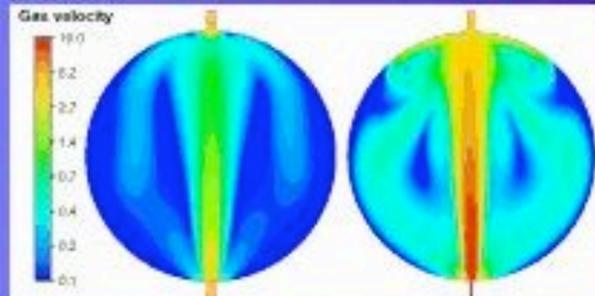


Mineralogical Association of Canada

Laser Ablation ICP-MS in the Earth Sciences: Current Practices and Outstanding Issues

Editor

Paul Sylvester



Short Course Series
Volume

40

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Robert Raeside



**Laser Ablation–ICP–MS in the Earth Sciences
CURRENT PRACTICES AND OUTSTANDING ISSUES**

Mineralogical Association of Canada
Short Course Series Volume 40

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VARIAN

APPENDIX A5: ANALYSIS MANAGEMENT SYSTEM (AMS) FOR REDUCTION OF LASER ABLATION ICP-MS DATA

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INTRODUCTION

This paper describes the Analysis Management System (AMS) software that has recently been developed to facilitate the reduction of data from laser ablation inductively coupled plasma-mass spectrometric (LA-ICP-MS) analysis of various materials. While the AMS software is applicable to analysis of any material, it was developed to address analytical and data reduction issues that are unique to the analysis of materials that are small (resulting in a short-lived signal) and/or heterogeneous at the scale (both spatial and temporal) of the analysis. Specifically, the AMS software is designed and optimized for reduction of data from fluid and melt inclusions in geologic samples (Roedder 1984).

In developing the AMS software, our goals were to develop an easy to use tool for reduction of LA-ICP-MS data from fluid and melt inclusions. To accomplish this we have automated many of the time-consuming steps commonly involved in LA-ICP-MS data reduction, such as normalization to 100% oxides. In addition, the AMS can provide more accurate and precise results by minimizing systematic errors, by allowing the use of multiple standards, and by incorporating an improved drift correction methodology.

The AMS software has the option to provide output as a single file that contains the ICP-MS data, the various options for data reduction, and the results, and is thus highly transportable. Moreover, AMS is a stand-alone software package that eliminates the need to purchase and learn to use commercial software. AMS software is written in Java and runs on Unix, Macintosh or Windows platforms. AMS is compatible with data formats used by most of the major ICP-MS manufacturers (*e.g.*, Perkin Elmer, Agilent, or any other instrument that has CSV-formatted output). AMS outputs the results in Excel, PDF, HTML or CSV formats. It is fully configurable by individual users and allows editing of XML text files to include, for example,

additional standards or new mass conversion values. Importantly, the software incorporates well established algorithms for data analysis (Gunther *et al.* 1998, Halter *et al.* 2002, Heinrich *et al.* 2003, Langerich *et al.* 1996).

Both the software and source code are available on the Fluids Research Laboratory website at: <http://www.geochem.geos.vt.edu/fluids/laICP-MS/ams.shtml>. AMS software is a community project in which interested persons can submit requests to enhance the software or report bugs. AMS software is an Open Source application under GPL.

In the following sections we describe those features of the AMS software that make it unique or which represent improvement over existing LA-ICP-MS data reduction packages. This is accomplished by describing features for using multiple standards, applying drift corrections, visualizing fractionation patterns during the analysis and finally by providing working examples of the reduction of LA-ICP-MS data from fluid and melt inclusions.

USE OF MULTIPLE STANDARDS AND DRIFT CORRECTION

AMS supports the use of multiple standards for a single analysis. In addition, AMS supports multiple standard runs (at different times) for each standard. A typical data reduction in AMS may include 6 or more spectra from 2 or more standards (*e.g.*, NIST 610, 612, *etc.*) that bracket (in time) the actual FI or MI analysis. The uncertainty in standard concentrations has been shown to be one of the largest sources of error in LA-ICP-MS analysis. If multiple standards (as a multi-point calibration) are used during the analysis of a sample with “known” element concentrations, such as the G-Probe-2 microprobe standard (Potts *et al.* 2008), the error in element abundance is commonly less than if only a single standard was used (Fig. A5-1).

If the FI or MI analysis is bracketed by standard runs, AMS can also correct for any

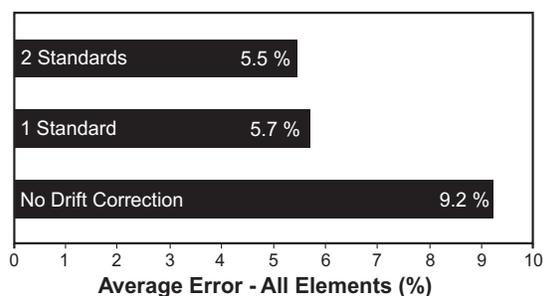


FIG. A5-1. Comparison of the average error in concentration (20 analyses) of 35 elements [Al, B, Ba, Ca, Ce, Cr, Cs, Cu, Eu, Fe, Gd, Hf, K, La, Mg, Mn, Na, Nb, Nd, Ni, Pb, Rb, Sc, Si, Sm, Sr, Ta, Tb, Th, Ti, U, V, Y, Zn, Zr] in the G-Probe-2 standard for data reduction involving only a single point standard with no drift correction (No Drift Correction), a single standard with drift correction (1 Standard), and two standards with drift correction (2 Standards).

instrumental drift (if present). AMS calculates the time-dependent drift in concentration for each element by calculating the element concentration using each standard run as a single point calibration. AMS then does a regression of element concentration vs. the time during the analytical sequence when the standard was analyzed. If the correlation (R^2) is greater than a user-specified value, AMS uses the regression value; otherwise AMS uses the concentration of the element calculated using the standard run closest in time (either before or after) to the analysis time. Analysis times are inferred from the ICP-MS data file time stamp.

