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X-RAY FLUORESCENCE SPECTROSCOPIC TECHNIQUE AS A TOOL  
FOR THE ANALYSIS OF SOME ELEMENTS PRESENT IN THE RAW  
MATERIALS OF CEMENT

A DISSERTATION SUBMITTED FOR THE PARTIAL FULFILMENT OF  
THE B.Sc. HONS. DEGREE

By

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## ACKNOWLEDGEMENT

I am very grateful to my supervisor Dr. Abd-Elrazig Elfaki for his keen supervision and valuable advices that helped in accomplishing this project.

I could hardly find the suitable words to express my thanks to my Co-supervisor Dr. Mustafa M. Osman for his sincere efforts and apperciable advice which have enriched this study. To him again my thanks with deep respect.

Also a lot of thanks are due to members of the department of Physics who help me in carrying out the practical work. My great thanks also to ustaz Abdalla Ahmed for typing of this manuscript.

TO MY MOTHER AND DEAR FAMILY

### ! ABSTRACT

Thirteen different samples of raw materials used in manufacturing of cement were collected from south ELGabalain area where Rabak cement raw materials available. The samples were crushed, ground and sieved. The concentration of the following elements and compounds were determined using X-Ray Fluorescence technique (XRF) :  $\text{TiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{MnO}_2$ ,  $\text{CaO}$ ,  $\text{SrO}$ ,  $\text{K}_2\text{O}$ ,  $\text{CO}$ ,  $\text{Cu}$ ,  $\text{Zn}$ ,  $\text{As}$ ,  $\text{Y}$ ,  $\text{Zr}$ , and  $\text{Nb}$ . XRF technique is found to be the most suitable one because it is non destructive , more rapid and simultaneous multi element determined tool.

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CHAPTER ONE  
INTRODUCTION

## 1.1 GENERAL INTRODUCTION:-

Since the pre-historical ages, the Man began to seek for loading to protect his life. He lived in caves and then he built houses with mud and other available materials which may stand better. Greeks and Romans were found that certain calcitic deposits if finely ground, yield a mixture which can resist the action of water. Such efforts continued by many other people and they developed the industry of cement.

Cement may be defined as "adhesive" substances capable of uniting fragments or masses of solid matter to a compact unit. It can be prepared from one chemical compound or a mixture of compounds. The raw materials of cement industry are lime or lime stone, clay and gypsum. The main components of these raw materials for the Portland cement ( which is commonly used in Sudan ) are the followings :-

Calcium oxide ( $\text{CaO}$ ), mainly obtained from the lime or lime stone and few percentages from clay.

Silicon dioxide ( $\text{SiO}_2$ ); is commonly derived from the clay. Also the aluminum trioxide ( $\text{Al}_2\text{O}_3$ ) and iron oxide ( $\text{Fe}_2\text{O}_3$ ) are from the clay.

The above four elements combined together in



the following formulae to form the stable structures responsible for the strength of cement specially when mixed with water.

- 1 - Tricalcium silicate ,  $\text{Ca}_3\text{SiO}_5$  ( C3S )
- 2 - Dicalcium silicate ,  $\text{Ca}_2\text{SiO}_4$  ( C2S )
- 3 - Tricalcium aluminate,  $\text{Ca}_3\text{Al}_2\text{O}_6$  ( C3A )
- 4 - Tetracalcium aluminoferrate  $\text{Ca}_4\text{AlFeO}_5$  ( C4AF )

The Portland cement contains MgO up to 5% ,  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  from 0.5 to 1.3% Also  $\text{TiO}_2$  occurs to small extent, about 0.2 to 0.3 % . Other trace elements may be present depending on the type of the raw material used e.g. Mn. Cr. .... etc.

#### 1.2 DEPARTING OF THE CEMENT FROM EQUILIBRIUM :-

The cement might depart from the equilibrium state. The reactions during burning between the lime and clay which resulted in the production of what is known as clinker may not be complete, thus would lead to a deficiency in the amounts of the most basic lime compounds formed and to the presence of the corresponding amount of uncombined lime in the clinker. The amount of uncombined lime would be a measure of the inefficiency of the burning. If the cooling process of the clinker is too rapid for equilibrium to be maintained between solid and liquid phases there will be a deficiency of C3S and small amount of free  $\text{CaO}$  will separate when the liquid finally solidifies. The

compounds content of cement in which the equilibrium is assumed to be frozen from the clinker temperature can be calculated, but if clinkering temperature of  $1400^{\circ}\text{C}$  is assumed, approximate correction for the deviation of the values can be obtained. Also a deficiency in CaO might occur leaving the other elements freely present as oxides and not as calcium silicates (1).

From the above mentioned facts about the importance of each element in the cement and its role in the property of the strength of the cement arises the importance of their precise analysis and evaluation. This is why the importance of using a proper technique in the analysis will be of great help such as the x-ray fluorescence technique.

### 1.3 X - RAY FLUORESCENCE ANALYSIS ( XRF ) : -

XRF spectroscopy is being increasingly employed particularly where large number of samples are required to be analysed. The sample is irradiated with a primary high-energy x-ray beam and the secondary (fluorescent) x-ray emitted will be detected. Each element has its characteristic x-ray spectrum and thus it is possible to be identified and determined.

#### 1.3.1 EMISSION OF CHARACTERISTIC X-RAY SPECTRA :-

When a sufficient energetic X-ray photon interacts with an atom, several phenomena take place. One interaction involves the transfer of the photon energy to one of electrons in the atom (e.g K-shell electron)

causing its ejection from the atom (photoelectric).

### 1.3.2 X-RAY TRANSITION :-

The distribution of electron in the ionized atom is then out of equilibrium and within an extremely short time returns to the normal state by transition of electrons from outer shells to inner shells. Each such electron transfer, for example from L-shell to the K-shell represents a loss in the potential energy of the atom which appears in the form of a photon of energy equal to the difference between the binding energies of the initial and final shells. There is some relation between the intensity and the concentration. If you consider a beam of X-ray photons impeding onto a homogeneous sample of a uniform thickness to ionize an atom in a layer of known thickness, the characteristic radiation emitted due to ionization process measured by the spectrometers system as intensity.

### 1.3.3 APPLICATION OF XRF:-

On condition that there is no change in the total absorption or enhancement over the range of the calibration line

$$C_i^x = C_i^s \left( \frac{I_i^x}{I_i^s} \right) \left( \frac{\text{Total absorption } \mu \text{ by } x}{\text{Total absorption } \mu \text{ by } s} \right) \left( \frac{\text{Total enhancement of } \lambda_i \text{ by } x}{\text{Total enhancement of } \lambda_i \text{ by } s} \right)$$

where

$C_i^x$  concentration of an element

$C_i^s$  concentration of standard of element  $i$

$X_i$  = the measured wave length

$I_i^x$  and  $I_i^s$  = the measured intensity of  $X_i$  in the specimen and standard respectively

The above equation shows that a linear relationship will exist between characteristic x-ray line intensity and elemental concentration .

Since the total absorption range is dependant upon the concentration range, it is clear that by limiting the calibration ranges of all interfering elements, one can limit the absorption range. Finally if the matrix of the calibration standard is matched with that of the unknown specimen, a direct correlation between measured x-ray intensity and elemental composition can be made. This is the basis of the so called in-type or type-matching analysis, which is by far the most commonly employed method of quantitative analysis<sup>(2,3)</sup>.

#### 1.3.4 COMPONENTS OF THE INSTRUMENT:-

1) X-RAY SOURCES :-

A - RADIOACTIVE SOURCE :-

Radio isotopes are commonly used. The monochromatic radioactive sources such as  $^{109}\text{Cd}$  and  $^{56}\text{Fe}$ .

The separation and collection of analytical lines is performed using (Si - Li) detectors<sup>(4)</sup>.

#### B - TUBE EMITTED SOURCE :-

Here the excitation is obtained from the anode of X-ray tube. Electron emitted from a heated cathode are accelerated through a potential (V) focused to strike the anode.

2- The (Si - Li) detector, is a useful one. It's energy dispersive systems provide a wide flexibility for analyzing the elements from sodium to uranium (Na - U)

#### 3) ELECTRONICS :-

The function of the ; preamplifier is to collect the charge pulses from the detector & provide the low driving impedance necessary to pass the signal through a co-axial cable to the main amplifier. The height of this pulse is proportional to the energy of X-ray .

#### 4) AMPLIFIER :-

Is applied for increasing the amplitude of the preamplifier output signal

#### 5) MULTI CHANEL PULSE HEIGHT ANALYZER ( MCA ) :-

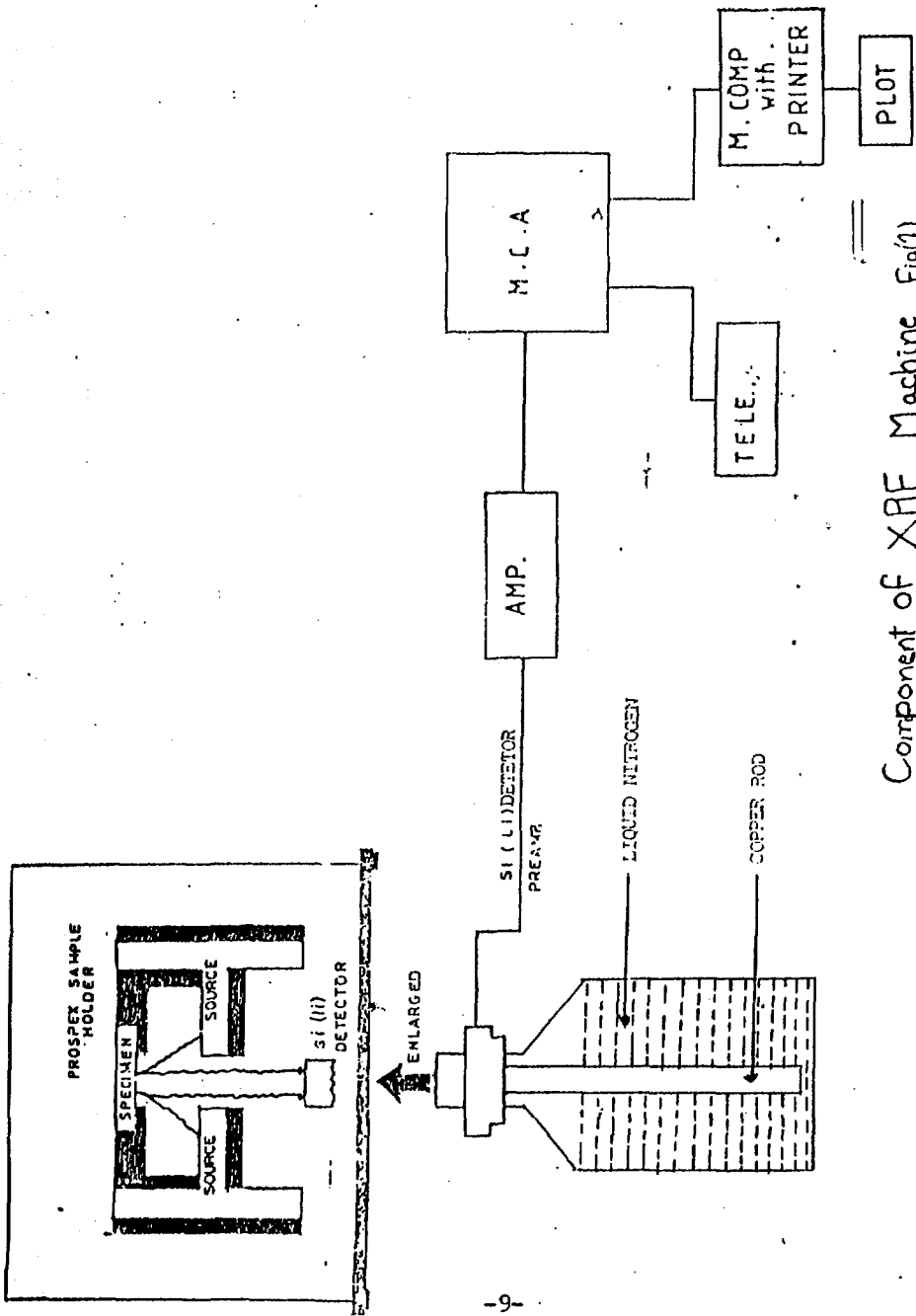
The purpose of the MCA, is to measure the height of each amplifier output, and to represent this amplitude by an integral number.

