Borehole X-Ray Fluorescence Spectrometer (XRFS): User's Manual, Software Description, and Performance Report

built by APL-UW under a NASA contract from the Langley Research Center

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Technical Report

APL-UW TR 0703

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CAUTION   CAUTION   CAUTION

**This device produces
X-rays when energized.**

CAUTION   CAUTION   CAUTION

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Acknowledgments

Contents

User's Manual

Overview

The X-ray fluorescence spectrometer (XRFS) is designed to be deployed down a pre-drilled hole for exploration and elemental analysis of subsurface planetary regolith (Figure 2 and Figure 10). The spectrometer excites atoms in the regolith and causes them to emit their characteristic X-rays. These characteristic X-rays produce peaks in the X-ray spectrum. By measuring the energy of the X-rays, elements are identified. By measuring the intensity of the peaks, the amount of each element can be determined. A software package operates the spectrometer, acquires the data, and analyzes the spectrum to provide elements and their weight fractions. It also provides a user interface to control the measurements and display the results.

Figure 1. X-ray fluorescence spectrometer **Head Unit** designed to be deployed down a pre-drilled hole to analyze subsurface elements.

The spectrometer consists of two main subsystems packaged in three physical units. The main subsystems are the X-ray source and the energy-dispersive X-ray detector. The source provides the X-rays to excite the specimen of regolith being investigated. The energy-dispersive X-ray detector detects the emitted X-rays, determines their energy (the energy-dispersive function), and counts the X-rays at each energy. Together these two subsystems measure the X-ray spectrum of the specimen.

Quick Start Guide

Figure 2. (from left to right) Borehole XRFS **Head Unit**, X-ray warning light, umbilical cable, X-ray **Interface** and **Control** units, and laptop computer.

Setup

To set up the instrument:

1. Remove the units from the case. The instrument consists of the **XRF Interface Unit**, the **XRF Control Unit**, and the **XRF Head Unit** (Figure 2). A laptop computer is used to control the instrument. There are three cables connecting the **Interface** and **Control** units, and the **Head Unit** has an umbilical cable (15 ft. long) permanently attached to connect it to the **Control Unit**. An X-ray warning light is also supplied. The instrument must be used in a radiation safety enclosure

or other personnel safety arrangement. An interlock cable connects to the safety interlock switch on the radiation safety enclosure to prevent accidental exposure.

2. Connect the cables as shown in Figure 3. All cables must be connected before turning on the main power.

3. Connect USB cable to the laptop computer and turn on the instrument with the key switch. The laptop should beep when it recognizes the connection to the instrument.

4. Wait one to two minutes for the detector to cool down. The instrument is now ready to collect a spectrum.
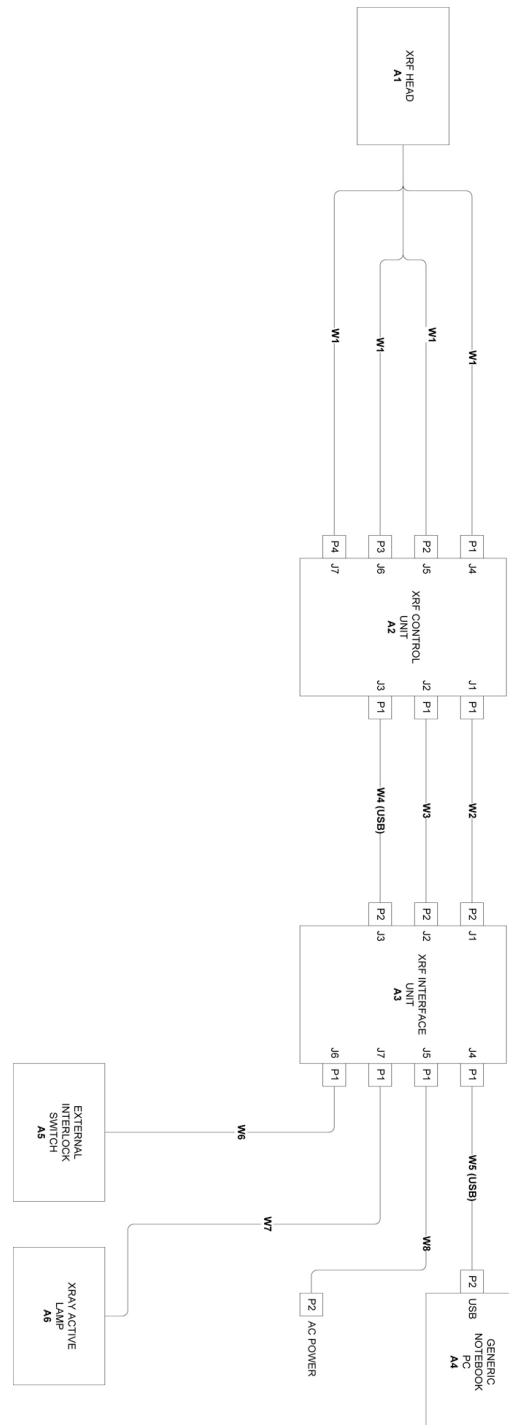
Figure 3. Connection diagrams for **Interface**, **Control**, and **Head** units of the XRF spectrometer.

Spectrum collection

1. Place the sample to be analyzed before the probe window.

2. Close the radiation safety enclosure and be sure the interlock switch is closed.

3. Start the program "BoreholeXRF"; the main screen appears (Figure 4).

4. Turn on the high voltage to 35 kV, and the emission current to 2 μA.

5. Set the preset time to the desired interval.

6. Click "Start".

7. The actual kV and μA values should be near the setting values (they may take one or two minutes to reach these values because of the slow ramp).

8. "Acquiring" will appear in red on the screen (Figure 5).

9. A spectrum will begin to appear in the plot box in the lower left corner of the interface (Figure 5).

10. To calibrate, click "Calibrate" and enter points in the form "channel number, energy in keV" in the window that opens (Figure 6). Then click "Compute," then "Calibrate," then "Close." You may calibrate before, during, or after spectrum collection.

11. To save the spectrum, click "Save."

12. To determine which elements are present in the sample, click the "Analyze" button. After several seconds, the plot box will show the background and spectrum fits and the elemental analysis will appear in the spectrum analysis box (Figure 7).

Software operation

Configuration settings:

- "load params panel" (Figure 8): Opening this window allows the user to change instrument control parameters (such as the ramp interval) and acquisition conditions (such as the atmosphere type).

- "load DP4 config panel" (Figure 9): Opening this panel allows the setting for the DP4 digital pulse processor to be changed. Refer to the AmpTek manual (Appendix A) on the DP4 for more information about these parameters.
- "on" and "off" under "X-ray control": These buttons turn the X-rays on and off.
- "setkV": This button allows the user to set the high voltage for the X-ray tube.
- "set µA": This button allows the user to set the emission current.
- "change preset time": Use this button to enter the desired interval of data collection in seconds, then click "OK." This command automatically clears the spectrum that is currently plotted (so save it first!). When the user clicks "Start" after entering this preset time, the program will collect a spectrum and stop automatically after the allotted time has elapsed. Note that this refers to accumulation time, not live time.

"Input Spectrum" functions:
- "Start": Click this button to begin taking a spectrum.
- "Stop": Click this button to stop spectrum collection before the preset time has elapsed.
- "Clear": This button clears the spectrum currently plotted.
- "Calibrate": The user may calibrate the energy of the spectrum any time before, during, or after collection. It is also possible to load and calibrate a previously saved spectrum. Click "Calibrate" to open the calibration window, enter the desired channel-energy pairs (in keV) in the form "channel, energy," click "Compute," "Calibrate," and "Close." It is also possible to type a desired energy-per-channel value and energy start value manually and then click "Calibrate," without pressing "Compute." The user may clear a previous calibration and return to the original channel values by pressing "Remove calibration."

- "Load": This button allows the user to bring up a previously saved spectrum. It is possible to enter a new spectrum label and operator, zoom in/out, calibrate, analyze and re-save any previously saved spectrum.

Plot controls: (Note: none of these buttons affect data collection.)
- "Restore": Restores the spectrum plot to its original scale and causes it to begin automatically adjusting the Y-scale to keep the entire spectrum in view.
- "← cursor": Moves the cursor one channel to the left.
- "cursor →": Moves the cursor one channel to the right.
- "X zoom in": Adjusts the X-scale so that it displays a smaller range of X values, centered around the cursor.
- "X zoom out": Adjusts the X-scale so that it displays a larger range of X values, centered around the cursor.
- "X shift left": Moves the view to the left approximately half of the plot range, so that the user is looking at slightly lower energies.
- "X shift right": Moves the view to the right approximately half of the plot range, so that the user is looking at slightly higher energies.
- "Y zoom in": Adjusts the Y-scale so that it displays a smaller range of Y values.
- "Y zoom out": Adjusts the Y-scale so that it displays a larger range of Y values.

"Analyze": Click this button (after calibrating) to run an automated analysis of the sample. It will return a list of elements present, their concentrations and uncertainty.

"Save spectrum": Allows the user to save the current spectrum and some configuration information to a file on the computer. These files are accessible by the "load" button in the Borehole XRF software, and can also be opened in a word processing program or text editor.

"Exit": Exits the spectrum collection program. The program will **not** prompt the user to save the current spectrum, so it is necessary to save (if desired) before exiting.
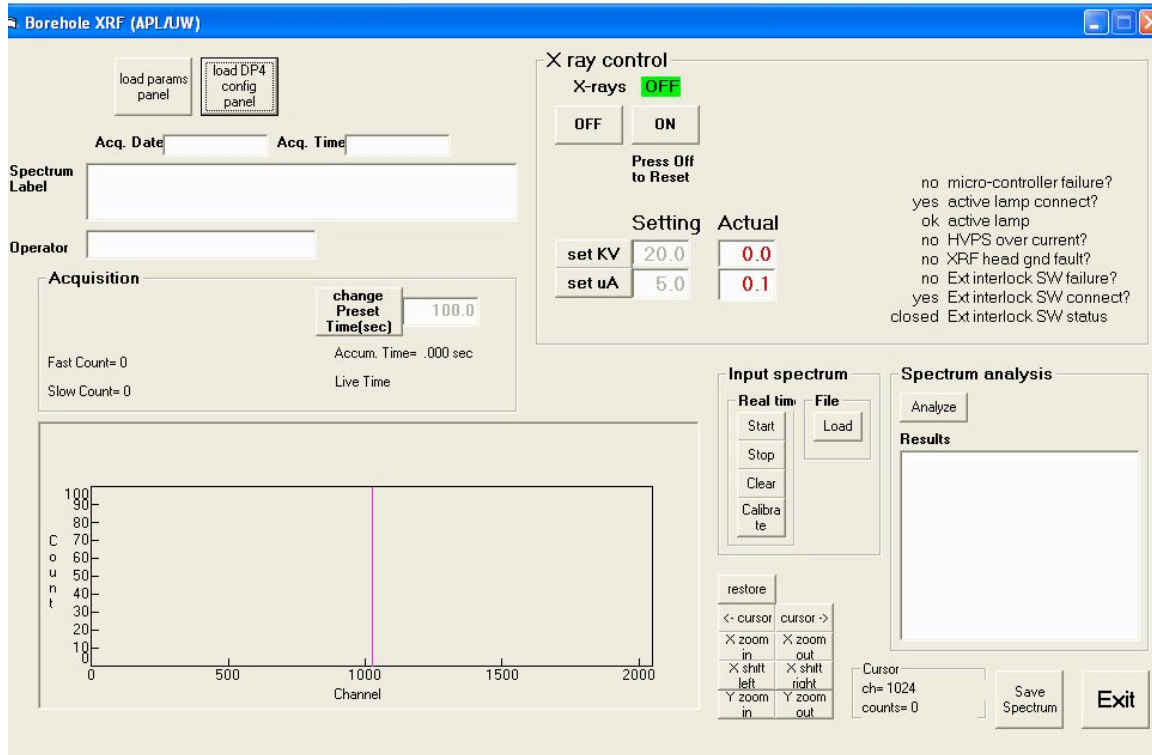

