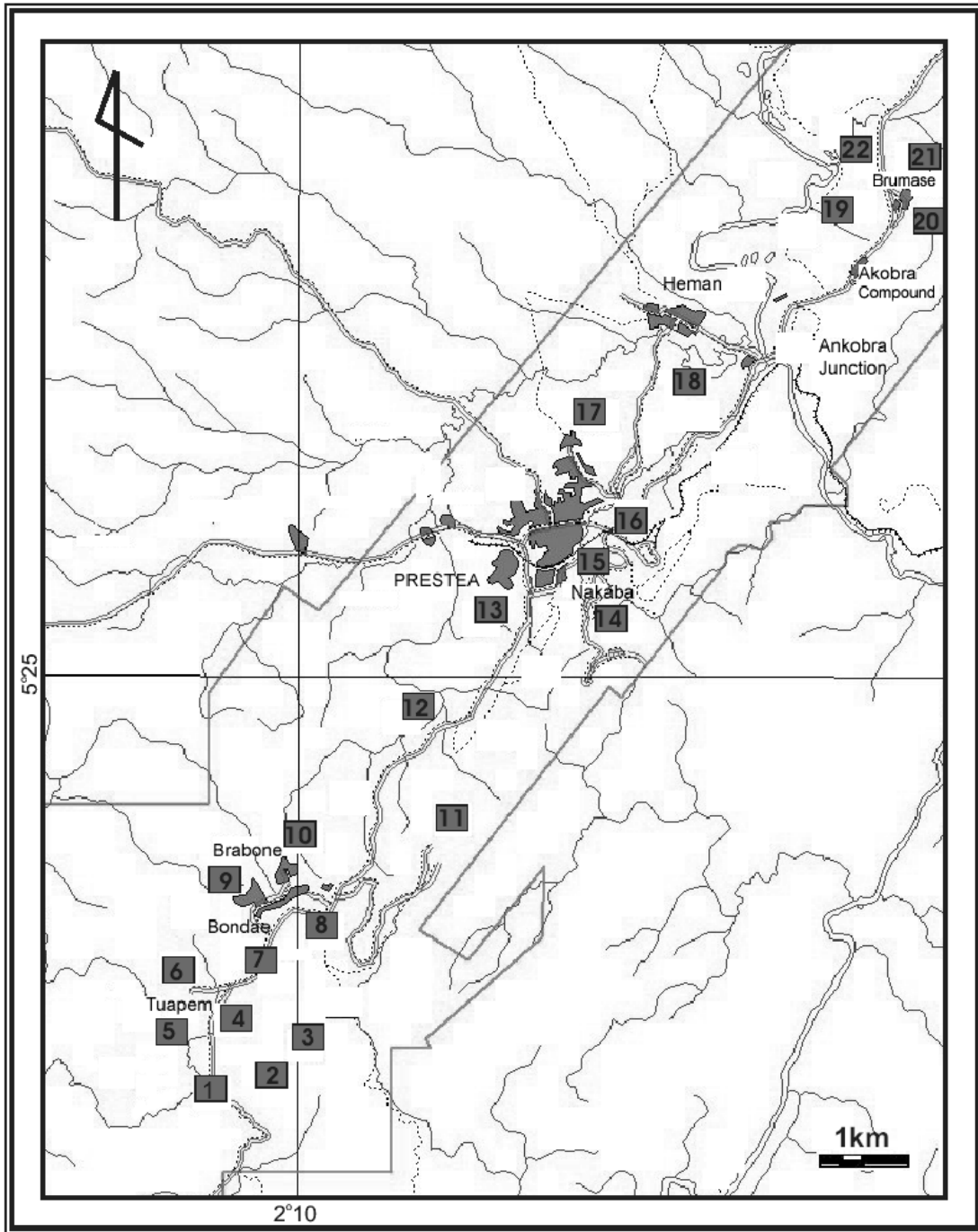


Figure 1: Map of Prestea showing sampling sites.

