

Geological Survey of Canada – Analytical Chemistry Laboratory

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Analytical Procedures

Sample crushing and/or grinding (METHOD PREP-100)

Rock samples were prepared for geochemical analysis by jaw crushing to 1.5 cm, sub-sampling and pulverizing in a Bico ceramic disc grinder followed by reduction to <100 mesh powder in a ceramic ball mill. The final product was a 20g vial of representative powder suitable for acid dissolution or fusion. Approximately 5g total of rock powder was used for the whole-rock and trace-metal analysis.

Major elements (X-ray fluorescence) (METHOD XRF-100)

Analysis was conducted by fused disk wavelength dispersive X-ray Fluorescence. For samples for which there was insufficient powder available to produce a fused disk major element determinations were completed using the ICPES-100 package.

Analyte	Calibration range (%)	Limit of determination (%)
SiO ₂	0 - 100	0.50
TiO ₂	0 - 3	0.02
Al ₂ O ₃	0 - 60	0.40
Cr ₂ O ₃	0 - 4	0.02
Fe ₂ O _{3 total}	0 - 90	0.10
MnO	0 - 1	0.01
MgO	0 - 50	0.10
CaO	0 - 35	0.10
Na ₂ O	0 - 10	0.50
K ₂ O	0 - 15	0.05
P ₂ O ₅	0 - 1	0.02
Ba	0 - 0.30	0.002
Nb	0 - 0.04	0.003
Rb	0 - 0.06	0.002
Sr	0 - 0.20	0.002
Zr	0 - 0.20	0.002

Sample required: 1.0 g.

Major elements (ICP-ES) (METHOD ICPES-100)

Analysis was completed by fusing the sample with a mixed lithium metaborate - lithium tetraborate flux, dissolution of the fusion melt followed by analysis by ICP emission spectrometry. This method was suitable for samples containing greater than 5% S or significant amounts of barite, percent levels of copper or lead or samples for which sufficient quantities were not available for the determination of major elements by XRF.

Analyte	Limit of determination (%)	Analyte	Limit of determination (%)
SiO ₂	0.5	CaO	0.01
TiO ₂	0.02	Na ₂ O	0.03
Al ₂ O ₃	0.2	K ₂ O	0.05
Cr ₂ O ₃	0.02	P ₂ O ₅	0.01
Fe ₂ O ₃	0.06	Ba	0.003
MnO	0.01	Loss on Ignition	0.1
MgO	0.04		

Sample required: 0.5 g

Supplementary whole rock determinations (volatiles)

Ferrous iron was determined using the Wilson Method (titrimetric). H₂O (total), CO₂ (total) and S were determined using combustion followed by infra-red spectrometry, CO₂ (carbonate carbon) was determined by acid evolution followed by infrared spectrometry and C (total non-carbonate carbon) was calculated. Loss on ignition was determined by gravimetry at 900° C.

Parameter	Sample required	Limit of determination, %	Method
FeO	0.2g	0.2	METHOD CHEM-100
H ₂ O _{total}	0.5g	0.1	METHOD CHEM-110
CO _{2total}	0.5g	0.1	METHOD CHEM-120
CO ₂	0.5g	0.1	METHOD CHEM-130
S _{total}	0.5g	0.02	METHOD CHEM-140

Trace elements

Determinations were based on the total dissolution of the sample using nitric, perchloric and hydrofluoric acids followed by a lithium metaborate fusion of any residual material. For ICPE-110 package analysis was done using ICP emission spectrometry. For the ICPMS-100 and ICPMS-110 packages, analysis was done using ICP mass spectrometry. Elements listed in ICPE-110 may be determined by ICPMS-100 or ICPMS-110 when analyte concentrations were too low for determination by ICP-ES.

Trace elements 1 (ICP emission spectrometry) METHOD ICPMS-110. Sample required: 1.0 g

Element	Limit of determination (ppm)	Element	Limit of determination (ppm)
Ag	5	Pb	10
Ba	10	Sc	0.5
Be	0.5	Sr	5
Co	5	V	5
Cr	10	Y	5
Cu	10	Yb	0.5
La	10	Zn	5
Ni	10	Zr	10
Mo	5		

Trace elements 2 (ICP mass spectrometry): Rare-earth elements + Y METHOD ICPMS-100.

Element	Limit of determination (ppm)	Element	Limit of determination (ppm)
Ce	0.1	Nd	0.1
Dy	0.02	Pr	0.02
Er	0.02	Sm	0.02
Eu	0.02	Tb	0.02
Gd	0.02	Tm	0.02
Ho	0.02	Y	0.02
La	0.1	Yb	0.05
Lu	0.02		

Trace elements 2 (ICP mass spectrometry): Other trace elements METHOD ICPMS-110. Sample required: 1.0 g.

Element	Limit of determination (ppm)	Element	Limit of determination (ppm)
Ag	0.1	Pb	2
Bi	0.5	Rb	0.05
Cd	0.2	Sn	0.5
Cs	0.02	Ta	0.2
Ga	0.1	Th	0.02
Hf	0.05	Tl	0.02
In	0.05	U	0.02
Mo	0.2	Zr	0.5
Nb	0.05		

Fluorine, chlorine, and sulphur (METHOD IC-100)

Fluorine, chlorine and sulphur were determined using a pyrohydrolysis method followed by ion chromatography. For this method, the upper limit of determination was 1%.

Sample required: 0.2 g.

