# PERFORMANCE OF A BOREHOLE X-RAY FLUORESCENCE SPECTROMETER FOR PLANETARY EXPLORATION

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## ABSTRACT

We have designed and constructed a borehole X-ray Fluorescence Spectrometer (XRFS) as part of the Mars Subsurface Access program [1].  It can be used to determine the composition of the Mars regolith at various depths by insertion into a pre-drilled borehole.  The primary requirements and performance metrics for the instrument are to obtain parts-per-million (ppm) lower limits of detection over a wide range of elements in the periodic table (Magnesium to Lead).   Power consumption during data collection was also measured.  The prototype instrument is complete and preliminary testing has been performed. Terrestrial soil Standard Reference Materials were used as the test samples. Detection limits were about 10 weight ppm for most elements, with light elements being higher, up to 1.4 weight percent for magnesium.  Power consumption (excluding ground support components) was 12 watts.

## INTRODUCTION & HISTORY

The requirement for elemental analysis during planetary exploration originated with the NASA Apollo Missions. With only Earth based visualization of the lunar surface and only conjecture as to the chemical composition and mechanical properties of the Lunar regolith, NASA realized a series of precursor missions to the moon were needed before astronauts could be sent there. The first missions were named "Ranger" and numbered nine spacecraft launched in the 1961 to 1965 time period by JPL to take close-up images of the lunar surface. Only three succeeded. The next missions to the moon were called "Surveyor" and besides the standard cameras, had instruments to measure soil mechanics and obtain an elemental analysis of the lunar soil. Five of the seven Surveyor were successful with the three (5, 6 & 7) having an alpha backscatter spectrometer (ABS), all surviving the trip. The ABS was designed to work in the vacuum lunar environment for the analysis of light elements that are most prevalent in planetary geology. After the Surveyor missions, the astronauts became the sample collection mechanism and returned the samples to Earth for analysis.

After Apollo, planetary exploration mainly used orbiters or fly-by type missions, so there was no need for an elemental analysis instrument. In the 70's, a decision was made to conduct a mission to Mars to test for the possibility of life.  At the insistence of planetary geologists, an additional instrument was added to the initial complement to perform elemental analysis of the Martian regolith. The ABS had the disadvantage that it had to operate in a $CO_2$ atmosphere on Mars and had to be heat sterilized for planetary quarantine reasons. The ABS was thus replaced by an X-Ray Fluorescence Spectrometer (XRFS). The XRFS instruments functioned through the full two year period that both

Viking Landers were operational. Elemental data obtained from the XRFS matches that obtained from the subsequent Mars Pathfinder and Mars Exploration Rovers (MER) despite the fact that data reduction was primitive by modern standards and the resolution of the proportional counters was 1000 to 1200 eV. The heat sterilization requirement dictated the use of proportional counter tubes rather than a solid state detector.

Since Viking did not find life on Mars, the planetary quarantine was relaxed and sterilization by means other that heat was allowed. A demonstration mission called "Pathfinder" was conducted to determine if a series of inflatable balls was sufficient to soft land a small rover onto the surface of Mars. The landing technique was successful and the Lander included an Alpha Proton X-ray Spectrometer (APXS) for elemental analysis using a solid state detector, along with a camera system and meteorology instruments. The APXS was subsequently redesigned using a new detector system and other design improvements, renamed Alpha Particle X-ray Spectrometer (APXS) and the APXS instruments now reside in the two Mars Exploration Rovers (MER) that are currently exploring Mars. The main limitation of the APXS is that the instruments still use a Cm-244 radioactive source for excitation, which gives considerably reduced elemental sensitivity above iron in the periodic chart. This provided the main motivation for the use of an X-ray tube for source excitation in the present instrument. An X-ray tube produces much higher source intensity for rapid data collection and the target material can be changed to match any particular required elemental analysis sensitivity.

**INSTRUMENT DESIGN**

The Mars Borehole instrument design was based on a previous instrument developed as part of the Department of Defense Site Characterization and Analysis Penetrometer System (SCAPS) to add the capability to measure heavy metal contamination to a suite of subsurface instruments deployed via cone penetrometry [2]. This new instrument will be deployed down a pre-drilled Mars borehole and must have a maximum diameter of 30 millimeters to be compatible with existing drills. The XRFS sensor assembly consists of an XRFS enclosed head assembly that will be deployed down the borehole and a rover based electronics control assembly consisting of power supply and control electronics for the XRFS instrument. PC based software provides the control, data readout and quantitative calculations needed for interpretation of the XRFS spectra. A study was undertaken to determine the minimum diameter that could be practically constructed given the available time, funding, and components. The final design achieved a 27.1 millimeter diameter.

