A STATISTICAL ANALYSIS OF THE RESULTS OF THE 'XRF' ANALYSIS OF LEAD CONTAMINATED SOIL AND SEQUENTIAL EXTRACTION BASED ON T-TEST.

Piga Luigi[1]*, Adewole O[2]
*[1,2]Environmental Engineering, Università di Roma

*Corresponding Author:-
Email: tolexaddy@gmail.com

Abstract:-

A statistical analysis and hypothesis testing was carried out on the randomly selected samples from experimental results of the 'XRF' analysis and sequential extraction of soils contaminated with lead using lead nitrate of known amount.
*Looking at our previous experimental results, we found that the concentration of the solid contaminated samples: OL S 1-2, & OL S 3-2 (2,080.26, & 2,080.41mg/kg) respectively in that order are very close to the concentration of lead also obtained using the **'XRF'** analysis for the liquid solutions obtained from the sequential extraction procedures of soil samples: 'OL S 1-2, & 32 as (2,110mg/kg, & 2,090mg/kg) respectively in that order.*
An hypothesis testing was conducted within the underlying assumptions based on the null hypothesis and our claim for equal means.
The p-value found was greater than the α-value, and also corroborating this fact, the t-statistics calculated absolute value is less than the critical value at the 5% significant level as presented and shown in our table of results.
We can quite well agree from our previous results findings, and corroborated by the findings of the statistical testing, we can confident agree the results from the 'XRF' are reliable enough, without any need to do the sequential extraction for obtaining the concentrations.

Keywords: - *'Sequential extraction, lead contaminated soil & transport, 'XRF' analysis, Hypothesis testing, t-test.*

1. INTRODUCTION

Theoretical Framework *

To describe EDTA (Ethylene diamine tetra acetic acid) and its various protonated forms, chemists distinguish between $EDTA^{4-}$, the conjugate base that is the ligand, and H_4EDTA, the precursor to that ligand. At very low pH (very acidic conditions) the fully protonated H_6EDTA^{2+} form predominates, whereas at very high pH or very basic condition, the fully deprotonated $EDTA^{4-}$ form is prevalent. In this case, the term EDTA isused to mean $H_{4-x}EDTA^{x-}$, whereas in its complexes $EDTA^{4-}$ stands for the tetra-deprotonated ligand.

2. Experimentation & Sampling

The previous activity was about the determination of lead concentration in contaminated soil. With a nominal concentration, a sequential extraction procedure was carried out to extract the lead in artificial contaminated soil.

The initial concentration of lead in the non-contaminated soil was determined. We have an idea of the nominal lead concentration of the contaminated soil.

Subsequently the samples from sequential extraction were experimentally analysed with the 'XRF & AAS', and the concentration of the solid sample was determined by 'XRF' analysis method, both before contamination, and after contamination with lead.

The deductions, findings, and observations are reported in subsequent sections and a statistical sampling of randomly selected values was conducted based on the t-test within underlying assumptions.

2.2 Analysis & presentation of results

Table 1: Table of Values obtained: "based on XRF analysis" 17, 13 & 14 -06- 2016, 'Sequential extractions' 13-07-16 &Predicted based on numerically random generated Monte Carlo.

Samples		Analysis ame: OLS	'XRF' Solid (mg/kg)	'MC'	'XRF' Liquid (mg/kg)	'Predicted: based on MC'
1		1-1	2035.82	2046	1943	1979
2		1-2 *	2080.26	2092	2110	2114
3		1-3	2116.75	2109	1920	1921
4			2006.78	2027	-	
5			2014.29	2027	-	
6		2-3	1833.72	1828	1825	-
7		3-1	2077.79		-	
8		3-2 *	2080.41	2091	2090	2060
9		3-3	2034.9	2028	1816	1886
	1	1.1 (NC)	138.199	-	306	-
	2	1.2 (NC)	-	-	312	-

Table 1b: Table of Values of the (new) contaminated samples, and residues from acqua regia attack & 'XRF'

Analysis (new) Samples	'XRF' Solid (mg/kg)	'Predicted (mg/kg)	'XRF' Liquid (mg/kg)	'Predicted (mg/kg)	'XRF' (residues) Liquid (mg/kg)
1	1855.0	1875	1890.2	1893	37.4
2	1724.4	1718	1836.6	1838	42.3
3	1804.6	1804	1729.9	1727	35.5

Table2: Table of Values obtained (new): "Randomly selected values from XRF analysis & sequential extractions"

XRF 'Solid'	XRF 'Liquid'
2080.26	2110
2116.75	1920
1833.72	1825
2080.41	2090
2034.9	1816
1855	1890.2
1724.4	1836.6
1804.6	1729.9

3. Descriptive statistics

Table 3: Table of values of descriptive statistics

Variables	Mean (mg/kg)	Std.Dev (mg/kg)	Kurtosis	Skewness
1	1941.255	53.9442	-2.0386	-0.1624
2	1902.213	47.4890	-0.5827	-0.7151

'XRF' Sol: 1
'XRF' Liq: 2

3.1 Hypothesis Testing:

We make our claim, and do the testing based on the 'null hypotheses from the underlying assumptions.