Element	Limit of detection (ppm)
F	50
Cl	100
S	50



Analytical Chemistry Laboratories - Determination Limits and Accuracy Estimates
Laboratoire de Chimie Analytique - Estimation de la limite de quantification et de l'exactitude

	Chemistry			Ion Chromatography			XRF-FD Majors			ICP-OES Majors		
	D.L.	Absolute (%)	Relative (% of conc.)	D.L. (ppm)	Absolute (%)	Relative (% of conc.)	D.L. (%)	Absolute (%)	Relative (% of conc.)	D.L. (%)	Absolute (%)	Relative (% of conc.)
FEO	0.2	0.2	5	---	---	---	---	---	---	---	---	---
FE2O3	---	---	---	---	---	---	---	---	---	---	---	---
H2OT	0.1	0.1	5	---	---	---	---	---	---	---	---	---
CO2T	0.1	0.1	3	---	---	---	---	---	---	---	---	---
CO2	0.2	0.2	5	---	---	---	---	---	---	---	---	---
C	---	---	---	---	---	---	---	---	---	---	---	---
ST	0.02	0.02	5	---	---	---	---	---	---	---	---	---
F	---	---	---	50	50	5	---	---	---	---	---	---
Cl	---	---	---	100	100	5	---	---	---	---	---	---
Sic	---	---	---	50	50	5	---	---	---	---	---	---

SiO2	---	---	---	---	---	---	0.4	0.4	1	0.5	0.5	1
TiO2	---	---	---	---	---	---	0.05	0.05	1	0.02	0.02	1
AL2O3	---	---	---	---	---	---	0.2	0.2	1	0.2	0.2	1
Cr2O3	---	---	---	---	---	---	0.02	0.02	1	---	---	---
FE2O3T	---	---	---	---	---	---	0.1	0.1	1	0.06	0.06	1
MNO	---	---	---	---	---	---	0.02	0.02	1	0.01	0.01	2
MGO	---	---	---	---	---	---	0.1	0.1	1	0.04	0.04	1
CAO	---	---	---	---	---	---	0.1	0.1	1	0.01	0.01	1
NA2O	---	---	---	---	---	---	0.1	0.1	1	0.03	0.03	1
K2O	---	---	---	---	---	---	0.05	0.05	1	0.05	0.05	1
P2O5	---	---	---	---	---	---	0.02	0.02	1	0.01	0.01	1
S	---	---	---	---	---	---	0.02	0.02	5	---	---	---

	ICP-OES Traces			XRF-FD Traces			ICP-MS Traces			ICP-MS REEs		
	D.L. (ppm)	Absolute (ppm)	Relative (% of conc.)	D.L. (ppm)	Absolute (ppm)	Relative (% of conc.)	D.L. (ppm)	Absolute (ppm)	Relative (% of conc.)	D.L. (ppm)	Absolute (ppm)	Relative (% of conc.)
Ag	40	40	10	---	---	---	0.1	0.1	---	---	---	---
Ba	20	20	10	30	30	3	---	---	---	---	---	---
Be	0.5	0.5	5	---	---	---	---	---	---	---	---	---
Bi	---	---	---	---	---	---	0.2	0.2	---	---	---	---
Cd	---	---	---	---	---	---	0.2	0.2	---	---	---	---
Co	5	5	5	---	---	---	---	---	---	---	---	---
Cr	10	10	5	---	---	---	---	---	---	---	---	---
Cs	---	---	---	---	---	---	0.02	0.02	---	---	---	---
Cu	10	10	5	---	---	---	---	---	---	---	---	---
Ga	---	---	---	---	---	---	0.1	0.1	---	---	---	---
Hf	---	---	---	---	---	---	0.05	0.05	---	---	---	---
In	---	---	---	---	---	---	0.05	0.05	---	---	---	---
Mo	40	40	10	---	---	---	0.2	0.2	---	---	---	---
Nb	---	---	---	20	20	3	0.05	0.05	---	---	---	---
Ni	10	10	5	---	---	---	---	---	---	---	---	---
Pb	40	40	10	---	---	---	1	1	---	---	---	---
Rb	---	---	---	20	20	2	0.05	0.05	---	---	---	---
Sb	---	---	---	---	---	---	0.2	0.2	---	---	---	---



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Sc	0.5	0.5	5	---	---	---	---	---	---	---	---	---
Sn	---	---	---	---	---	---	0.5	0.5	---	---	---	---
Sr	10	10	10	20	20	2	---	---	---	---	---	---
Ta	---	---	---	---	---	---	0.05	0.05	---	---	---	---
Te	---	---	---	---	---	---	0.2	0.2	---	---	---	---
Th	---	---	---	---	---	---	0.02	0.02	---	---	---	---
Tl	---	---	---	---	---	---	0.02	0.02	---	---	---	---
U	---	---	---	---	---	---	0.02	0.02	---	---	---	---
V	5	5	5	---	---	---	---	---	---	---	---	---
Zn	5	5	5	---	---	---	---	---	---	---	---	---
Zr	40	40	10	20	20	2	0.5	0.5	---	---	---	---
Ce	---	---	---	---	---	---	---	---	---	0.1	0.1	---
Dy	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Er	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Eu	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Gd	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Ho	---	---	---	---	---	---	---	---	---	0.02	0.02	---
La	10	10	5	---	---	---	---	---	---	0.1	0.1	---
Lu	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Nd	---	---	---	---	---	---	---	---	---	0.1	0.1	---
Pr	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Sm	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Tb	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Tm	---	---	---	---	---	---	---	---	---	0.02	0.02	---
Y	5	5	5	---	---	---	---	---	---	0.02	0.02	---
Yb	0.5	0.5	5	---	---	---	---	---	---	0.02	0.02	---

Notes:

- 1: Fe₂O₃ is a calculated value.
- 2: C is a calculated value.
- 3: -999 concentration - value not determined
- 4: FD - fused disc
- 5: OES - Optical Emission Spectroscopy
- 6: D. L. - Determination limit