The final design is shown in Figure 1. The excitation source was a silver anode X-ray tube and the energy dispersive X-ray detector was a 7 $mm^2$ Si-PIN diode. This detector was chosen mainly because of the availability of a preamplifier compatible with the size restrictions. It has a good peak to background ratio and a 12 micron thick Beryllium window for light element sensitivity. A digital pulse processor from the detector manufacturer converted the detector output to an energy spectrum. The energy calibration is linear and determined from the location of the iron characteristic emission and silver elastic scatter peaks. Since the borehole diameter is not controlled to very

good precision, the collimation and beam definition geometry was optimized to allow for varying distance to the measurement volume at the borehole wall. The excitation beam is larger than the area viewed by the detector, making the signal less sensitive to the wall distance.
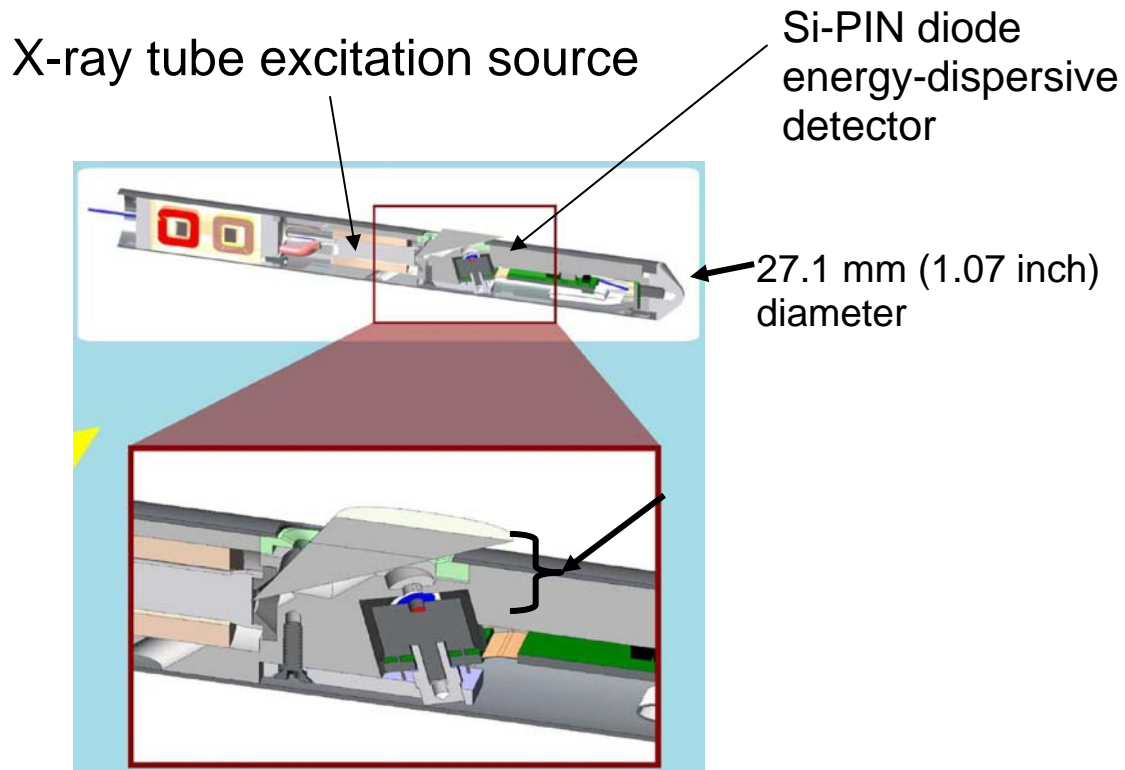
X-ray tube excitation source

Si-PIN diode energy-dispersive detector

27.1 mm (1.07 inch) diameter

Figure 1. Engineering drawing of the final design of the downhole assembly. The enlarged area shows the X-ray tube and the detector.

The performance requirement was to detect the elements magnesium through zirconium (atomic numbers 12 through 40 in the periodic table) and the elements cadmium through lead (atomic numbers 48 through 82 in the periodic table). The performance was determined by measuring the detection limits of these elements in a light element matrix.