#### 6) COMPUTER :-

Most quantitative fluorescence spectrometers,

include small digital computer. It is to perform fitting of spectra and determining the net areas of the peaks of the interest and finally for quantitative analysis of samples.

Measurements of energies of the peaks in X-ray spectrum indicate what kind of atoms are present. The number of a counts or the area of the peak is related to the number of atoms in the sample allowing the quantitative measurements to be made, if the X-ray production and detection efficiency are known. This method gives information about the samples irrespective to their state of chemical combination or phases at which they exist<sup>(5,6)</sup>.



Component of XRF Machine Fig(1)

CHAPTER TWO

METHOD AND RESULTS



## 2.1 SAMPLE PREPARATION :-

About thirteen different samples were collected from south EL Gabalain area where the Rabak raw materials for cement lies ( see Table one ). The samples were crushed and ground to 100 um in grain size. One gram of each sample was weighed and pressed into a pellet of 2.5 cm in diameter and 1 mm in thickness applying a pressure of fifteen ton. The pellet then subjected to the XRF system

## 2.2 MEASUREMENTS FOR THE DIFFERENT ELEMENTS USING (XRF)

Each sample pellet was loaded on the top of the Si - Li detector and a source of  $^{109}\text{Cd}$  was used for the excitation of the different elements in the sample. Time collection was 1000 seconds and Canberra Multi Channel Analyser ( MCA ) was used for the accumulation of the spectra( see Fig 1 ). The data were transefered to the computer and the net area for each element was measured by the Axil program. Three different standards were measured under the same conditions of the samples, using the type matching method the concentration of each elements was calculated . The results of the analysis are shown in table (2). This results are concluded from the spectra (2-17) and tables (3-19), (see appendix).

TABLE ( 1 ) Shows the discription of the samples collected.

SAMPLE NUMBER	DISCRIPTION	SAMPLE NUMBER	DISCRIPTION
L1	Dolomite, whitish	L8	Dolomite, grey
L2	Calcite, grey	L9	Clay, dark grey
L3	= , pinkish	10	Iron pellets brown
L4	= , white	11 & 12	Calcite rejects different colour
L5	= , grey	13	Granit contacte the calcite,
L6	= =		
L7	= , pinkish		

TABLE NUMBER (2) SHOWS THE ELEMENTAL ANALYSIS FOR DIFFERENT COLLECTED SAMPLES.

SAMPLE NUMBER	L1	L2	L3	L4	L5
TiO <sub>2</sub>	---	---	---	---	00.01
Fe <sub>2</sub> O <sub>3</sub>	00.98	00.95	00.93	00.89	00.87
MnO	00.01	00.01	00.01	00.02	---
CaO	31.52	43.01	41.02	44.15	39.28
SrO	00.02	00.11	00.02	00.08	00.08
K <sub>2</sub> O	00.06	00.21	00.12	00.16	00.15
Co	10	09	--	10	--
Cu	03	04	04	04	03
Zn	09	05	06	12	12
As	--	--	--	--	--
Rb	02	05	07	--	04
Y	--	--	--	--	--
Zr	02	08	02	05	10
Nb	01	04	--	--	--

TABLE NUMBER (2) CONTINUE.

SAMPLE NUMBER	L6	L7	L8	L9
TiO <sub>2</sub>	---	---	00.21	00.70
Fe <sub>2</sub> O <sub>3</sub>	01.12	01.15	01.41	05.73
MnO	00.01	00.01	00.03	1171*
CaO	44.58	39.80	34.59	05.52
SrO	00.09	00.02	00.01	140*
K <sub>2</sub> O	00.17	00.14	00.21	00.91
Co	09	07	11	---
Cu	01	04	02	01
Zn	10	07	10	33
As	13	09	--	21
Rb	--	03	01	32
Y	--	01	03	29
Zr	04	03	02	65
Nb	--	--	01	22

TABLE NUMBER (2) CONTINUE.

SAMPLE NUMBER	10	11	12	13
TiO <sup>2</sup>	00.25	00.16	00.12	01.87
Fe <sub>2</sub> O <sub>3</sub>	37.35	01.88	02.10	33.10
Mno	715*	327*	461*	8701*
CaO	---	18.56	17.11	00.15
SrO	190*	175*	191*	08*
K <sub>2</sub> O	01.10	01.30	01.31	00.35
Co	71	49	37	490
Cu	04	04	06	05
Zn	49	18	17	06
As	16	15	16	24
Rb	13	14	18	02
Y	17	17	19	47
Zr	30	31	27	41
Nb	15	06	09	12

\*- CONCENTRATION IN MICROGRAM PER GRAM (PPm)

CHAPTER THREE

DISCUSSION AND CONCLUSION

### 3.1 DISCUSSION AND CONCLUSION:-

The aim of these work is to analysed some samples collected from different locations around ELgabalain area. These samples are generally used in manufacturing of cement. The most important element which is calcium shows relatively high concentrations in samples L2, L5, L6 and L7 which may confirm the classification of the mentioned samples as calcites. On the other hand, samples L1 and L3 have relatively low concentration of calcium, therefore they may be classified as dolomites.

The concentration of other elements show acceptable values according to literature (1).

The concentration of calcium in the two reject sample is relatively low which might indicate the predominancy of dolomite.

Sample L8 and L11 are small grain in nature because they are reject from the calcite ore.

XRF technique is proved to be the best method for these analysis because it is a time saving technique and non destructive method as well as it gives acceptable results for many metals present in the same sample simultaneously. Therefore it covers large number

of elements, depending on the sources and the conditions of the samples.

Many information and conclusions can be drawn from the accompanied spectra and tables, but we just<sup>detect</sup> the major component which is calcium as an example.



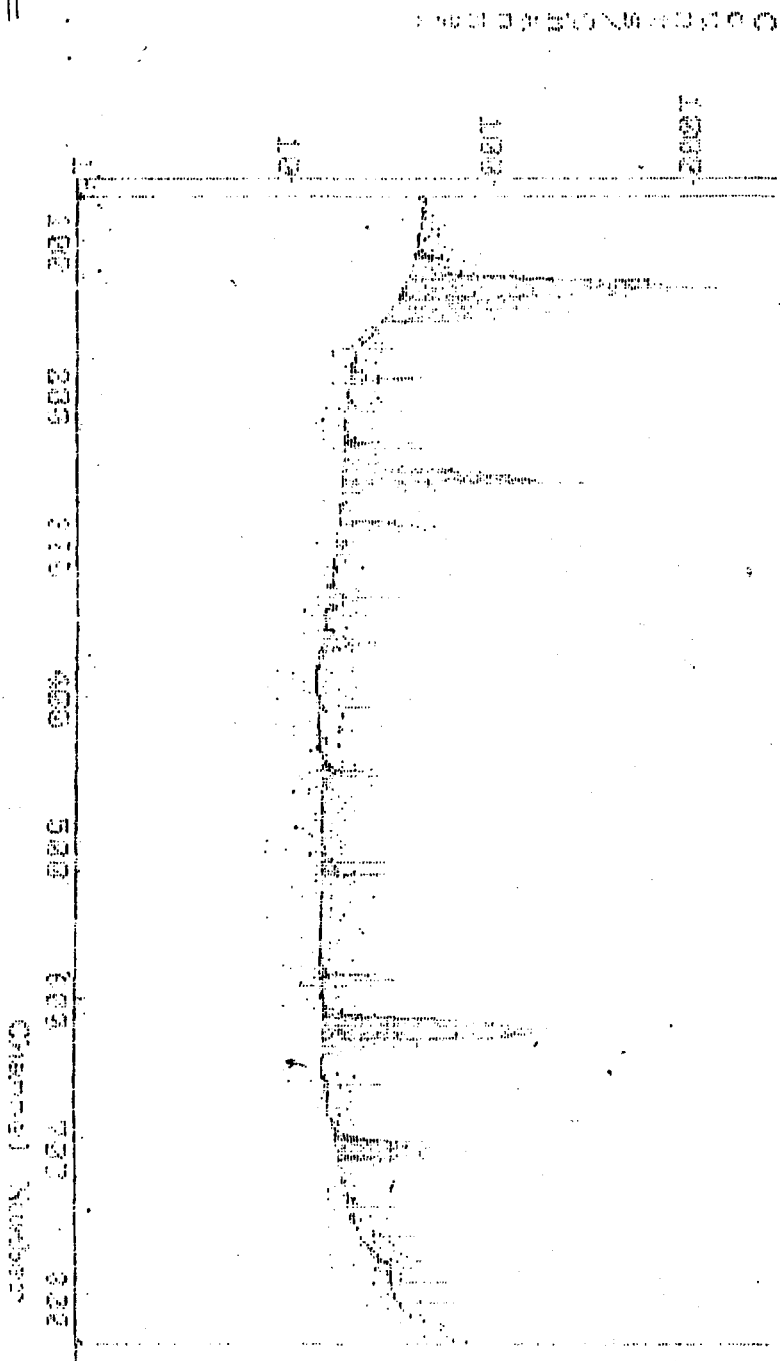
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- 2- Osman M.M, Ph.D Thesis, Khortum university (1988).
- 3- Ron Jenkins, Gould, R.W. and Dule Gedck, Quantitative X-ray Spectroscopy, Marcel Dekker. INC, New York and Basel (1981)
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- 5- Kafi. E.T., M.Sc., Thesis, Khartoum university (1993).
- 6- AbdAlla. M.D, M.Sc. Thesis, Khartoum uinversity (1984)

