Development of a Mass Spectrometer System for the Measurement of Inert Gases in Meteorites

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ABSTRACT

The study of the inert gases in meteorites has provided many clues as to the origin and evolution of the solar system. Particularly crucial and complex have been the gases krypton and xenon. To accurately measure the isotopic compositions of these gases requires a mass spectrometer of high sensitivity and resolution.

In this project a previously unused and largely untested mass spectrometer system was brought to the point where it was ready for routine sample analyses. This involved, among other things, focusing the ion beam for optimal peak shape and sensitivity, documenting the instrument's response to a series of characteristic tests such as multiplier gain checks, and interfacing the instrument to a computer to run the sample analyses.

Following this testing and setting up, three iron meteorite samples were to be analyzed for argon, krypton, and xenon. The three samples have been shown in prior work by the author to possibly contain primordial heavy inert gases. Although these analyses have not yet been carried out, it is anticipated that they will be completed in the near future.

Center Research Advisor: Don Bogard

INTRODUCTION

There were two primary objectives which I hoped to carry out during my 1983 summer research fellowship. The first was to help bring the new mass spectrometer in the gas analysis laboratory to the point where it was set up for routine analyses of meteorite samples. Following this, I wished to analyze the inert gases argon, krypton, and xenon in three iron meteorite samples to confirm, with reduced error bars, prior work which indicated possible primordial heavy inert gases. In the theory section, I will discuss the impetus for the iron meteorite sample analyses, while in the results section I will discuss the progress toward achieving the original two objectives.

THEORY

Meteorites are classified into three macroscopic groups: irons, stony-irons, and stones. These three broad classifications are further broken into many finer divisions based on mineralogical and structural differences. Many investigators of stony meteorites have found unusual elemental isotopic ratios relative to those seen in the earth's atmosphere, the sun, and bulk meteorite compositions. These data have provided clues to the solar system's origin and evolution. In seeking explanations for the anomalies seen, studies of stony meteorites have burgeoned, with increasingly more precise techniques and data. Meanwhile, the iron meteorites have been more-or-less ignored in the search for primordial inert gas anomalies. This treatment is not totally without justification, since iron meteorites clearly represent samples which have undergone a significant amount of metamorphism.

Thus they might well be expected to have lost, or at least appreciably altered, original inert gas compositions. Nevertheless, this is a conclusion reached more by assumption than by experimental confirmation.

Those studies of the inert gases in iron meteorites which have been carried out were generally done with a different objective in mind than the detection of possible primordial anomalies. Because of this, the samples selected for study and the procedures used were inappropriate for the objective I hoped to achieve. First of all, measurements of inert gases in iron meteorites have been almost exclusively restricted to the light gases helium, neon, and argon. This was done because the light gases are expected to have much greater concentrations than the heavy gases, krypton and xenon. The reason for this is due to the elemental composition of iron meteorites being approximately 90% iron and 10% nickel. Assuming that all the gas in these meteorites is the result of spallation induced by galactic cosmic rays, the target elements iron and nickel can yield the gases lighter in weight than themselves, helium, neon, and argon, but not the gases heavier than themselves, krypton and xenon. Thus spallogenic krypton and xenon will only be produced from heavy trace elements.

On one hand that is favorable for my objective, since it means that spallation produced krypton and xenon may not overwhelm a small primordial gas component. On the other hand, it means the concentration of the gases is probably going to be extremely low. This expected low concentration requires an extremely sensitive mass spectrometer with sufficient resolution to clearly separate the isotopes of xenon. The new mass spectrometer at Johnson Space Center was well suited for this investigation.

RESULTS

For the following discussion of adjustments made in the mass spectrometer system, I will first very briefly outline its overall operation. Neutral gas atoms are released from a heated sample, enter the mass spectrometer and are ionized in the source region by bombardment with electrons from a filament. The ions are now collimated and accelerated through a series of slits in parallel plates with a voltage drop. The resulting ion beam enters the field of an electromagnet where it is split due to the varying charge to mass ratios in the beam. Those ions whose charge to mass ratio is such that they will enter the analyzer slit impinge on an ion collector. The signal thus received is then amplified and recorded on a strip chart.

