

# Determination of Chromium and Other Trace Elements from North Western Nigeria by Proton Induced X-ray Emission Technique

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## Abstract

Proton induced X-ray emission (PIXE) was used in the analyses of geological samples to determine accurate deposits of chromium and other trace elements. Fourteen samples were collected from sites suspected to have chromium deposits, then prepared to the required standard to be interrogated by the above technique. Samples were irradiated at Centre for Energy Research and Development, Ile-Ife, Nigeria. From the Spectra and results generated, it suggests significant deposits of Chromium in some regions of Nigeria. Gold deposit was observed to be significant in some areas. Concentrations of Pb, Si, P, S, K, Ca, Ti, V, Cr and Mn elements were determined as well.

**Keywords:** proton induced X-ray emission (PIXE), chromium

## 1. Introduction

The need for chromium in steel industries cannot be overemphasized, particularly now that the Ajaokuta Steel Industries in Nigeria is about to resume production after a long time in comatose. Chromium is a relatively common element with an average concentration of 100 ppm. It is the 21st most commonly occurring element in the earth's crust. For its production one does not require the pure metal. Pure chromium is obtained by reduction of chromium oxide with aluminium, by electrolysis, or via chromium iodide. About 10% of chromium consumed originates from recycling; mainly from scrap steel (Saager, 1984). Chromium compounds are used in the chemical industry in various fields. The metal industry uses most of the chromium in the form of master alloys, preferably in special steels (stainless steel). In the galvanizing industry other metals are coated with a chromium layer in an electrolytic process in which chromium is deposited from a sulfuric acid-chromate solution (El-Taher, 2010). Additional applications of chromium compounds are found in the following: building industry (as pigments), printing industry (photomechanical reproduction processes), oil industry (as anti-corrosives), textile industry (chromium mordant for textiles and chrome dyeing processes), match industry and fireworks (additive to the inflammable mixture). In the cassette tape industry chromium oxide is used in a specially crystallized form.

This work is concerned with the use of proton induced X-ray emission to achieve an accurate knowledge of the chromium content and other trace elements in North Western Nigeria (Ahmed, 2004).

## 2. Materials and Analytical Methods

The aim of this study was to characterize fourteen rock samples that were collected from different regions of North Western Nigeria.

Samples were crushed to small pieces using a mechanical crusher. The crushed samples were dried at 105°C to constant weight. The dried samples were ground to form fine powder. Then the powdered samples were sieved using a standard set of sieves to a diameter range of less than 125. Every powdered sample was shaken using an electric shaker to be sure that the sample was homogenized. From each of the fourteen bulk samples, pellets were made by hydraulic press (3 ton). Standards (NIST 278, BCS 355) were irradiated by thick target proton induced X-ray emission (CERD) with 2.5 MeV and 3.0 MeV proton beams accelerator for accuracy and validation. The proton beam was collimated. Characteristic X-ray were measured by three detectors, one detector was placed at 45° for PIXE, the other at 135° for Rutherford backscattering spectroscopy (RBS) and the third detector was for proton induced gamma ray emission (PIGE). Table 1 display the quality control from the standard reference

material (SRMs) (Graham et al., 2002). Thereafter the pellets were subjected to same analytical conditions as the SRMs, subsequently analyzed by GUPIX (a program for the non-linear least-squares fitting of PIXE spectra).

Table 1. Elemental concentrations (ppm) in selected reference materials using PIXE

Analyte	NIST 278 (Obsidian rock)		BCS 355 (Tin ore)	
	Reported value	This work	Reported value	This work
Si	341436	342392.3±9039	5000.0	5152.6±484
Cl	NA	609.1±140	NA	321.3±187
K	3453	3465.8±148	-NA	3215.6±562
Ca	7025	7057.4±114	2630	2688.5±528
Ti	1468	1475.9±30	3700	3693.7±76
Mn	403	378.8±15	NA	1795.6±39
Fe	14268	14321±72	170800	170282.8±255
Cu	5.9	6.4±7.3	850	848.8±35
Zn	55	56.4±17.3	590	589.5±32
Rb	127.5	127.8±21	-	-
Zr	NA	265.3±48	-	-
Cr	-	-	NA	60.7±25
As	-	-	1400	1398.5±50
Sn (K)	-	-	314200	314901.4±4881
Sn (LA)	-	-	314200	314969.2±1134
Ba	1140	1144.1±229	-	-
Ce	62.2	65±32	-	-
Cd	-	-	NA	1070±577
W	-	-	3500	3491.7±152

NA: Not analyzed

### 2.1 PIXE Calculations

The formula for calculating concentration  $[Y(Z)]$  in PIXE is given as:

$$Y(Z) = \frac{N_{av} \omega_z b_z t_z \epsilon_z^i \Omega / 4}{A_z} N_p C_Z \int_{E_0}^{E_f} \frac{\sigma_z(E) T_z(E)}{S_M(E)} dE \quad (1)$$

Where  $N_p$  is the number of protons,  $N_{av}$  Avogadro's number, and  $\sigma_z(E)$  the K-shell ionization cross section for the proton energy  $E$  corresponding to depth  $x$ . The number of K X-rays in a particular spectral line is then obtained via the fluorescence yield  $\omega_{k,z}$  and line intensity fraction  $b_{k,z}$ .