An example of the method used for drift correction is shown in Figure A5-2 for Rb. Sample G-Probe-2 (Potts *et al.* 2008) was analyzed 20 times over a period of about 200 minutes, with the NIST 610 and 612 standards analyzed at the beginning, near the middle, and at the end of the session. Shown on the

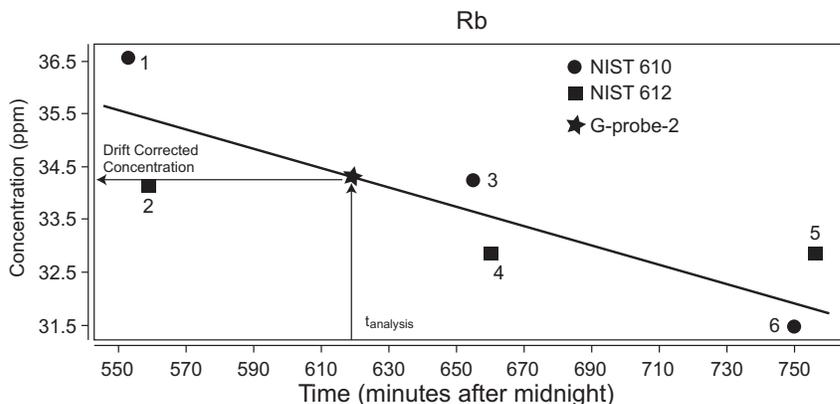


FIG. A5-2. Concentration of Rb in sample G-Probe-2 that would be obtained for the analysis conducted at ≈ 618 min using six single point standards (labeled 1 to 6), compared to the concentration that is obtained if the six single point standards are used to define an instrumental drift correction line.

diagram are six concentrations for Rb in the G-Probe-2 sample that would have been obtained if only a single point calibration was used. For example, if the NIST 610 standard labeled “1” had been used as a single point standard, the concentration predicted for the unknown analyzed at time $t=618$ min would have been about 36.5 ppm. Similarly, if the NIST 610 standard labeled “6” had been used to determine the composition of Rb in the analysis conducted at 618 min, the estimated concentration would have been about 31.5 ppm. However, by analyzing multiple standards periodically during the entire analytical session and using these data to define a drift correction defined by the linear regression line through the standards data, a corrected concentration of approximately 34.6 ppm is obtained. The results show clearly that the use of only a single point reference standard, collected either before or after the analytical session, that did not take into account instrumental drift could lead to a less accurate result for the unknown.

ELEMENTAL FRACTIONATION DURING ANALYSIS

Elemental fractionation can be a major issue during LA-ICP-MS analyses (Gunther & Hattendorf 2001). AMS provides a method to estimate the amount of fractionation for each element in a homogeneous sample volume through a series of fractionation plots (FP). When the option to generate the FP is on (see below), the signal region of interest (SROI) selected by the operator is divided into 10 slices and data reduction is performed on each slice. After the analysis the results are plotted as concentration vs. time, allowing a visual estimate of how SROI selection affects the outcomes of the calculations (Fig. A5-3).

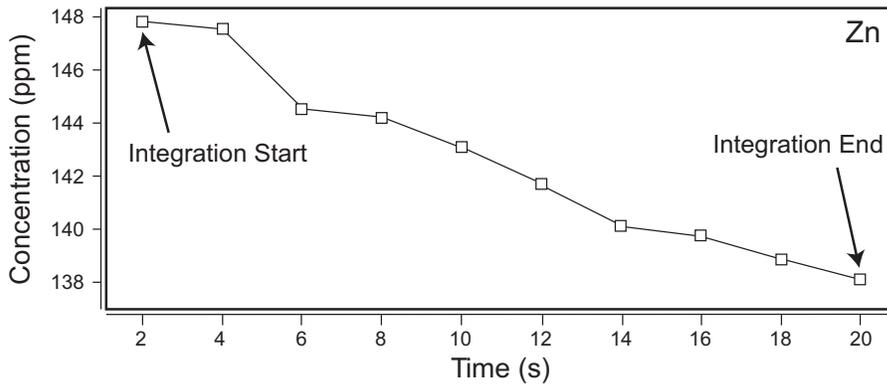


FIG. A5-3. Fractionation plot produced by AMS showing the concentration of zinc as a function of time in the signal region of interest.

The ideal output (*i.e.*, complete absence of fractionation) would be a flat line ($\Delta C/\Delta t = 0$) where the concentration (C) in each of the 10 slices is the same. Additional plots also display the standard/element signal ratios. The signal ratio plots show the stability (or lack thereof) of the intensity ratios during the analysis.

