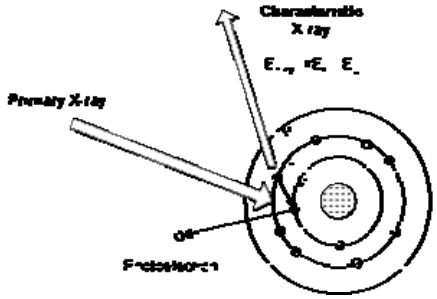
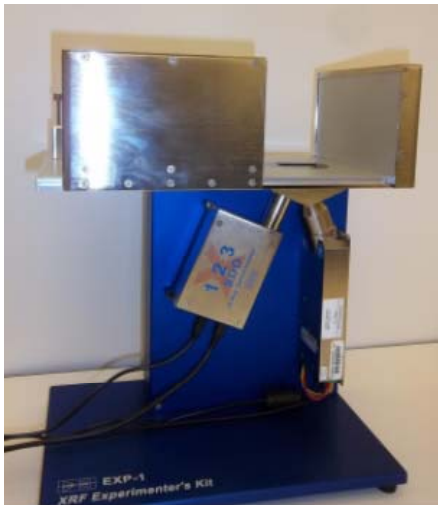
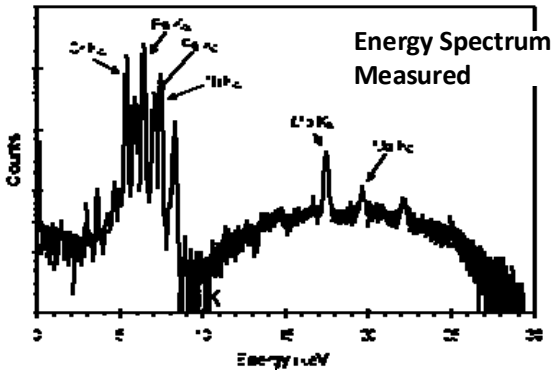
Amptek XRF Kit

Application Notes



Characteristic X-rays produced





Energy Spectrum Measured

Analysis Software Processes Spectrum

Analysis Results

	Intensity (cps)	Concentration (%)
Cr	419.8 ± 7.9	19.7 ± 0.4
Mn	24.0 ± 2.4	1.0 ± 0.1
Fe	1124.5 ± 12.9	63.0 ± 0.7
Co	3.9 ± 1.4	0.2 ± 0.1
Ni	168.6 ± 5.0	12.5 ± 0.4
Cu	2.9 ± 0.9	0.2 ± 0.0
Mo	479.6 ± 8.5	3.6 ± 0.1

Amptek, Inc
14 DeAngelo Dr, Bedford MA 01730
781-275-2242 x 132
www.amptek.com

1 WARNINGS AND PRECAUTIONS

These warnings and precautions MUST be read prior to installation and first operation. Serious hazards exist in this equipment, some which are potentially LIFE THREATENING. Only qualified and experience personnal should operate, maintain, or repair this system.

X-RAY SOURCES GENERATES X-RAY RADIATION DURING NORMAL OPERATION AND PRESENT A SAFETY HAZARD.

The EXP-1 fixture includes several safety features, which must be in place for safe operation:

- Radiation shielding reduces the dose outside the enclosure.
- Interlocks prevent the Mini-X from operating with shielding open.
- Warning lights and a beeper warn when the Mini-X is generating X-rays.
- See section 3 of this document for more important information on radiation safety.

IT IS VITAL THAT THE EXP-1 BE ASSEMBLED CORRECTLY AND THAT THE USER NOT DISABLE OR TAMPER WITH THE SAFETY FEATURES OF THE EXP-1.

HIGH VOLTAGES ARE PRESENT IN THE MINI-X AND THE X-123.

The Mini-X voltage (up to 50 kV) is potentially life threatening.

THE DETECTOR AND X-RAY TUBE BOTH CONTAIN THIN, FRAGILE BERYLLIUM WINDOWS. If the windows are damaged, the units will be destroyed and cannot be repaired. Do not touch the windows! Do not drop anything on the windows!

DO NOT DROP OR CAUSE MECHANICAL SHOCK TO THE X-123. Components inside the AXR are mechanically fragile and may be damaged if the unit is dropped.