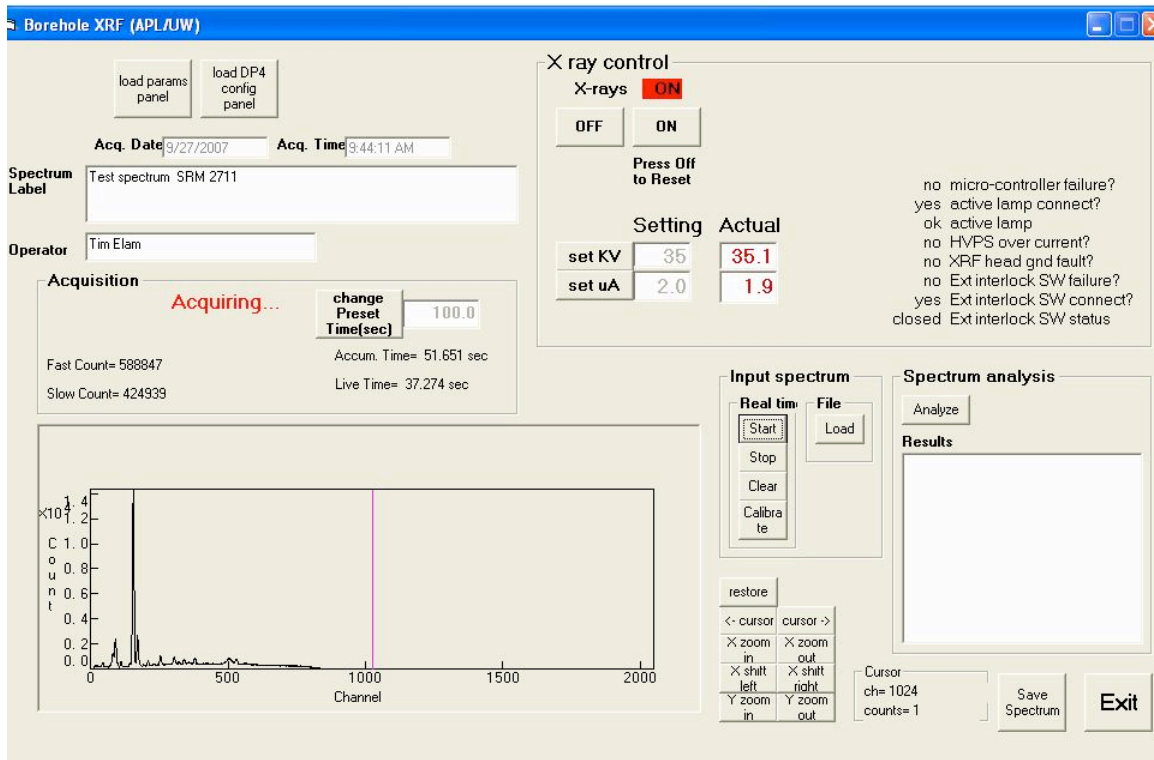Figure 4. Main screen view upon starting the program "BoreholeXRF"

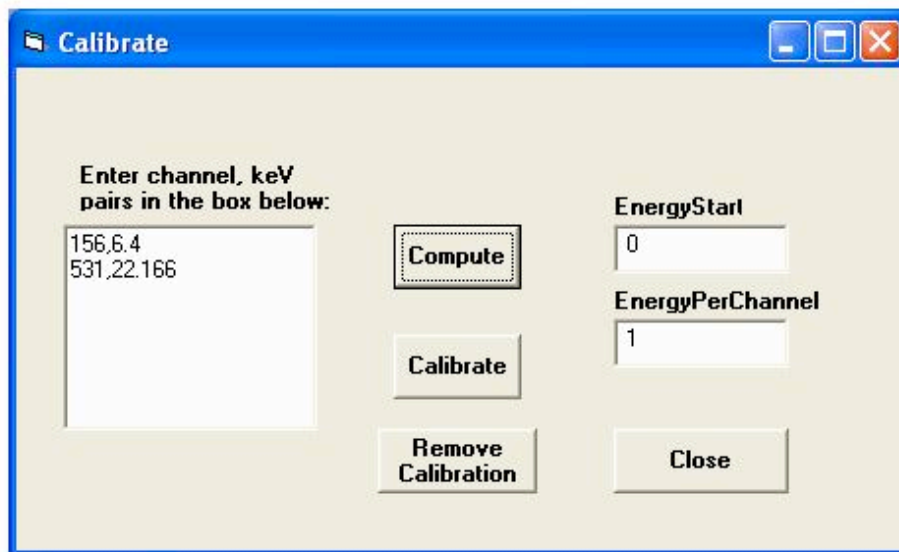Figure 5. View of laptop display during spectrum acquisition

Figure 6. View of calibration control window
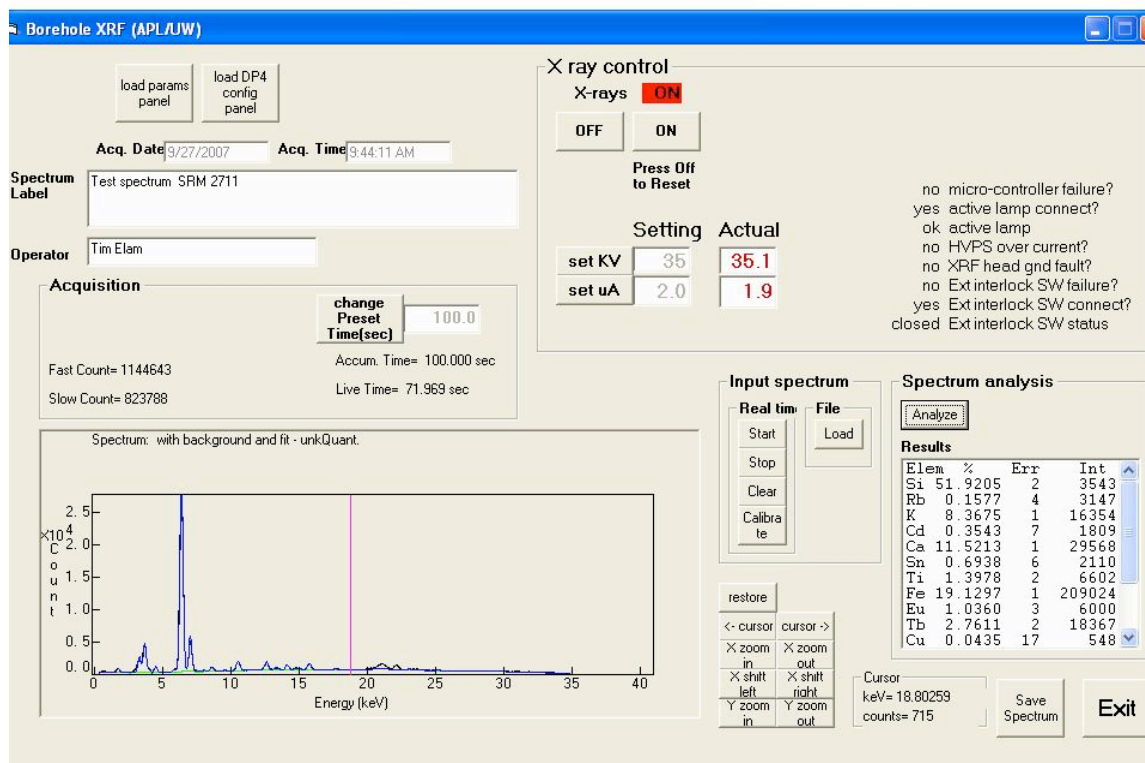
Figure 7. View of spectrum analysis display
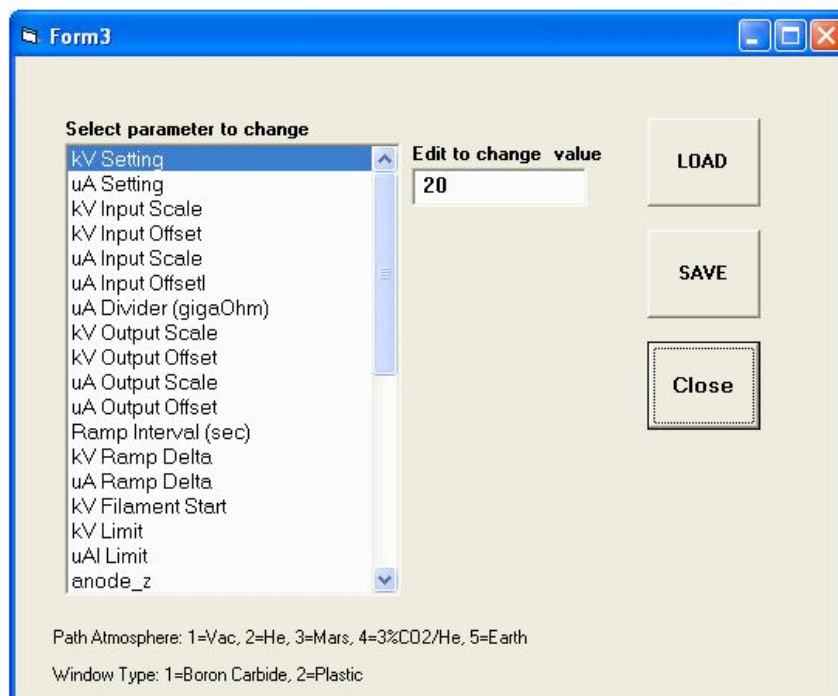
Figure 8. View of configuration setting "load parameters panel"
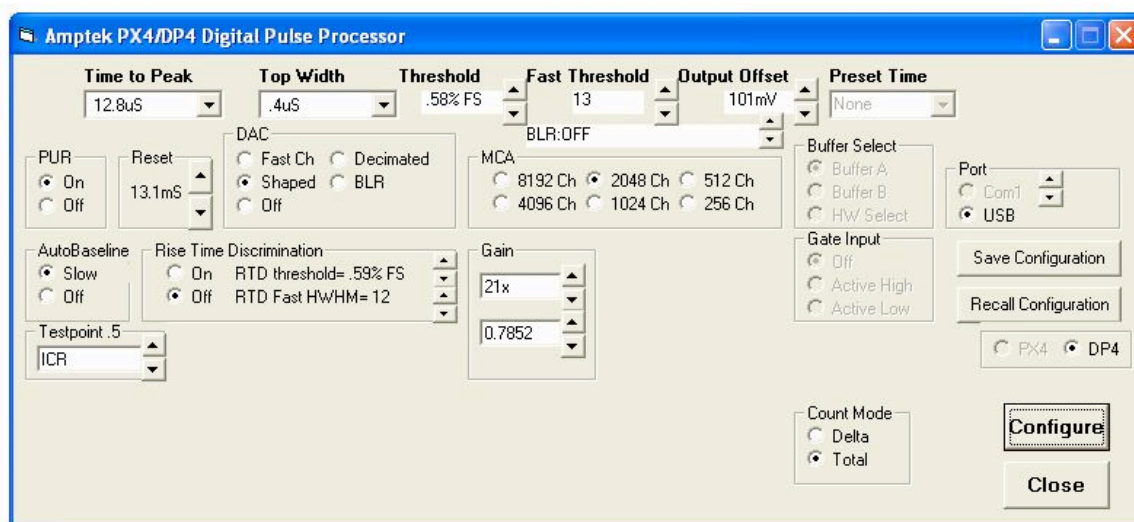
Figure 9. View of configuration setting "load DP4 configuration panel"

Borehole XRFS Software Installation

Before connecting hardware, copy all files from CD-R folder titled "BoreholeXRF As

Shipped Bin Sept 27.2007."

