

	<b>Geochemistry</b> <b>Lakefield Laboratory</b>	<b>Doc Type</b> <b>Method Summary</b> <b>Method Code</b> <b>GC_XRF76V/R</b> <b>Service</b> <b>Testing</b> <b>Issued Date</b> <b>December 2021</b>
<b>Natural Resources</b>	<b>Preparation and Determination of Major Element Oxides, LOI and Rare Earth Oxides in Oxide Process Control Products by Borate Fusion and Xray Fluorescence Spectrometry</b> [SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , MgO, CaO, Na <sub>2</sub> O, K <sub>2</sub> O, P <sub>2</sub> O <sub>5</sub> , MnO, TiO <sub>2</sub> , Cr <sub>2</sub> O <sub>3</sub> ; V <sub>2</sub> O <sub>5</sub> ; LOI; additions BaO; Ce <sub>2</sub> O <sub>3</sub> ; Nd <sub>2</sub> O <sub>3</sub> , La <sub>2</sub> O <sub>3</sub> ; Pr <sub>2</sub> O <sub>3</sub> , Sm <sub>2</sub> O <sub>3</sub> ; Nb <sub>2</sub> O <sub>5</sub> , ThO <sub>2</sub> , Ta <sub>2</sub> O <sub>5</sub> ; SnO <sub>2</sub> ; SrO; ZrO <sub>2</sub> ; HfO <sub>2</sub> ; Y <sub>2</sub> O <sub>3</sub> ; WO <sub>3</sub> ; U <sub>3</sub> O <sub>8</sub> ; Co; Ni ; XRF]	<b>Approved by</b> <b>K. Williams</b>

**1. Parameter(s) measured, unit(s):**

Silicon Dioxide (SiO<sub>2</sub>), Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>), Iron(III) Oxide (Fe<sub>2</sub>O<sub>3</sub>), Magnesium Oxide (MgO), Calcium Oxide (CaO), Sodium Oxide (Na<sub>2</sub>O), Potassium Oxide (K<sub>2</sub>O), Phosphorus Pentoxide (P<sub>2</sub>O<sub>5</sub>), Manganese Oxide (MnO), Titanium Dioxide (TiO<sub>2</sub>), Chromium (III) Oxide (Cr<sub>2</sub>O<sub>3</sub>), Vanadium Oxide (V<sub>2</sub>O<sub>5</sub>), LOI, in %

Barium Oxide (BaO), Cerium (III) Oxide (Ce<sub>2</sub>O<sub>3</sub>), Neodymium Oxide (Nd<sub>2</sub>O<sub>3</sub>), Lanthanum Oxide (La<sub>2</sub>O<sub>3</sub>), Praseodymium Oxide (Pr<sub>2</sub>O<sub>3</sub>), Samarium Oxide (Sm<sub>2</sub>O<sub>3</sub>), Niobium Pentoxide (Nb<sub>2</sub>O<sub>5</sub>), Thorium Dioxide (ThO<sub>2</sub>), Tantalum Pentoxide (Ta<sub>2</sub>O<sub>5</sub>), Tin Dioxide (SnO<sub>2</sub>) Uranium Oxide (U<sub>3</sub>O<sub>8</sub>), Cobalt (Co), Nickel (Ni), Strontium Oxide (SrO), Zirconium Dioxide (ZrO<sub>2</sub>), Hafnium Oxide (HfO<sub>2</sub>), Yttrium Oxide (Y<sub>2</sub>O<sub>3</sub>), Tungsten Trioxide (WO<sub>3</sub>) in % can be added as additions Fluoride (F), Chloride (Cl), Sulfide (SO<sub>3</sub>) in % can be added as additions, but are not accredited and are only available for certain sample types.

**2. Typical sample size:**

0.2 to 0.5g

**3. Type of sample applicable (media):**

Metallurgical test products. Intended for oxide minerals only, with levels of sulfide <2% by weight. Fluoride can be added for fluorspar and fluorite material. Chloride and sulfide can be added for potash samples.