### **Sample preparation**

The lichen samples were carefully removed from the bark of the trees with stainless steel knife. The samples were then washed with deionised water, air dried in the laboratory and grounded into fine powder using an agate mortar and homogenised. Fifteen replicate samples of the lichen samples and the standard reference material IAEA lichen 336 were prepared. Five replicate samples each for short, medium and long irradiation were prepared. Between 150 –200 mg were prepared, wrapped in a thin polyethylene capsule and heat-sealed. They were then packed in 7ml polyethylene capsules and heat-sealed. The elemental gold comparator standard used in this work was made from plasma emission spectroscopy standard solutions supplied by Sigma chemical products (SCP) Canada Ltd. The standard has certified purity of 99.999% and has a concentration of 1000 ppm. A working stock solution of 10 ppm was prepared by dilution. A volume of 100 µl was taken from the working standard solution and pipette into a small 1.2 ml polyethylene vial half filled with finely grounded sucrose (obtained from Koch Light Laboratories, USA). Ten replicate standards were prepared. Few drops of deionised, distilled water, DDW were added to form a homogenous mixture and air dried before heat-sealing the cap.

### **Irradiation and Counting**

Irradiation of the samples was done using the GHARR-1 reactor operating at half full power of 15 kW with a neutron flux of  $5 \times 10^{11} \text{ ncm}^{-2} \text{ s}^{-1}$ . The scheme for irradiation was chosen according to the half-lives of the element of interest. For the short-lived elements with half-lives up to 20 minutes, irradiation time was 60 seconds. For the medium-lived elements, i.e., those with half-lives between 20 minutes and 3 days, the irradiation times were limited to 10 and 60 minutes. For the long-lived, that is elements with half-lives greater than 3 days, the irradiation time was 6 hours. The samples were sent into the reactor by means of a pneumatic transfer system. At the end of the irradiation, the capsules were returned from the reactor and allowed to cool down until the level of activity was within the acceptable limit for handling. The samples were then placed on top of an N-type High Purity Germanium Detector (HPGe) and the counts were accumulated for a pre-selected time to obtain spectra intensities. For the short and medium irradiation, 600s counting time was chosen. For the long irradiation, counting time ranged between 2 and 20 hours.

### **Qualitative and Quantitative analysis**

The PC-based gamma ray spectroscopy system was used for the measurement. The spectroscopy system consisted of an N-type HPGe detector model GR 2518, a HV power supply model 3103, a spectroscopy amplifier model 2020 and an ACCUSPEC multi-channel analyser (MCA) emulation software card ( all manufactured by Canberra Industries Inc.). The efficiency of the detector is 25%. It operates in a bias voltage of (-3000V) and has a resolution of 1.8 keV for  $^{60}\text{Co}$  gamma ray energy of 1332 keV. The spectra intensities for the samples were obtained by means of the MCA card. The samples and the comparator standard were counted at a distance of 7.2 cm from the top of the detector surface to quantify the elements. The concentrations of the elements were obtained using equation (3). The full energy

efficiency determination at this position for use was achieved by using IAEA mixed radionuclides standard solution containing  $^{241}\text{Am}$ ,  $^{109}\text{Cd}$ ,  $^{57}\text{Co}$ ,  $^{203}\text{Hg}$ ,  $^{113}\text{Sn}$ ,  $^{85}\text{Sr}$ ,  $^{137}\text{Cs}$ ,  $^{65}\text{Zn}$  and  $^{60}\text{Co}$ . The Efficiency parameters for the NAA calculation were obtained by fitting the efficiency curve with a polynomial function [15].

## Results And Discussions

The computed EPI values of Hogdahl Convention and the Westcott formalism are shown in table 1. It can be seen that there were little variations between the EPI values. Table 2 shows the analysis of IAEA 336 lichen certified reference material. The precision was calculated as a percentage relative standard deviation of five replicate measurements and was found to be within 10%.

In order to evaluate the pollution status, the index pollution (IP) factor was employed. [16]

$$IP = C_s / C_c \quad (8)$$

where

$C_s$  is the average concentration of element in the samples

$C_c$  is the average concentration of elements in the control samples.

