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The Varian Mat GD150 for Argon Analyses in Connection with K-Ar Dating

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ABSTRACT

A Varian Mat GD 150, a relatively inexpensive gas source mass spectrometer is discussed from the viewpoint of its suitability for argon analyses in connection with K-Ar dating. It is shown that the instrument is satisfactory for this type of research.

Introduction

In 1964 FARRAR et al. reported on their successful adaptation of the Associated Electrical Industries Limited MS10 mass spectrometer for argon analyses. Their report clearly demonstrated that it was possible to establish a K-Ar dating laboratory capable of measuring precise geologic “ages” employing a relatively inexpensive gas source mass spectrometer. Since the above report other commercial instruments have become available, including the Varian Mat GD 150. It is the purpose of the present communication to describe the Varian Mat GD 150 as it has been set up in our laboratory with particular emphasis to the usage of this instrument for the measurement of argon extracted from rocks and minerals.

Description of the instrument

The mass spectrometer is an all metal, bakeable, 180° analyser having a 2-cm and 5-cm radius of curvature collector as well as a total ion collector. The sample is admitted directly into the ionization box; ions being produced in a NIER (1947) type electron impact source. A particular feature of the ion source, not always found on small commercial instruments, is its electric lens system consisting of draw out plates, lenses, and a focusing plate. The potential on each of these plates and half-plates is variable within certain limits giving the operator some flexibility in adjusting the ion beam for optimum intensity, peak shape and resolution. A permanent magnet of 0.385 Wb M^{-2} deflects the ions through 180°. Mass selection is achieved by varying the ion accelerating voltage either manually or automatically (electronically).

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The amplifiers (two), potential sources for accelerating voltage and ion optics are solid state apart from a high voltage tube and two electrometer tubes. The noise level of the amplifiers corresponds to an ion current of $2 \cdot 10^{-15}$ A. On our instrument the 5-cm collector port has been adapted to the head of a Cary vibrating reed electrometer.

The pumping system is a standard high vacuum one consisting of a liquid nitrogen cold trap, two mercury diffusion pumps and a rotary pump.

Performance of the instrument

Baking the mass spectrometer overnight at 250°C (filament current = 2 A) is sufficient to reduce the final pressure in the analyser to the order of 1×10^{-9} Torr. After baking, running the filament for a few hours at a higher trap current ($225\ \mu\text{A}$) than normally used ($80\ \mu\text{A}$) results in the dynamic background in 34–43 mass region being reduced to an undetectable level ($< 2 \cdot 10^{-15}$ A). Operated statically ($80\ \mu\text{A}$ trap current) there is a slowly rising ion current at mass 40 ($2 \cdot 10^{-15}$ A min^{-1}) and 38 ($1 \cdot 10^{-15}$ A min^{-1}). There is no detectable ion current ($< 1 \cdot 10^{-15}$ A) at mass position 36 even when the analyser tube is closed from the pumps for more than one hour.

After preliminary checks with various combinations of source exit slits (0.06, 0.15, 0.25 mm) and collector slits (0.35, 0.50 mm), an exit slit of 0.15 mm and a collector slit of 0.50 mm was selected as offering the best compromise of sensitivity, peak shape and resolution. The static sensitivity for argon is $6 \cdot 10^{-5}$ A cm^{-3}STP ($80\ \mu\text{A}$ trap current, ion repeller voltage = 4.3 V). An indication of the peak shape and resolution for the argon 36, 38 and 40 masses can be seen in Figure 1. This mass spectrum was recorded by feeding the output from the amplifier supplied with the instrument to a 10-inch strip chart recorder. The argon sample was a mixture of a few-hundred-mg of an Alpine mica, 1.5×10^{-6} cm^3 STP of Ar^{38} "tracer" and atmospheric contamination.

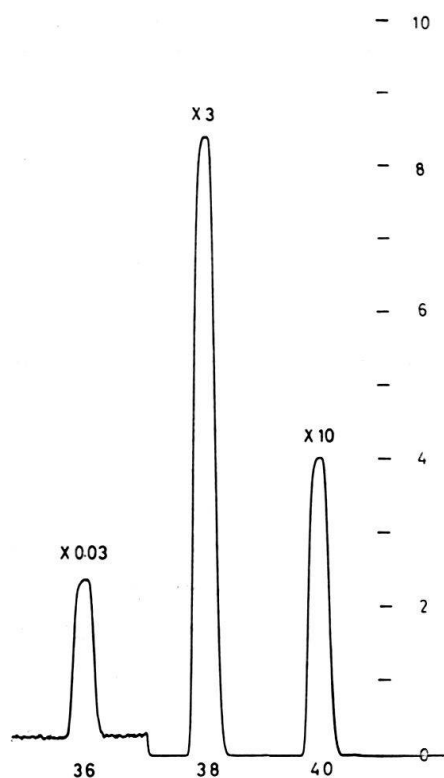


Fig. 1. Recording of typical argon measurement.