Place files in C:\Program Files\BoreholeXRF

(Note: the files MUST be in exactly this location to operate correctly.)

Necessary software files from the folder "BoreholeXRF As Shipped Bin Sept 27.2007":

- xrayxsct.dat
- XRFanalysis.dll
- APL_UW_XraySettings.xcg
- asycfilt.dll
- BoreholeXRF.exe
- cbw32.dll
- COMCAT.DLL
- COMCT232.OCX
- Comdlg32.ocx
- dp4.cfg
- MSCOMM32.OCX
- msvbvm60.dll
- oleaut32.dll
- olepro32.dll
- usbdrvd.dll

Install the driver for the Measurement Computing DAQ module.

- Load the Measurement Computing "MCC DAQ Software" CD
- Install InstaCal for Windows, TracerDAQ, and Hadware manuals
- Install Shield Wizard for InstaCal – click "Next"
- Destination Folder – click "Next"

- Ready to Install – click "Install"

- Completed – click "Finish"

- Install Shield Wizard for TracerDAQ – click "Next"

- Destination Folder – click "Next"

- Ready to Install – click "Install"

- Completed – click "Finish"

- User's Guides Setup – select "USB," then click "Install"

  (Driver is installed. This takes a few seconds.)

- MCC DAQ message box – "You must restart your system..." – click "Yes"

After system has restarted, connect Borehole XRF hardware to USB port and turn power

on. "Found New Hardware Wizard" should appear.

"Can Windows connect to Windows Update..." – choose "No, not at this time." – click

"Next"

Install software for DP4 Digital Pulse Processor (see also page 19 of Appendix A)

- Select "Install from a list or specific location" – click "Next"

- Select "Don't search, I will choose the driver to install" – click "Next"

- Hardware type – Select "Human Interface Devices" – click "Next"

- "Select the device driver..." – Click "Have disk..."

- Insert the AmpTek CD into the CD drive

- Click "Browse..."

- "Install From Disk" file dialog appears

- Navigate in the file dialog to:

  My Computer\AMPTEK\USB_Driver\Win2k_XP\apausb2k.ini

- Click "Open"

- Back at the "Install From Disk" dialog – click "OK"

- Back to "Select the device driver..." dialog – click "Next"

  (Driver is installed. This takes a few seconds.)

- Completing installation – click "Finish"

Run the InstaCal program

- From the menu bar, select: Start -> Programs -> Measurement Computing -> Instacal
- "Plug and Play Board Detection, USB-1408FS (Serial# 150)" should be selected
- Click "OK"
- Under the Install menu item, choose "Configure..."
- Change No. of Channels from "4 Differential" to "8 Single Ended"
- Click "OK"
- Under the File menu item, choose "Exit"

Run the Borehole XRF program by double-clicking on the file "Borehole XRF.exe"

At this point the software main screen should appear; it will obtain a spectrum and bring up all dialogs.

You may want to put a shortcut to the "Borehole XRF.exe" file on the desktop or some other convenient location.

Instrument Design

The instrument is designed to be deployed down a pre-drilled borehole and has a maximum diameter of 27.1 mm to be compatible with existing drills (Figure 1 and Figure 10). The XRFS sensor assembly consists of an XRFS enclosed head assembly that is deployed down the borehole and an electronics control assembly consisting of a 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.

The excitation source is a silver anode X-ray tube (Comet NA, Stamford, CT) [see Appendix B]. The energy dispersive X-ray detector is a 7-mm^2 Si–PIN diode (Amptek, Inc., Bedford, MA) [see Appendix C]. 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 (Amptek, Inc., Bedford, MA) converts the detector output to an energy spectrum [see Appendix C]. The energy calibration is linear and determined from the location of the iron characteristic emission and silver elastic scatter peaks. Because the borehole diameter cannot be controlled with precision, the collimation and beam definition geometry are 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.

The performance requirement is 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).
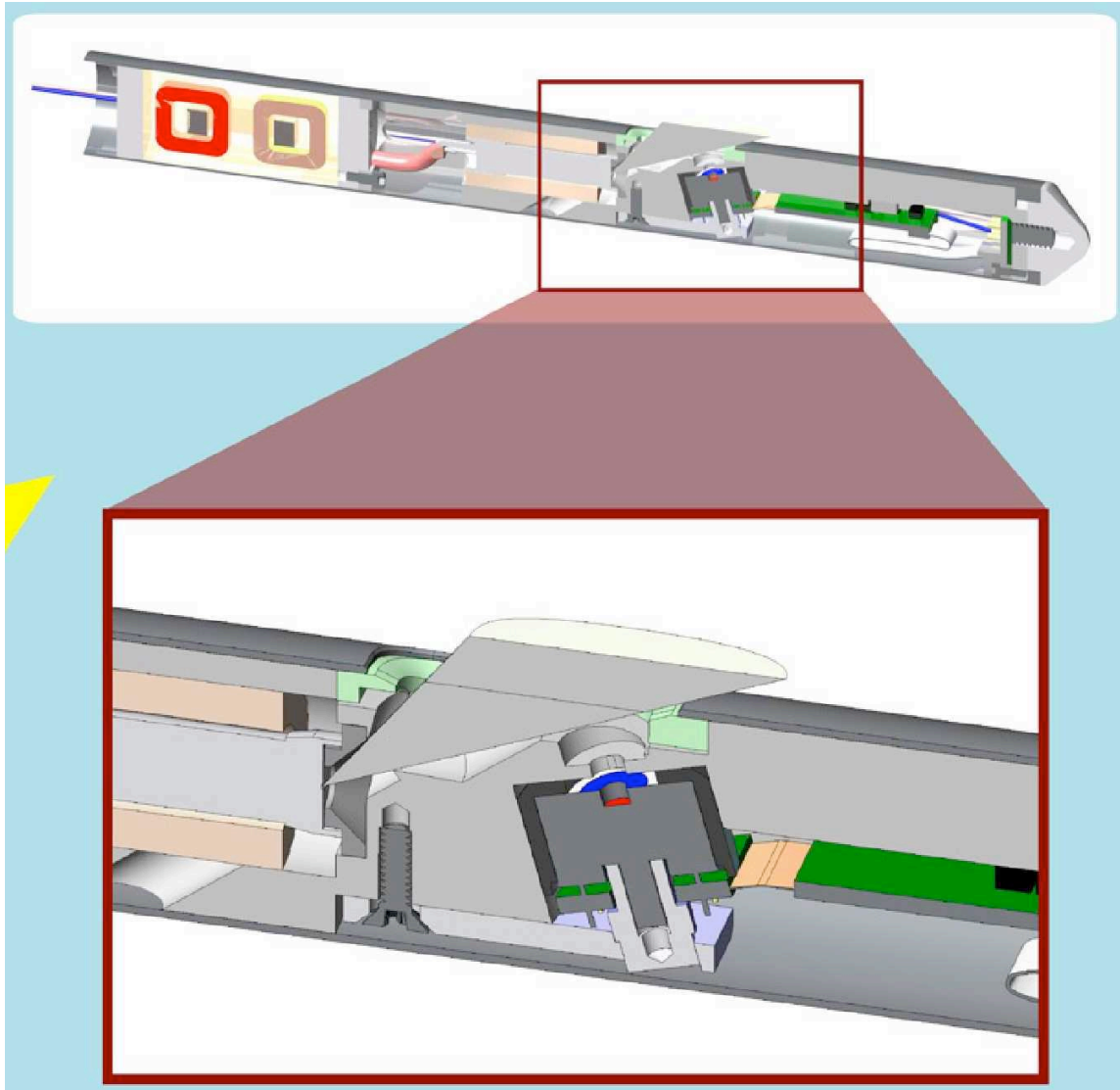

Figure 10. Engineering drawing of the final design of the downhole assembly. The enlarged area shows the X-ray tube and the detector.

Major hardware subsystems

The X-ray source is a miniature but otherwise conventional X-ray tube. It generates X-rays by bombarding a metal anode with high-energy electrons. The electrons are

produced in a hot filament and accelerated to high energy by a high voltage. The filament heater power controls the beam, or emission, current. The X-ray output is proportional to this current. The electron beam energy is controlled by the high voltage applied to the X-ray tube. This voltage determines the X-ray spectrum emitted by the tube and is one of the main parameters used to control the spectrometer. The X-ray tube has a very high vacuum inside and the X-rays exit via a thin window. Other parameters that influence the X-ray spectrum of the tube are the angles that the electron beam makes with the anode and the exit window, the material and thickness of the exit window, and, of course, the anode material.

The X-ray detector is based on a silicon diode that is reverse biased to provide a thick region of high-resistivity silicon with an electric field across it. The X-rays are absorbed in this region and produce electron–hole pairs in the silicon. The high electric field separates the electron–hole pairs and produces a pulse of charge at the electrodes of the diode. This pulse is amplified and its amplitude measured. Its amplitude is proportional to the energy of the absorbed X-ray. A digital pulse processor separates this pulse from the noise, determines its amplitude, digitizes the amplitude, and counts the pulses with matching amplitudes to collect a spectrum.

The silicon diode is taken to about –60ºC by Peletier cooling to reduce the noise and allow better resolution of the pulse amplitude. The energy resolution in the spectrum is limited by the electronic noise in the diode and is typically about 150 electron volts. The digital pulse processor is optimized for detecting and discriminating X-ray pulses from this diode from the background noise. The count rate (the maximum rate that X-rays can strike the detector) is limited to about 10,000 per second by the speed of the pulse processing. The count rate is determined by the material being measured and the strength of the X-ray source. The rate is typically adjusted by controlling the beam current in the X-ray tube, as described above.

Hardware physical units

The subsystems are packaged into three units: the **XRF Head** that goes down the borehole and makes contact with the material being measured, the **XRF Control Unit**, and the **XRF Interface Unit**.

The **X-ray Head** contains the X-ray tube and the silicon diode X-ray detector. It also has a filament isolation transformer for the X-ray tube to isolate the filament heating power from the high voltage. It contains a preamplifier for the detector to amplify the pulses before they travel over the connecting cable. The **X-ray Head** is as small as possible to go down the smallest pre-drilled hole and measure the composition of the regolith at various depths. The 15-ft. umbilical cable is permanently attached to the **Head Unit**; it connects the **Head** and **Control** units.

The **XRF Control Unit** contains all of the essential electronics to operate the X-ray tube and detector. It constitutes the electronics that would be required for a future spacecraft instrument. For the X-ray tube, there is the high voltage power supply (HVPS), the filament driver and regulator, isolation amplifiers to provide monitor signals for the voltage and current, and an over-current protection circuit. For the detector, the unit contains a power supply board and the digital pulse processor board.

The **XRF Interface Unit** contains the hardware necessary to adjust and monitor the X-ray tube voltage and current from the host computer, several interlock sensors for personnel safety, and the low voltage power supplies for the electronics. This unit contains all of the support equipment that is necessary to operate the spectrometer on the ground. There are several cables connecting the **XRF Interface** and **XRF Control** units. The **XRF Interface Unit** also connects to the host computer via USB, to the personnel safety outerlocks, and to the main power line.