If we generalized angle  $\alpha$  and  $\theta_{TO}$  for proton impact and X-ray take off en route to the detector, the X-ray intensity from the element of the path indicated suffers a transmission factor.

When specimens are thick enough to stop the beam, then we have  $E_f = 0$  in Eq. 1. The matrix (M) effect (i.e., those due to proton slowing and X-ray attenuation) are contained in the integral, which we denoted by  $I_z(M)$  where M denotes the specimen (SP) or the standard (ST).

$$\frac{C_z(SP)}{C_z(ST)} = \frac{Y_z(SP) I_z(ST)}{Y_z(ST) I_z(SP)} \quad (2)$$

Standards are usually single elements or very simple compounds containing the elements of interest or their near neighbors in the chart of nuclides.

The merit of this ratio process is its cancellation of instrumental factors such as solid angle, efficiency, and calibration factors for charge integration (Aung, 2002). This is important given the practical difficulties in obtaining accurate knowledge of the detector's lineshape and intrinsic efficiency at the low X-ray energies characteristic of the light elements that are so often the major elements in environmental specimens (Sven, 1995).

### 3. Results and Discussion

We have reported data only for Pb, Si, P, S, K, Ca, Ti, V, Cr and Mn elements; however trace elements like Au were significantly detected in the pellets. Cr is found in all the samples; however we should be quick to point out that it is only at sites of samples C, D, I, J, k, L, M, N and Q that Cr can be mined profitably while sites A, B and D require further research to be undertaken before exploitation. Figure 2 clearly indicated energypeaks for chromium as well as other elements. Lead (Pb) was unusually above the normal in this region; that probably explains the recent epidemic of Lead poisoning in children. World Health Organization (WHO) had to intervene to arrest the scourge. From Figure 1, the three energypeak of Pb displayed in the spectra shows that Lead is quite significant compared to other elements in this pellets. Pb energypeaks were replicated in all the spectra as the one discussed above. Only in two samples that Lead was found to be below detection. Further research is recommended for the promising sites. Table 2 depicts the concentrations of the all elements alongside research errors.

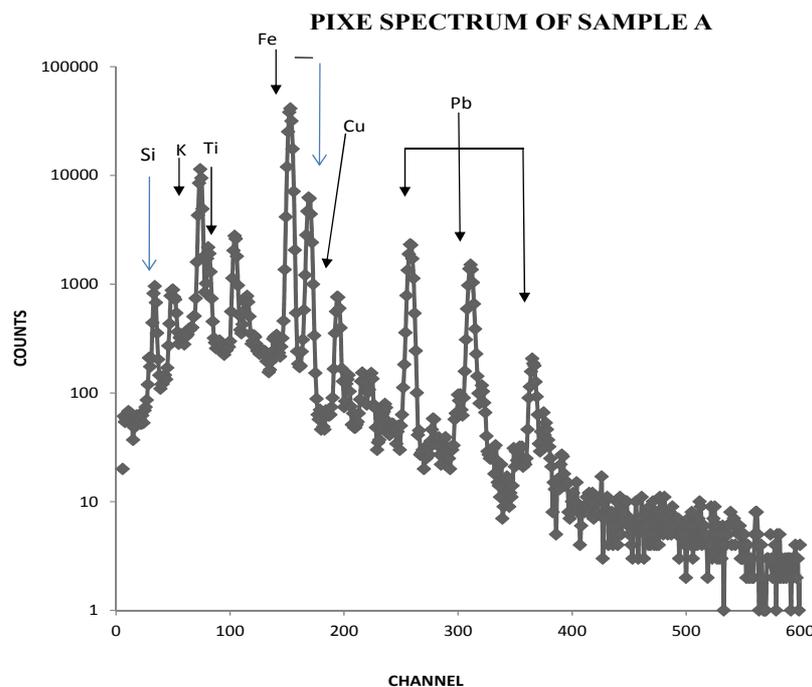


Figure 1. X-ray spectrum of Sample A (Pellet A)

