# Certificate of Analysis

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Type of analysis:	Surface Composition
Description of sample/s:	Powders
Identification of sample/s:	Pb; Wo
Analysed for:	University of Pretoria Pretoria
Location of analysis:	NMISA, Building 5, CSIR Campus, Meiring Naude Road, Pretoria, 0184
Date sample/s received:	22 February 2021
Date/s sample/s analysed:	22-23 February 2021

#### 1. TECHNIQUE

X-ray photoelectron spectroscopy (XPS) consists of irradiating a surface with X-rays to extract photoelectrons. Knowing the energy of the X-ray photons and measuring the kinetic energy of the extracted electrons, one can determine the binding energy of the extracted electrons. This quantity is unique and can be used to identify the elements from which the electrons were extracted. The technique can detect all elements except hydrogen and helium, as well as compounds, because the binding energy of an element differs from compound to compound. It is primarily a surface technique, as the escape depth of the photoelectrons ranges from 2 to 10 nm. The detection limit of XPS is approximately 0,1at%. Information from subsequent layers beneath the outer surface can be obtained by sputtering the surface with energetic argon ions, while monitoring the binding energy regions of specific elements.

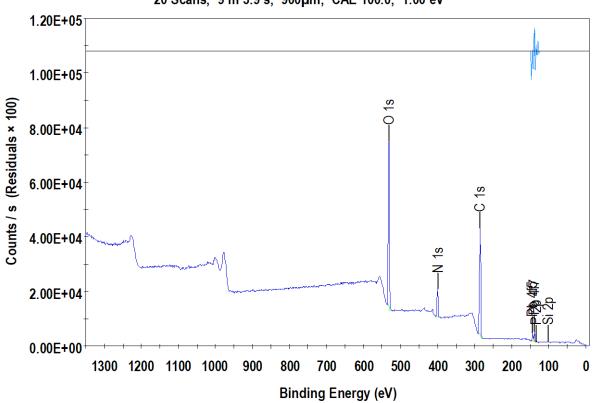
## 2. EXPERIMENTAL SETUP

Parameter	Value
Instrument Brand	Thermo
Model	ESCAlab 250Xi
X-rays	Monochromatic Al kα (1486.7eV)
X-ray Power	300W
X-ray Spot Size	900 μm
Pass Energy (Survey)	100 eV
Pass Energy (Hi-res)	20 eV
Pressure	<10 <sup>-8</sup> mBar

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# 3. RESULTS

Pb - Survey 20 Scans, 9 m 3.9 s, 900μm, CAE 100.0, 1.00 eV

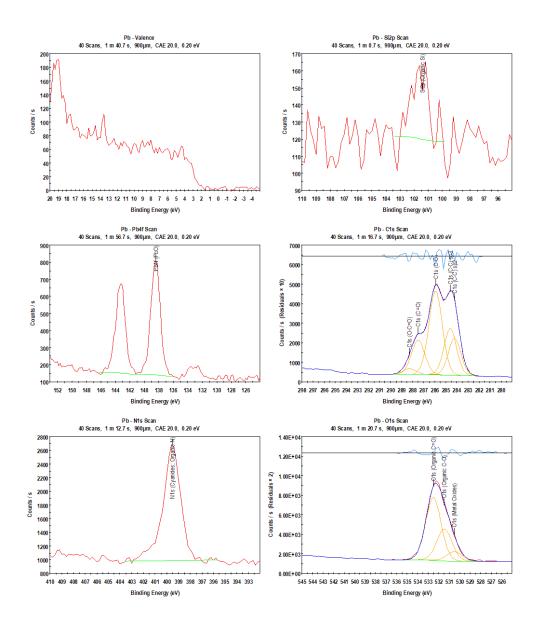


## **Elemental ID and Quantification**

Name	Peak BE	Atomic %
C 1s	285.7	62.0
O 1s	532.1	29.9
N 1s	399.8	7.1
P 2p	133.4	0.5
Si 2p	101.8	0.3
Pb 4f7	138.6	0.2