Spectrum: LINE1.SPE

Iteration 3: ChiSquare =

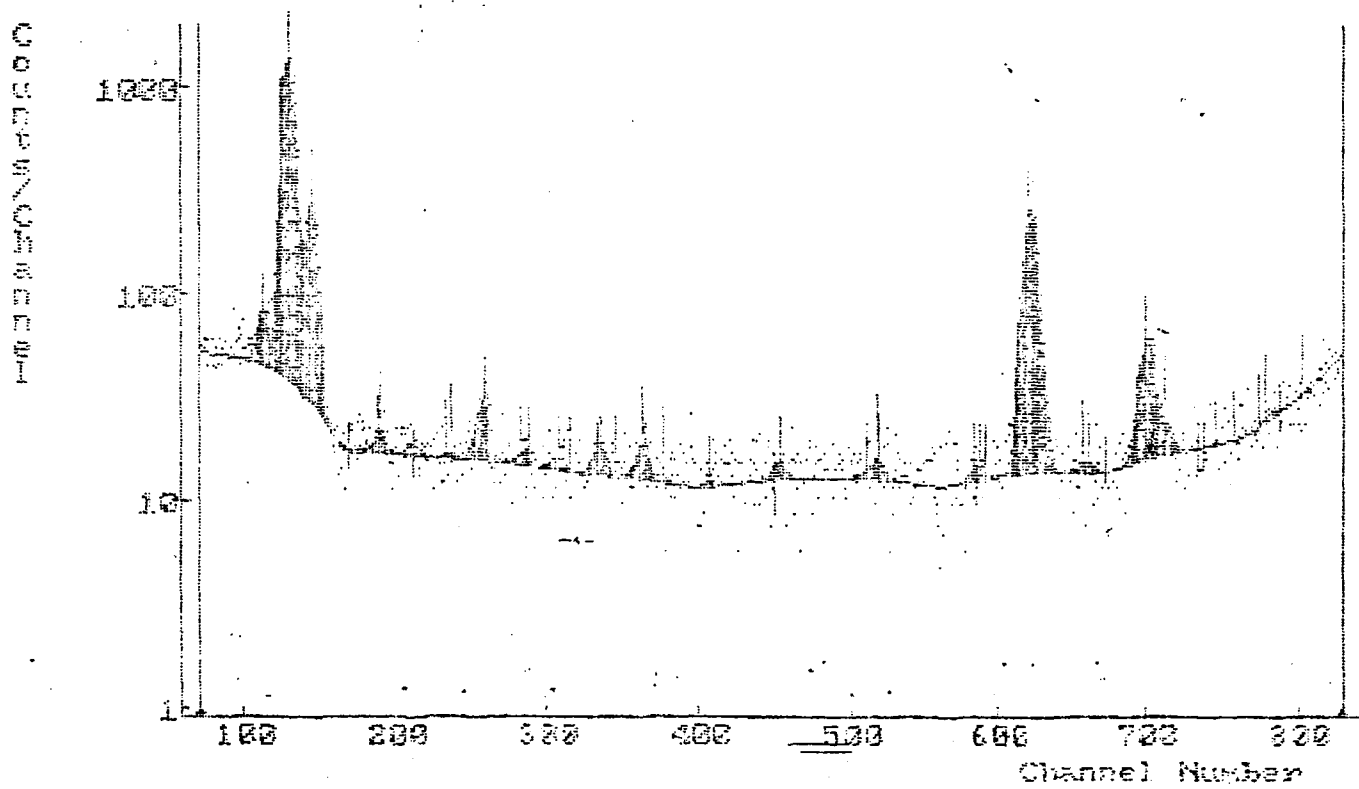
1.0; DIF = -.2



Spectrum LINE2.SPE

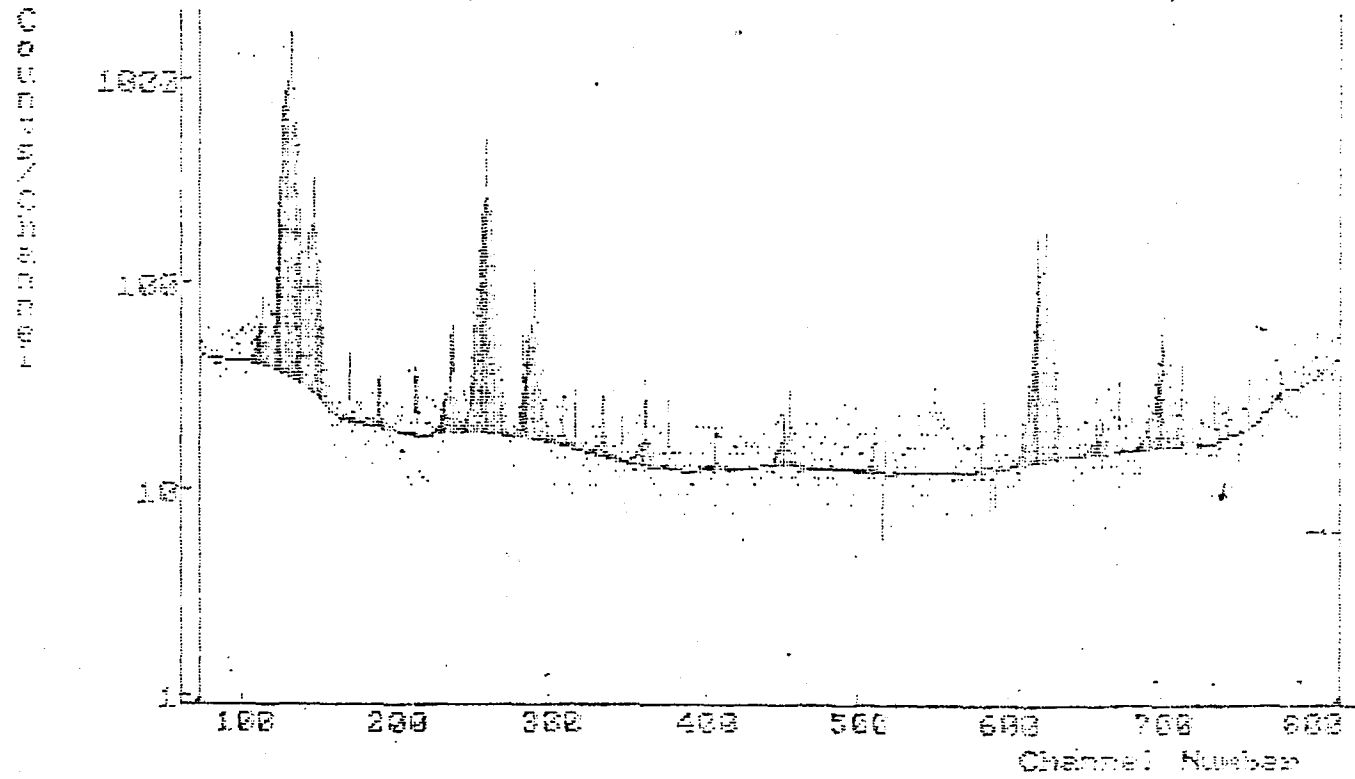
Iteration 2: ChiSquare =

1.2; Dif = -.8



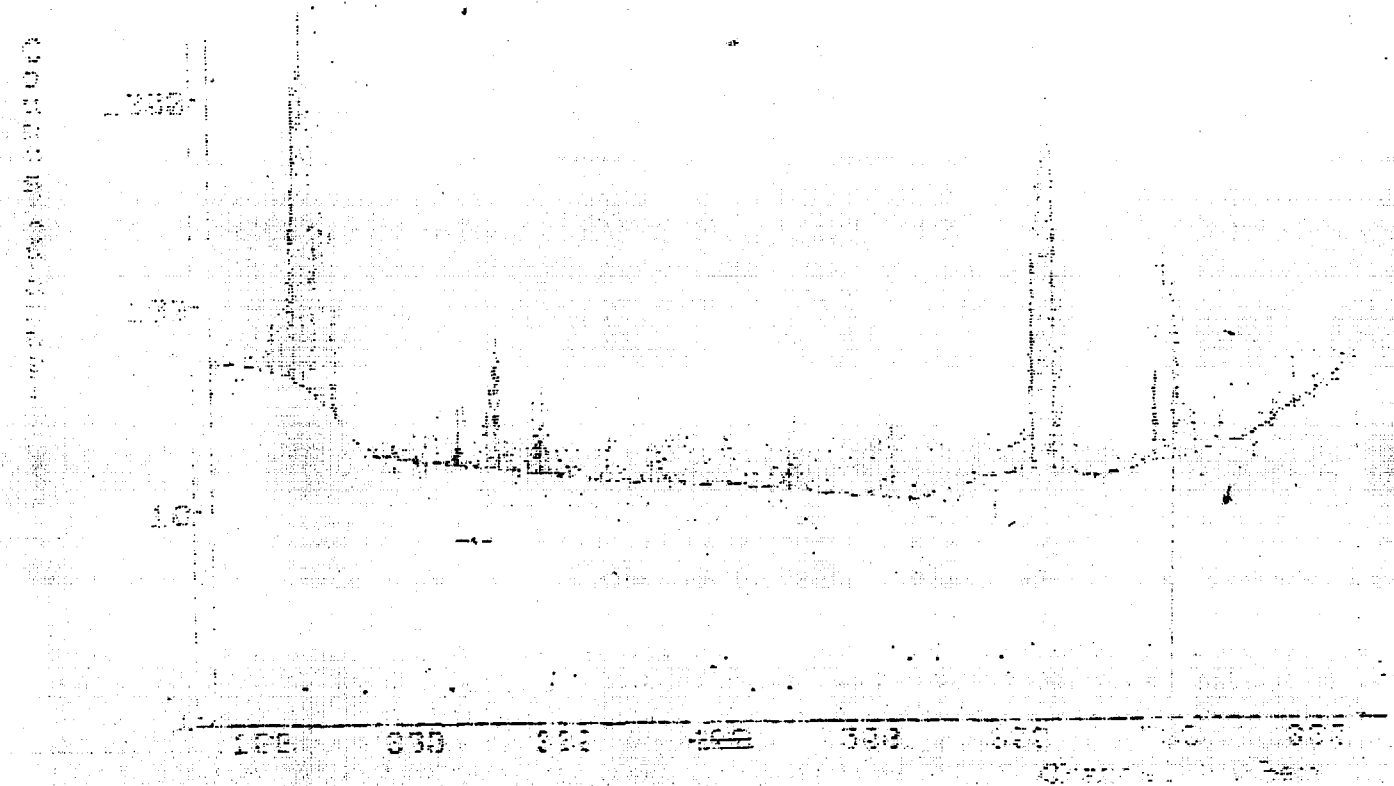
Spectrum LIME3.SPE

Iteration 2: ChiSquare = 1.3; Dif = -0.7



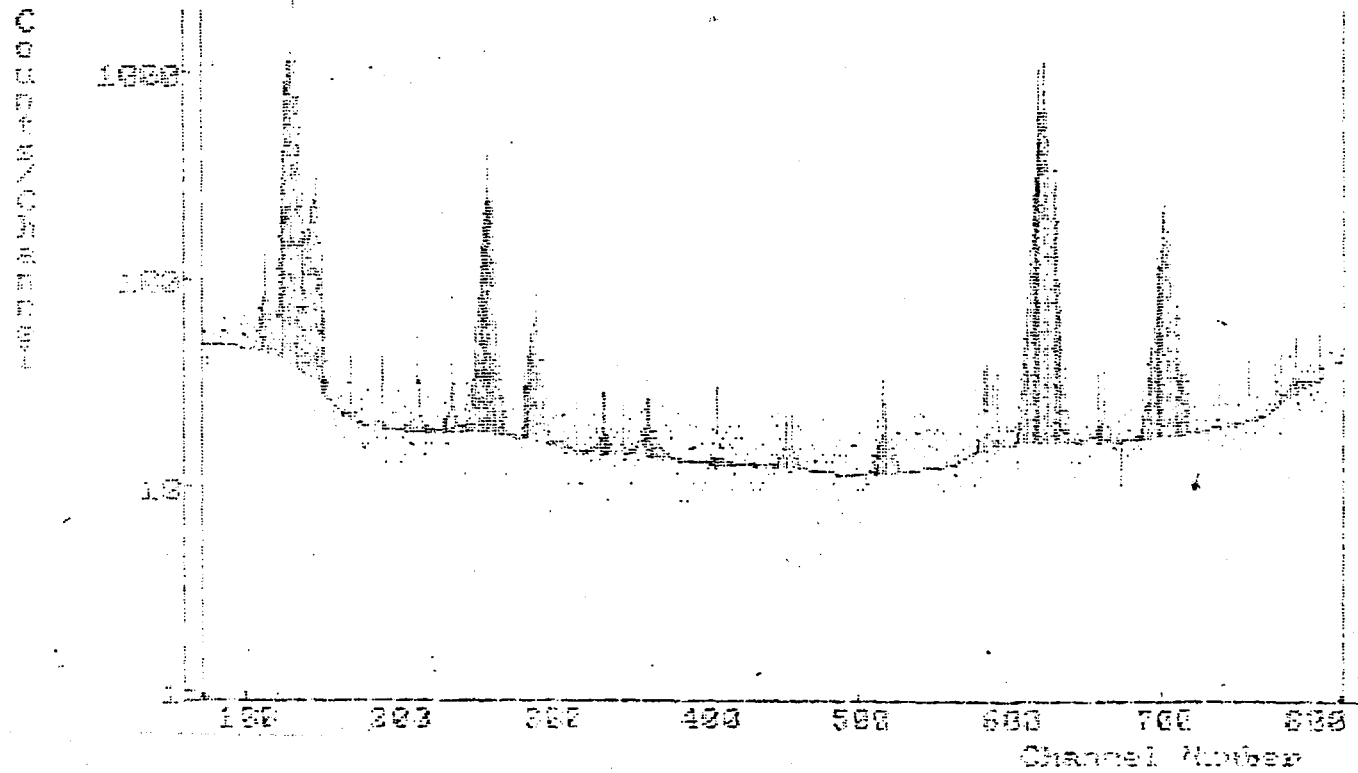
Spectrum LINE4.SPE

Iteration 2: ChiSquare = 1.4; Dif = -0.5



Spectrum LINES.SPE

Iteration 3: ChiSquare = 1.2: Dif = -.1

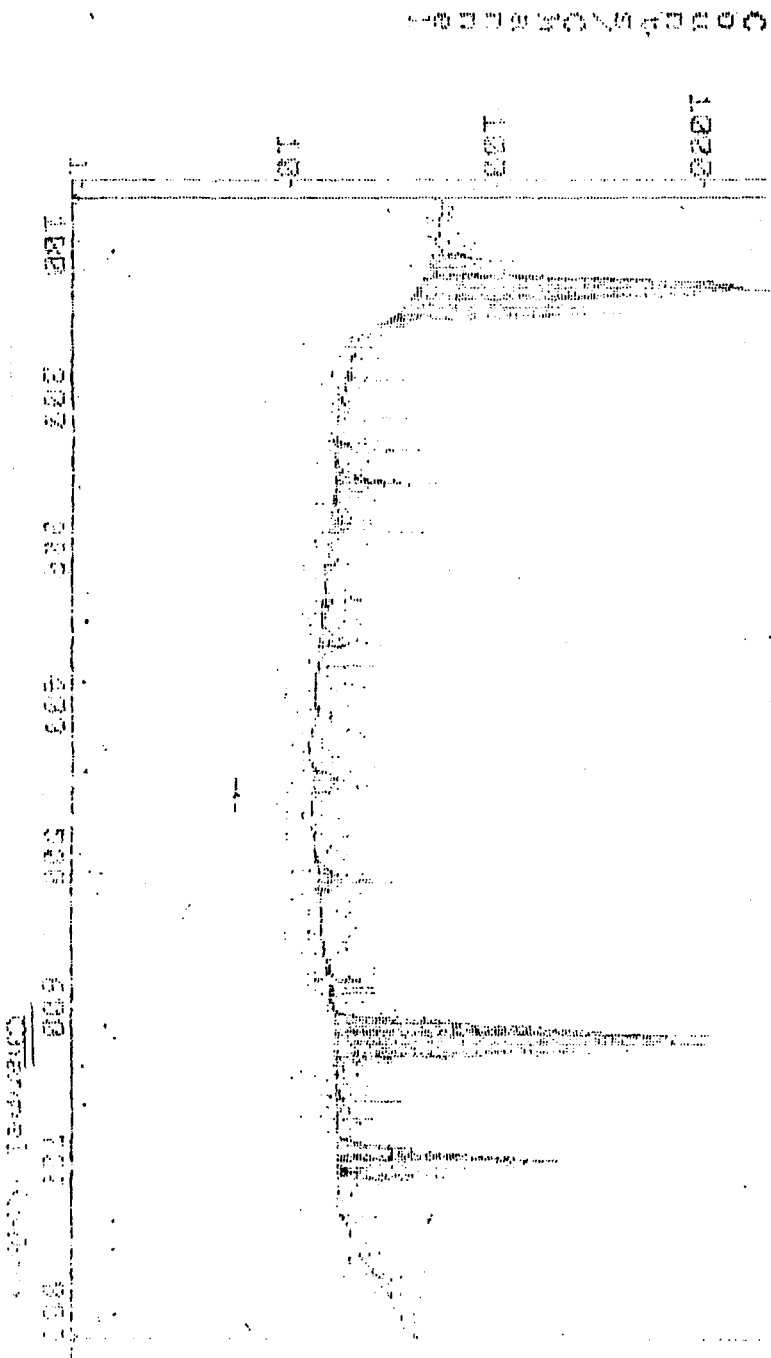


Spectrum LINE6.SPF

Iteration 3:

