

Machinations: Multiple collection in solid-source mass spectrometry

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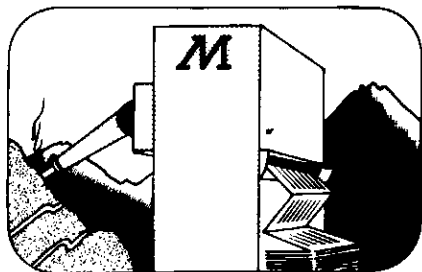
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Features



Machinations

This is the first article of a new feature series designed to bring to the attention of the non-specialist reader innovations in equipment and laboratory procedures in the Earth Sciences. We are indebted to Charlene E. Miall for suggesting the title.

Readers are invited to submit articles for this feature. They should be about 1000 words in length and may be accompanied by a short references list and two or three illustrations. They should explain how the new methodology permits the acquisition of new kinds of data or contributes to the resolution of a particular problem.

—Editor

Multiple collection in solid-source mass spectrometry

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Advances in solid-source mass spectrometry within the last decade have increased precision and sensitivity to such an extent that isotopic ratio and abundance measurements can be made on most elements found in geological materials. This largely resulted from instrument automation (and as a consequence reduction in operator bias), and the ability to massage and refine the data using on-line computers. Although the basic design of isotope ratio measuring machines has hardly changed since As-

ton's pioneer work at the turn of the century, some recent and exciting refinements in solid-source mass spectrometry are now paving the way for a new generation of mass spectrometers.

Solid-source mass spectrometry is fairly straightforward. Samples are thermally ionized, the ions are accelerated and focussed, and the resulting beam travels down a flight tube that lies between the pole pieces of a magnet. The magnetic field separates the ion beam into several, corresponding to the different isotopes of the element being analysed. The intensities are then determined in sequence by some sort of measuring device (Fig. 1a). The measurement of each ion beam involves changing the strength of the magnetic field so that the appropriate beam falls onto the collector.

In conventional machines, individual isotopes of the same element are measured sequentially, one after the other. In the case of Sr, for example, which has four isotopes (^{86}Sr , ^{87}Sr , ^{88}Sr and ^{84}Sr), four consecutive measurements are needed to determine the isotopic ratios. Because each isotopic ratio must be measured a large number of times in order to obtain the desired precision, a considerable period of time, of the order of a few hours, is required to collect such information.

The approach now being taken in solid source mass spectrometry is quite different.

The aim is to measure as many isotopes of the same element as required at the same time. Instead of one collector there are several, each specific for a particular isotope (Fig. 1b). This enables more information to be collected during a run.

Multi-collection has already been successfully tried in gas source machines, particularly in K-Ar geochronology (Stacy *et al.*, 1981). It is only now, however, that a new generation of solid-source mass spectrometers is emerging, each with its own bank of collectors carefully tailored to meet individual needs.

A new multi-collector, *Finnigan-MAT 261* extended geometry machine, the first of its type in North America, has recently been installed at Carleton University in Ottawa. Its main features are (i) a magazine that enables up to 13 samples to be loaded at one time; (ii) complete computer (HP 9845) control of the machine; and (iii) the multi-collector system. The latter is only the second in the world to be fitted to a solid-source mass spectrometer.

The arrangement of the nine fixed collectors in the Carleton machine is shown in Figure 2. This array is used routinely to measure three of the isotopes of Sr, three of Nd and four of Pb. With the arrangement shown in Figure 2 measurements can be made in a variety of ways during the

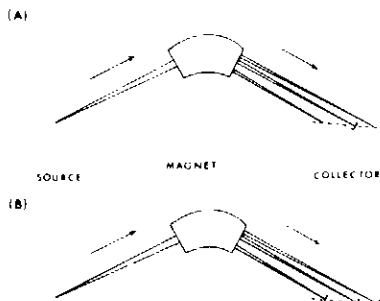


Figure 1 (a) Conventional mass spectrometer with one single collector. (b) Multi-collector machine with a three-collector array. The collectors are positioned on the focal plane, which is inclined.