FLUID INCLUSION ANALYSIS AND DATA REDUCTION

The logical steps followed during analysis of fluid and melt inclusions are shown in Figure A5-4. The analysis of fluid or melt inclusions starts with detailed petrographic examination of each sample to determine the relationship of the inclusions to the

host mineral phase and to the petrogenesis of the sample (Bodnar 2003, Bodnar & Student 2006) (Fig. A5-4, box 1). After the fluid inclusions have been selected, the freezing point depression or the halite disappearance temperature of the inclusions is determined by microthermometry (Fig. A5-4, box 2). These temperatures are entered into AMS, which then uses published algorithms (Bodnar 1993, Bodnar *et al.* 1989, Sterner *et al.* 1988) to calculate an NaCl-equivalent bulk salinity of the inclusion (Fig. A5-4, box 3). The bulk salinity of the inclusion, in conjunction with elemental concentration ratios determined by LA-ICP-MS analysis, is used to calculate the absolute element concentrations in the inclusion.

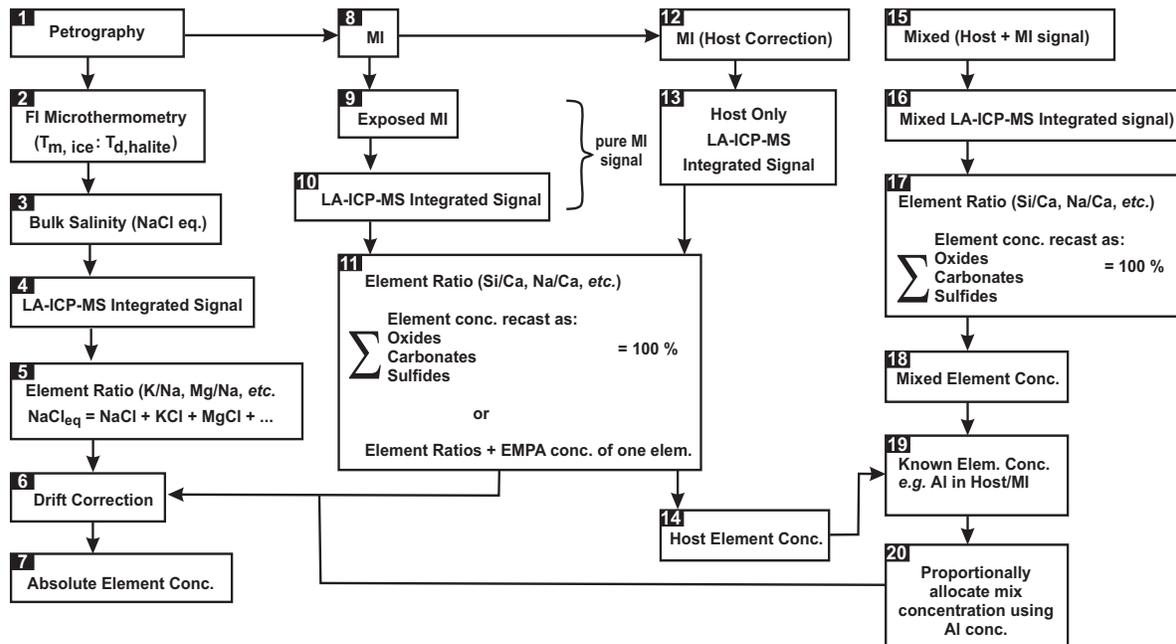


FIG. A5-4. Flow chart showing the sequence of tasks involved in the analysis and data reduction for fluid and melt inclusions using AMS software.

AMS uses the LA-ICP-MS signal from the FI (Fig. A5-4, box 4) and a known external standard (e.g., NIST 610) to calculate elemental concentration ratios (Fig. A5-4, box 5). AMS uses the integrated signal areas after background subtraction for the FI and standard to determine elemental intensity ratios using equation (1) (from (Longerich *et al.* 1996)). The use of an external standard eliminates the effects of element-specific instrumental sensitivity (Longerich *et al.* 1996).

$$\frac{C_{Ca}^{Sample}}{C_{Na}^{Sample}} = \frac{C_{Ca}^{Stand}}{C_{Na}^{Stand}} \times \frac{I_{Ca}^{Sample}}{I_{Na}^{Sample}} \times \frac{I_{Na}^{Stand}}{I_{Ca}^{Stand}} \quad (1)$$

The absolute concentrations of individual elements in the inclusion are determined from the calculated element ratios and the bulk salinity obtained by microthermometry using mass balance constraints (Heinrich *et al.* 2003) (Fig. A5-4, box 7), with or without a drift correction (Fig. A5-4, box 6).

After the laser ablation ICP-MS spectra have been collected, one launches the options window in AMS (Fig. A5-5) to define the parameters used

during the analysis, including which standards were used, how many standard analyses were conducted, and the method that will be used to calculate concentrations. In the example shown in Figure A5-5, four analyses of NIST 610 (two at the beginning of the session and two at the end) were used for the external standards. During microthermometry, the ice phase melted at -18°C , and this value is entered in the “Method” section and used to calculate the bulk salinity. The host correction options shown in the lower left are not required for this fluid inclusion analysis because the host is quartz and we did not measure Si in the FI. The instrument settings used during the analysis included concentrations in counts, with a dwell time of 0.01 sec and drift correction confidence interval (R^2) of 0.9. In this example, the “Generate fractionation plots” option was not selected. At the conclusion of the data reduction process, the results window (see below) will be shown and the results will be written to an Excel file, as defined by the output options.