ChiSquare =

1.21 Df = 8



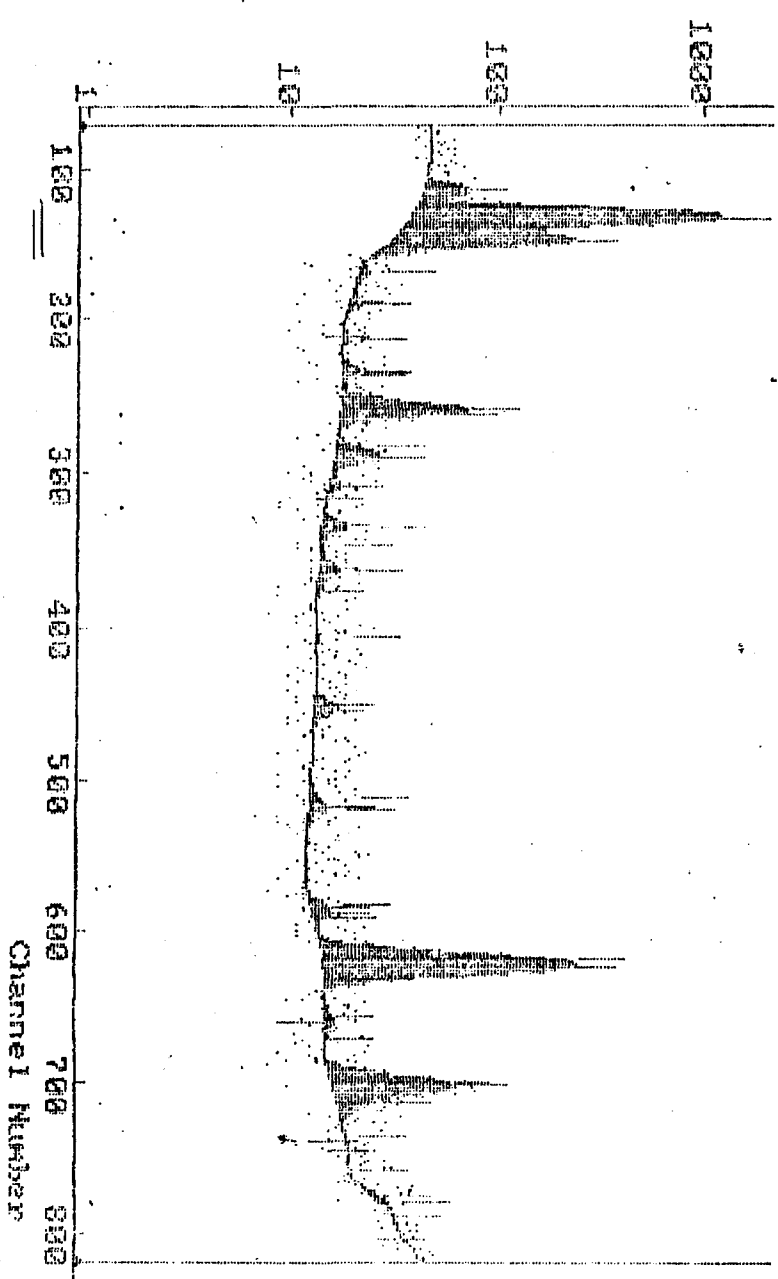
Spectrum LIME7.SPE

Iteration 3

ChiSquare =

1.3; DIF = -.0

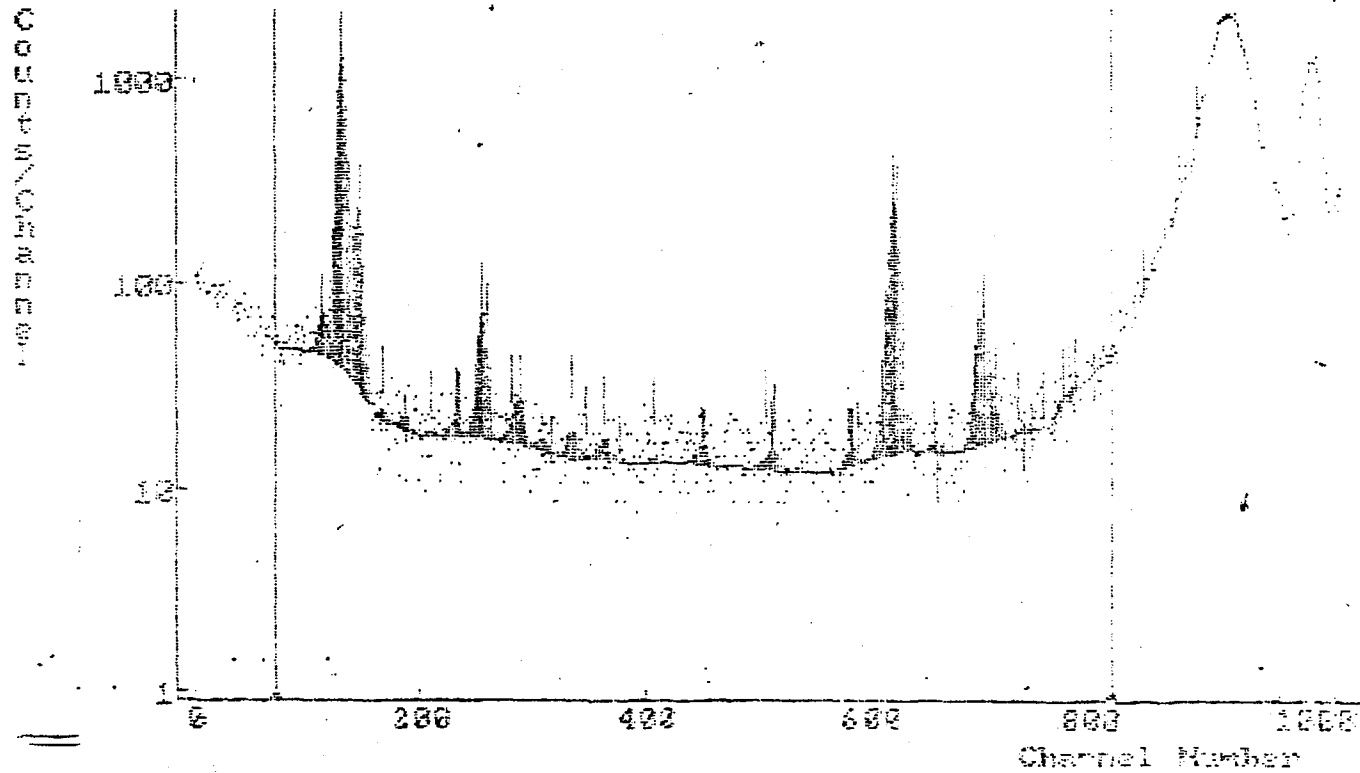
COMPLETION





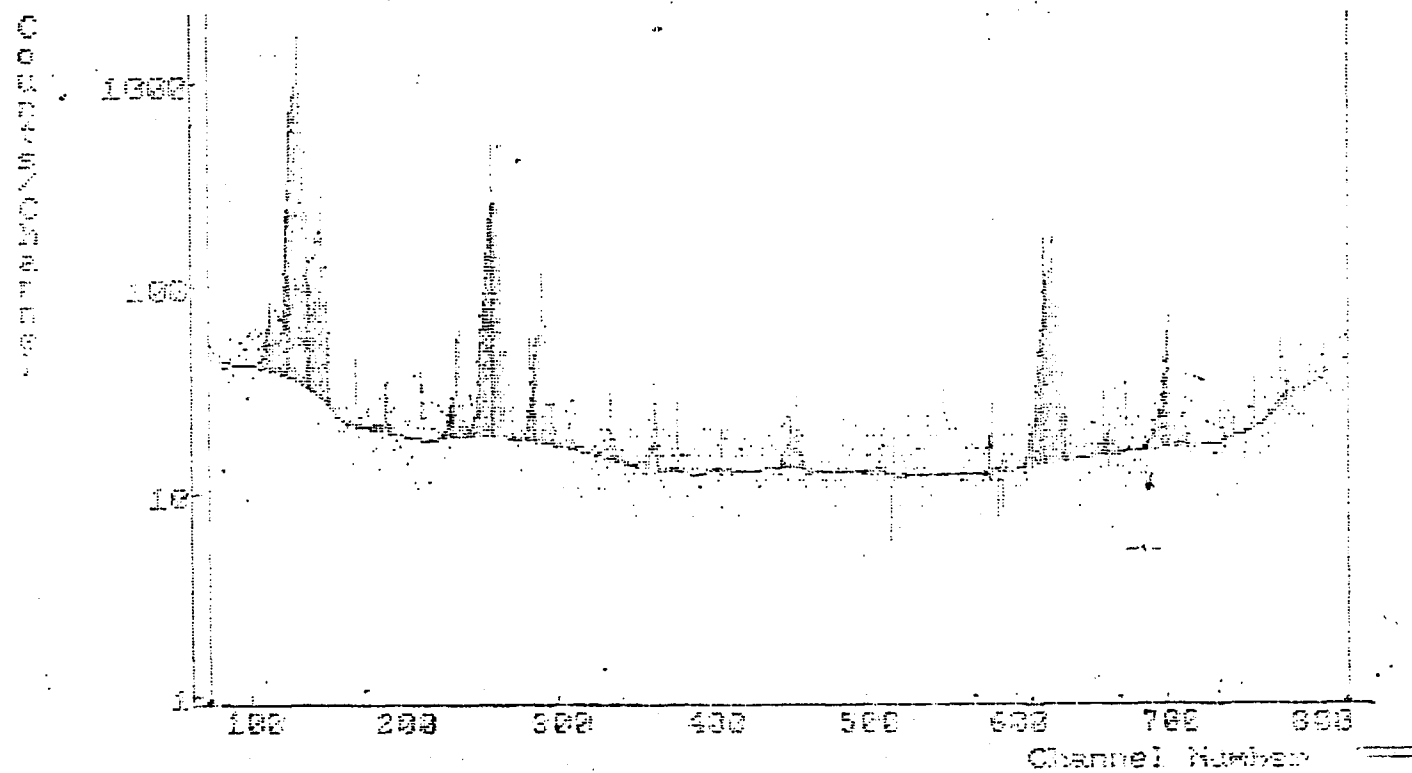
Spectrum LINE7.SPE

Iteration 3: ChiSquare = 1.3; Dif = -.0