Figure 2. Probe in Radiation Enclosure with Interlocks

Figure 2 shows the constructed probe in a leaded acrylic plastic enclosure for radiation protection with corresponding safety interlock. The dimensions of the probe are approximately 27 mm diameter and 250 mm in length. The 1/2 inch thick acrylic walls of the enclosure reduces the emitted radiation flux to normal background levels and yet still allows standard probe operation for performance determination and for elemental analysis of reference standards.

**MEASUREMENTS**

Standard Reference Materials (SRMs) from the National Institute of Standards and Technology were used for the characterization tests. SRMs numbered 2709, 2710, 2711, 97B and 2702 were used. These SRMs are a set of selected soils with varying amounts of the basic soil elements and extra elements in the form of contaminants. Concentrations ranged from tens of percents for the basic soil components to below one part per million. This provided a wide range to evaluate the instrument.

Samples were received from NIST as fine powders. The samples were poured into specimen cups as received and presented to the instrument without further preparation. Mars environmental conditions were simulated on a laboratory bench-top using a glove bag. Eight millibar carbon dioxide partial pressure (on Mars) was chosen as representative of the Mars atmosphere. A gas mix of 3 volume percent carbon dioxide with helium making up the balance at Earth ambient pressure and gravity provides the same carbon dioxide density typical of Mars atmosphere. All measurements were made in this atmosphere and in pure helium. Temperature was at ambient or at 0°C for the frozen samples.

**PERFORMANCE**

Figure 3 shows typical spectra from the borehole instrument. The specimen was a terrestrial soil, SRM 2709, measured in the Mars simulated atmosphere. The silver target X-ray tube was operated at 35 kV and 2 microamps. No filter or other optics was used in the incident beam. The detector has an internal collimator to restrict the beam to the center of the diode. Data collection time was 1000 seconds for the upper spectrum and 100 seconds for the lower spectrum. Note that the majority of the information is still available even with the 100 second data collection time. This short data collection will greatly facilitate the measurement of multiple strata in a borehole with vertical resolution of about 1 cm.
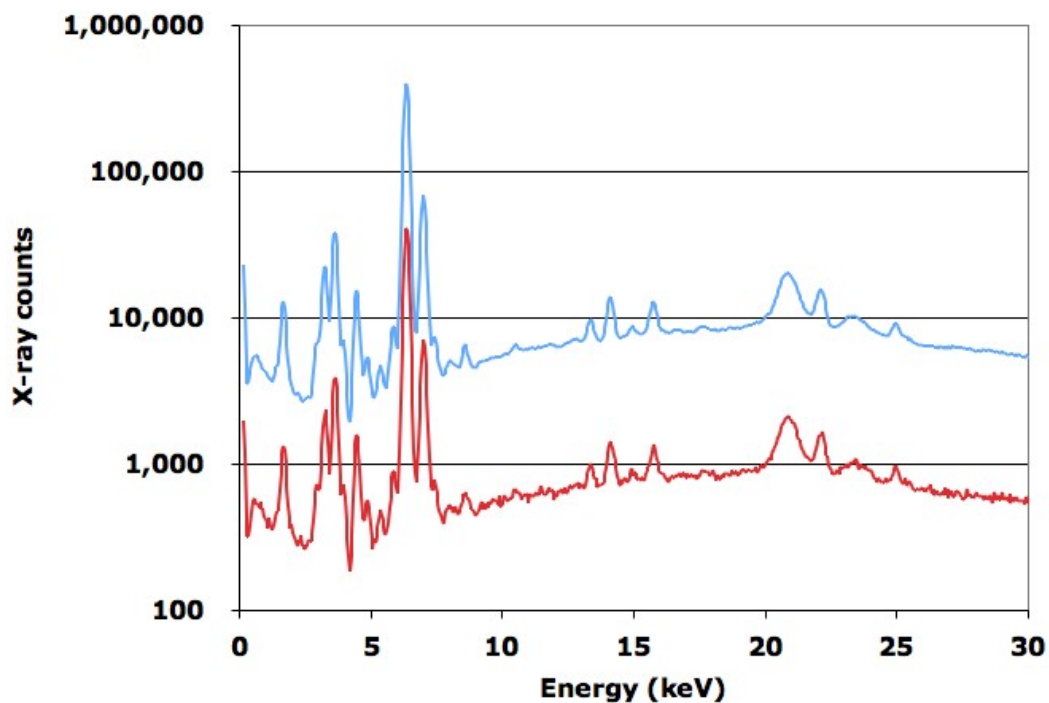


Figure 3. Spectra from borehole instrument. Upper curve is 1000 seconds data collection time, and lower curve is 100 seconds.