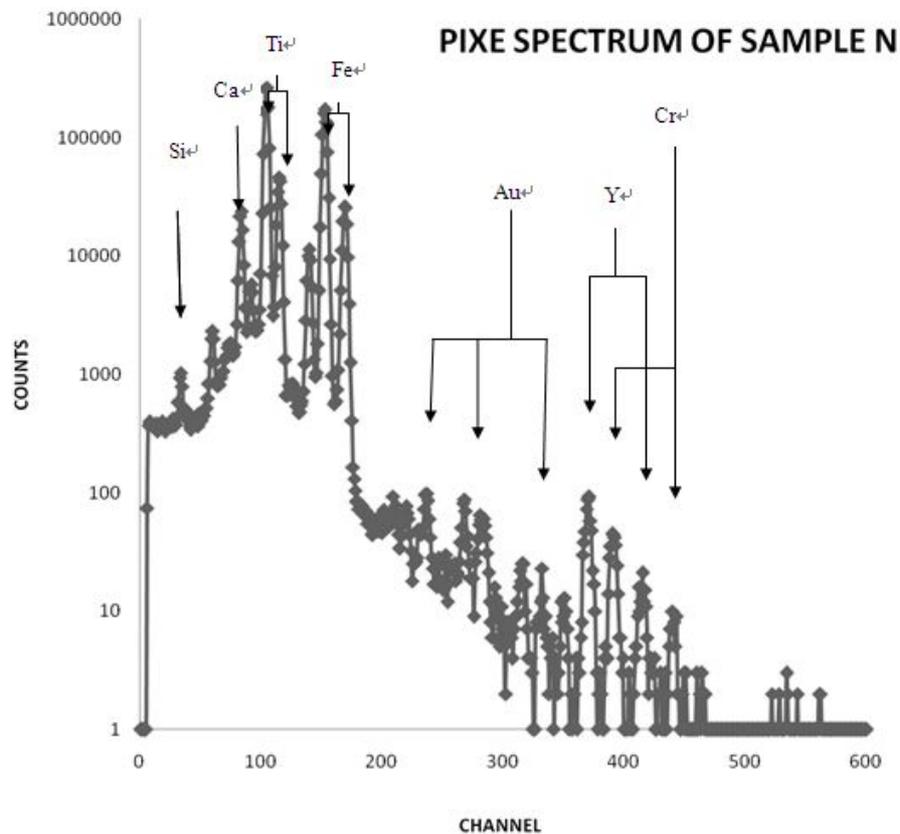


Figure 2. X-ray spectrum of sample N

Table 2. Average concentration of elements in the samples (Pellets)

	Fb	Si	P	S	K	Ca	Ti	V	Cr	Mn
A	46460.2±483	377416.5±9473	2077.8±1043	8376.2±552	20708.1±128	289.1±66	2310.8±43	74.5±29	49.2±16	188.1±18
B	15932.7±287	550391.8±9522	1586.4±730	2388±370	BDL	99.9±29	235.2±19	BDL	52.3±11	BDL
C	5379.9±170	394457.3±8047	2075.5±707	612.4±250	4525.3±55	320.2±32	819.7±23	55.4±18	403.9±17	3944.5±85
D	128.2±60	582448.6±13338	BDL	BDL	1274.2±68	31327.5±122	129814±206	BDL	260.8±22	6023.6±52
E	50332.1±438	343277.2±8033	BDL	8760.2±498	19458.1±111	208.2±62	2397.3±36	53±24	77.9±16	BDL
G	655.5±86	341083.5±12825	BDL	BDL	1401.5±60	4581.4±57	232362.9±232	BDL	346±29	11958.6±67
I	11216.7±208	365260.6±7707	1652.1±763	3850.2±322	22414.7±110	384.9±62	2905.4±33	262±24	206.3±14	113±13
J	32087.7±347	367964.5±7948	3448.4±841	5997.6±480	15251.4±96	422.5±51	2715.1±35	104.9±25	118±16	1265.9±24
K	BDL	243974.5±16249	BDL	BDL	631±68	8054±69	360244.9±288	BDL	663.1±38	20015±90
L	18591.8±268	411887.3±8082	2514.6±732	3910.4±340	10626.1±79	370±42	915.3±28	67.9±21	102.2±13	1639.8±13
M	40918±393	334759.7±7867	4704.3±922	8514±467	29259±129	323.5±76	3668.7±40	209.3±28	113.4±15	201.3±16
N	165.2±82	539773.8±16733	BDL	BDL	1281±82	44195.4±141	233525.2±280	1492.5±493	290±36	14859.6±85
P	BDL	358373.9±15088	BDL	BDL	1511.2±71	9172.8±79	231040±	BDL	295±34	10994.2±75
Q	59.2±54	169638.3±10416	BDL	BDL	681±45	8490.2±58	136234.4±164	BDL	2455.1±32	9017.9±61

From the values tabulated in Table 1, the effectiveness and accuracy of PIXE method (within  $\pm 5.0\%$ ) was evaluated and validated. Figure 1 depicts the X-ray spectrum of some of the elements in sample A in their

various channels. It can be deduced from this figure that concentrations of elements can be speculated from the heights of energypeaks.

#### 4. Conclusion

From the obtained results, it appears that PIXE can provide useful data with a satisfactory accuracy and precision. Table 2 shows that sample Q has promising concentration of Cr compared to other samples. This site is recommended for further investigations. Samples A, B and E are below the average concentration of 100ppm. Pb, Ca, Ti, and Mn are the major elements after Cr. Particularly Lead (Pb) is observed to be oddly high in some of the samples. This confirms the rife cases of Lead (Pb) poisoning being experienced in North Western Nigeria. Many children have died, and many others are suffering debilitating illnesses associated with Lead (Pb) poisoning.

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