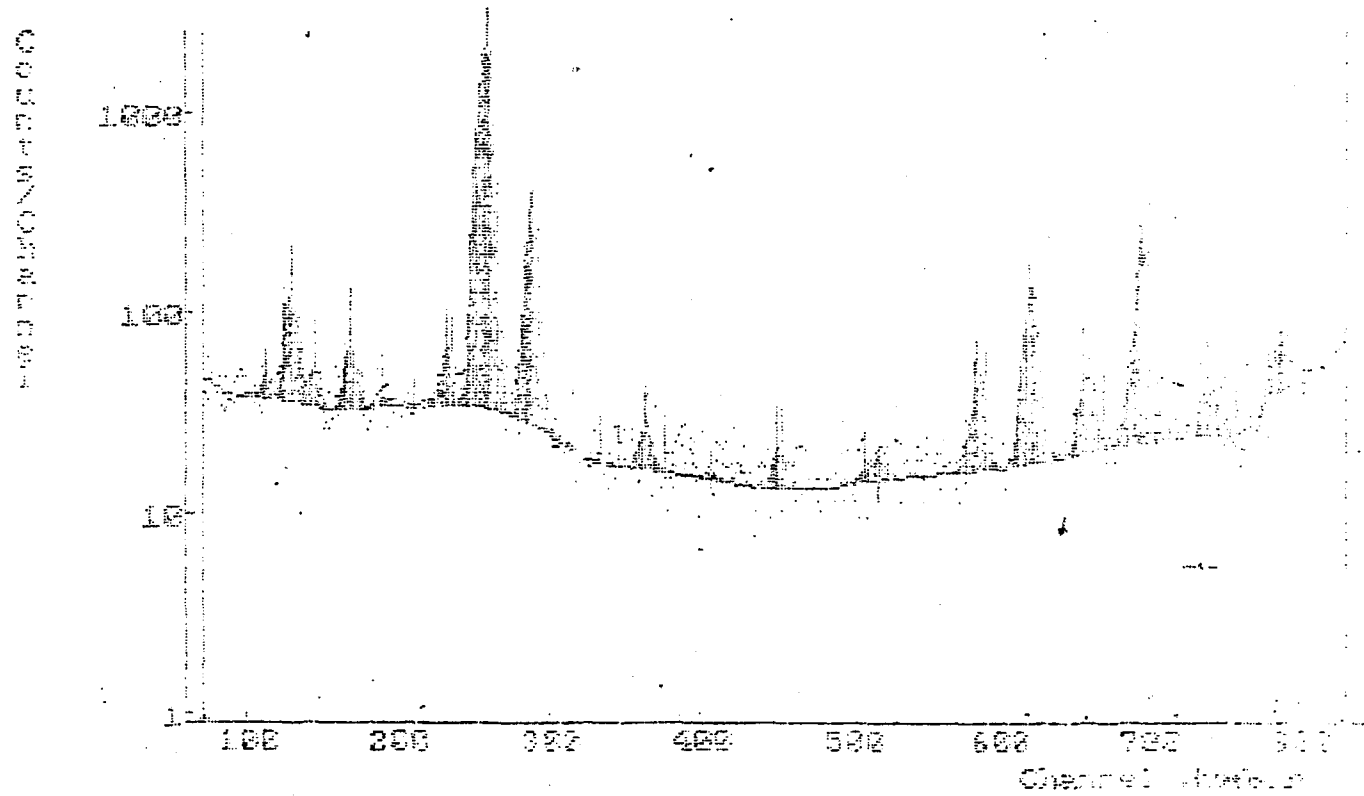


Spectrum LINE8.SPE

Iteration 2: ChiSquare = 1.3; Dif = -.4

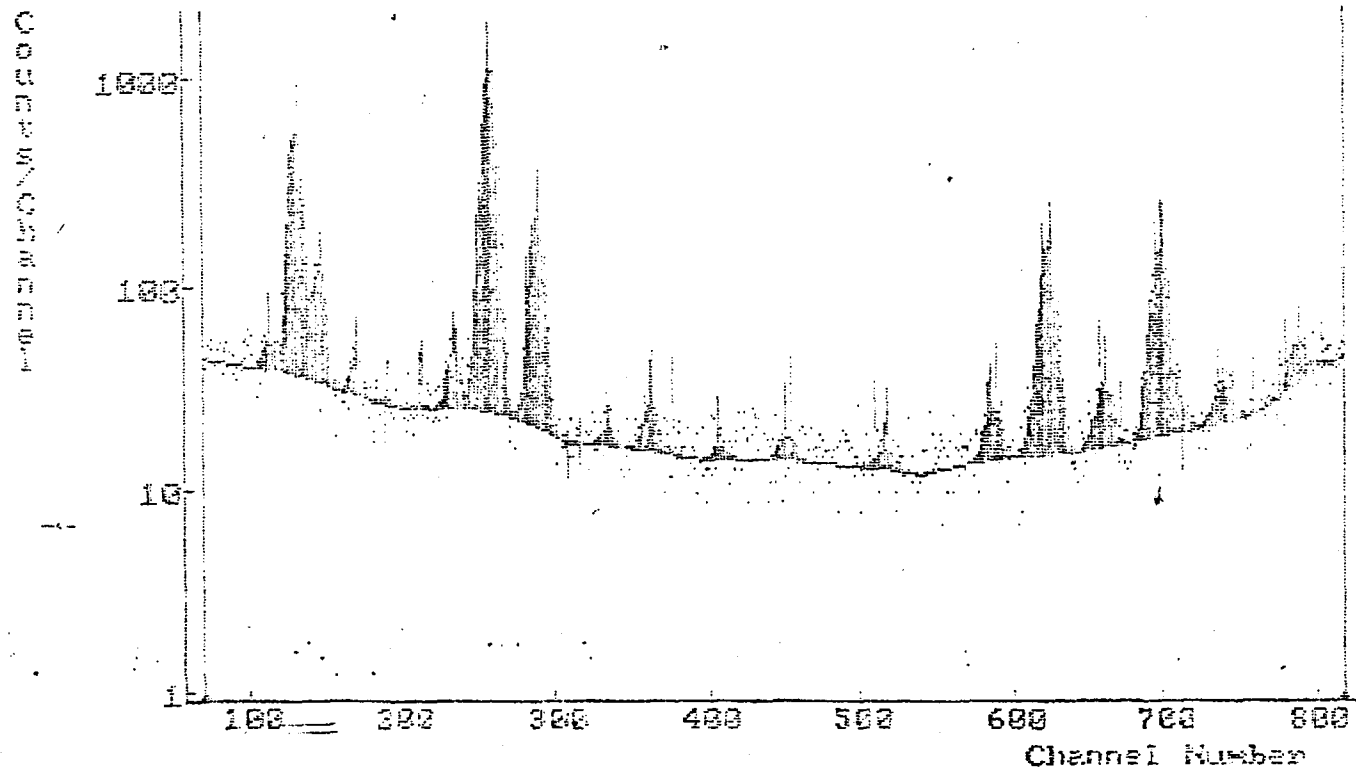


Spectrum, CLAY.SPE (9) Iteration 3: ChiSquare = 1.2; Dif = -1.

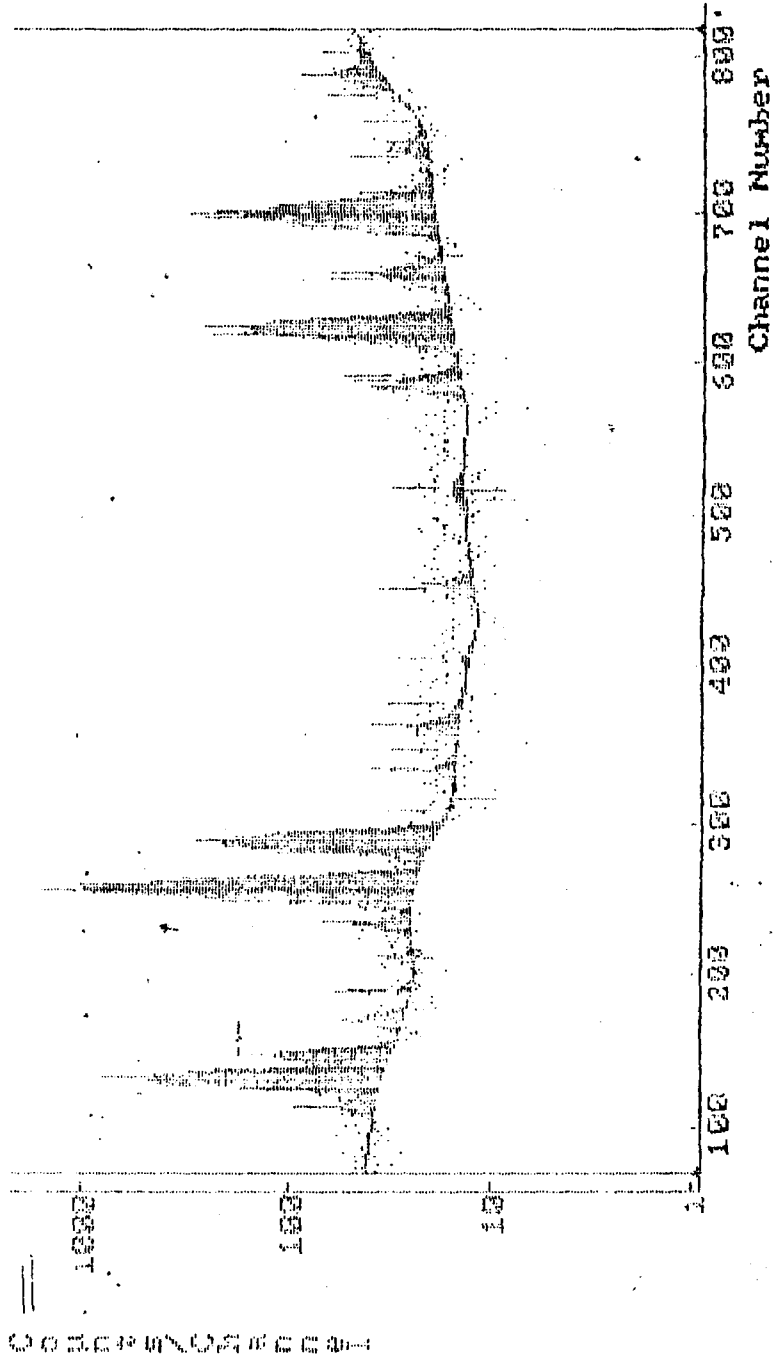


(14)