It's assumed the two samples are of unequal variance, assuming the normal distribution, then from a randomly selected pool of the population, or entire sample, within our assumptions, we conduct the statistical and hypothesis testing.
Two hypothesis are set comprising; the 'null and alternative' hypotheses.

Null Hypothesis:
$\mu_1 = \mu_2$ (Equal means)
Alternative Hypothesis:
$\mu_1 \neq \mu_2$ (Unequal/different means)

3.2 Hypothesis Testing Results & Tables of Results

We found from the tables above the results of the hypothesis testing carried out based on the t-test and our assumptions within a statistical significant level of 5%. The two sample t-test was based on unequal variances having assumed equal means,

Table of values 4: Results of hypothesis testing conducted

t – Stat	P (T<=t) '1 tail'	P (T<=t) '2 tails'	t Critical (1 tail)	t Critical (2 tails)	df	N
0.5432	0.2978	0.5955	1.7613	2.1448	14	8

4. Discussion

The above table is obtained based on the 'XRF' analysis. Our samples were soil samples both contaminated, and non-contaminated with 'nitrate di piombo: $Pb (No_3)_2$ '.
Samples serial number, 1-9 are soil samples contaminated with lead nitrate, while the remaining two samples 1 & 2 last bottom of the table 1 in that sequence were non-contaminated with lead.
The solid samples of all the contaminated soils with, lead nitrate were analysed with 'XRF' analysis, and also the non-contaminated soil samples, and the results are displayed in the above table.
Out of the 9 soil samples obtainmed with lead, we initially selected soil samples: 'OL S 1-2, & 3-2 indicated by the astericks *, with 2 soil samples:' OL S' non-contaminated for sequential procedures, arriving at a total of 4 samples:' OL S' altogether comprising contaminated (C), and non-contaminated (NC).

We've randomly selected the samples for hypothesis testing for the two groups of variables comprising; 'XRF & Sequential extraction' from the table of values presented in figures 1 & 1b respectively.

5. Conclusion

Our previos results and other presentations show that the results presented are quite close, we can confidently conclude that the **'XRF analysis'** procedure is very good enough, and appropriate in obtaining the lead concentration of the contaminated solid soil sample, without necessarily carrying out the sequential extraction procedures.

We've endearvoured to conduct a statistical testing based on the two sample t-test within our underlying results from randomly selected results obtained from the 'XRF' and sequential extraction procedures assuming unequal variances.

Our hypothesis testing result findings indicate we can accept the null hypothesis and our claim for equal means because the p-value found was greater than the α-value, and also corroboratng this fact, the t-statistics calcuated value was less than the critical value at the 5% significant level as presented and shown in our table of results.

Conclusively, since the values of the lead concentrations obtained for the soil samples from the 'XRF' analysis of the solid contaminated soil samples, and the solutions of the sequential extractions of the contaminated solid soil samples, quite well agree from our previous results findings, and corroborated by the findings of the statistical testing, we can confidently agree the results from the 'XRF' are reliable enough, without any need to do the sequential extraction for obtaining the concentrations.

References
[1]. Jennifer M. Jones, Sequential extraction method: a review and evaluation, Environmental Geochemistry and Health, Volume 15, Numbers 2-3, September, 1993
[2]. Z.S. Ahnstrom and D.R. Parker, Development and Assessment of a Sequential Extraction Procedure for the Fractionation of Soil Cadmium, Soil Science Society of America Journal 63:1650-1658 (1999)
[3]. Barnett and Lewis, Outliers in Statistical Data 3rd Ed., John Wiley and Sons, 1990.
[4]. Snedecor, George W and Cochran, Wiliam G, Statistical Methods,8th Edition, Iowa State University Press, 1989.
[5]. M. Bucheli-Witschel, M.; T Egli, T. (2001), "DAB: Environmental Fate and Microbial Degradation of Amino polycarboxylic Acids", FEMS Microbiology Reviews, 25 (1): 69–106. Advanced Engineering Mathematics, Erwin *Kreyszig, 2011.*