- The detector window is made from thin beryllium (0.0005 in / 12 µm thick) which is extremely brittle and can shatter very easily. Do not permit any object to come in contact with the window. Do not touch the window because the oil from your fingers will cause it to oxidize. The window cannot be repaired. If the window shatters, the detector assembly must be replaced.
- In the EXP-1, it is possible to drop a sample or tools through the holder and shatter the detector window. Be very careful when positioning the sample. We recommend covering the holder with a thin, protective membrane (discussed below).
- BERYLLIUM WINDOWS DAMAGED BY IMPROPER HANDLING WILL NOT BE COVERED BY THE WARRANTY.
- RADIATION DAMAGE to the detector will occur if it is exposed to a high flux environment. Damage to the detector will be permanent if the flux from an X-Ray Tube, a strong nuclear radiation source, or an accelerator is not attenuated. A RADIATION DAMAGED DETECTOR WILL NOT BE COVERED UNDER WARRANTY.
- No operator serviceable parts are inside the X-123 or the Mini-X. Refer servicing to Amptek, Inc. To prevent electrical, shock do not remove covers.
- For the latest information about this analyzer, including firmware upgrades, application software upgrades, application information, and product information, go to

  http://www.amptek.com

2 EXP-1 FIXTURE DESCRIPTION

2.1 OVERVIEW

The Experimenter's Kit combines three separate, standard Amptek products and a unique mechanical fixture. The components are:

Amptek's X-123 spectrometer (which includes a detector and the DP5 digital signal processor). This has its own acquisition and control software, DPPMCA.EXE.

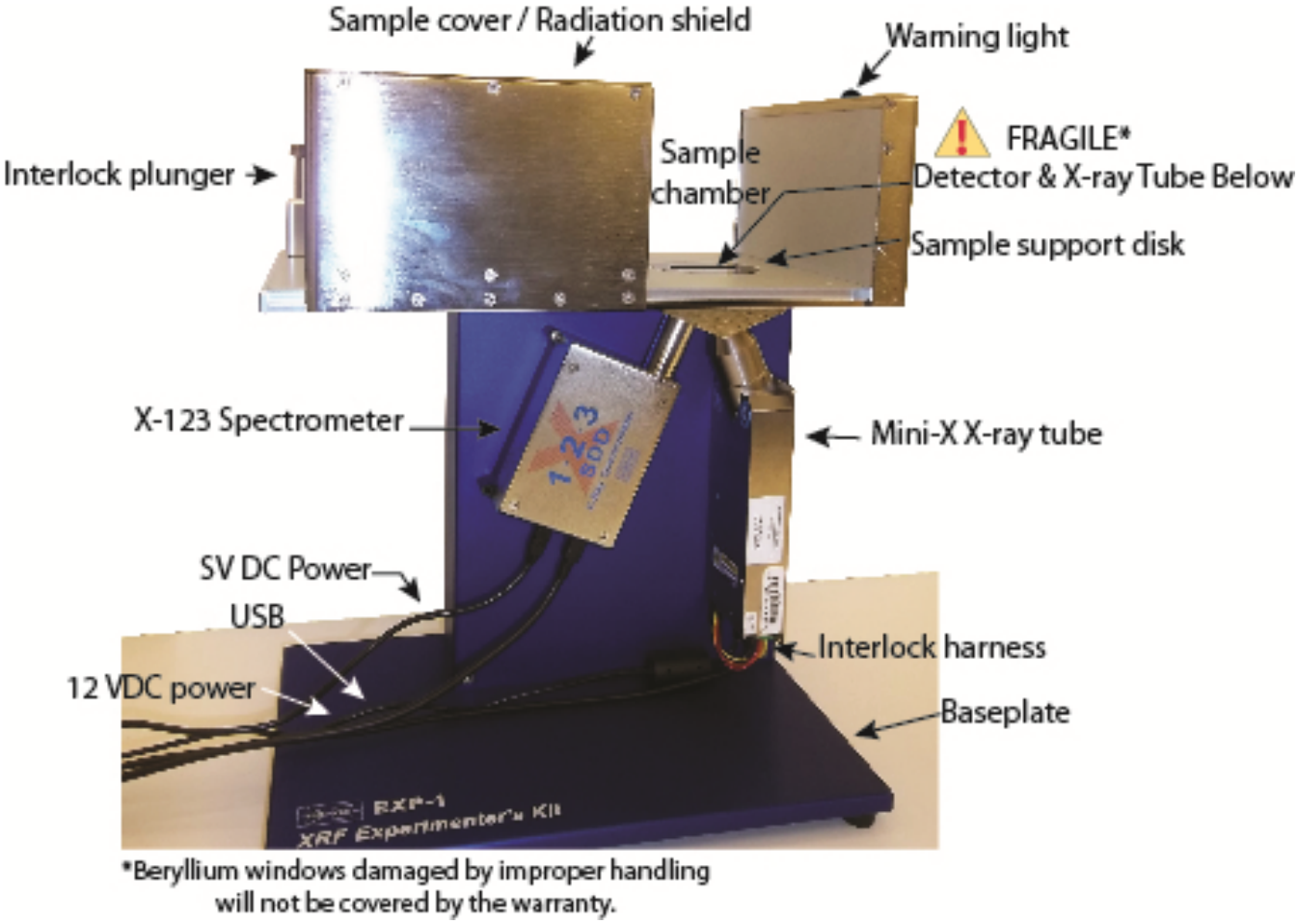
Amptek's Mini-X X-ray tube (which includes a collimator with a place to mount filters). This has its own acquisition and control software, Mini-X.EXE.