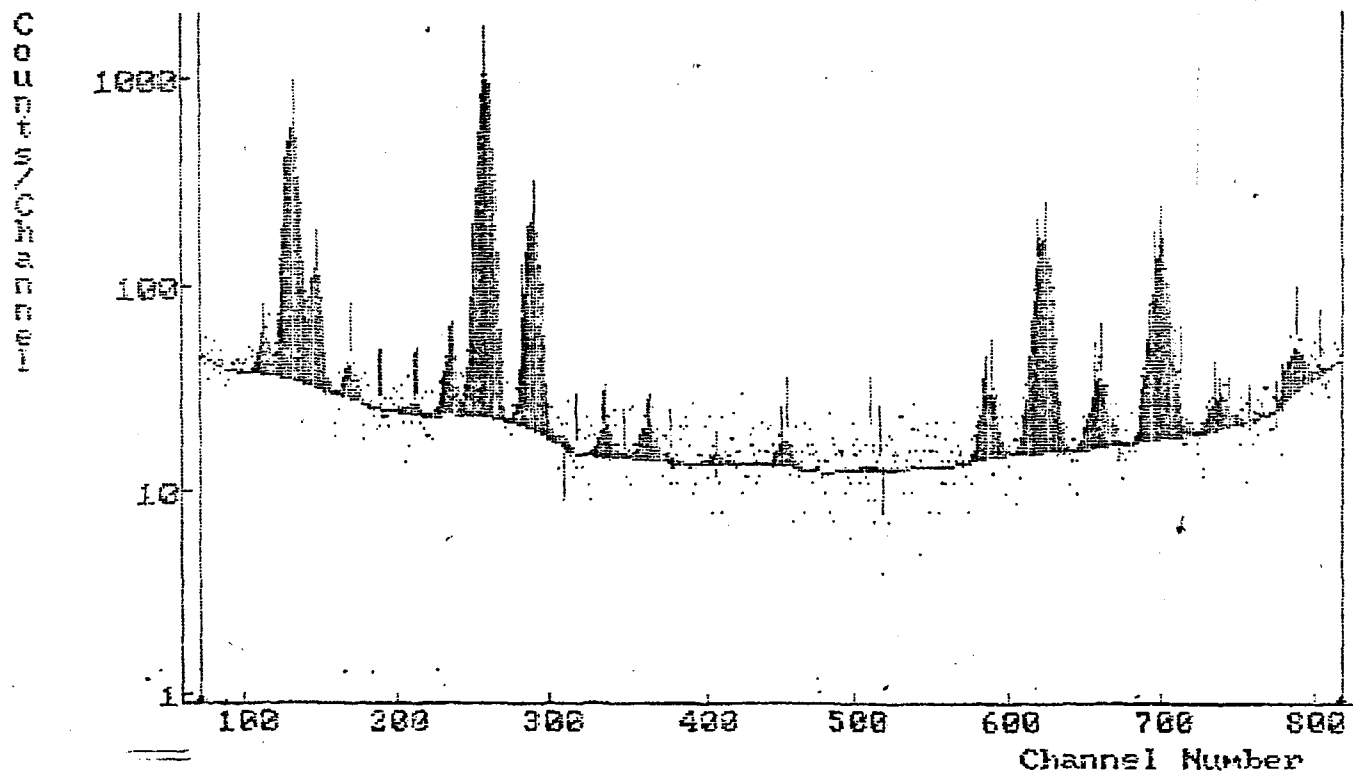
Spectrum FEPFLETT.SPE(0) Iteration 2: ChiSquare = 1.1; Dif = -1.0



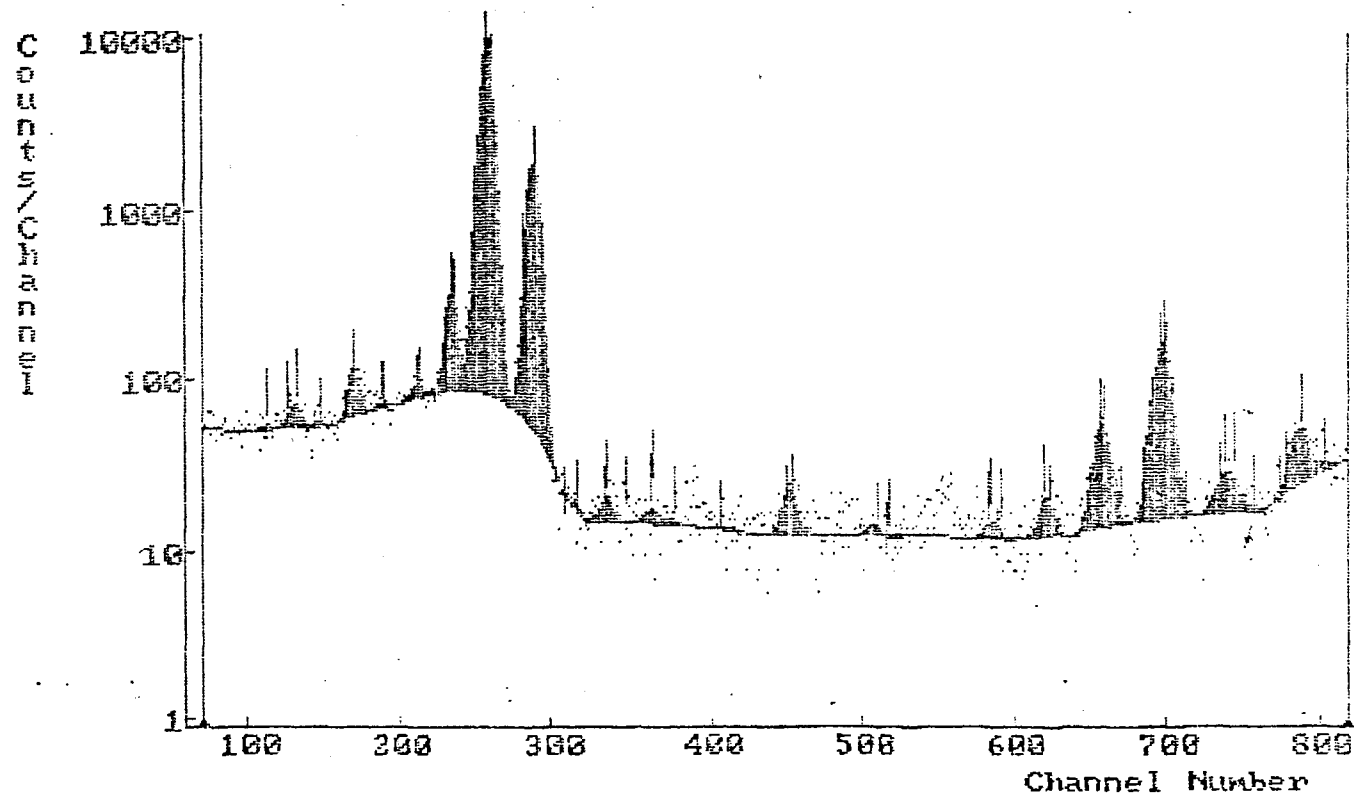
Spectrum MLEDC1.SIF(1) Iteration 2: ChiSquare = 1.1; DIF = -.4



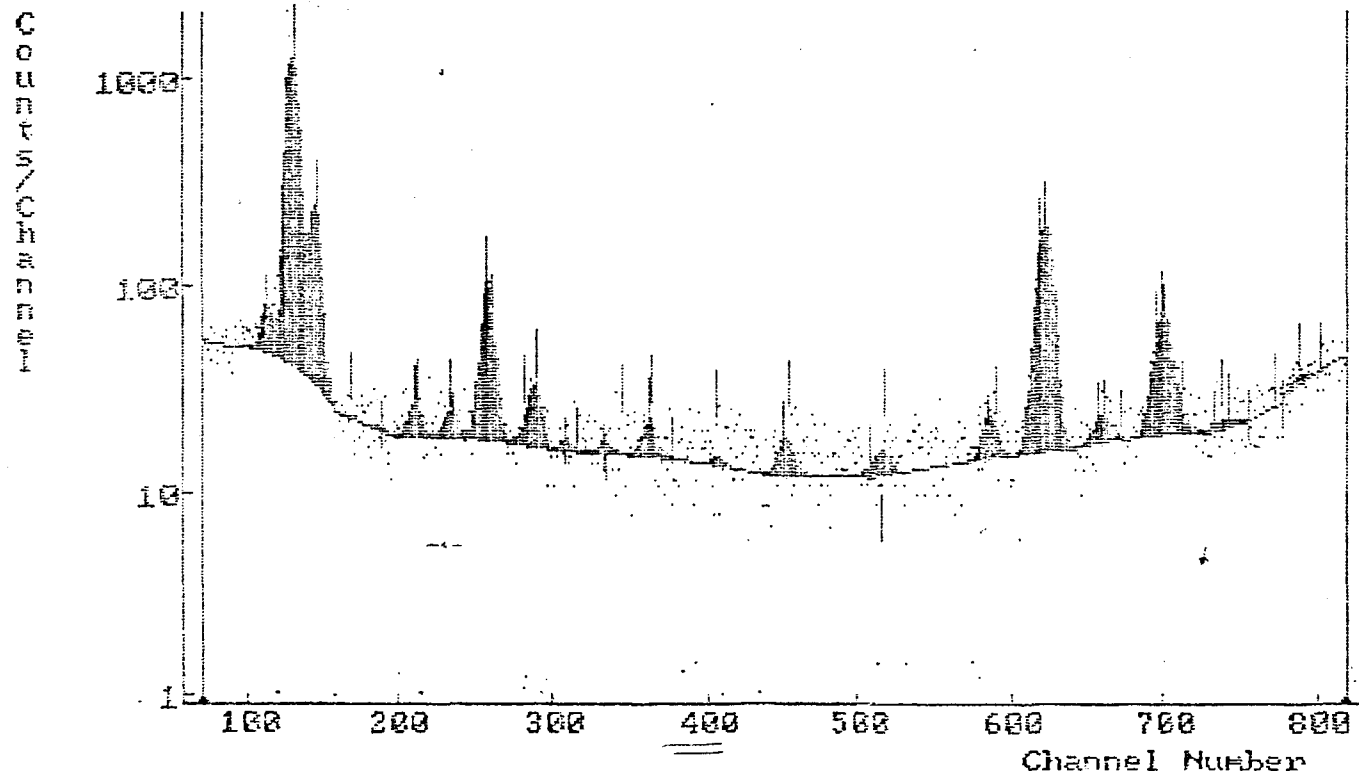
Spectrum REJECT2.SPE (2) Iteration 2: ChiSquare = 1.2; Dif = -1.6