The 40 and 38 masses contributed negligibly to each other. The contribution of the 38-ion current to the 36-ion current is $< 6.10^{-5}$ times the height of the 38-ion current.

The mass spectrometer does exhibit a "memory" effect which, for routine sample analysis can be minimized or virtually eliminated in the usual fashion by choosing the amount of sample so the isotopic composition of the argon admitted to the mass spectrometer is always similar. In those cases where a memory effect is noted a typical variation of the (40/38) ratio is $(0.05-0.1)\% \text{ min}^{-1}$ for samples having a (40/38) ratio between 0.1 and 10.

FARRAR et al. (1964) reported the absence of a memory effect for the MS 10. This was attributed to the low accelerating voltage (100 V) for argon isotopes and consequent smaller likelihood of embedding argon ions into the walls of the spectrometer tube which, in turn, may be readmitted to the analyser tube during a later analysis. More recently, REX and DODSON (1970) have described their experience with a MS 10 employing a more powerful ($0.41 \text{ Wb } M^{-2}$) magnet than was used by FARRAR et al. ($0.183 \text{ Wb } M^{-2}$). Using the stronger magnet REX and DODSON (1970) noted a memory effect. They reported a variation of $0.05\% \text{ min}^{-1}$ for the (40/38) ratio of a "spiked" sample analysed after the mass spectrometer had been used for running atmospheric argon samples. This (40/38) variation is similar to the one noted above for the GD 150.

Apart from the above mentioned "memory" effect, there is no indication of other variations in the measured isotopic ratios for purified argon samples. The measured ratios are, however, affected by the setting of the ion repeller voltage, the latter being variable between 0 and 10 V positive with respect to the ionization box. For our instrument, a setting of 4.3 V gives a minimum change in the measured ratios with a change in ion repeller voltage. A change of 0.3 V at 4.3 V produces a change in the measured ratios of less than 0.05%.

With an ion repeller voltage of 4.3 V and a trap current of $80 \mu\text{A}$, the value measured for the (40/36) ratio of atmospheric argon is 292.4. This value has been repeated over a period of several months with a standard error of 0.4%. The value 292.4 for the (40/36) ratio is an average of values measured by leaking a small fraction of argon into the analyser tube from a large reservoir, necessitating multiplying the measured ratios by $\sqrt{40/36}$; and of values measured by equilibrating a small amount of argon between the analyser and inlet system. Within the stated precision, there is no difference between the (40/36) ratio values measured in these two ways. Comparing the value 292.4 with the value 295.5 determined by NIER (1947) indicates the mass discrimination of the instrument with the above source conditions.

In the Table are given the results of argon concentration determinations of a number of interlaboratory standards. The argon was extracted in vacuo using radio frequency induction heating. The extraction and purification lines were conventional in design being constructed of pyrex and employing all-metal bakeable valves throughout. After two separate purifications with titanium sponge the samples were admitted directly into the mass spectrometer. Nearly pure Ar^{38} ($> 99.98\%$) was used as a spike, this being metered into the system via a double tap reservoir system. Calibration of the spike was against known amounts of atmospheric argon. From the Table it can be seen that the values for argon concentrations of U. S. G. S. P-207 and M. I. T. B3203 agree with the accepted values within 1%; for the Bern muscovite and biotite the measured values are within the range reported by other laboratories.

Table 1. Analyses of standard minerals (values in 10^{-6} cm³ STP/g).

Standard sample	Reported value(s)	Measured with GD150
U. S. G. S. P-207	28.07 ^a)	27.79, 28.08
M. I. T. B3202	387.7 ^b)	389.1, 392.1
Bern muscovite 4M	5.75–6.61 ^c)	6.23, 6.27, 6.43, 6.35, 6.35, 6.30, 6.38, 6.20
Bern biotite 4B	5.01–5.31 ^c)	5.43, 5.35, 5.29, 5.30

^a) LANPHERE, M. A. et al. (1967).

^b) HURLEY, P. M. et al. (1962).

^c) JÄGER, E. (1969).

The mean value for the Bern muscovite 4M is 6.31×10^{-6} cm³ STP with a standard deviation of 0.5%. This is well within the scatter reported by other laboratories (JÄGER 1969) for this sample.

Conclusions

In terms of static sensitivity (6.10^{-5} A cm⁻³ STP), absence of a background correction at the critical mass 36 position, resolution of argon isotopes, reproducibility of results, long-term stability of electronic components, lifetime of the filament (in excess of one year when operated continuously), the Varian Mat GD150 is a suitable instrument for measuring argon concentrations from terrestrial samples. A useful improvement for our instrument in terms of daily maintenance and running costs would be the conversion of our present pumping system to an ion pumping system.

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