The software described here interacts mainly with the data acquisition board used to control the X-ray tube high voltage supply and with the digital pulse processor board for the detector. These functions are described more fully beginning on page 26.

Typical Operation

A typical X-ray spectrum of terrestrial soil is shown in Figure 11. There are three significant features. First are the large peaks in the spectrum between 3 and about 15 keV. These peaks are from the elements in the sample and are the main features of interest. Second are the peaks between 15 and 20 keV. These are the characteristic peaks from the X-ray tube anode material (silver in this case) that have been scattered toward the detector by the sample. They can provide additional information but are not as straightforward to interpret. The third feature is the background under the peaks. The background is small in an XRF spectrum from a good spectrometer, allowing detection of even very small peaks from elements at very low concentrations (the minimum detection limit). However, it must be modeled and removed by the analysis algorithms to provide accurate measurements of the peak intensities.
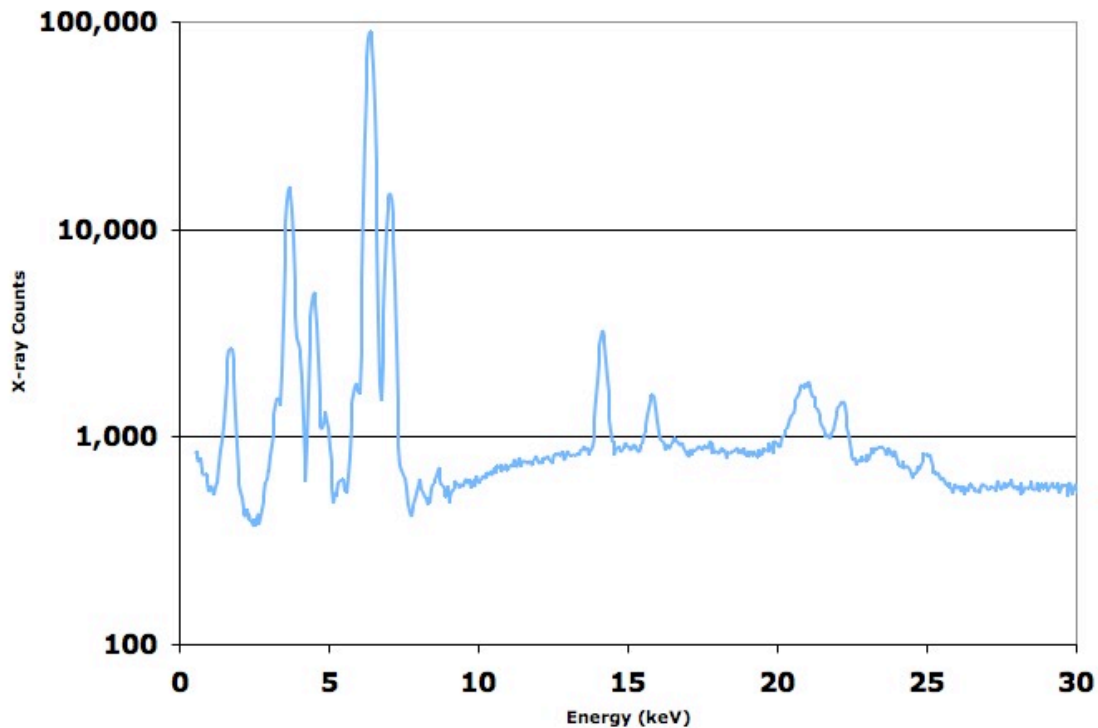
Figure 11. Fluorescence spectrum taken with the borehole XRFS. The soil sample was JSC1A Lunar Simulant.

Typical operation of the spectrometer by a user involves these steps. When power is turned on, the X-ray source is off (not producing X-rays) and the detector starts to cool down. The user places a specimen in front of the measurement window on the side of the XRF head (or places the head in a borehole).

The user then closes a radiation safety enclosure. When the safety enclosure is closed, a safety interlock switch closes, allowing the X-rays to be turned on. The voltage and current for the X-ray tube are set at this time, or previous settings are read in and used. The user chooses a data acquisition time, clears the spectrum in the digital pulse processor (DPP), and starts data acquisition. The spectrum is displayed as it is collected and the user will typically check the total count rate and make sure the spectrum looks correct (perhaps examining some regions more closely using zoom and pan). The user may be looking for particular elements, and will thus focus on the chosen elements. The

user will then stop the data acquisition, change any data acquisition parameters to optimize the spectrum, clear the spectrum, and collect the desired data.

The data are displayed as a function of the X-ray energy. To do this, the detector pulse height must be calibrated to match the X-ray energies. This is typically done using X-ray peaks from known elements. The calibration must be checked (typically daily) and may need to be repeated at irregular intervals.

Once data are collected, they can be stored in a file and/or analyzed further. Further analysis consists of modeling the background, finding any peaks in the spectrum and associating them with the corresponding elements, determining the net intensity of the peaks, and converting this net intensity into weight fractions of the elements. The background and a reconstruction of the spectrum using the extracted net intensities are displayed. This allows the user to quickly and visually evaluate the analysis of the spectrum. The element list and weight fraction of each element, together with estimated uncertainties, are also displayed.

The conversion from peak intensity to weight fraction is accomplished using a physical model of the interaction of X-rays with the material being analyzed. This model requires a complete description of the instrument to give accurate results. This description is more information than is typically changed by the user, such as fixed angles and distances within the components. It is also more information than the software needs to control the instrument. This information is read in from a parameter file and is usually not changed. It can be initially entered and changed via second-level dialogs that are invisible unless needed.

The user can also enter information about the material being analyzed and can change the instrument description information to be stored with the spectrum if desired. The data

acquisition parameters, such at X-ray tube voltage and current, are automatically stored with the spectrum.

The spectrum is stored in a file along with the information about the parameters under which it was acquired and enough of a description of the instrument to allow later analysis if necessary. The file format used is the standard format for energy-dispersive spectra adopted by the Microscopy Society of America and the European Microscopy Society.[1] Additional keywords were added to provide a complete description of the measurement conditions including instrument configuration (see Table 1).

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[1] European Microscopy Society standard format, Version 1.0, see files emmff.doc and emmff.src, at http://www.amc.anl.gov/ANLSoftwareLibrary/02-MMSLib/XEDS/EMMFF/
There is a proposed format based on XML that is not yet standard. See file EMSA_MAS_V2_XML_MM8_2002.pdf

Hardware Description

XRF Interface Unit

Low voltage power supplies (COTS)

+/– 15 VDC for op-amps
+15 VDC, 1.5 amp for HV module
+5 VDC for interlocks and detector
Detector requires 0.5-A steady-state with 1-A startup surge lasting 30–60 sec.

DAQ and control board (COTS)

OnTrak ADR2000A

USB to serial adaptor (COTS)

Targus PA088

USB hub (COTS)

D-Link, Model DUB-H4, high-speed USB 2.0 4-port hub

Safety interlocks and control (APL-UW)

X-ray on/off

Purpose. Turns the X-ray tube on and off, including ramping filament up and down and safety disabling the high voltage module.

Background. This signal controls the main functions of the X-ray tube power supply system. It is disabled whenever one of the safety signals (see below) is absent. It will shut down and latch in the "off" condition whenever one of the safety signals disappears.

Operation. Logic circuit responds to a binary signal from the DAQ, tests all of the safety condition signals, and provides signals for HV disable, filament voltage ramp control, and status to the DAQ board.

Warning light and fail-safe

Purpose. Controls the warning light (115 V AC external lamp), turning it on and off with the X-rays. Provides a safety signal that indicates when the lamp is working.

Background. One of the federal safety requirements for X-ray systems is a warning light that turns on whenever the X-ray producing system is energized (defined as high voltage on). The light must be fail-safe in that the X-rays will not come on if the light bulb is burned out.

Operation. Solid-state relay to turn the power to the external socket on and off. A current detector (full-wave bridge rectifier in parallel with Zener diodes driving a 5-V DC relay) indicates that the external lamp is drawing current (i.e., connected and not burned out). The current detector does not provide a signal unless the lamp is energized, a delay must be provided that allows the lamp to be turned on, then X-rays must be turned off if the current detector does not indicate lamp operation. The delay is typically about 100 milliseconds.

Electrical interlock status

Purpose. An external signal provided by a user that indicates that all of the X-ray shielding is in place.

Background. Another federal safety requirement is that the enclosure that protects human operators from radiation exposure be interlocked to the high voltage supply. This interlock must disable the high voltage if the shielding is opened, to prevent accidental radiation exposure.

Operation. An external signal. It disables the high voltage power supply and prevent X-rays from being turned on (or shut them off if they are on). The signal is typically an external switch closure. It also disables the X-rays in case of a short to ground to prevent shorts from giving a false OK signal. A TTL or other logic signal is OK if +5 V or similar is available on the external connector to facilitate use with un-powered mechanical switches on a radiation enclosure.

Filament connector engaged

Purpose. To insure that the filament connector is inserted before the high voltage or X-rays are turned on.

Background. The commercial, miniature high voltage connectors used have only a contact for the negative high voltage, not for the positive return path (ground in this case). The ground return path is via the filament connector. If the high voltage is energized with the high voltage connector in place and the filament connector dangling, then a shock hazard condition can be produced.

Operation: A simple logic circuit that passes through the external filament connector via two extra pins.

Over-current signal

Purpose: See over-current cutoff under high voltage power supply board in the **XRF Control Unit** above. This signal is passed through to the DAQ and should remain after the X-rays are turned off until reset via the DAQ (usually by the X-rays off signal).

Background. This is just an interface to the over-current cutoff from the high voltage power supply board.

Operation. Logic circuit that is part of the X-ray on circuit.

Ground failure detect

Purpose. Insures that a safe return path for the high voltage exists and avoids potential shock hazards.

Background. If high voltage is applied to the X-ray tube and a connection between the X-ray tube anode and the return current path to the power supply ground fails, then a potential shock hazard exists. This circuit tests the ground return path by applying a voltage to the X-ray tube anode via a resistor, then testing to be sure that voltage is shorted to ground.

Operation. Applies a voltage through a pair of resistors, to the X-ray tube anode over the umbilical cable. One resistor is in the power supply and one is in the **XRF Head Unit** near the X-ray tube anode. This will produce a known voltage if the X-ray tube is properly grounded. If the umbilical cable lead is shorted, then the voltage will be zero. Provides a signal if the correct voltage is present, and disable the X-rays if not.

XRF Control Unit

High voltage power supply board
HV module (COTS)

Filament driver and regulator (APL-UW)

Purpose. Provides AC drive voltage for filament isolation transformer to heat filament. Regulates filament voltage to achieve emission current set point.

Background. The electron beam in an X-ray tube is generated from a hot filament at high negative voltage. The electrons emitted from the hot filament are accelerated by the high voltage and strike a metal anode at ground potential. The metal anode emits X-rays. The filament is heated by a current passing through the filament wire. Since the filament is at high negative potential, the heater current must be isolated by a filament transformer operating at 6 kHz. The electron beam current is regulated by the temperature of the filament, which is controlled by the filament heater current.

Operation. 6-kHz AC is generated by an oscillator, whose output voltage is controlled by a feedback loop. The output goes to a power audio amplifier chip to produce enough power and voltage to drive the filament transformer (which is located in the XRF Head). The feedback loop compares the current signal from the HV module to a set point and adjusts the filament heater voltage. The feedback has upper and lower limits (via a Zener diode), integration of the error signal (via a capacitor), and some linear gain for stability (via a resistor), all in the feedback leg of an op-amp. This regulator reverts to the "filament off" condition on power-up and wherever X-rays are turned off.