Spectrum GRANIT.SPE (13) Iteration 4: ChiSquare = 1.8; Dif = .0

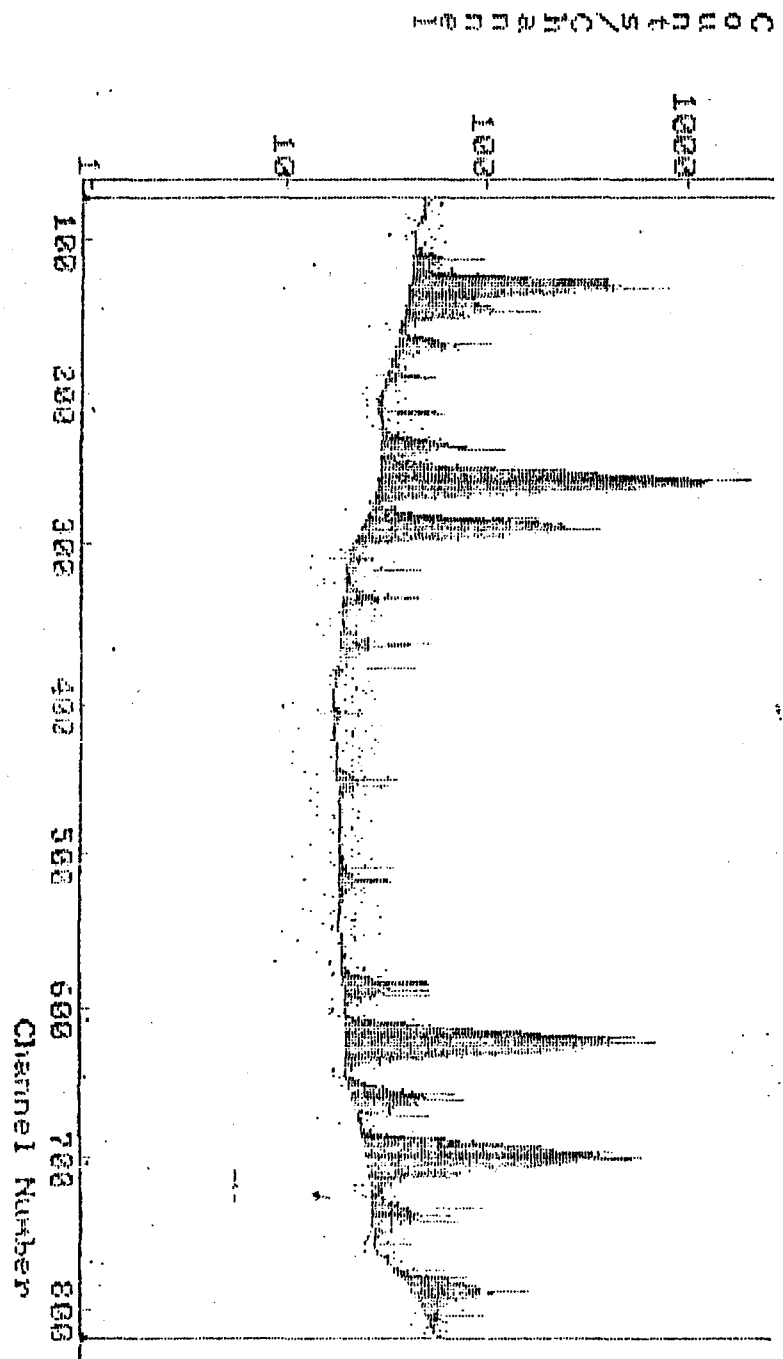


Spectrum STANDRD1.SPE Iteration 3: ChiSquare = 1.2; Dif = -.1





Spectrum STANDRD2.SPE Iteration 3: ChiSquare = 1.1; DIF = .0



Spectrum STANDARD3.SPE Iteration 3: ChiSquare = 1.4; DIF = .0

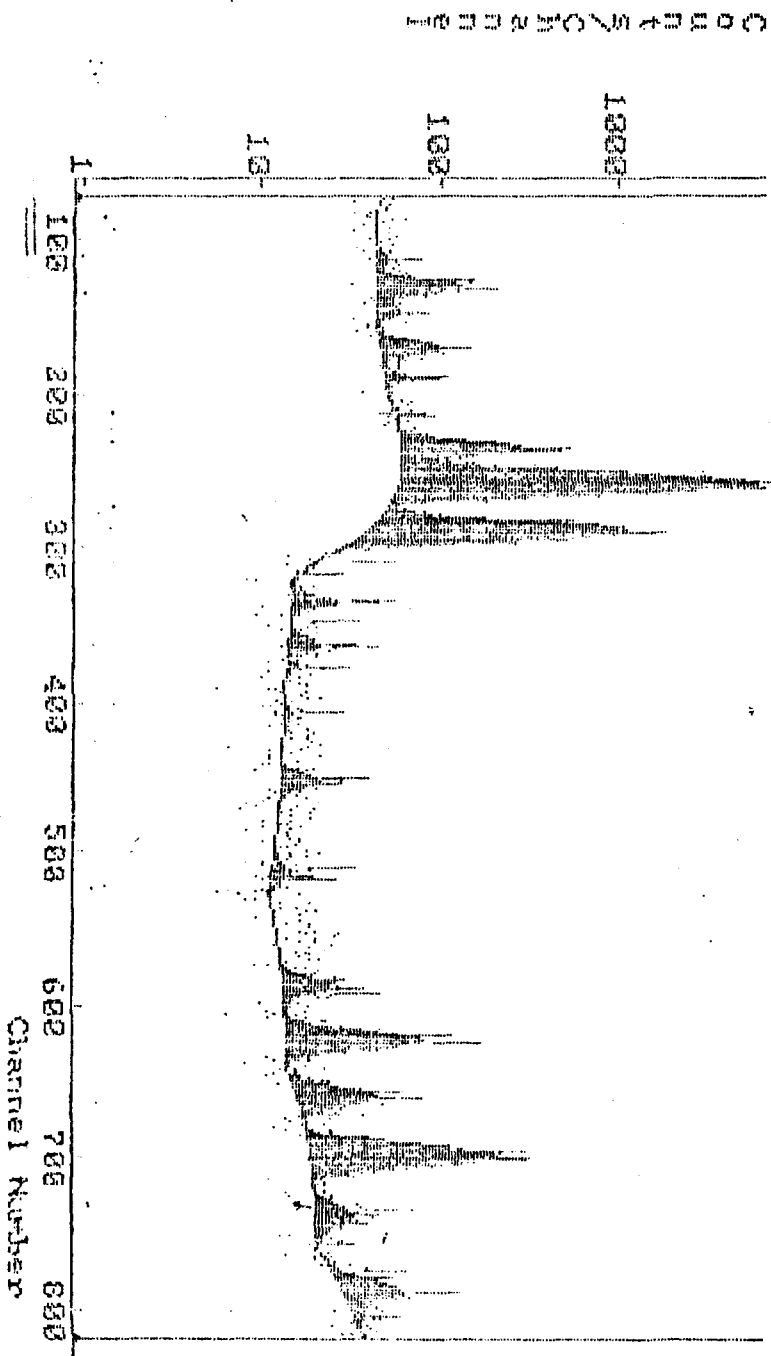


Table (2)

AXIL IBM-PC V3.00 09-14-1994 08:17:37  
Spectrum: LINE1.SPE 1000s

Fitting Region: channels 70 - 830; ChiSqr = 1.0

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K -Ka	3.313	155. ±	27.	1.92
Ca-Ka	3.691	7758. ±	84.	.66
Ti-Ka	4.509	2. ±	18.	.89
V -Ka	4.950	41. ±	19.	.63
Cr-Ka	5.412	14. ±	18.	1.36
Mn-Ka	5.895	42. ±	18.	1.02
Fe-Ka	6.399	1557. ±	43.	.82
Co-Ka	6.925	13. ±	20.	.50
Ni-Ka	7.472	37. ±	17.	1.60
Cu-Ka	8.041	35. ±	17.	.70
Zn-Ka	8.631	57. ±	17.	.71
As-Ka	10.532	48. ±	17.	1.52
Br-Ka	11.908	45. ±	18.	.68
Rb-Ka	13.375	16. ±	18.	1.06
Sr-Ka	14.142	2000. ±	49.	1.34
Y -Ka	14.932	9. ±	18.	.77
Zr-Ka	15.746	48. ±	28.	1.07
Nb-Ka	16.583	33. ±	22.	.87
Mo-Ka	17.443	60. ±	26.	1.01

AXIL IBM-PC V3.00 09-14-1994 08:21:35  
Spectrum: LINE2.SPE 1000s

Fitting Region: channels 70 - 830; ChiSqr = 1.2

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K -Ka	3.313	357. ±	32.	3.17
Ca-Ka	3.691	13966. ±	113.	1.73
Ti-Ka	4.509	13. ±	16.	1.09
V -Ka	4.950	48. ±	18.	.50
Cr-Ka	5.412	15. ±	17.	.97
Mn-Ka	5.895	5. ±	16.	.86
Fe-Ka	6.399	159. ±	20.	1.68
Co-Ka	6.925 *	8. ±	16.	.97
Ni-Ka	7.472	24. ±	15.	1.71
Cu-Ka	8.041	86. ±	16.	1.06
Zn-Ka	8.631	65. ±	16.	.67
As-Ka	10.532	33. ±	15.	1.60
Br-Ka	11.908	39. ±	16.	1.52
Rb-Ka	13.375	29. ±	16.	1.54
Sr-Ka	14.142	3115. ±	58.	1.27
Y -Ka	14.932	18. ±	17.	1.50
Zr-Ka	15.746	66. ±	30.	.65
Nb-Ka	16.583	3. ±	19.	1.23
Mo-Ka	17.443	24. ±	23.	1.09

ACTL 1B11-PC V3.00 09-14-1994 09:46:10  
 Spectrum: LINE3.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.3

Line	Ener. (KeV)	Peak area	st.dev.	Chi_sq
K-Ka	3.313	240. ±	21.	3.07
Ca-Ka	3.691	9396. ±	70.	1.18
Ti-Ka	4.509	5. ±	13.	.73
V-Ka	4.950	24. ±	13.	.96
Cr-Ka	5.412	0. ±	12.	2.78
Mn-Ka	5.895	119. ±	15.	.84
Fe-Ka	6.399	2568. ±	38.	2.21
Co-Ka	6.925	-5. ±	16.	1.08
Ni-Ka	7.472	20. ±	12.	1.75
Cu-Ka	8.041	28. ±	12.	.67
Zn-Ka	8.631	57. ±	12.	.93
Ga-Ka	10.532	63. ±	12.	1.04
Br-Ka	11.908	-5. ±	10.	1.27
Rb-Ka	13.375	11. ±	11.	.96
Sr-Ka	14.142	1267. ±	29.	.65
Y-Ka	14.932	38. ±	13.	2.29
Zr-Ka	15.746	113. ±	19.	.56
Nb-Ka	16.583	4. ±	14.	1.25
Hf-Ka	17.443	55. ±	18.	1.44

ACTL 1B11-PC V3.00 09-14-1994 08:24:57  
 Spectrum: LINE4.SPE 1000s

Fitting Region: channels 70 - 830; ChiSqr = 1.4

Line	Ener. (KeV)	Peak area	st.dev.	Chi_sq
K-Ka	3.313	326. ±	23.	3.54
Ca-Ka	3.691	14454. ±	85.	1.89
Ti-Ka	4.509	0. ±	12.	1.01
V-Ka	4.950	7. ±	12.	1.60
Cr-Ka	5.412	13. ±	12.	.98
Mn-Ka	5.895	35. ±	12.	.75
Fe-Ka	6.399	391. ±	18.	1.23
Co-Ka	6.925	74. ±	12.	.66
Ni-Ka	7.472	22. ±	11.	1.52
Cu-Ka	8.041	36. ±	11.	1.12
Zn-Ka	8.631	13. ±	11.	2.53
Ga-Ka	10.532	15. ±	12.	.70
Br-Ka	11.908	43. ±	11.	1.62
Rb-Ka	13.375	7. ±	11.	1.06
Sr-Ka	14.142	7670. ±	63.	1.02
Y-Ka	14.932	7. ±	12.	1.33
Zr-Ka	15.746	157. ±	28.	1.94
Nb-Ka	16.583	29. ±	15.	1.54
Hf-Ka	17.443	10. ±	17.	1.23

AXIL IBM PC V3.00  
Spectrum: LINE5.SPE

02-14-1994 09:10:58  
1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.2

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K -Ka	3.313	350. ±	32.	3.96
Ca-Ka	3.691	12077. ±	105.	2.14
Ti-Ka	4.509	15. ±	18.	1.24
V -Ka	4.950	12. ±	17.	.58
Cr-Ka	5.412	25. ±	18.	1.86
Mn-Ka	5.895	47. ±	19.	1.03
Fe-Ka	6.399	2396. ±	51.	1.29
Co-Ka	6.925	-36. ±	20.	.89
Ni-Ka	7.472	12. ±	16.	1.74
Cu-Ka	8.041	34. ±	16.	1.12
Zn-Ka	8.631	69. ±	17.	.74
As-Ka	10.532	33. ±	16.	.88
Br-Ka	11.908	92. ±	17.	.68
Rb-Ka	13.375	32. ±	18.	1.11
Sr-Ka	14.142	8915. ±	95.	.76
Y -Ka	14.932	49. ±	20.	.77
Zr-Ka	15.746	411. ±	45.	1.40
Nb-Ka	16.583	9. ±	20.	1.51
Mo-Ka	17.443	-1. ±	24.	1.11

AXIL IBM PC V3.00  
Spectrum: LINE6.SPE

02-14-1994 09:15:23  
1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.2

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K -Ka	3.313	350. ±	33.	3.77
Ca-Ka	3.691	14866. ±	117.	1.47
Ti-Ka	4.509	-3. ±	17.	1.08
V -Ka	4.950	9. ±	17.	1.09
Cr-Ka	5.412	18. ±	17.	1.75
Mn-Ka	5.895	33. ±	17.	1.38
Fe-Ka	6.399	137. ±	20.	1.93
Co-Ka	6.925	32. ±	17.	.72
Ni-Ka	7.472	20. ±	16.	1.24
Cu-Ka	8.041	14. ±	16.	1.49
Zn-Ka	8.631	57. ±	17.	.89
As-Ka	10.532	69. ±	17.	1.12
Br-Ka	11.908	47. ±	17.	.86
Rb-Ka	13.375	4. ±	18.	1.53
Sr-Ka	14.142	8756. ±	94.	1.36
Y -Ka	14.932	19. ±	19.	1.13
Zr-Ka	15.746	120. ±	41.	1.01
Nb-Ka	16.583	3. ±	19.	1.03
Mo-Ka	17.443	18. ±	24.	.99