Once all the options have been set, the ICP-MS

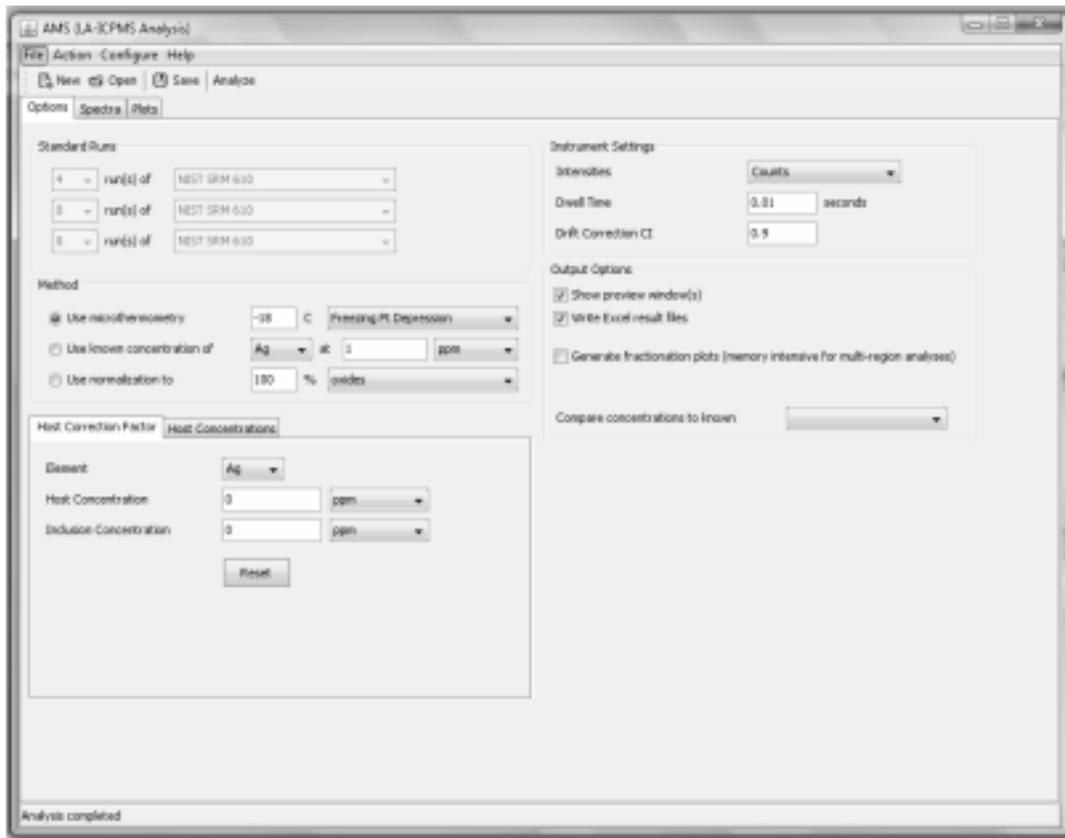


FIG. A5-5. Options Window to define the conditions that were used during data collection and methods to be used for data reduction in AMS.

data are loaded into AMS to define the signal regions of interest (SROI). The shaded region on the left of Figure A5-6 represents the SROI over which the background signal will be integrated to determine average background intensities. AMS automatically makes a “best guess” of the background and sample SROIs based on signal intensities from the current cycle compared to the average intensity for the three previous cycles.

However, these may be modified manually after visual inspection of the spectra. When the laser is turned on to begin the ablation process, the Si signal intensity increases significantly as the laser ablates through the quartz host overlying the fluid inclusion. After a few seconds the laser has ablated into the inclusion and the intensities of those elements contained in the inclusion (*i.e.*, Na, Ca, K, Sr, Ba, *etc.*) increase rapidly and then tail off as the material is quickly removed from the inclusion. The shaded region on the middle-right side of Figure A5-6 represents that portion of the signal that will

be integrated to obtain signal intensities from the inclusion. As noted above, AMS selects this region automatically but the operator may adjust the limits of integration manually. The process of defining the SROIs is repeated for each analysis as well as for the standard runs.

Once the background and fluid inclusion signal integration ranges have been defined, clicking on the “Analyze” button produces the results window showing the elements measured; concentration of the elements in ppm (Con. (ppm)); limits of detection (LOD (ppm)); concentration in weight percent of the chloride salt containing the element (Weight %); counts per second above background for the sample (Sample (cps)); background counts per second for the sample (Bkg (cps)); counts per second above background for the standard (Std (cps)); background counts per second for the standard (Bkg (cps)) (Fig. A5-7). Additional information to identify the sample are included in the header of the results window. The results are