Isolation and amplification of HV monitor signal (APL-UW)

Purpose. To condition the signals from the HV module to achieve convenient gain and to protect the remainder of the circuits from spikes due to high voltage arcs.

Operation. Op-amps with diode and capacitor spike suppression at their inputs.

Over-current cutoff (APL-UW)

Purpose. To protect all of the hardware from a long-term overload condition.

Background. X-ray tubes sometimes develop arcs or plasma discharges. If they are brief, they usually clear themselves and are not a problem. But if they last for several seconds, they can overheat themselves or other components. This protection circuit serves as a backup to the software over-current protection. The HV module is also current-limited, but that only protects the module, not the X-ray tube.

Operation. Compares the emission current signal from the HV module to an on-board set point. If the emission current exceeds the set point for more than 5 sec, turn off the X-rays.

Detector power board (COTS)

AmpTek PC4-3

Detector pulse processor board (COTS)

AmpTek DP4. The detector system is completely isolated as well as electrically and magnetically shielded from the X-ray tube power supply, with one common ground point at the +5 volt power supply. The signals from the X-ray detector at the preamp output are pulses of about 10 microseconds duration and about 1 mV amplitude. Their amplitude must be measured to within a few percent to obtain a useable X-ray spectrum. Electronic noise is the major limitation and is minimized. Magnetic shielding is accomplished with co-netic foil.

Software Description

X-ray Tube Control (XTC)

Description

This module has two main purposes: to display and allow the user to change the parameters related to the X-ray tube, and to control the high-voltage power supply (HVPS) for the X-ray tube. The controls for this module are located on the main screen in the upper right corner of the interface (Figure 4).

The main parameters for the X-ray tube are the high voltage (kV) and the beam emission current (μA). Typical values are 35 kV and 2 μA. The user must also input a complete description of the X-ray tube for proper operation of the quantitative analysis software. These parameters are in a separate dialog that appears on request but is usually invisible (Figure 8).

The HVPS has a series of safety interlocks to prevent accidental exposure of personnel to high electrical voltages and X-ray radiation. The status of these interlocks is clearly visible to the user and turn red if any fail (Figure 12).

Control of the HVPS requires turning the X-ray on and off under user control, and responding to any changes in the interlock status by turning off the X-rays. The X-ray tube voltage and current settings are converted from the display units (kV and μA) to the DAC integer values and sent to the DAC using its commands. The actual values are read from the DAC and converted to the display units. When the X-rays are turned on, the X-ray tube must be ramped up to the operating conditions gradually (see ramp-up under functions).
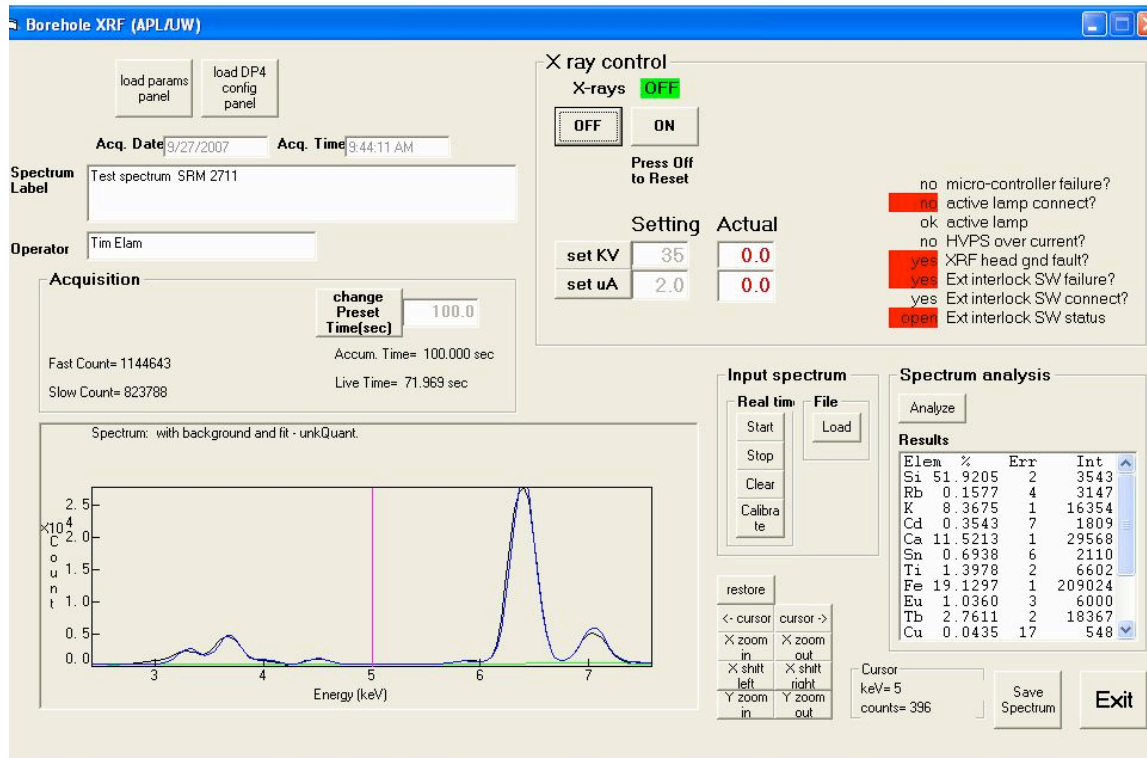
Figure 12. Borehole XRF interface indicating interlock failure

Functions

Set and display X-ray tube voltage (kV)

Set and display X-ray tube emission current (μA)

Check limits for X-ray tube parameters

Check and display status of safety interlocks

- X-ray on/off
- Warning light and fail-safe
- Electrical interlock
- Filament connector engaged
- Over-current signal
- Ground failure detect

Turn X-rays on and off

Ramp-up X-ray tube gradually to full operation

- Bring up kV to no more than 10 kV

- Bring up emission current to no more than 5 μA

- Raise kV and μA gradually together to specified values

Communicate with HVPS

- USB port or other communication parameters

- Commands to ADC and DAC

- ADC and DAC conversion constants

Set and display X-ray tube and instrument description

- X-ray tube type (side or end window)

- Anode material

- Be window thickness

- Electron incident angle

- Takeoff angle

- Aperture size and distance

- Filter material and thickness (if any)

- Path length from X-ray tube to specimen

- Angle that X-rays from tube strike specimen (incidence angle)

Detector Data Acquisition (DET)

Description

The X-ray detector acquires the spectrum; its associated electronics are commercial off-the-shelf. The manufacturer (Amptek, Inc., Bedford, MA) also supplies a library of communications and control routines that operate over a USB interface. The main function of the DET module is to drive these functions to acquire the spectrum (once the X-ray tube is operating and the user requests data be collected). As with the X-ray tube,

there are several data acquisition parameters that the user can change. Some of these appear on the main screen and some in a separate dialog (Figure 9).

The signal from the X-ray detector is analyzed by a digital pulse processor (DPP) that is specialized for energy-dispersive X-ray detector pulses. Many of the parameters for this DPP are software changeable but require specialized commands and some tuning procedures. The parameters are loaded at startup from a database.

One of the auxiliary functions of the detector data acquisition module is to check, set, and maintain the detector energy calibration (Figure 6). This calibration relates the channels in the spectrum (which are proportional to the pulse amplitude from the detector) to X-ray energy. The calibration is determined using the peaks of known elements, either in the spectrum from the material of interest if they are known or from a calibration sample. The energy calibration procedure consists of finding the location of the peaks, identifying the element associated with the peak, and including the peak positions and element energies in a calibration function. The function used is linear. The energy calibration will usually not change much day-to-day, so a stored calibration can be used. Any changes in the DPP tuning will change the calibration, so the DPP setup and calibration will force a re-calibration if any DPP parameters are changed.

Functions

> Communicate with detector digital signal processor (COTS code)
>
> Set and display data acquisition parameters
>
> - Live time (seconds, calculated in DPP)
> - Real time (seconds)
> - Count rate (counts per second, display only)
> - Dead time (%, display only)
> - Total counts (display only)

- Chamber atmosphere (Earth ambient, Mars ambient, pure helium, vacuum)

Energy calibration (eV per spectrum channel)

- Set and display calibration constants
- Calculate energy vs. channel (linear or quadratic function)

Set and display detector parameters

- Aperture size and distance
- Path length from specimen to detector
- Energy resolution
- Window material and thickness
- Dead layer material and thickness
- Active layer material and thickness
- Angle that X-rays exit specimen toward detector (emergence angle)

Set and display digital pulse processor parameters

- (See manufacturer's manual, Appendix A)

Control digital pulse processor setup

Save and Load Parameters (PAR)

Description

This module handles all the parameters from other modules. The functions in this module are called at startup and shutdown, and by the other modules whenever any parameters are changed.

The module saves the parameters to a file and reads them from a file. The name and location of the parameter file are set and displayed by this module via a dialog (Figure 8). No other parameters are modified or displayed by this module. The file format is determined and controlled by this module.

Functions

      Set and display parameter file name

      Save all parameters to file

      Load all parameters from file

Save and Load Spectrum (SSF)

Description

The spectrum is stored in a file that contains the spectrum data (counts per channel), the energy calibration that relates channels to X-ray energy, the parameters under which it was acquired, and the description of the instrument. Older files can be read in by the software and displayed and analyzed just as newly collected data are handled. All of the information necessary to display the spectrum and to allow later re-analysis if desired is stored in the spectrum file.

Functions

      Set and display spectrum file name

      Save data and all relevant parameters to file

      Load data and all relevant parameters from file

File format

The file format is the standard format for energy-dispersive spectra adopted by the Microscopy Society of America and the European Microscopy Society.[2] Additional

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[2] European Microscopy Society standard format, Version 1.0, see files emmff.doc and emmff.src, at http://www.amc.anl.gov/ANLSoftwareLibrary/02-MMSLib/XEDS/EMMFF/
There is a proposed format based on XML that is not yet standard. See file EMSA_MAS_V2_XML_MM8_2002.pdf

keywords (Table 1) were added to this format to allow inclusion of the instrument

parameters used to analyze the spectrum.