AXIL IBM-PC V3.00 09-14-1994 09:23:36  
 Spectrum: LINE7.SPE 1000s

Fitting Region: channels 70 - 820; ChiSq = 1.3

Line	Ener. (KeV)	Peak area	St. dev.	Chi_sq
K -Ka	3.313	230. ±	30.	3.25
Ca-Ka	3.691	11755. ±	105.	.82
Ti-Ka	4.509	24. ±	18.	2.46
V -Ka	4.950	28. ±	18.	2.31
Cr-Ka	5.412	5. ±	17.	1.10
Mn-Ka	5.895	36. ±	18.	1.70
Fe-Ka	6.399	572. ±	29.	1.83
Co-Ka	6.925	14. ±	18.	1.78
Ni-Ka	7.472	23. ±	16.	1.22
Cu-Ka	8.041	31. ±	16.	.67
Zn-Ka	8.631	42. ±	16.	.65
As-Ka	10.532	45. ±	16.	.99
Br-Ka	11.908	33. ±	16.	1.56
Rb-Ka	13.375	31. ±	17.	.56
Sr-Ka	14.142	3002. ±	58.	1.25
Y -Ka	14.932	19. ±	18.	1.08
Zr-Ka	15.746	137. ±	31.	3.54
Nb-Ka	16.583	7. ±	19.	1.28
Mo-Ka	17.443	35. ±	21.	1.00

AXIL IBM-PC V3.00 09-14-1994 09:32:03  
 Spectrum: LINE8.SPE 1000s

Fitting Region: channels 70 - 820; ChiSq = 1.3

Line	Ener. (KeV)	Peak area	St. dev.	Chi_sq
K -Ka	3.313	239. ±	21.	3.10
Ca-Ka	3.691	9390. ±	70.	1.21
Ti-Ka	4.509	5. ±	13.	.73
V -Ka	4.950	24. ±	13.	.96
Cr-Ka	5.412	0. ±	12.	2.78
Mn-Ka	5.895	119. ±	15.	.84
Fe-Ka	6.399	2563. ±	33.	2.21
Co-Ka	6.925	4. ±	15.	1.03
Ni-Ka	7.472	21. ±	12.	1.75
Cu-Ka	8.041	23. ±	12.	.67
Zn-Ka	8.631	57. ±	17.	.73
As-Ka	10.532	63. ±	12.	1.03
Br-Ka	11.908	5. ±	10.	1.27
Rb-Ka	13.375	10. ±	11.	.95
Sr-Ka	14.142	1265. ±	29.	.65
Y -Ka	14.932	33. ±	18.	2.29
Zr-Ka	15.746	112. ±	18.	.95
Nb-Ka	16.583	4. ±	13.	1.25
Mo-Ka	17.443	56. ±	18.	1.44

AXIL IBM PC V3.00 09-14-1994 10:05:25  
 Spectrum: GRANIT.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.8

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	27. ±	27.	1.19
Ca-Ka	3.691	191. ±	30.	1.36
Ti-Ka	4.509	594. ±	39.	2.57
V-Ka	4.950	-38. ±	33.	1.29
Cr-Ka	5.412	146. ±	36.	.98
Mn-Ka	5.895	2544. ±	64.	2.19
Fe-Ka	6.399	107893. ±	310.	4.21
Co-Ka	6.925	116. ±	64.	5.46
Ni-Ka	7.472	-9. ±	19.	3.01
Cu-Ka	8.041	71. ±	17.	1.04
Zn-Ka	8.631	35. ±	16.	2.34
As-Ka	10.532	121. ±	18.	1.14
Br-Ka	11.908	-9. ±	15.	.41
Rb-Ka	13.375	41. ±	16.	1.04
Sr-Ka	14.142	110. ±	19.	2.33
Y-Ka	14.932	563. ±	29.	.92
Zr-Ka	15.746	2136. ±	50.	1.46
Hf-Ka	16.583	110. ±	23.	.72
Ho-Ka	17.443	-4. ±	24.	1.01

AXIL IBM-PC V3.00 09-14-1994 08:11:01  
 Spectrum: CLAY.SPE 1000s

Fitting Region: channels 70 - 830; ChiSqr = 1.2

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	91. ±	24.	1.06
Ca-Ka	3.691	935. ±	37.	1.29
Ti-Ka	4.509	344. ±	29.	1.22
V-Ka	4.950	13. ±	24.	.99
Cr-Ka	5.412	14. ±	23.	.69
Mn-Ka	5.895	338. ±	30.	1.21
Fe-Ka	6.399	21266. ±	141.	1.79
Co-Ka	6.925	57. ±	40.	1.39
Ni-Ka	7.472	21. ±	19.	1.20
Cu-Ka	8.041	11. ±	17.	.61
Zn-Ka	8.631	104. ±	19.	1.67
As-Ka	10.532	23. ±	18.	1.80
Br-Ka	11.908	49. ±	18.	2.01
Rb-Ka	13.375	338. ±	26.	1.14
Sr-Ka	14.142	1411. ±	43.	.65
Y-Ka	14.932	317. ±	28.	1.32
Zr-Ka	15.746	3288. ±	63.	.79
Hf-Ka	16.583	201. ±	23.	1.30
Ho-Ka	17.443	-7. ±	29.	1.21

AXIL IBH-PC V3.00

09-14-1994

09:51:07

Spectrum: REJECT1.SPE

1000s

Fitted Region: channels 70 - 820

ChiSqr = 1.1

Line	Elem	(KeV)	Peak area	st. dev.	Chi_sq
K-Ka		3.313	156 ±	26	1.64
Ca-Ka		3.691	4319 ±	67	.53
Ti-Ka		4.509	160 ±	23	1.65
V-Ka		4.950	18 ±	20	1.52
Cr-Ka		5.412	22 ±	20	.91
Mn-Ka		5.895	130 ±	23	1.52
Fe-Ka		6.399	10761 ±	101	1.16
Co-Ka		6.925	32 ±	31	1.80
Ni-Ka		7.472	1 ±	15	1.05
Cu-Ka		8.041	41 ±	16	.69
Zn-Ka		8.631	25 ±	17	1.19
As-Ka		10.532	60 ±	16	1.28
Br-Ka		11.908	27 ±	15	1.86
Rb-Ka		13.375	131 ±	21	1.15
Sr-Ka		14.142	1045 ±	47	.58
Y-Ka		14.937	321 ±	24	.77
Zr-Ka		15.746	1690 ±	40	.81
Nb-Ka		16.583	17 ±	22	1.44
Hf-Ka		17.443	11 ±	26	.85

AXIL IBH-PC V3.00

09-14-1994

09:56:05

Spectrum: REJECT2.SPE

1000s

Fitted Region: channels 70 - 820

ChiSqr = 1.2

Line	Elem	(KeV)	Peak area	st. dev.	Chi_sq
K-Ka		3.313	187 ±	26	1.45
Ca-Ka		3.691	4925 ±	71	1.13
Ti-Ka		4.509	127 ±	23	.75
V-Ka		4.950	20 ±	19	.55
Cr-Ka		5.412	32 ±	19	1.61
Mn-Ka		5.895	193 ±	24	.83
Fe-Ka		6.399	10539 ±	99	1.31
Co-Ka		6.925	6 ±	29	.93
Ni-Ka		7.472	21 ±	16	1.41
Cu-Ka		8.041	50 ±	17	.96
Zn-Ka		8.631	31 ±	17	.80
As-Ka		10.532	51 ±	16	1.16
Br-Ka		11.908	1 ±	13	2.65
Rb-Ka		13.375	201 ±	22	.62
Sr-Ka		14.142	1305 ±	43	1.03
Y-Ka		14.937	325 ±	24	1.10
Zr-Ka		15.746	1334 ±	46	1.05
Nb-Ka		16.583	107 ±	23	1.20
Hf-Ka		17.443	6 ±	24	1.26



AXIL IBM-PC V3.00 09-14-1994 10:01:02  
 Spectrum: PEPellet.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.1

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	148. ±	26.	.85
Ca-Ka	3.691	5104. ±	72.	1.09
Ti-Ka	4.509	133. ±	23.	1.02
V-Ka	4.950	-1. ±	19.	2.30
Cr-Ka	5.412	15. ±	19.	1.09
Mn-Ka	5.895	219. ±	25.	1.44
Fe-Ka	6.399	11510. ±	104.	1.88
Co-Ka	6.925	10. ±	31.	.90
Ni-Ka	7.472	8. ±	16.	.86
Cu-Ka	8.041	45. ±	17.	.64
Zn-Ka	8.631	141. ±	19.	.54
Ga-Ka	10.532	54. ±	16.	1.53
Br-Ka	11.908	37. ±	16.	1.18
Rb-Ka	13.375	131. ±	20.	.90
Sr-Ka	14.142	1918. ±	47.	.77
Y-Ka	14.932	208. ±	23.	1.06
Zr-Ka	15.746	1196. ±	48.	1.23
Nb-Ka	16.583	136. ±	24.	.82
Mo-Ka	17.443	-23. ±	25.	1.22

AXIL IBM-PC V3.00 09-14-1994 10:09:48  
 Spectrum: STANDRD1.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.2

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	334. ±	32.	2.73
Ca-Ka	3.691	12451. ±	108.	.75
Ti-Ka	4.509	9. ±	18.	.88
V-Ka	4.950	16. ±	18.	1.86
Cr-Ka	5.412	34. ±	19.	.73
Mn-Ka	5.895	55. ±	19.	.82
Fe-Ka	6.399	897. ±	34.	.47
Co-Ka	6.925	25. ±	19.	.40
Ni-Ka	7.472	19. ±	16.	1.00
Cu-Ka	8.041	21. ±	16.	1.52
Zn-Ka	8.631	89. ±	18.	1.56
Ga-Ka	10.532	71. ±	16.	2.06
Br-Ka	11.908	45. ±	16.	1.95
Rb-Ka	13.375	106. ±	20.	1.85
Sr-Ka	14.142	2324. ±	52.	1.44
Y-Ka	14.932	74. ±	21.	1.32
Zr-Ka	15.746	355. ±	34.	.45
Nb-Ka	16.583	18. ±	21.	1.04
Mo-Ka	17.443	-5. ±	24.	1.17

AXIL IBH-PC V3.00 09-14-1994 10:13:02  
 Spectrum: STANDRD2.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.1

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	159 ±	28	1.41
Ca-Ka	3.691	3903 ±	65	1.90
Ti-Ka	4.509	271 ±	29	.62
V-Ka	4.950	-3 ±	23	.90
Cr-Ka	5.412	8 ±	21	1.33
Mn-Ka	5.895	321 ±	29	1.42
Fe-Ka	6.399	12732 ±	110	1.41
Co-Ka	6.925	109 ±	36	1.31
Ni-Ka	7.472	22 ±	19	1.63
Cu-Ka	8.041	103 ±	20	.89
Zn-Ka	8.631	98 ±	20	.54
As-Ka	10.532	57 ±	19	1.43
Br-Ka	11.908	37 ±	19	.87
Rb-Ka	13.375	169 ±	24	.54
Sr-Ka	14.142	4976 ±	74	.60
Y-Ka	14.932	336 ±	29	1.65
Zr-Ka	15.746	3962 ±	72	.69
Nb-Ka	16.583	224 ±	29	.69
Mo-Ka	17.443	18 ±	31	.73

AXIL IBH-PC V3.00 09-14-1994 10:18:43  
 Spectrum: STANDRD3.SPE 1000s

Fitting Region: channels 70 - 820; ChiSqr = 1.4

Line	Ener. (KeV)	Peak area	st. dev.	Chi_sq
K-Ka	3.313	64 ±	26	1.03
Ca-Ka	3.691	846 ±	39	.90
Ti-Ka	4.509	535 ±	36	1.36
V-Ka	4.950	40 ±	30	.65
Cr-Ka	5.412	41 ±	30	1.14
Mn-Ka	5.895	2401 ±	59	1.36
Fe-Ka	6.399	69846 ±	242	3.12
Co-Ka	6.925	56 ±	55	2.67
Ni-Ka	7.472	-15 ±	18	3.06
Cu-Ka	8.041	113 ±	19	1.58
Zn-Ka	8.631	73 ±	18	.93
As-Ka	10.532	100 ±	18	1.12
Br-Ka	11.908	24 ±	15	.96
Rb-Ka	13.375	107 ±	21	1.43
Sr-Ka	14.142	885 ±	35	1.46
Y-Ka	14.932	376 ±	27	.75
Zr-Ka	15.746	2535 ±	55	.63
Nb-Ka	16.583	169 ±	26	1.18
Mo-Ka	17.443	50 ±	27	1.05