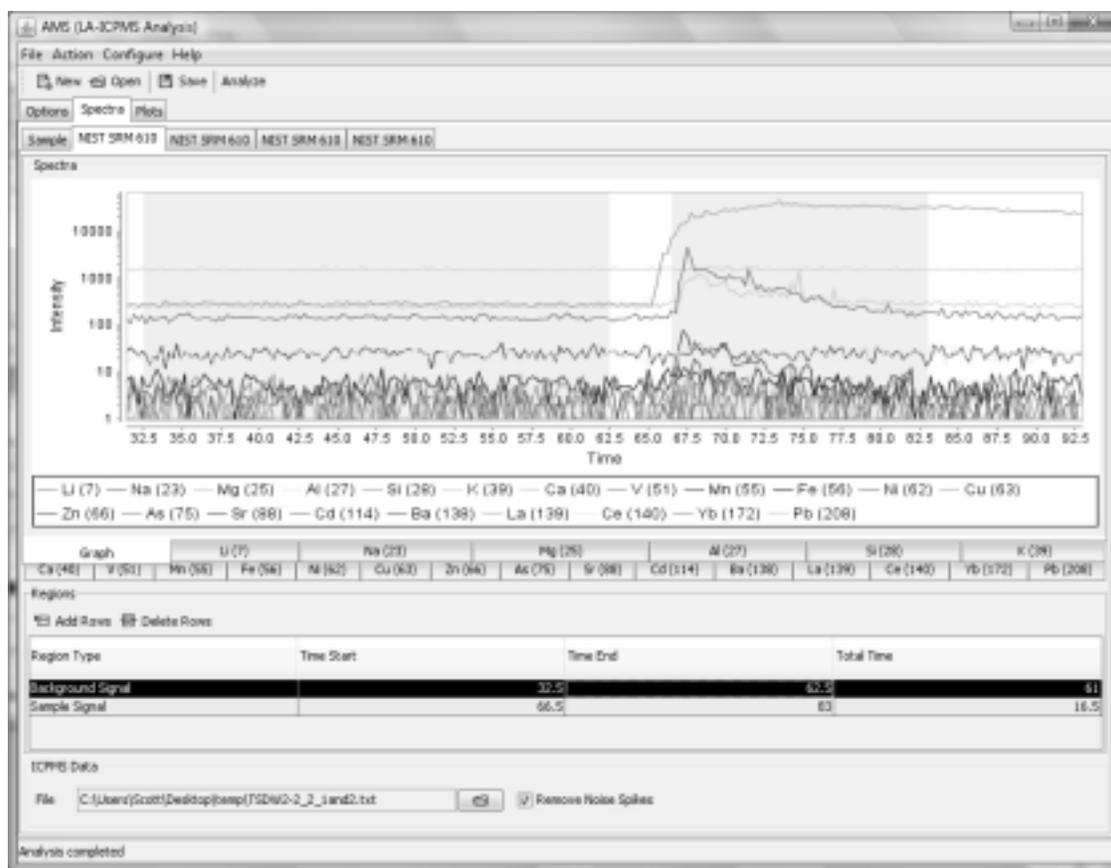


FIG. A5-6. ICP-MS time-resolved spectrum showing signal intensities during the analysis of a fluid inclusion. The relatively flat region on the left side is the background signal before the laser is turned on, the increase in intensity of Si at about 65 sec represents ablation of the host quartz above the inclusion, followed a few seconds later by the signal from the inclusion.

LA-ICPMS - Print Preview

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LA-ICPMS Laboratory
Department of Geosciences

Sample: TSDW2-2_5_1.net
Date: 05/03/2008 02:11
Internal Standard: Na
Standard Method: Microthermometry
Region: 130.D : 165.D seconds

Host Correction Factor:
70% NaCl eq.

Element	Con. (ppm)	LOD (ppm)	Weight %	Sample (cps)	Bkg (cps)	Std. (cps)	Bkg (cps)	Mix (cps)
Li (7)	-	72.889	-	39	386	64,527	464	-
Na (23)	63,645.169	183.998	16.18	255,730	14,123	64,988,899	13,996	-
Mg (25)	111.459	83.663	0.04	39	95	26,514	77	-
Al (27)	72.711	66.011	0.04	164	2,178	3,980,434	2,138	-
K (39)	2,142.967	220.384	0.41	12,147	151,528	430,483	148,138	-
Ca (40)	15,862.041	72.145	4.17	103,984	25,712	92,258,134	23,226	-
Mn (55)	-	14.764	-	51	2,466	728,064	2,713	-
Fe (56)	-	9.26	-	0	519	543,429	625	-
Ni (62)	-	141.136	-	0	16	5,758	14	-
Cu (63)	-	31.745	-	14	189	89,112	160	-
Zn (66)	-	46.768	-	0	297	89,999	306	-
As (75)	-	21.749	-	0	30	41,642	32	-
Sr (88)	481.291	0.567	0.09	6,792	6	1,183,978	9	-
Cd (114)	-	15.759	-	4	233	110,957	285	-
Ba (138)	14.807	0.397	0	221	4	1,186,553	2	-
La (139)	-	0.239	-	1	1	1,040,457	2	-
Ce (140)	-	0.218	-	0	1	1,183,943	1	-

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FIG. A5-7. Results window showing the composition of a fluid inclusion in quartz calculated with AMS. See text for description of each of the data columns.

available as PDF, Excel, CSV or HTML files and/or in hard copy.

The composition of the inclusion in this example calculated by AMS is 16.18 wt% NaCl, 4.17 wt% CaCl₂, 0.41 wt% KCl, and trace amounts of Mg, Al and Sr chlorides.

MELT INCLUSION ANALYSIS AND DATA REDUCTION

One of the major advantages of AMS is in the interpretation of laser ablation ICP-MS spectra for melt inclusions. If the melt inclusion is homogeneous (*i.e.*, glassy) and exposed at the surface then the analysis and data reduction are straight forward and identical to the analysis of a solid phase. However, many melt inclusions are partly to completely crystallized and/or not exposed at the host crystal surface (Bodnar & Student 2006). In both cases it is necessary to use a laser spot that is somewhat larger than the melt inclusion, resulting

in a mixed signal that includes signal from the MI as well as from the host. The mathematical process for separating the contributions of the MI and the host is conducted within AMS and requires no manual calculations.