Table 1. Keywords for XRFS spectrum output and parameter files added to the standard format
for energy-dispersive spectra

Keyword	Description	Default value
kVsetting	X-ray tube kiloVolts Setting	20.00000000
uAsetting	X-ray tube microAmps Setting	5.00000000
kVscaleIn	kV Input Scale	-9.56999969
kVoffsetIn	kV Input Offset	0.00000000
uAscaleIn	uA Input Scale	24.50000000
uAoffsetIn	uA Input Offset	0.01000000
uAdividerR	uA Divider resistance (gigaOhm)	0.40500000
kVscaleOut	kV Output Scale	0.09380000
kVoffsetOut	kV Output Offset	-0.14000000
uAscaleOut	uA Output Scale	0.04100000
uAoffsetOut	uA Output Offset	0.10000000
RampInterval	Ramp Interval (sec)	1.00000000
kVdelta	kV Ramp Delta	1.00000000
uAdelta	uA Ramp Delta	5.00000000
kVstart	Minimum kV for Filament Start	10.00000000
kVlimit	kV Limit	40.00000000
uAlimit	uA Limit	25.00000000
anode_z	X-ray tube anode atomic number	47.00000000
kv	X-ray tube kiloVolts during acq.	20.21008301
tube_inc_angle	X-ray tube electron incident angle	90.00000000
tube_takeoff_angle	X-ray tube takeoff angle	51.11999893
tube_be_window	X-ray tube Be window (mm)	0.50000000
filter_z	Incident beam Filter atomic number	1.00000000
filter_thick	Incident beam Filter thickness (micron)	0.00000000
excit_angle	Incident beam Excitation angle (deg)	38.86999893
emerg_angle	Fluorescence Emergence angle (deg)	74.12000275
solid_angle	Solid Angle (sterdian)	0.00000850
path_type	Atmosphere Path type	2.00000000
inc_path_length	Incident path length (cm)	0.94000000
emerg_path_length	Emergence path length (cm)	1.97000003
window_type	Probe Window type	2.00000000
window_thick	Probe Window thickness (micron)	0.00000000
minimum_energy	Minimum analysis energy (eV)	1000.00000000

Spectrum Processing (SP)

Description

This module calls another written in C++ to handle all of the computations. The parameters needed by the physical model contained in the code are provided by the spectrum processing module to the C++ module. The results of the spectrum analysis are provided to the user in an on-screen list and to the spectrum display module. This includes the calculated background and peak fits, the list of elements found in the spectrum, and the weight fractions of each element with associated uncertainties (Figure 7). Net intensities of the associated peaks for each element are also displayed with the intensity error from the Poisson statistics of the spectrum in the list on the lower right corner of the interface.

The element identification and association of peaks with elements is fully automated but is not entirely reliable. The quantitative results can be copied to the clipboard and made available outside the program to prepare reports using the results of this instrument.

Functions

   Background calculation and removal
   Peak search
   Element identification (associate peaks with elements)
   Net peak intensity determination and calculated peak fits
   Quantitative analysis (converting peak intensity to element weight percent)
   Copy results to display

Spectrum Display (SD)

Functions

   Plot spectrum vs. X-ray energy (Figure 5)
   Overlay calculated background and peak fits

Display markers at characteristic element emission line energies

Scale, zoom, and pan

Instrument Performance Report

The purpose of this instrument is elemental analysis of regolith strata in a pre-drilled borehole to investigate the subsurface of Mars and possibly other bodies within the solar system. As such the primary performance criterion is the ability to quantify the elements present in a particular stratum in an acceptable time and with sufficient accuracy to obtain useful scientific information. For the purposes of this study, the detailed performance of the sensor was evaluated by measurements on the actual prototype. The main performance metric is the minimum detection limit (MDL). Improvements in the ability to detect an element imply improvements in the ability to quantify the amount present. Though there are some subtleties in this, the performance is dominated by the number of X-rays present in the spectrum, which is dominated by the source strength given the constraints on geometry and the available detectors for this instrument. The performance was evaluated by measuring the detection limits of target elements in a light element matrix.

The ability to accurately quantify a particular element is mainly limited by the precision with which its X-ray emissions can be measured. This is determined by the statistical variations in X-ray intensity due to the Poisson nature of their arrival times. In a given time interval the number of X-rays that are detected has an intrinsic variance (the square of the standard deviation) equal to the number of X-rays. This means that the relative standard deviation is one over the square root of the number. For a given geometry and sample composition, the number of X-rays detected from a particular element is proportional to the source strength and the measurement time.

Detecting an element depends on both the number of X-rays collected from that element and the background present even in the absence of that element. Because the background is also subject to the same variations, the MDL is usually taken as three times the

standard deviation of the background (converted to elemental concentration by an appropriate calibration coefficient). This is equal to three times the square root of the background counts in the spectrum. Both the desired signal and the background are proportional to the source strength. The background arises from scatter of the continuum from an X-ray tube and the detector peak-to-background ratio.

Materials and Methods

Standard Reference Materials

Standard Reference Materials (SRMs) numbered 2709, 2710, 2711, 97B and 2702 from the National Institute of Standards and Technology were used for the characterization tests. 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 the National Institute of Standards and Technology 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 was chosen as representative of the Mars atmosphere. A gas mix of three volume percent carbon dioxide with helium making up the balance at Earth ambient pressure and gravity provided the same carbon dioxide density typical of Mars atmosphere. All measurements were made in this atmosphere.

Determining the Minimum Detection Limit (MDL)

A spectrum is collected of a known sample containing the element for which the MDL is desired. It is best to use a sample with a known element concentration less than 100 times the MDL. The largest peak from the element is found (usually K α or L α) and the background is determined by a linear fit to the spectrum on either side of the peak. To determine the total background counts the number of channels under the peak is multiplied by the average counts in the channels on either side of the peak. The gross peak counts are similarly determined by summing the counts in all channels under the peak. The net counts from the element are the gross counts in the peak minus the total background counts. Next the square root of the total background counts is multiplied by three, then multiplied by the ratio of the known element concentration to the net counts from the element. This yields the MDL in the same units as the known concentration. Note that this procedure assumes a linear relationship between net counts and concentration, which is a good assumption at low concentrations near the MDL. All MDLs given in this instrument performance report were calculated using this procedure.
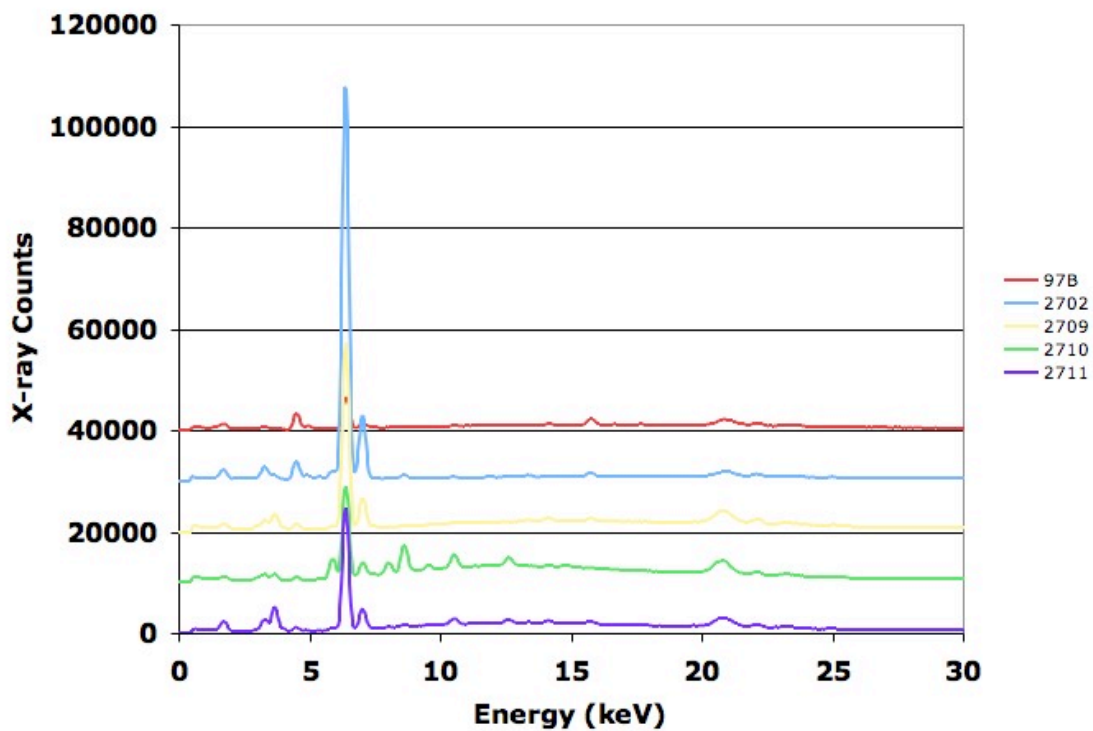
Figure 13. Raw spectra of five SRMs acquired with the borehole XRFS.

Test Plan Summary

- Determine the MDL for the elements Mg, Zr, Cd, and Pb

- Measure power consumption during spectrum collection

- Dry, water saturated, and frozen sample

- Variation with distance to probe (in case borehole diameter is not constant)

- Measurement stability vs. time

- Calibration linearity

Results

Figure 14 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 µA. No filters or other optics were 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 sec for the upper spectrum and 100 sec for the lower spectrum. Note that the majority of the information is still available even with the 100-sec 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.
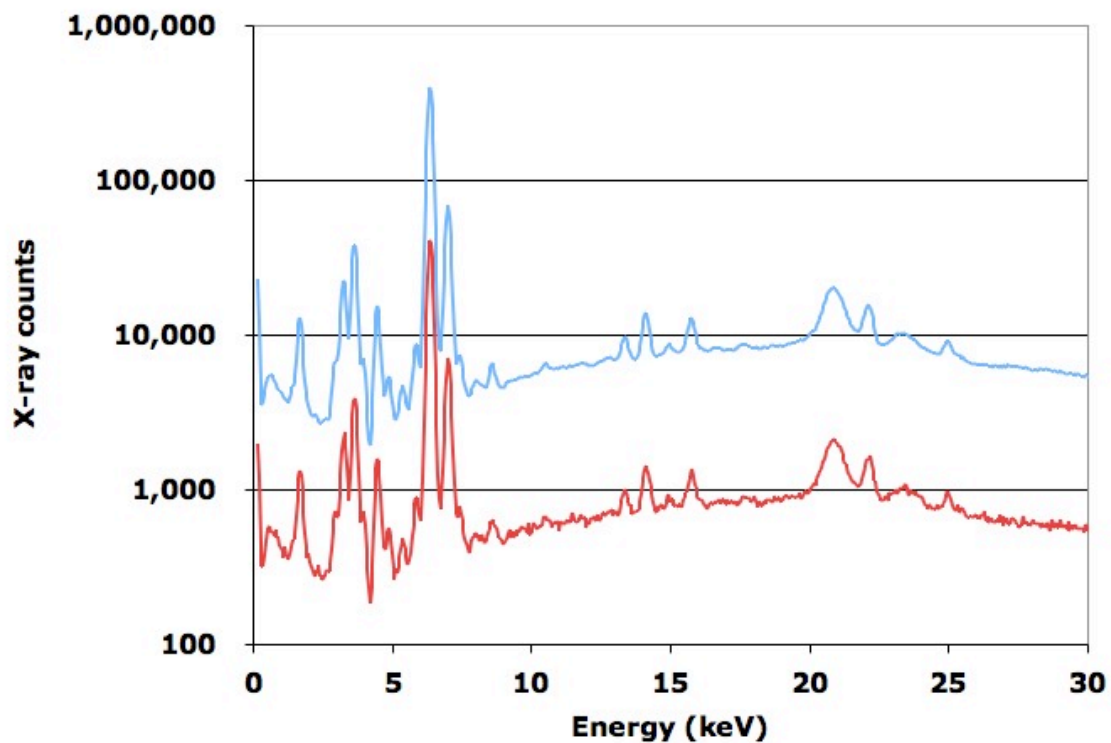

Figure 14. Spectra from borehole XRF spectrometer. Upper curve is 1000-sec data collection time, and lower curve is 100 sec.