Figure 4 gives a comparison with the APXS spectra on the Pathfinder and MER rovers [3]. Note that the Pathfinder spectrum (lower curve) has little usable data above the iron peaks at 6.4 keV. The spectrum acquisition times for both APXS curves were many hours. The scale is counts per second, so 1 corresponds to about 72,000 total counts.
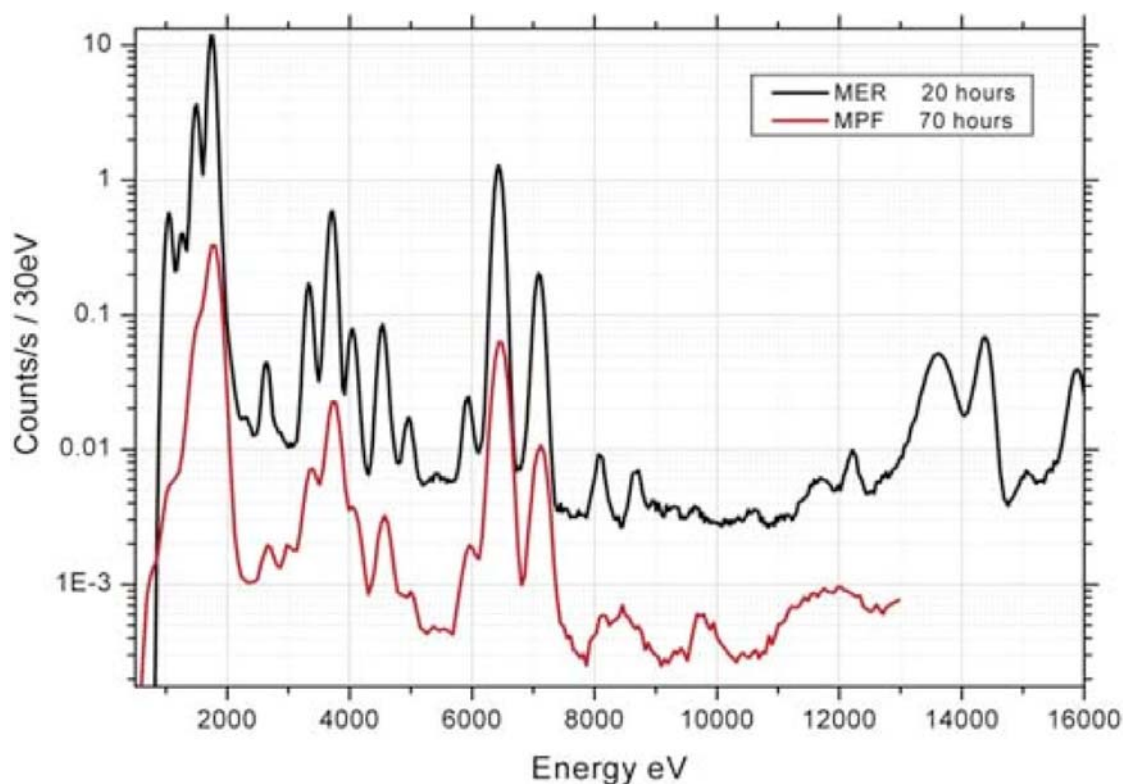
Figure 4. Spectra from the APXS instrument. Reproduced from reference 3.

Using the Borehole spectrometer, the detection limits for a number of elements in parts per million are presented in Table I. They are computed using the three sigma method and assuming a linear relationship between net counts and the certified concentration. The background was linearly interpolated from the counts on either side of the peak. Detection limits for each SRM are given, along with the average values. SRM 2710 has rather high concentrations of many of the elements, so the linear concentration relationship may not hold. This causes the detection limits to be larger in this material. They were included in the averages since they have the effect of raising the detection limits. None of the SRMs contained Mg at a level that gave an unambiguous peak. A compound with magnesium as a major element was used to determine the magnesium detection limit. Talc, or magnesium silicate hydroxide, is a readily available magnesium compound (as baby powder, obtained from a local pharmacy) and was used for this purpose. Lowering the X-ray tube voltage to 20 kV decreased the magnesium detection limit from about 3% to the 1.4% value in the table. The ability to change the excitation conditions is another strong argument for using an X-ray tube.