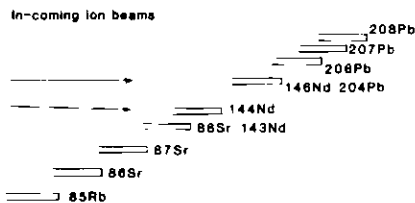


Figure 2 Arrangement of the nine collectors in the Carleton machine; with this spacing the Sr collector can be used to measure Nd, and that for Nd to measure Pb. The separation between adjoining collectors is about 5.3 mm in the Sr mass range, and about 2.2 mm in the Pb range.

same run using either single, double or multiple collection (Blenkinsop, 1983). A summary of the significant features of each collecting mode is given in Table I. It can well be appreciated that with the increased number of collectors their alignment is critical; all have to be squeezed into the mass spectrometer and each carefully positioned to a tolerance of about 0.01 mm. Because the isotopes are measured simultaneously it therefore becomes possible to plot the outputs from all channels, using the computer, by sweeping each beam across its appropriate collector. From the plots a comparison can be made of respective peak shape. An example for Sr is shown in Figure 3.

Because each collector is allied with its own amplifier, significant variations among amplifiers can limit both the accuracy and precision of the measurements; no matter how good the amplifiers are, no two are going to behave in the same way. The gain of the temperature-compensated amplifiers (to 0.01°C) is determined by feeding a known signal into each. Done by computer, each calibration takes about one minute per channel. Figure 4 illustrates the variation in amplifier gain of three selected channels during a 24-hour period and shows that the channels track each other. For the precision we are after, the stability of the amplifiers has to be ± 10 ppm over thirty minutes, the time required for a complete run.

Table II summarizes some multi-collector measurements of two standards, one Sr, one Nd. The first 13 Sr values (a full sample magazine) in Table II were measured automatically during a 24-hour period. Shown in Table III are results from one run in which the ion beam was measured using the single, double and multi-collector modes. Although the results from each of the different modes agree within analytical precision, it is apparent that the double and multiple collector results are considerably better.

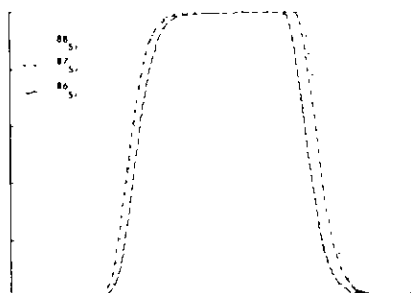


Figure 3 Simultaneous traces of the normalized Sr, Sr and Sr peaks. Important points to note are the extent of the common "peak flat", the similarity of the three peak shapes, and the coincidence of the peaks. The latter verifies the alignment of the collectors.

Resolution of such problems as mantle evolution and the origin of the continental crust requires measurements of isotopic ratios, such as ¹⁴³Nd/¹⁴⁴Nd and ⁸⁷Sr/⁸⁶Sr, to better than 3 or 4 parts in 100,000. This precision can easily be exceeded with our new machine. With this increased capability we should be able to refine our work related to the geochemical evolution of the sub-continental upper mantle. Already we have shown, from initial Sr isotopic ratio measurements of Canadian carbonatites and related rocks of various ages, that the Sr was derived from a mantle source with a uniform Rb/Sr ratio (0.018 ± 0.002). This source remained unchanged, at least as far as Rb and Sr were concerned, from about 2,700 m.y. through to 120 Ma B. P. (Bell *et al.*, 1982). The mean time at which such a depleted source region formed from a more primitive mantle, similar in chemical composition to bulk earth, can be estimated. The figure we obtained of about 2,800 Ma B. P. is very similar to the mean age of the Superior Province crustal rocks. In our model (summarized in Fig. 5) we suggest that at about 2,800 Ma B. P. the primitive mantle differentiated into continental crust, leaving behind a residual, LIL-depleted mantle zone with its own characteristic isotopic signature. The latter remained coupled to the continental crust until at least 120 Ma B. P. Pb isotope ratio measurements from the same material used for the Sr study also indicated a LIL-depleted source with a low U/Pb ratio (Grunenfelder *et al.*, 1982).

Our next step, using the new analytical facility, is to pin down the Rb/Sr ratio of the depleted mantle source still further, to evaluate the degree of chemical homogeneity of this anomalous mantle zone and to assess its lateral extent under both the Canadian Shield and other continents. Preliminary Sr isotope measurement of carbonatites from Africa using our new machine, although consistent with our model, suggest possible mixing between Sr from the depleted sub-continental upper mantle

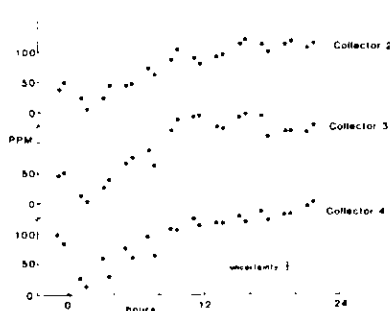


Figure 4 Gain variations for three selected channels during a 24-hour period. Although there is noticeable fluctuation, the variation in all three channels is the same.

