

Ontario Geological Survey - Geoscience Laboratories

Contact Information

Geoscience Laboratories

933 Ramsey Lake Road
Sudbury, ON, P3E 6B5, Canada

Tel: (705) 670-5632; 866-GEO LABS

Fax: (705) 670-3047

Email: geoscience.labs@ndm.gov.on.ca

Analytical Procedures

Closed Beaker Digestion –CT4

Four acids (hydrofluoric, hydrochloric, perchloric and nitric acid) were used in a closed digestion vessel to ensure total dissolution of all solids. This method was preferred for the determination of rare earth elements (REE: La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu), high field strength elements (HFSE: Zr, Nb, Hf, Ta) and large ion lithophile elements (LILE: Rb, Sr, Cs, Ba) plus Y, U, and Th. The sample size was 0.5 g.

Two solutions were used for the complete digestion of the pulverized geological sample: T4A (which contains 1:1:10 of HCl: HClO₄:HF) and T4B (which contains 25:50:1 of DDW:HCl: HClO₄). 0.200 grams of sample was weighted in a 60 mL Savillex closed digestion vessel, to which 15 mL of T4A solution was added. The vessel was capped tightly and placed in a well ventilated oven at 120°C for seven days. After seven days the samples were removed from the oven, allowed to cool, transferred to 50 mL PTFE beakers, and placed on a (MAKE) Hotplate at 120°C for 24-48 hours or until the cake has dried. Once dry, 15 mL of T4B was added and were again placed on the Hotplate at 120°C for 24-48 hours and allowed to go to dryness. Eight drops of HCl, 1 mL of HNO₃ and a few drops of HF were added to redissolve the digestion cake, and were further brought into solution using DDW. The beakers were replaced on the hotplate to bring all the cake into solution, then allowed to cool. The hotplate acid digestion takes place under a well ventilated fumehood.

1 mL of a 10 ppm Re Ru internal solution was added to a clean Class A volumetric flask using a 1000 µL Eppendorf pipette. The beaker contents were added followed with two rinses of 10% HNO₃. The solution was brought to the final volume of 100 mL and mixed thoroughly before transferring to a 12 mL snap-cap disposable (BRAND?) test tube.

ICP Analysis IM-100/101

The Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) IM-100 package was designed as a general technique that gives accurate data for minor elements and many of the more abundant trace elements.

The ICPMS IM-101 package was designed as a specialized analysis for the accurate determination of the rare-earth elements (REE: La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu) plus Y, high field strength elements (HFSE: Zr, Nb, Hf, Ta, Th, and U) and large-ion lithophile elements (LILE: Rb, Sr, Cs, and Ba) contained in most geological samples. Since more instrument time was spent per element and a larger number of corrections for potential interferences were applied, the data for these elements may be considered more accurate and precise than those obtained using the IM-100 package.

Quantitative analysis was done on the Perkin-Elmer ELAN 5000 ICP-MS equipped with random-access autosampler, and the calibration solutions consist of three well-characterized certified reference materials and a blank (IM-100) or a calibration solution prepared from calibration solutions of precisely known trace element concentrations and an appropriate blank in matrix-matched solutions (IM-101).

The lower limits (LL) and upper limits (UL) for both IM-100 and IM-101 by ICP-MS are listed as follows:

	IM-100/CT4		IM-101/CT4	
	LL	UL	LL	UL
	ppm	ppm	ppm	ppm
Al	500	120000		
Sb	0.05	2		
Ba	0.6	1400	0.8	800
Be	0.3	12		
Bi	0.05	0.5		
Cd	0.03	0.3		
Ca	200	100000		
Ce	0.06	250	0.07	200
Cs	0.007	40	0.007	600
Cr	8	400		
Co	0.1	120		
Cu	0.7	140		
Dy	0.01	20	0.008	20
Er	0.009	6	0.008	20
Eu	0.007	5	0.005	5
Gd	0.01	20	0.009	20
Ga	0.1	40		
Hf	0.05	30	0.1	30
Ho	0.003	2	0.003	10
Fe	500	100000		
La	0.02	100	0.02	100
Pb	0.4	40		
Li	0.5	140		
Lu	0.005	1.5	0.003	5
Mg	500	80000		
Mn	2	1400		
Mo	0.07	5		
Nd	0.04	200	0.03	100
Ni	0.8	300		
Nb	0.09	100	0.2	80
Pb	200	50000		
K				
Pr	0.01	50	0.006	25
Rb	0.07	250	0.05	150
Sm	0.01	30	0.01	30
Sc	0.6	50		
Na				
Sr	1	1400	0.5	1200

	IM-100/CT4		IM-101/CT4	
S				
Ta	0.07	20	0.17	20
Tb	0.004	1.5	0.003	5
Tl	0.01	2		
Th	0.03	60	0.06	100
Tm	0.005	2.5	0.003	5
Sn	0.07	10		
Ti	17	20000		
W	0.04	30		
U	0.004	10	0.007	20
V	1	300		
Yb	0.02	8	0.01	20
Y	0.03	120	0.02	120
Zn	3	150		
Zr	1	600	4	1200