Amptek's XRS-FP X-ray analysis software. Its primary purpose to is to analyze the spectra, i.e. to do quantitative analysis of elemental composition from the spectra. It can also do control and acquire data from the Mini-X and the X-123

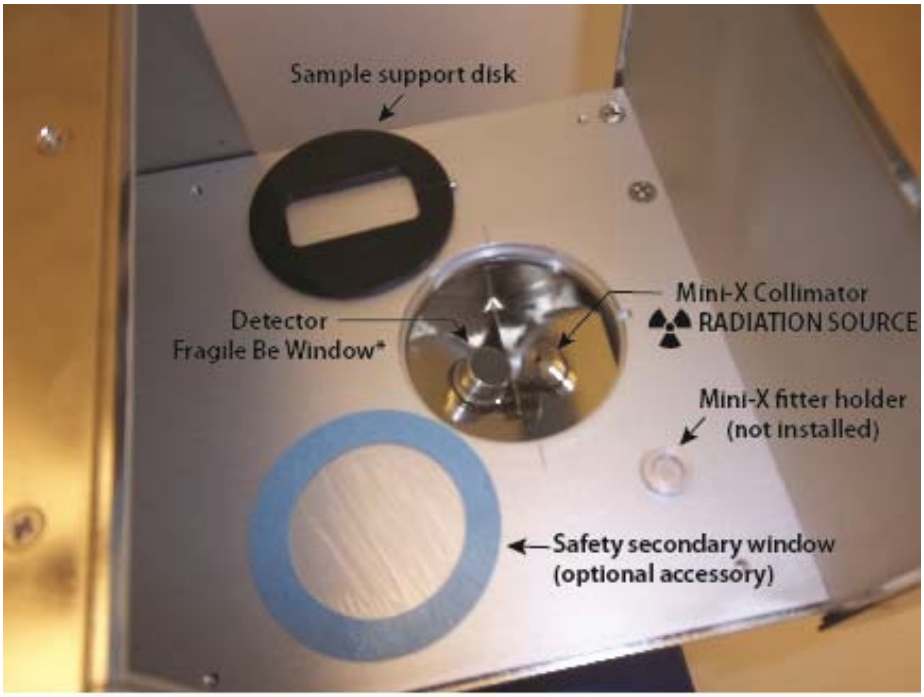
The EXP-1 mechanical fixture which holds the first two items in place and provides radiation shielding, safety interlocks, and a sample chamber.

Mechanical Components

The photo illustrates the major mechanical components in the EXP-1. These include (a) the stand and baseplate, (b) the sample chamber with shielding, (c) the X-123 spectrometer, and (d) the Mini-X X-ray tube. Both the X-123 and the Mini-X include an AC/DC power supply and a USB cable to the computer running the data acquisition software and the analysis software.



The figure below shows the sample chamber and its main components. At the bottom of the sample chamber is a hole, 2.25" in diameter; below this is located the detector and the X-ray tube collimator. In normal operation, the plastic sample support disk is placed onto the shelf in this hole and the sample placed on the support disk.



*Beryllium windows damaged by improper handling will not be covered by the warranty.