**Table 2:** Analytical Results and Reported values of IAEA 336 Lichen Certified Reference Material ( $\mu\text{g/g}$ ).

Element	This work	Reported value	Element	This Work	Reported value
Al	$675 \pm 49$	680 (570 – 790)	K	$1850 \pm 60$	1840 (1640 – 2040)
As	$0.65 \pm 0.02$	0.63 (0.55 – 0.71)	Mn	$63.3 \pm 10$	63 (50 – 70)
Br	$130 \pm 3.9$	12.9 (11.2 – 14.6)	Th	$0.12 \pm 0.0020$	0.14(0.12-0.16)
Dy	$4.0 \pm 0.32$	3.9 (3.2 – 5.3)	V	$1.48 \pm 0.04$	1.47(1.25-1.69)
Eu	$1.12 \pm 0.06$	1.0 (0.9 – 1.3)	Zn	$32.2 \pm 1.5$	30.4(27.0-33.8)
Cl	$1910 \pm 20$	1900 (1600 – 2200)	Na	$3235 \pm 3.7$	320 (280 – 360)
Cr	$0.98 \pm 0.02$	1.06 (0.89 – 1.23)	Sb	$0.074 \pm 0.008$	0.073 (.063 – .083)
Fe	$429 \pm 30$	430 (380 – 480)	Ti	$3300 \pm 170$	3000(2600–37000)
Ba	$6.9 \pm 0.9$	6.4 (5.3 – 7.5)			

All the elements identified were normalised by the control. If the IP factor is less than 1.2, it is referred to as grade 1, clean area. If the IP is in the range of 1.2 – 2, it is a pollution grade II, a light polluted area. If the IP factor is in the range of 2 to 3, it is considered pollution grade III, a medium polluted area. If the IP factor is greater than 3, the area is considered heavy pollution area, grade IV [16]. The concentrations of seventeen elements from fifteen sampling points in and around Prestea township and

**Table 3.** Elemental concentrations ( $\mu\text{g}$ ) in lichen samples of 15 sampling sites and their IP factors

Element	Control	SITE 4		SITE 6		SITE 8		SITE 9		SITE 10		SITE 11		SITE 12	
		Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF
Al	2500	6700	2.68	2567	2.63	22960	9.18	26720	10.7	18410	7.36	14260	5.70	7279	2.91
As	0.78	55.0	70.5	51.0	65.4	168	215	201	258	122	156	89.7	115	52.9	67.8
Br	5.50	25.2	4.58	6.5	1.18	10.7	1.95	1108	2.09	9.5	1.73	24.9	5.53	7.5	1.36
Ct	2.20	20.0	9.09	9.6	4.36	36.9	16.8	38.5	17.5	13.4	6.09	60.7	3.05	6.71	3.05
Cl	240	735	3.06	12.0	0.05	318	1.33	330	1.38	214	0.89	326	1.36	3.75	1.56
Fe	1050	4650	4.43	5920	5.64	21190	20.18	25700	24.5	2770	2.64	2676	2.55	3760	3.58
K	3250	6200	1.91	3630	1.12	9350	2.88	7650	2.35	4560	1.40	3692	1.14	37800	1.16
Mn	560	151	0.27	60.1	0.11	175	0.31	160	0.29	84	0.15	324	0.58	471	0.84
Na	165	250	1.52	785	4.76	2560	15.5	3100	18.79	1350	8.18	950	5.76	169	1.02
Sb	0.33	1.23	3.73	1.22	3.70	7.22	21.9	4.2	12.7	4.25	12.88	1.46	4.42	1.32	4.00
Ti	221	260	1.18	577	2.6	1773	8.02	2420	11.0	1415	6.40	1622	7.34	600	2.71
V	2.20	14.4	6.55	12.0	5.45	47.8	21.7	51.9	23.6	32.6	14.82	20.4	9.27	13.3	6.05
Zn	156	166	1.06	154	0.99	110	0.71	118	0.76	210	1.35	138	0.88	225	1.44
Dy	0.86	Nd	Nd	0.44	0.51	1.10	1.28	1.35	1.57	0.75	0.87	Nd	Nd	1.98	2.3
Ba	120	Nd	Nd	74.3	0.62	105	0.88	172	1.43	103	0.85	Nd	Nd	108	0.90
Eu	0.31	0.18	0.58	0.12	0.39	0.38	1.23	0.38	1.23	0.11	0.35	0.05	0.16	0.07	0.23
Lu	0.14	0.15	1.02	0.13	0.93	0.31	2.21	0.24	1.71	0.17	1.2	0.07	0.50	0.11	0.79