At the beginning of the summer, the ion beam was obviously not well focused since the shape of the peaks made when scanning over a mass was poor and the sensitivity low. The desired peak shape is one which has a flat top and steep sides. The flat top ensures that when one measures the height of the peak, the exact place on the peak selected will not be critical; this is especially important for peak jumping where a computer is running the sample analysis. The steep sides make it possible to clearly resolve adjacent mass peaks. The focus of the instrument is affected by a number of variables, and the adjustment of one parameter will usually have an effect on the others. Thus the proper focus was only reached after a long series of trial and error adjustments. Among the coarse adjustments are a magnet in the source region, the main electromagnet, rotation of the collector slit, and the collector slit width. The source slit width is not adjustable on this machine. Finer adjustments included the level and

difference of the source divider; the deflection plate voltage, repeller, and dynode #1 of the electron multiplier; and the repeller of the emission regulator.

The best focus for one mass may not be optimal for another. The original focusing was done with ^{40}Ar since that is always in the background of the system. However, after it became evident that the sensitivity of the instrument was such that measuring argon would not be a problem in typical lunar or meteorite samples, the focusing was readjusted for xenon, which is generally of much lower concentrations. Part of this readjustment involved an analysis of the five inert gas peak heights as a function of the electron accelerating voltage. This voltage is the potential difference between the filament and the ionization chamber walls. The peak sensitivity for krypton and xenon came with the electron voltage at 43.5 V, and this value was adopted for all gases for convenience and because double ionization of ^{40}Ar (which would then contribute to the ^{20}Ne peak) would be small.

Constant sweep rate scans of the krypton and xenon mass regions after the final focusing adjustments are presented in Figures 1 and 2. As the magnetic field increases, the charge to mass ratios differ by less and less, so the excellent resolution at xenon is one of the instruments clear strengths. Thus the machine is set up in such a way that the sensitivity in measuring the isotopes of krypton and xenon is optimized (while still retaining a good peak shape) at the slight expense of the generally far more abundant inert gases helium, neon, and argon.

Since the concentrations of the heavy inert gases are so low (^{132}Xe on the order of 10^{-11} cm^3/g STP), it is naturally vital to maintain as low a pressure and clean a system as possible. This is accom-

plished by enclosing the all metal mass spectrometer in a hood and baking it with resistance heaters when necessary. It is always possible that during these bakeouts the metal flanges and copper seals may expand and contract in such a way as to produce a leak. The leaks are detected by blowing ^{4}He over the various flanges while monitoring the ^{4}He peak on the recorder. One large copper seal and a gold valve ring had to be replaced (the gold ring by one of paladium) this summer, and the machine subsequently baked out at approximately 150 ^{o}C for several days. Another small leak was plugged by applying leak sealant to the affected region. The background gases (in particular benzene, which interfers with ^{78}Kr) were monitored and found to be steadily, albeit slowly, decreasing, so that by the end of the summer there was a much cleaner machine with an ionization gauge pressure of about 2.5×10^{-9} torr.

Other tests run on the machine were multiplier gain tests and sensitivity tests. The results of these were used primarily to compare the new mass spectrometer in the lab to the old one. In the multiplier gain test, a peak is first measured by having the beam strike a Faraday cup, rather than the electron multiplier. The gain of the multiplier is then computed by comparing the multiplier signal at various multiplier voltages to that of the Faraday cup. In the sensitivity tests, inert gases of known amounts and isotopic compositions are admitted to the mass spectrometer to determine the instrument's response.

After the focusing problems were largely overcome, the next major phase of bringing the new mass spectrometer into routine operation was the interface with the computer. Under the computer's control, scans of the peaks can be made much more swiftly and accurately. However, because the vibrating reed electrometer and the magnet regulator

on the new instrument differed from that of the old, the software had to be modified. In the interfacing process many problems arose from the different characteristics of the hardware. The magnet regulator in particular had to be modified to damp out oscillations which were keeping it from coming to rest on a peak by the time the computer was integrating the peak's height. The nonlinearity of the electrometer presented difficulties in measuring the signal from widely different peak heights (for example, ^{38}Ar relative to ^{40}Ar in atmospheric composition) while on a volt range setting sufficiently high to keep the larger peak on scale.