CAUTION: THE DETECTOR HAS A THIN (12 MICRON) BERYLLIUM WINDOW ON THE ENTRANCE APERTURE. IF YOU DROP ANYTHING ONTO THIS WINDOW, YOU WILL BREAK THE WINDOW AND DAMAGE THE DETECTOR. THE DETECTOR WILL NEED TO BE REPLACED AND REPLACEMENT FOR DAMAGE IS NOT COVERED BY WARRANTY.

Because it is important to avoid damaging or contaminating the window, we recommend placing a very thin polymer film across the opening. Such films are commercially available (sources and part numbers are given below). These films are inexpensive, so if one is damaged or contaminated, it can easily be replaced. For powdered or liquid samples, standard sample cups are also available. One can place the sample in the cup and then cover it with a thin polymer film. One source is

http://www.chemplex.com/spectrofilmr-self-sticking-safety-xrf-secondary-film

As shown in the included mechanical drawings, the sample chamber is a multilayer structure, made of brass (which provides the primary radiation shielding) and lined with aluminum (to attenuate the Cu and Zn K X-rays from brass). The top of the chamber is made of 1/4" brass, to stop the primary beam from the Mini-X. The sides and bottom of the chamber are made from 1/8" brass, to stop the scattered radiation. The aluminum lining is 1/16" thick. The attached drawings illustrate the tube to sample to detector geometry, when the EXP-1 is used with Amptek's X-123 and the Mini-X.

Experimenter's Kit Documentation

The X-123, Mini-X, and XRS-FP Analysis software in the Experimenter's Kit come with extensive, separate documentation (quick start guides, use manuals, software installation guides, and application notes). These can all be found on the Amptek Installation CD and Amptek's website. The DPPMCA software also has a help file with much information on the X-123 configuration parameters. Please refer to these documents to understand the individual components in the kit. The "Experimenter's Kit" documents focus primarily on assembling and initial operations of the kit rather than offering details on the various components.

3 SAFETY INTERLOCK AND ALARM

Radiation safety is an important consideration with the EXP-1. This section describes how radiation safety works WHEN USED WITH THE MINI-X. The customer must adapt the components in the EXP-1 to any other X-ray tube used. There are several key points:

- There are three interlock switches: one underneath the Mini-X (to make sure the tube is installed) and two on the plunger beside the sample chamber (to make sure the chamber is closed). Power to the X-ray tube flows through these switches; if any is open, the Mini-X will not produce X-rays.
- The plunger interlock has two switches in series, to provide a failsafe interlock. If either switch fails to close, the interlock still turns off the X-ray tube, assuring safety.
- When the interlock is opened, it resets the Mini-X circuits: it resets the logic and latches the bias and current to zero. After the interlock is closed, a software command must be sent to turn the Mini-X HVPS back on. Closing the hardware interlock alone will not restart the X-rays; two steps are required, first closing the switches and then sending the command. This prevents accidentally producing X-rays.
- There are several alarms, for redundancy. There is a flashing red lamp, on the top of the sample chamber, and an audible beeper, inside the Mini-X. There is also a flashing red LED on the bottom side of the Mini-X. Also, the Mini-X tab in the Windows application bar flashes when the Mini-X is used. When the Mini-X is enabled, via software command, all of these alarms are enabled.

Radiation Safety Standards

Amptek's complete Experimenter's Kit has been designed to meet the standards for an "enclosed beam x-ray system" defined by ANSI/HPS N43.2-2001, *Radiation Safety for X-ray Diffraction and Fluorescence Analysis Equipment*, and by *Radiation Safety Requirements for Analytical X-ray Equipment*, Part H of *Suggested State Regulations for Control of Radiation*, published by the Conference of Radiation Control Program Directors.