Element	SITE 13	SITE 14		SITE 15		SITE 16		SITE 17		SITE 18		SITE 20		SITE 22	
		Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF	Conc.	IPF
Al	5640	2.26	14260	5.70	18300	7.32	8810	3.52	1340	0.54	1830	0.73	2940	1.18	2618
As	51.3	65.8	98.0	126	120	154	47.0	60.3	14.0	17.9	3.36	4.31	11.0	14.10	1807
Br	18.6	3.38	23.0	4.18	5.5	1.0	5.9	1.07	4.2	0.76	15.5	2.82	5.7	1.04	8.1
Cr	17.7	8.05	18.5	8.41	25.0	11.36	11.2	5.09	4.1	1.86	3.78	1.72	5.5	2.50	3.72
Cl	365	1.52	326	1.36	325	1.35	1220	5.08	215	0.90	260	1.08	171	0.71	336
Fe	4870	4.64	8362	7.96	11670	11.10	6910	6.58	1050	1.00	1270	1.21	2010	1.91	4770
K	8790	2.70	5737	1.77	4850	1.49	9095	2.80	3650	1.12	2475	0.76	2880	0.89	2865
Mn	100	0.18	374	0.67	313	0.56	200	0.36	168	0.30	445	0.79	253	0.45	94.0
Na	560	3.39	946	5.73	1250	7.58	571	3.46	2350	14.2	340	2.06	280	1.70	257
Sb	0.96	2.91	1.32	4.00	4.60	13.9	1.71	5.18	0.69	2.10	0.29	0.88	1.02	3.09	1.08
Ti	483	2.91	1149	5.20	1390	6.29	850	3.85	150	0.68	110	0.50	369	1.67	411
V	10.4	4.73	23.4	10.64	29.5	13.4	14.4	6.55	2.51	0.95	2.90	1.32	4.53	2.06	4.16
Zn	33.3	0.21	139	0.89	185	1.19	231	1.48	256	1.64	65	0.42	54.5	0.35	205
Dy	1.32	1.53	Nd	Nd	1.27	1.48	0.39	0.45	0.76	0.88	Nd	Nd	1.71	1.99	Nd
Ba	112	0.93	Nd	Nd	135	1.13	Nd	Nd	110	0.92	Nd	Nd	140	1.17	Nd
Eu	0.15	0.48	0.09	0.29	0.18	0.58	0.14	0.45	0.18	0.58	0.11	0.35	0.11	0.35	0.13
Lu	Nd	Nd	0.13	0.93	0.16	1.14	0.10	0.77	0.11	0.79	0.10	0.71	0.12	0.86	0.08

control values of Akyem Tafo are shown in tables 3. It is clear from tables 3 that Prestea and its surrounding towns and villages are heavily polluted with As, Cr, Sb, V and Fe by the gold mining activities with As being the major pollutant. The IP factors for As in all the fifteen (15) sampling sites are greater than 3, indicating heavy pollution. The effect of this As pollution on the health of the people can be very vital. The most polluted Arsenic areas are sites 8,9, and 10 around the Bondae shaft area. The other highly polluted Arsenic areas are found at sites 14 and 15 near Prestea main processing plant.

Sampling points near the shaft and the processing plant have higher IP factors than those far away. Apart from As, Cr, Fe, Sb, V, Na and Ti which have almost all IP factors for all the sites greater than 1.2, the rest of the elements have almost all the IP factors less than 1.2. The IP factor for the metal pollutants decreases with increase in distance from the processing plant and the shaft, meaning the processing of the gold ore is the source of pollution.

## Conclusion

The  $k_0$  NAA standardization method using a low power research reactor has proven to be a quick and effective method for monitoring air pollution with biological indicators. The results also show that one can use either the Hogdahl convention or Westcott formalism for quantification in the  $k_0$ -NAA when using low power research reactors. There were no significant differences between the EPI values of the two conventions. Lichen samples were used as biological indicators to assess atmospheric pollution due to mining activities in and around Prestea, (a gold mining town) in Ghana. The results obtained show that, mining activities in and around the township releases heavy metal pollutant such as As, Cr, Sb, and V into the atmosphere with As being the major pollutant.

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