CONCLUSIONS

At the time of this report, the mass spectrometer and the computer interfacing are very close to being ready for sample analyses. Already sensitivity checks and blank background measurements have been completed in anticipation of the melting of the first of the iron meteorite samples, Babb's Mill. I expect that in the following weeks the other two, Braunau and Sierra Gorda, will also be run. Although the results of these measurements cannot be reported here, it is hoped that they will soon be included in a paper with due credit to the NASA/ASEE summer faculty program for their support.

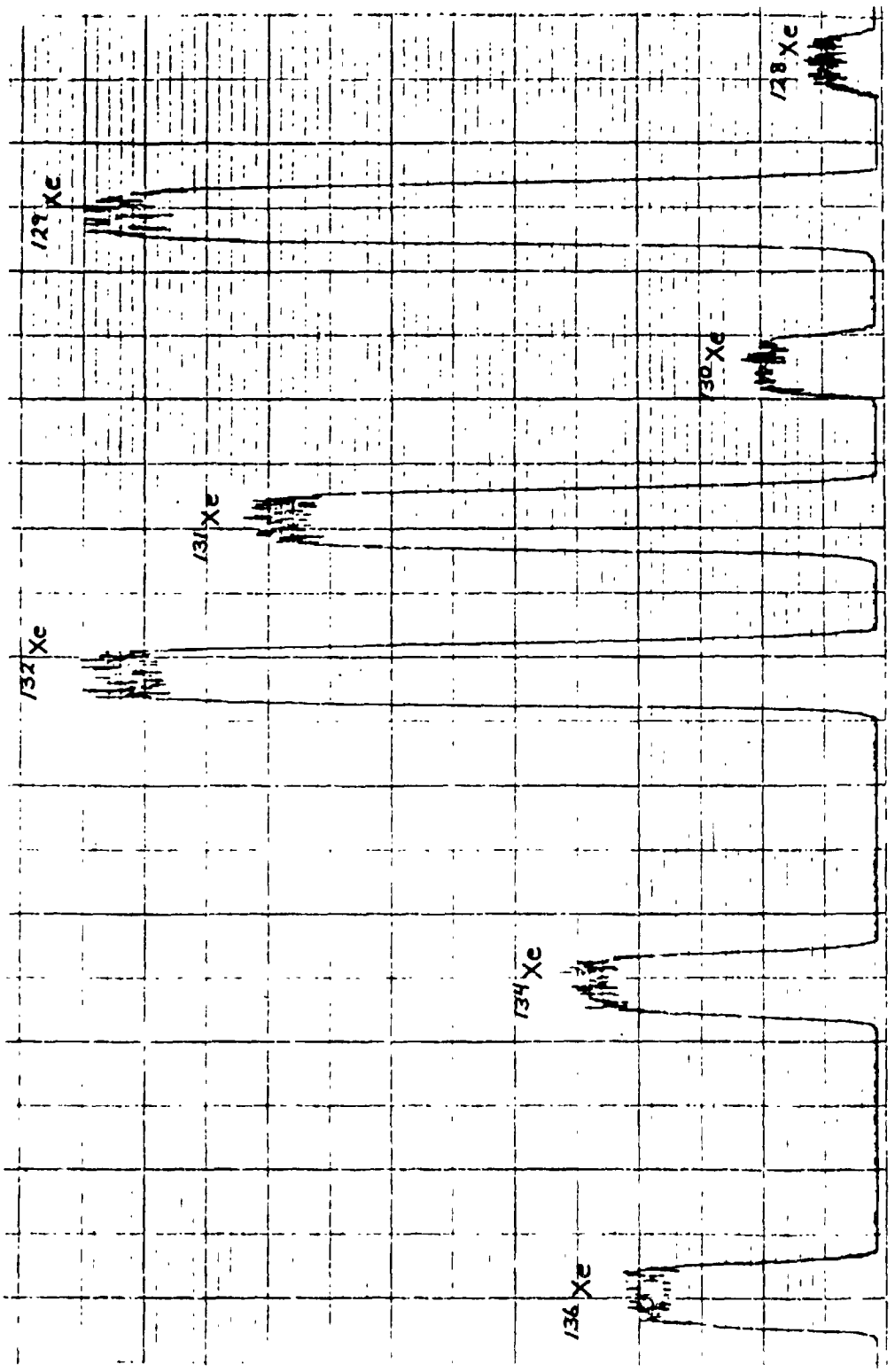

Figure 1. Constant sweep rate scan of xenon with atmospheric composition.