**4. Sample preparation technique used:**

Samples are crushed and pulverized according to client specified instructions or default preparation procedures. This method is used to report, in percentage, the whole rock suite (SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, MnO, TiO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>) and Ni, Co as well as the rare earth oxides (La<sub>2</sub>O<sub>3</sub>, Ce<sub>2</sub>O<sub>3</sub>, Nd<sub>2</sub>O<sub>3</sub>, Pr<sub>2</sub>O<sub>3</sub>, Sm<sub>2</sub>O<sub>3</sub>), other major element oxides BaO, SrO, ZrO<sub>2</sub>, HfO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub>, ThO<sub>2</sub>, Ta<sub>2</sub>O<sub>5</sub>, SnO<sub>2</sub>, WO<sub>3</sub>, U<sub>3</sub>O<sub>8</sub>) and other majors (Cl, F, SO<sub>3</sub>). Sample preparation entails the formation of a homogenous glass disk by the fusion of the sample and a lithium tetraborate/lithium metaborate mixture. The LOI is determined separately and gravimetrically at 1000°C.

## 5. Method of analysis used:

The prepared disks are analyzed by wavelength dispersion X-ray fluorescence (WD-XRF). The LOI is included in the matrix correction calculations, which are performed by the XRF software.

## 6. Data reduction by:

Computer, on line, data fed to Laboratory Information Management System with secure audit trail.

## 7. Figures of Merit:

This method has been fully validated for the range of samples typically analyzed. Method validation includes the use of reference materials, replicates, duplicates and blanks to calculate accuracy, precision, linearity, range, limit of detection, reporting limit, specificity and measurement uncertainty.

Element	Report Limit %	Upper limit %	Element	Report Limit %	Upper limit %
SiO <sub>2</sub>	0.01	100	Sm <sub>2</sub> O <sub>3</sub>	0.05	100
Al <sub>2</sub> O <sub>3</sub>	0.01	100	BaO	0.02	100
MgO	0.01	100	La <sub>2</sub> O <sub>3</sub>	0.01	100
Na <sub>2</sub> O	0.01	100	Nd <sub>2</sub> O <sub>3</sub>	0.02	100
K <sub>2</sub> O	0.01	100	Ta <sub>2</sub> O <sub>5</sub>	0.01	100
CaO	0.01	100	SnO <sub>2</sub>	0.01	100
P <sub>2</sub> O <sub>5</sub>	0.01	100	HfO <sub>2</sub>	0.01	100
TiO <sub>2</sub>	0.01	100	ZrO <sub>2</sub>	0.01	100
Cr <sub>2</sub> O <sub>3</sub>	0.01	100	Y <sub>2</sub> O <sub>3</sub>	0.01	100
V <sub>2</sub> O <sub>5</sub>	0.01	100	SrO	0.02	100
Fe <sub>2</sub> O <sub>3</sub>	0.01	100	ThO <sub>2</sub>	0.01	100
MnO	0.01	100	U <sub>3</sub> O <sub>8</sub>	0.01	100
Ni	0.01	100	WO <sub>3</sub>	0.02	100
Co	0.01	100	Nb <sub>2</sub> O <sub>5</sub>	0.01	100
Ce <sub>2</sub> O <sub>3</sub>	0.02	100	F	0.1	50
Pr <sub>2</sub> O <sub>3</sub>	0.03	100	Cl	0.01	60
LOI	-10	100	SO <sub>3</sub>	0.01	2.5

\* A negative LOI indicates a gain on ignition

## 8. Quality control:

Quality control materials include method blanks, replicates and reference materials and are randomly inserted with the frequency set according to method protocols at ~18% for process control analysis. Quality control materials will also include BRM (Barren reference materials, or preparations blanks) and preparation duplicates if samples have been taken through the sample reduction process. Calibration materials that cover the range upon method set-up; calibration check performed as scheduled.

## 9. Accreditation:

SGS Natural Resources conforms to the requirements of ISO/IEC 17025. Scopes of Accredited tests are site specific, please visit <https://www.scc.ca/en/search/laboratories>