Table I: Modes of Analyses

1. Single collector:
 - (i) Sequential measurement of ion beams
 - (ii) Amplifier gain not required—all signals measured on the one amplifier.
2. Double collector:
 - (i) Two ion beams measured simultaneously
 - (ii) Two such measurements needed
 - (iii) Three isotopes with equal mass separation measured—ratio of two of them must be known
 - (iv) Amplifier gains not needed—cancel out in calculation of ratio.
3. Multiple collector:
 - (i) Several ion beams measured simultaneously
 - (ii) Gains of amplifiers must be measured precisely.

Table II Measurements of Standards

⁸⁷ Sr/ ⁸⁶ Sr (NBS 987)	¹⁴³ Nd/ ¹⁴⁴ Nd (Nd alpha standard)
0.710242	0.511731
0.710250	0.511732
0.710276	0.511739
0.710211	0.511749
0.710255	0.511747
0.710248	0.511751
0.710238	0.511723
0.710227	0.511737
0.710242	0.511734
0.710260	0.511748
0.710244	0.710261
Average = 0.710245 0.511739	
2 _n = 0.000006 0.000006	
where $\bar{x} = \frac{\sum(x-x)^2}{N(N-1)}$	

Table III Comparison of single, double and multicollector Sr results from the same run using NBS 987

Mode of collection	⁸⁷ Sr/ ⁸⁶ Sr (atomic)	Time required (in minutes)
Single collector	0.71025 ± 2 175	
Double collector	0.71023 ± 160 0.71025 ± 1	
Multi-collector	0.71025 ± 130	
Error quoted as 2 _n .		

(SCUM) and that from a more primitive mantle below.

Geophysical evidence for a thickened, stable refractory root underlying most continents and unaffected by convective disruption agrees with our interpretation of the isotopic data (Jordan, 1978; Vitorello and Pollack, 1980). More refined measurements of a greater selection of mantle-derived material will, it is hoped, answer the question of how widespread the depleted sub-continental upper mantle is. Small isotopic differences, at a level that we can now measure, will be critical to understanding fully the evolution of the sub-continental upper mantle during the last 3,000 Ma.

Our experience so far suggests that the major advantages of data collection with the new system are that (i) isotopic ratios can be measured to higher precision; (ii) more samples can be measured in a given period of time; (iii) acceptable precision can be obtained on smaller samples; and (iv) effects of unstable ion beams can be minimized. Although multiple collection is still very much in its infancy, there is little doubt that it will play an increasingly important role in solid-source mass spectrometry.

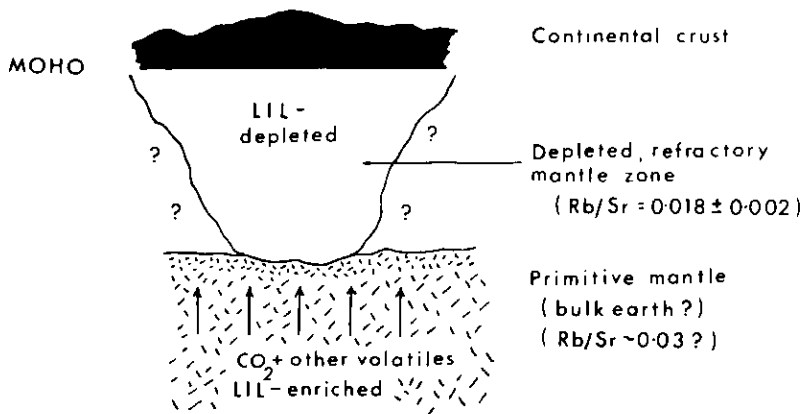


Figure 5 In our model continental crust is underlain by a complementary reservoir of a LIL-depleted mantle zone ($Rb/Sr = 0.018$) with a mean age of about 2800 Ma. Most of the Sr found in the Canadian carbonatites was obtained from such a depleted, refractory zone. The CO_2 , probably derived from a more primitive, deeper mantle source, scavenged Sr from the overlying depleted zone. Release of volatiles

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from the interface between the refractory zone and the more primitive, outgassing zone below is probably the result of such processes as orogenesis and large scale rifting. Carbonatites from other continents suggest that Sr can also be derived from the primitive mantle source. The vertical scale on the diagram has no real significance.

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