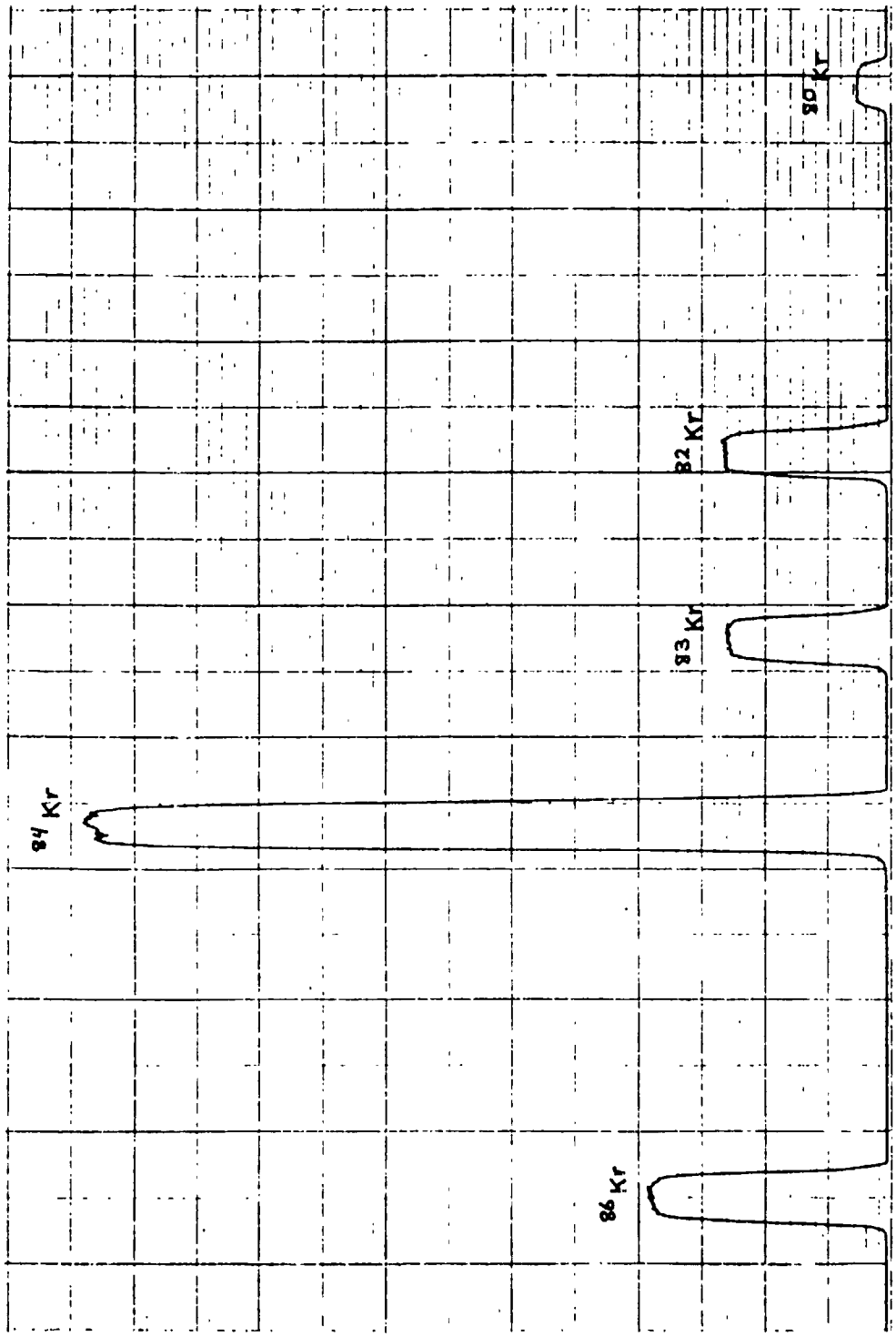

Figure 2. Constant sweep rate scan of krypton with atmospheric composition.