Table I.  Minimum detection limits for a number of elements.  See text for data acquisition conditions.

| Element | 2702 | 2709 | 2710 | 2711 | 97B | Average |
|---------|------|------|------|------|-----|---------|
| Mg | ND | ND | ND | ND | ND | 1.4%[*] |
| Ni | ND | 8.9 | 2.0 | ND | NP | 5.5 |
| Cu | 8.2 | 4.2 | 16.2 | 8.9 | NP | 9.4 |
| Zn | 8.0 | 6.6 | 16.9 | 8.3 | 6.4 | 9.2 |
| Pb | 8.8 | 3.3 | 22.0 | 12.4 | NP | 11.6 |
| Zr | NP | 4.5 | NP | 4.1 | 4.8 | 4.5 |

ND = Not Detected    NP = None Present    *20 kV excitation

Measured power consumption is given in Table II for the system components and the total power per spectra.  Ground support components like safety interlocks and the USB computer interface are not included, as these functions are either not necessary in a spacecraft or are expected to be provided.  The total power of 12 watts implies an energy requirement of 12 kiloJoules per spectrum for a 1000 second spectrum or 1200 Joules for a 100 second spectrum.  The 1000 second spectrum is comparable to the APXS energy per spectrum which although it uses lower power, requires it over much larger collection times and thus exceeds our energy per spectrum requirement.

Table II.  Power consumption during data collection.

| Function | Voltage | Current | Power |
|----------|---------|---------|-------|
| HV Power Supply | +14.84 V | 0.426 A | 6.31 W |
| X-ray tube control | +14.92 V | 0.150 A | |
| | -14.92 V | 0.149A | 4.46 W |
| Detector | +4.99V | 0.240A | 1.20 W |
| Total | | | 11.97 W |

Figure 5 provides the signal variation of spectrum counts with distance to the probe. Since the normalized Iron intensity is divided by total counts, this quantity is stable out to several millimeters, indicating that accurate quantification can be performed at these distances.

### Figure 5.
## Fractional Change of total spectrum counts & Iron intensity

**Out to 25 mm.**          **First 5 mm.**
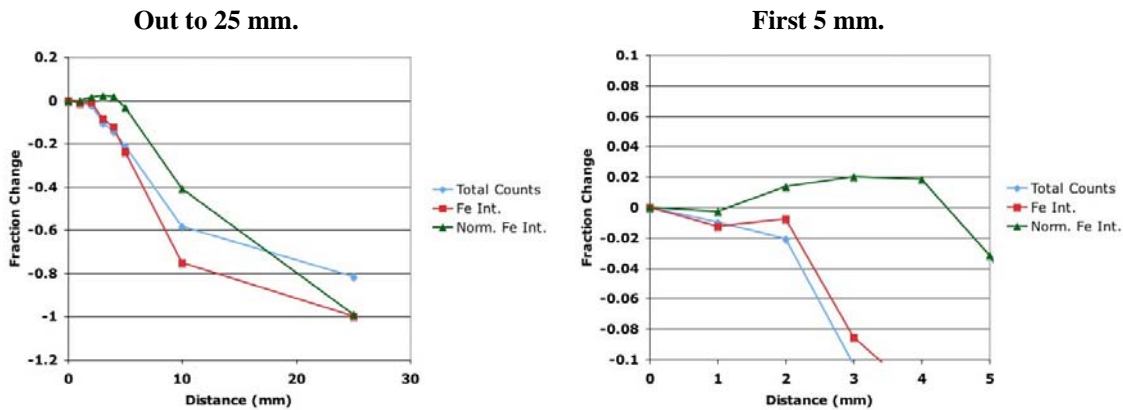


Entire spectrum varies less than 2% out to 2 mm.
Normalized Fe intensity varies less than 2% out to 4 mm.

## CONCLUSIONS

A borehole X-ray Fluorescence Spectrometer (XRFS) has been successfully constructed and tested.  Miniaturization has been performed to a diameter of 27.1 millimeters and components can be configured in a variety of XRFS instrument designs.  Modifications can be easily incorporated, such as an SDD detector, the use of a different target X-ray tube, or use of radioactive sources for excitation.  Performance is very good, with detection limits of about 10 ppm for many elements and detection of light elements down to magnesium at 1.4%.  Power consumption is 12 watts during data collection and the total energy per spectrum is comparable to or less than the previous planetary inorganic analysis instruments.  Adequate data can be collected in 100 seconds, facilitating investigation of strata with vertical resolution of about 1 cm in a reasonable time.

## ACKNOWLEDGEMENTS

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