The example below outlines the procedure for analyzing a glassy melt inclusion that is not exposed at the crystal surface. The laser diameter was set slightly less than the MI diameter so that when the laser ablated to the depth of the MI it would sample only MI material. As with analysis of fluid inclusions, the first step is to set the options in the options window. As described above for fluid inclusions, four analyses of the NIST 610 standard are used (two at the beginning of the session and two at the end) and the host material phase analysis will be normalized to 100% oxides. If the "Normalization to 100% oxides" option is selected, all elements that contribute significantly to the composition of the host must be included in the list

of elements measured. Next, the SROIs for the background and host are selected in the spectra window (Fig. A5-8). That portion of the signal collected before the laser has ablated to the depth of the MI is selected for the host SROI (shaded region between 45 and 55 seconds on Fig. A5-8), being careful to not include any of the mixed signal. Once the host SROI is selected the host concentration is calculated by clicking on the “Analyze” button at the top of the window.

Once the host composition has been determined, the next step is to set the host correction factor values on the options window (Fig. A5-9). In the example shown, the element aluminum is selected to determine the proportion of the mixed signal that comes from the melt inclusion

(Halter *et al.* 2002). The concentration of Al in the host determined in the previous step (31.14 wt%) is entered into the “Host Concentration” box. To complete the host correction, the concentration of this same element (Al) in the melt inclusion must be known. This value can be obtained in various ways. If the MI are glassy, inclusions that are exposed at the host crystal surface can be analyzed by electron microprobe or LA-ICP-MS to determine the major element compositions. These values can then be used for analysis of buried MI. If the inclusions are buried and crystallized, obtaining the composition of one (or more) elements to use to reduce the data is more complicated. The preferred method is to homogenize a few inclusions from the same Melt Inclusion Assemblage and then analyze these

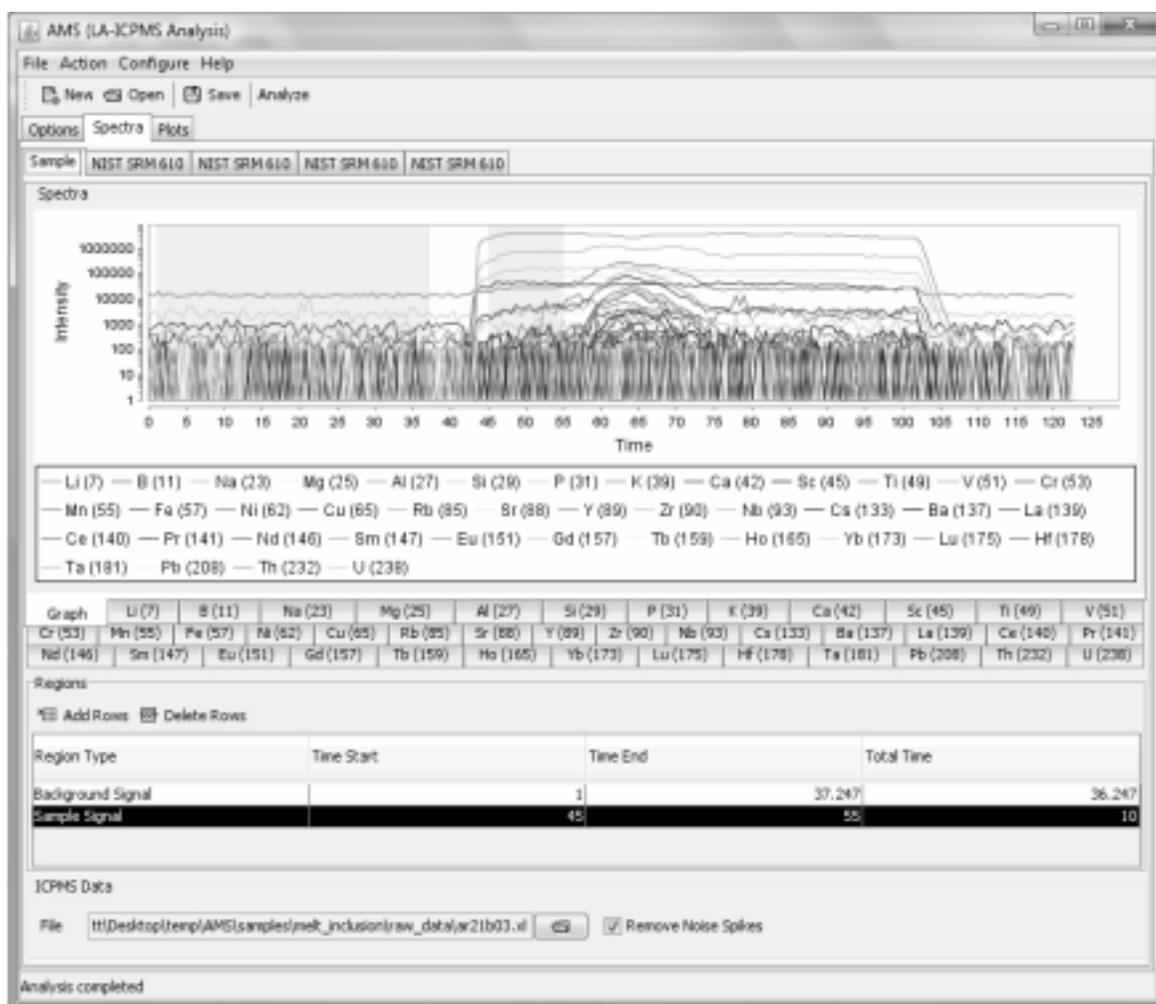


FIG. A5-8. ICP-MS time-resolved spectrum showing signal intensities during the analysis of a melt inclusion that is beneath the host mineral surface. The relatively flat region on the left side is the background signal before the laser is turned on. When the laser is turned on it begins to ablate the host phase only until it eventually reaches the buried melt inclusion. The region from 45–55 sec defines the host SROI in this example.