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### ANALYSIS OF THE SURFACE COMPOSITION OF POWDERS

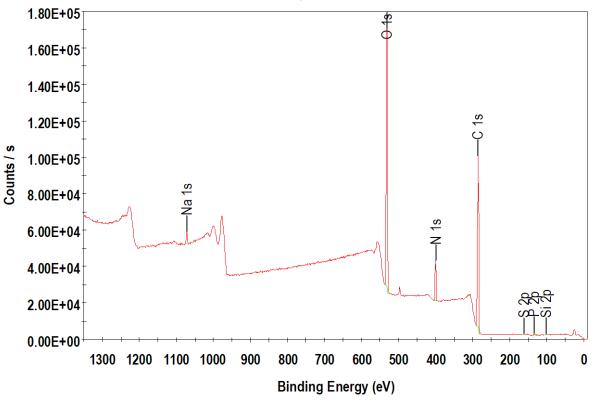


# **Chemical ID and Quantification**

Name	Peak BE	FWHM eV	Atomic %
Si2p (Organic Si)	101.4	0.0	0.2
Pb4f (PbO)	138.4	1.5	0.2
C1s (C-C) sp2	284.2	1.3	10.8
C1s (C-C) sp3	284.6	1.3	13.6
C1s (C-O)	285.9	1.4	26.1
C1s (C=O)	287.5	1.4	10.7
C1s (O-C=O)	288.3	1.4	1.7
N1s (Cyanides; Organic N)	399.5	1.5	7.4
O1s (Metal Oxides)	530.5	1.7	2.7
O1s (Organic C-O)	531.5	1.7	8.9
O1s (Organic C=O)	532.5	1.7	17.5

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\$Wo-Survey\$ 20 Scans, 9 m 3.9 s, 900 $\mu m,$  CAE 100.0, 1.00 eV

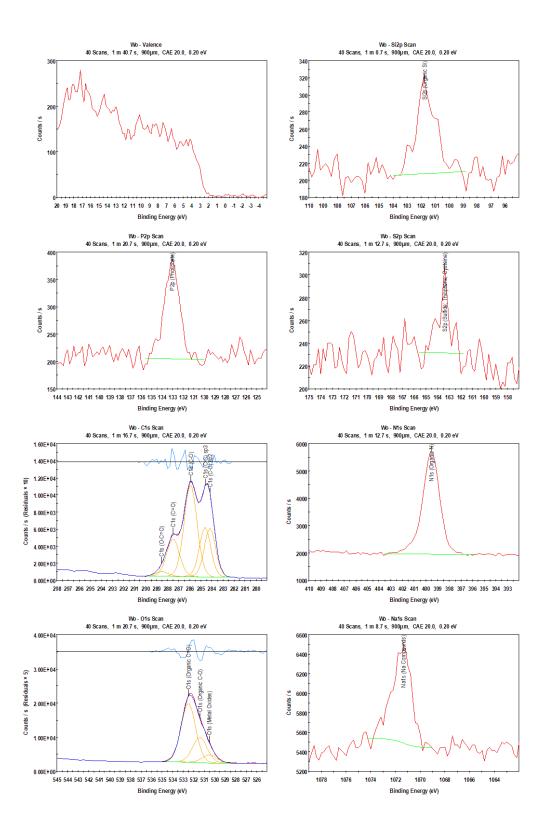


# **Elemental ID and Quantification**

Name	Peak BE	Atomic %
C 1s	285.7	61.4
O 1s	532.1	30.6
N 1s	399.8	6.6
Na 1s	1071.5	0.6
P 2p	133.2	0.4
Si 2p	101.8	0.3
S 2p	163.1	0.1

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### ANALYSIS OF THE SURFACE COMPOSITION OF POWDERS



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#### ANALYSIS OF THE SURFACE COMPOSITION OF POWDERS

### **Chemical ID and Quantification**

Name	Peak BE	FWHM eV	Atomic %
Si2p (Organic Si)	101.8	0.6	0.3
P2p (Phosphate)	133.1	1.6	0.4
S2p (Sulfide; Thiophene; Cysteine)	163.4	0.5	0.1
C1s (C-C) sp2	284.2	1.2	12.3
C1s (C-C) sp3	284.6	1.2	12.5
C1s (C-O)	285.9	1.3	26.1
C1s (C=O)	287.5	1.3	10.6
C1s (O-C=O)	288.6	1.3	1.3
N1s (Organic N)	399.5	1.6	6.9
O1s (Metal Oxides)	530.5	1.6	2.5
O1s (Organic C-O)	531.4	1.6	8.0
O1s (Organic C=O)	532.5	1.6	18.6
Na1s (Na Compounds)	1071.4	1.6	0.4

## 4. COMMENTS

#### 4.1 None

## 5. REMARKS

- 5.1 The sample(s) was/were analysed in the condition as received.
- 5.2 More information about XPS can be found at http://xpssimplified.com
- 5.3 The results in this report relate only to the sample(s) mentioned therein.
- 5.4 Only the results as stated in this certificate are considered to be correct and valid.
- 5.5 Due to the shape, position or intensity of a peak, the actual compound may be different than stated.
- 5.6 Other possible compounds may be identified using the NIST database at: <a href="http://srdata.nist.gov/xps">http://srdata.nist.gov/xps</a>

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