As a comparison, the APXS (alpha proton X-ray spectrometer) spectra used on the Pathfinder and MER rovers have little usable data above the iron peaks at 6.4 keV (Figure 15). 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.
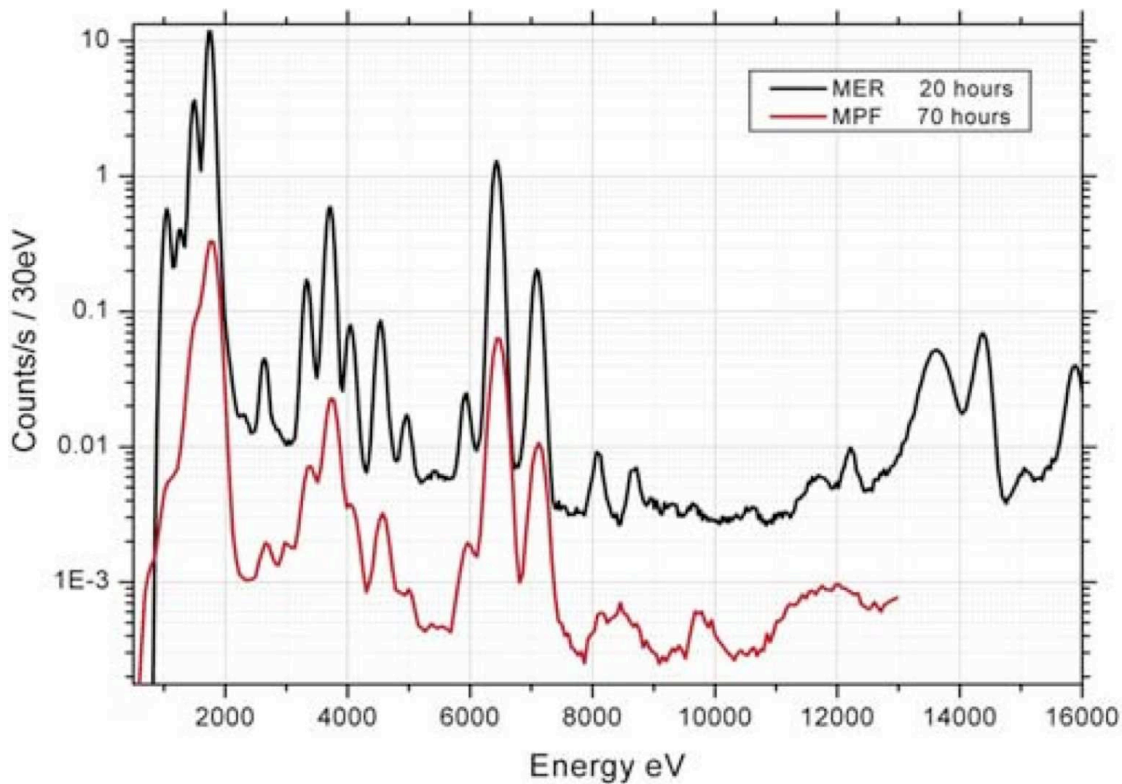

Figure 15. Spectra from the APXS instrument. Reproduced[3] with permission.

Detection limits for a number of elements in parts per million are presented in Table 2. 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

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[3] R. Reider, R. Gellert, J. Brückner, G. Klingelhölfer, G. Driebus, A. Yen, and S.W. Squyres, *J. Geophys. Res.,* **2003**, *108*, 8066–8078, DOI:10.1029/2003JE002150.

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, and including them avoids any bias toward lower values.

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 (Table 2). The ability to change the excitation conditions is another strong argument for using an X-ray tube.

Measured power consumption is given in Table 3 for the system components and the total. Ground support components including the 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 kJ per spectrum for a 1000-sec spectrum or 1200 J for a 100-sec spectrum. This is comparable to the APXS energy per spectrum, with larger power consumption but shorter collection times.

Table 2. Minimum detection limits for several elements

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

Table 3. 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	4.46 W
	-14.92 V	0.149A	
Detector	+4.99V	0.240A	1.20 W
Total			**11.97 W**

The effect on the measured spectrum from the presence of water is shown in Figure 16, where spectra from dry, water saturated, and frozen specimens of SRM 2702 are overlaid. There is almost no change in peak intensities, which is expected and indicates that good quantitative information can be obtained regardless of water content. Also, the presence of water will cause no significant degradation of detection limits. The region of the spectrum that has peaks from coherent (Rayleigh) and incoherent (Compon) scatter from the characteristic emission lines of the silver X-ray tube is shown in more detail in Figure 17. Note that the scatter is much larger in the saturated and frozen specimens. This increased scatter indicates presence of water and can be used to quantify the amount of water present.
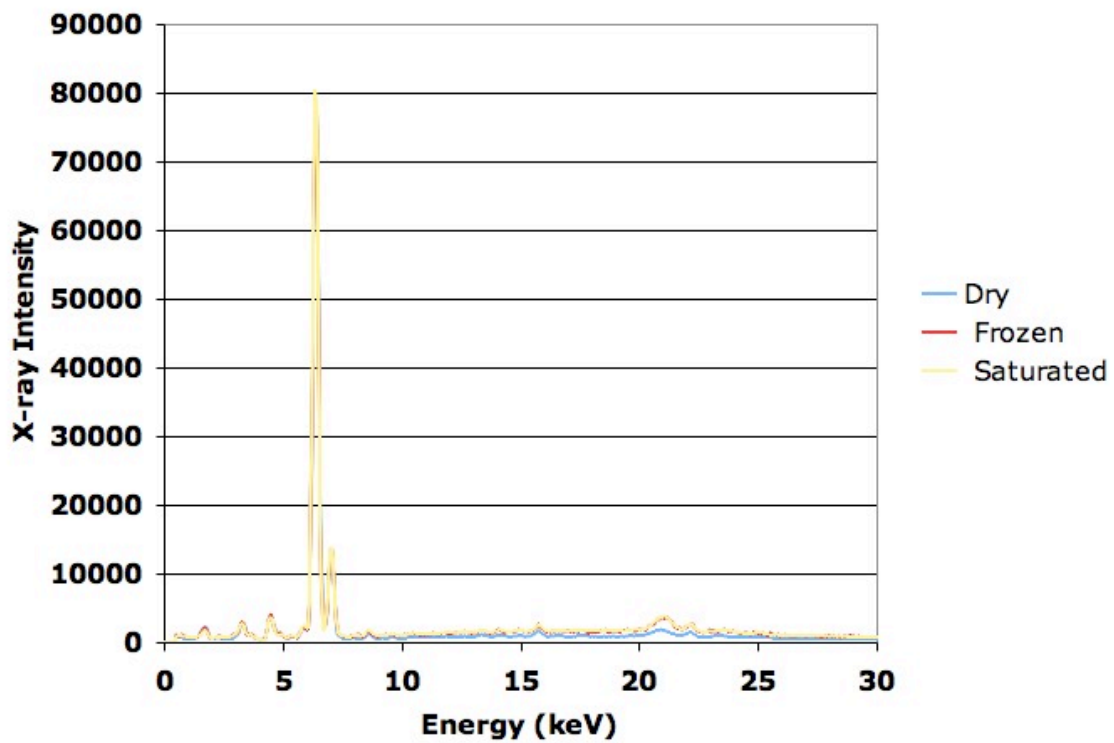
Figure 16. Spectra of dry, water saturated, and frozen samples of SRM 2702. (Frozen spectrum is 100 seconds to avoid thawing. It is multiplied by 10 for comparison.)
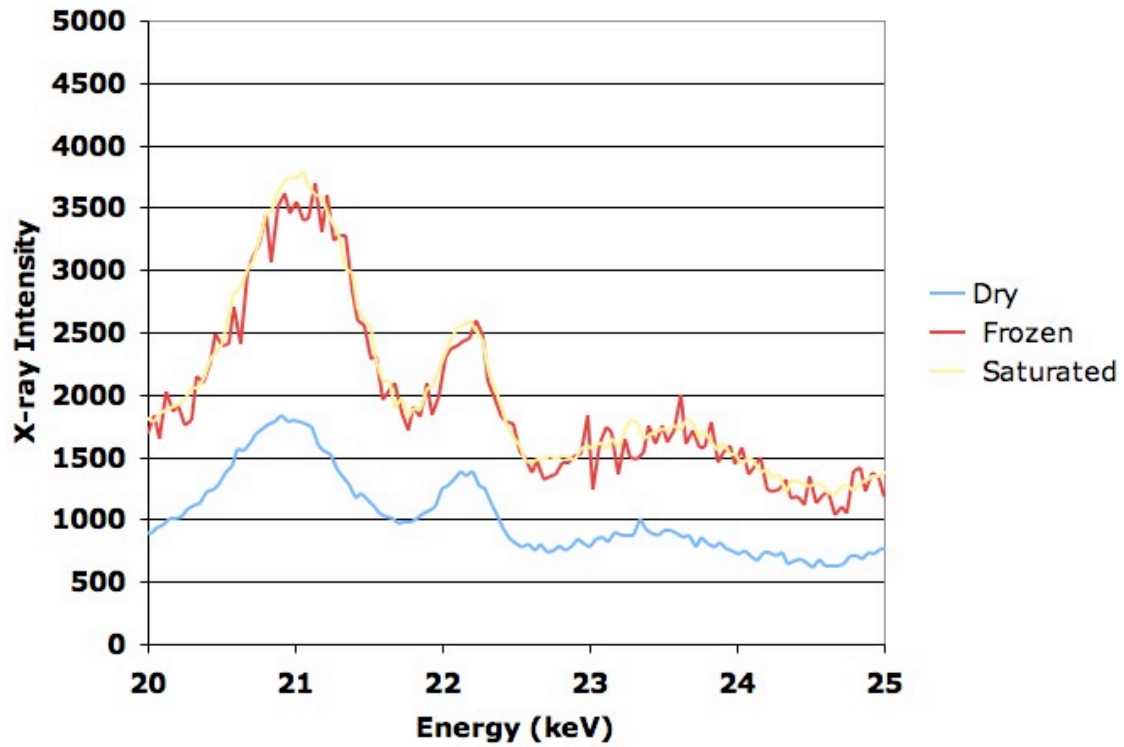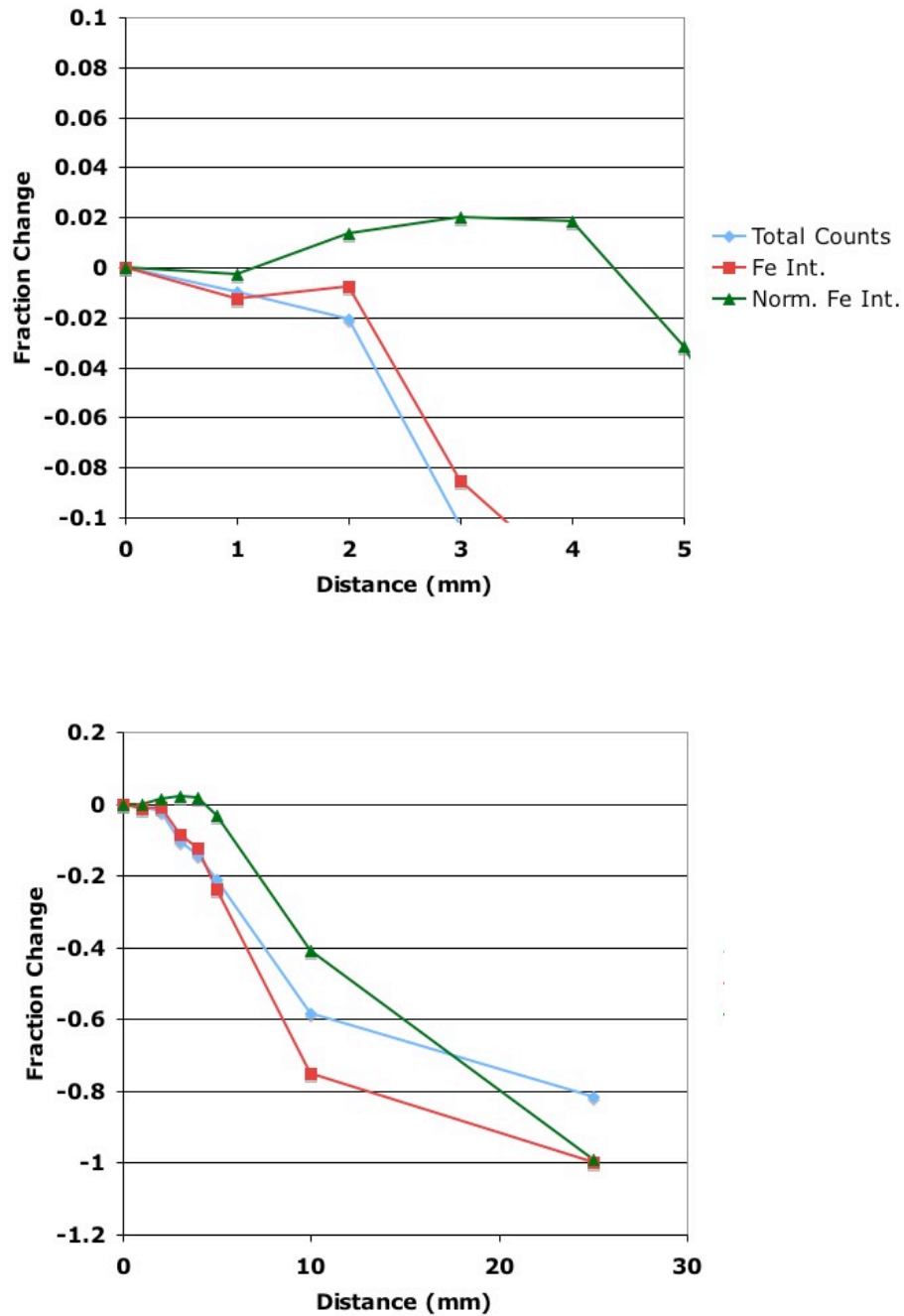
Figure 17. Spectra of dry, water saturated, and frozen samples of SRM 2702. (Frozen spectrum
is 100 seconds to avoid thawing. It is multiplied by 10 for comparison.)