APPENDIX: ANALYSIS MANAGEMENT SYSTEM (AMS)

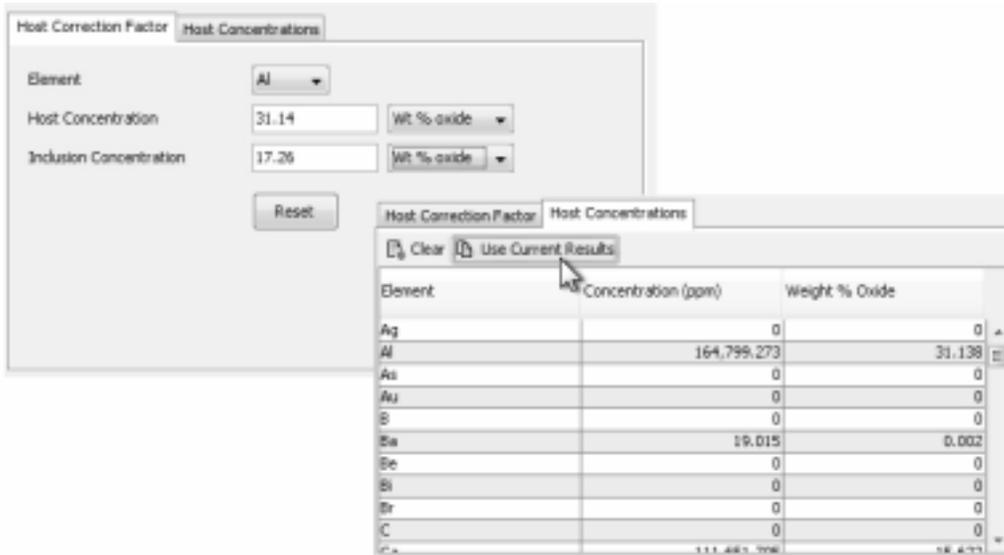


FIG. A5-9. Host correction window in AMS to enter the concentration of the element that will be used to deconvolve the mixed signal from the host and MI.

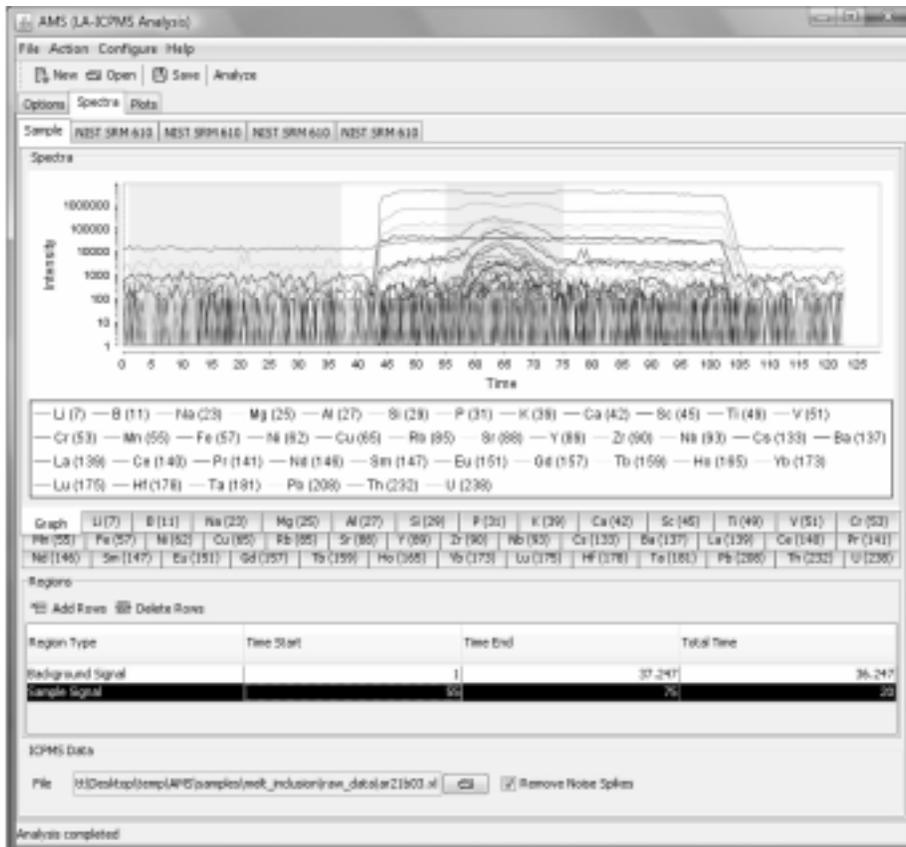


FIG. A5-10. ICP-MS time-resolved spectrum showing signal intensities during the analysis of a melt inclusion that is beneath the host mineral surface. The relatively flat region on the left side is the background signal before the laser is turned on. When the laser is turned on it begins to ablate the host phase only until it eventually reaches the buried melt inclusion. The region from 55–75 sec defines the mixed signal (host + MI) SROI in this example.

inclusions by electron microprobe or LA-ICP-MS to obtain concentration values that can be used to reduce data from the remaining inclusions. Alternatively, Halter *et al.* (2002) estimated the concentration of one or more elements in the MI based on whole rock analysis and assumptions concerning the relationship of the MI to the bulk rock composition. In the example here, the concentration of Al in the MI is estimated to be 17.26 wt% based on analyses of other MI in the same MIA that were exposed at the surface. Next, select the “Host Concentrations” tab and then click on “Use current results” to set the host concentrations – this uses the host concentrations determined in the previous steps.

Based on analyses of homogenized MI in this sample, we know that the MI contain about 4 wt% H₂O. Therefore, before analyzing the mixed signal region we return to the options window (*e.g.*, Figure A5-5) and enter “96” (rather than 100) in the box

“Use normalization to” in the Methods subsection. The final step before the results can be calculated is to return to the spectra window and select the mixed signal region (Fig. A5-10). Finally, click on the “Analyze” button to complete the analysis. The results are then displayed in the results window (Fig. A5-11).

The first eight columns in the results window (Fig. A5-11) are the same as for the fluid inclusion analysis, except that in column 4, Weight % refers to weight percent oxide of the element.. Column 9 (Mix (ppm)) shows the concentration of the element in the mixed (host + MI) signal. Column 10 (Host (ppm)) shows the concentration of the element in the host. It is important to emphasize that the concentrations shown in columns 2 to 4 are the concentrations corrected for the host phase contribution. The host correction factor (0.53) indicates that 53% of the mixed signal was from the melt inclusion.

LA-ICPMS - Print Preview

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SampleID: m21683 xl
Date: 05/04/2008 08:47
Internal Standard: Al (27)
Standard Method: Normalized(96.0%)
Region: 57.0 : 73.0 seconds

Host Correction Factor: 0.53
Wt% NaCl eq: -

Element	Con. (ppm)	LOD (ppm)	Weight %	Sample (cps)	Blk (cps)	Std. (cps)	Blk (cps)	Mix (ppm)	Host (ppm)	K
Al (27)	91,150.576	3.823	17.26	307,680,440	13,235	86,140,727	19,335	124,098.88	161,038.06	-
B (11)	12,733	7.888	0	1,797	783	260,308	1,000	7.8	2.34	-
Be(137)	165.96	0.654	0.02	212,667	390	3,095,881	667	98.81	23.05	-
Ce(42)	70,938.185	480.33	9.93	3,434,802	77,112	10,582,490	83,254	85,236.27	101,343.6	-
Ce(140)	39,663	0	0	474,398	0	22,948,248	0	30.07	19.26	-
Cr (53)	113,182	16.851	0.02	30,012	16,913	660,886	22,883	60	60	-
Ce(138)	0.902	0.183	0	9,375	625	22,118,346	167	0.46	0.01	-
Cu(63)	993,774	2,034	0.12	704,866	1,093	1,817,727	500	329.57	5.89	-
Eu(151)	0.933	0.073	0	5,338	136	15,455,593	0	0.5	0.01	-
Fe(57)	55,637.84	39,317	7.16	3,327,679	3,513	135,134	3,730	31,333.31	3,937.35	-
Gd(157)	3.86	0.373	0	5,845	313	4,414,545	0	1.62	0	-
Hf(178)	1.7	0	0	5,357	0	6,889,103	333	1.02	0.26	-
Ho(165)	0.314	0	0	3,571	0	28,192,559	0	0.17	0.02	-
K(39)	4,924,389	17,454	0.39	14,537,117	1,304,938	8,005,900	1,349,135	2,734.53	263.89	-
La(139)	7.838	0.07	0	66,833	313	23,195,417	167	4.04	0.64	-
Li(7)	26.51	0.481	0.01	28,137	78	2,670,525	500	16.62	5.47	-
Lu(175)	0.234	0	0	2,500	0	27,777,418	167	0.12	0	-
Mg(25)	30,094.1	4,038	4.99	5,736,134	489	514,262	0	16,331.13	804.9	-
Mn(55)	1,338,788	1,343	0.15	5,408,739	28,394	12,303,625	27,900	623.11	41.37	-
Na(23)	29,188.33	1.3	3.93	99,842,994	4,646	1,389,839,621	8,084	22,713.11	15,412.32	-
Nb(93)	0.918	0.065	0	5,381	234	14,564,258	167	0.49	0	-
Nd(146)	7.937	0.226	0	16,272	136	5,067,706	167	4.44	0.49	-
Ni(62)	34,081	4,965	0	3,858	136	235,119	167	18.38	0.69	-
P(31)	419,638	23,855	0.1	167,880	56,233	733,761	70,169	230.12	58.88	-

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FIG. A5-11. Results window showing the composition of a buried melt inclusion calculated with AMS. See text for description of each of the data columns.

SUMMARY

As described above, the AMS software automates many of the tedious tasks that previous were conducted graphically or manually, and greatly facilitates the reduction of LA-ICP-MS data. A video showing the data reduction steps described above for the melt inclusion is available at: <http://www.geochem.geos.vt.edu/fluids/laICP-MS/ams.shtml>.

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