Figure 18. Variation in fractional change of total spectrum counts and iron intensity with distance to probe. Tests conducted with SRM 2711. Note different scales.

The results of varying the distance from the probe to the sample are given in Figure 18. The intensity in the iron peak and the total counts in the spectrum are plotted as a function of separation between the probe body and the sample surface. The plot on the left shows the behavior in the first few millimeters and the plot on the right shows all of the data taken for this test. Note that both of these measurements are stable to within 2% for as much as 2 mm of separation. In addition, the normalized iron intensity, which is the ratio of the iron peak to the total counts, is plotted as the green line. This quantity is stable out to almost 5 mm, indicating that accurate quantitation can be performed even at this distance. This is important since the diameter of the borehole may not be constant and thus the distance between the probe and the regolith being measured may vary. Because of the design, these expected variations will not affect the results of they are less than 2 mm and can be compensated for out to 5 mm. Beyond 10 mm the spectrum is no longer a reliable measurement of the sample.
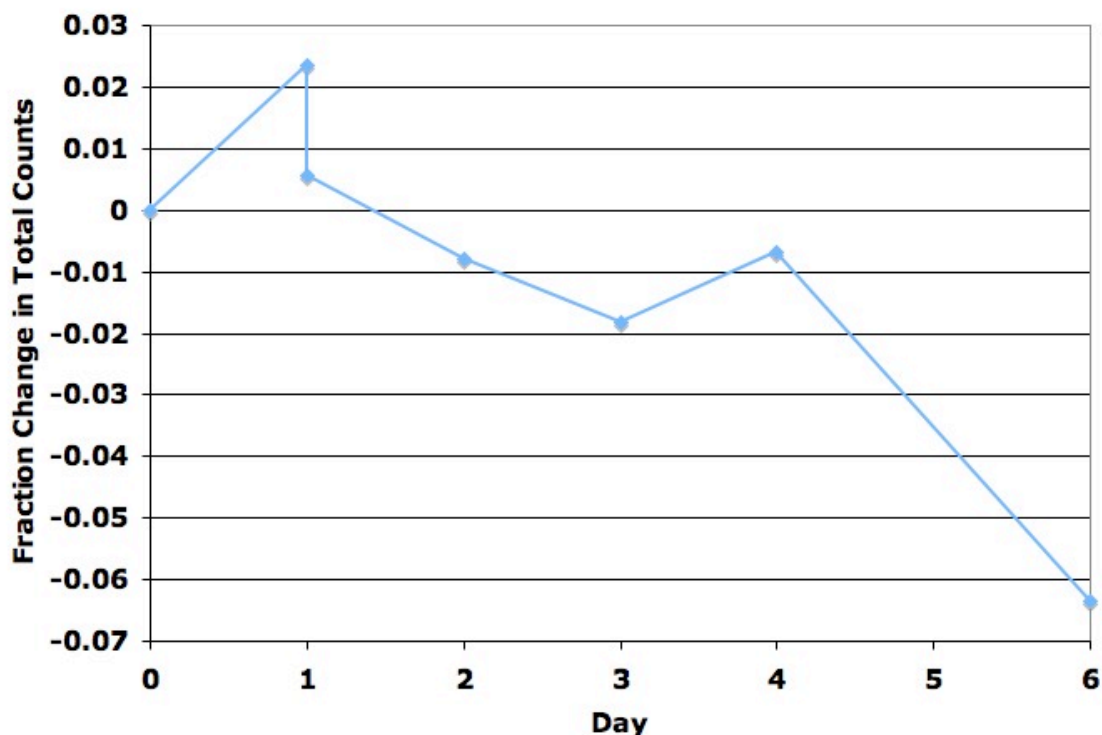
Figure 19. Variation in total spectrum counts over one week. (Point on Day 2 is after instrument was in continuous operation for 4 hr.)

Results of the measurement stability test are given in Figure 19. Stability is about 2% except for the final point. It is now known why this point is an outlier. The two points on day 1 were taken when the instrument was first powered on and after several hours of operation.

The calibration linearity was checked by plotting the composition measured by the instrument against the certified composition for all elements in all of the SRMs (below 10 weight percent) (Figure 20). Except for two outliers and several false positives (the points above the line near zero composition), the calibration is very good. The analysis algorithm used here is a "standardless" algorithm that relies entirely on the fundamental

parameters method to obtain the weight percent from the intensities in the spectrum. No standards were used in calculating these results. This is an advanced method that is not as good as careful use of type-specific standards, but was incorporated into the probe software because standards that are similar to the planetary regolith may not be available, especially if the subsurface regolith composition is unknown. Further work on the fundamental parameters analysis algorithm should improve the calibration performance. For the best results, appropriate standards with certified compositions can be used with an empirical correction algorithm.
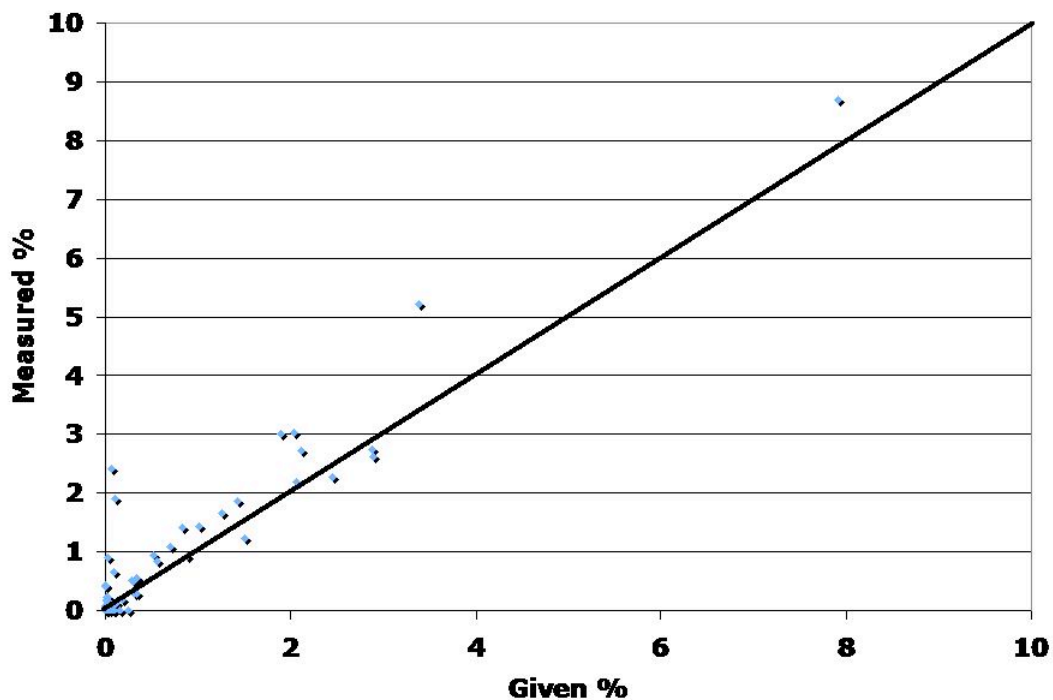

Figure 20. Measured vs. given composition for a wide range of elements in all five standard reference materials.

Conclusions

A borehole X-ray fluorescence spectrometer (XRFS) has been successfully constructed and tested. Miniaturization has been performed to a diameter of 27.1 mm 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 previous planetary inorganic analysis instruments. Adequate data can be collected in 100 sec, facilitating investigation of strata with vertical resolution of about 1 cm in a reasonable time.

Appendices

All appendices are available on the CD-R that accompanies this report.

Appendix A. **Digital Pulse Processor: User's Guide and Operating Instructions**

Appendix B. **X-ray Tube Product Documentation**

Appendix C. **Detector specification sheet**

Appendix D. **Borehole XRFS wiring diagrams**

Appendix E. **Borehole XRFS safety interlock/control board schematic**

Appendix F. **Borehole XRFS HVPS power and control board schematic**

Appendix G. **Borehole XRFS head unit and umbilical cable schematics**

Appendix H. **Borehole XRFS safety interlock/control board layout**

Appendix I. **Borehole XRFS HVPS power and control board layout**

Appendix J. **Bill of materials for safety interlock control**

Appendix K. **Bill of materials for HVPS power and control board**

Appendix L. **Borehole XRFS detector interface board schematic**

Appendix M. **Borehole XRFS detector interface board layout**

Appendix N. **Borehole XRFS safety controller software program by Peter Sabin**

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13. ABSTRACT *(Maximum 200 words)*

The X-ray fluorescence spectrometer (XRFS) is designed to be deployed down a pre-drilled hole for exploration and elemental analysis of subsurface planetary regolith. The spectrometer excites atoms in the regolith and causes them to emit their characteristic X-rays. These characteristic X-rays produce peaks in the X-ray spectrum. By measuring the energy of the X-rays, elements are identified. By measuring the intensity of the peaks, the amount of each element can be determined. A software package operates the spectrometer, acquires the data, and analyzes the spectrum to provide elements and their weight fractions. It also provides a user interface to control the measurements and display the results.

The spectrometer consists of two main subsystems packaged in three physical units. The main subsystems are the X-ray source and the energy-dispersive X-ray detector. The source provides the X-rays to excite the specimen of regolith being investigated. The energy-dispersive X-ray detector detects the emitted X-rays, determines their energy (the energy-dispersive function), and counts the X-rays at each energy. Together these two subsystems measure the X-ray spectrum of the specimen.

14. SUBJECT TERMS

Mars, spectrometry, regolith, X-ray fluorescence, XRF, elemental analysis, inorganic analysis, borehole

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