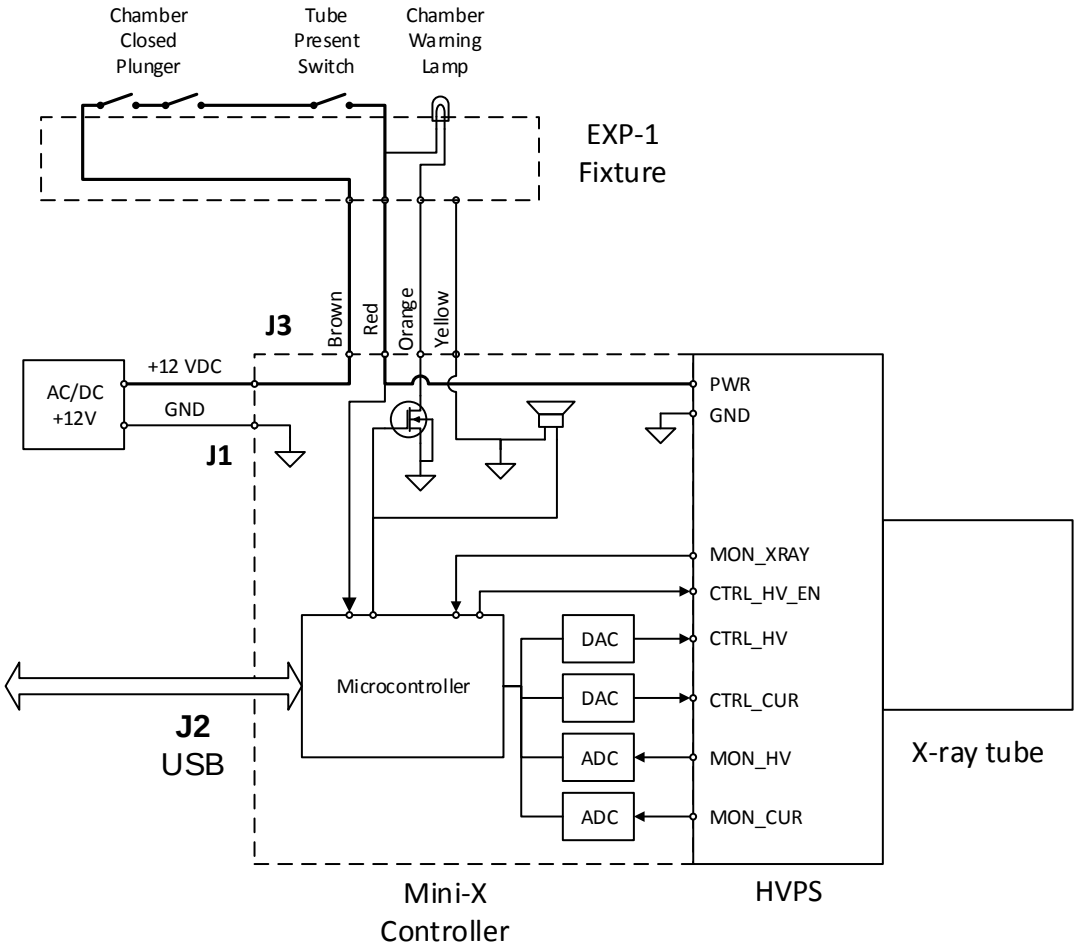
Note that your installation may be subject to federal, state and local regulations, administrative requirements, and other requirements that are different from those of these standards. The Radiation Safety Officer, the person in charge of the installation, and all equipment operators must be familiar with applicable operating procedures and regulations governing the x-ray installation. **The user is responsible for verifying that the Experimenter's Kit meets the standards of his/her institution.**

As a good resource for radiation safety, we recommend the following site:

http://www.ndt-ed.org/EducationResources/CommunityCollege/RadiationSafety/cc_rad-safety_index.htm

Detailed discussion

Because of the importance of radiation safety, the interlock and alarm system will be described in detail. As sketched below, the Experimenter's Kit includes several main components: (a) the X-ray tube, (b) a HV power supply (HVPS) (c) the control and monitor circuit, and (4) an interlock and lamp circuit in the EXP-1. The interface between the HVPS and the control circuit is critical to understanding the interlock and alarm system.



The interface to the HVPS includes the power (12 VDC), a digital control signal (CTRL_HV_EN), two analog control signals (CTRL_HV and CTRL_CUR), a digital monitor, and two analog monitors. For the HVPS to turn on, there must be power on the 12VDC pin, the control line must be enabled, and there must be valid voltages on the analog control lines. The HVPS then produces three outputs, read by the monitor circuit. Key issues for the safety interlock are the following:

1) The interlock carries the 12 VDC power supply current to the HV supply. If the interlock is opened, the HV supply is powered off so cannot operate. This is important because it guarantees that the HVPS will turn off when the interlock is opened: there is no other path to supply power to the HVPS.

   In the EXP-1, the 12 VDC current flows through two interlock switches, in series. One switch is at the "chamber closed plunger"; only if the sample chamber is closed and the plunger depressed will this switch close and enable the HVPS. The second switch is under the Mini-X and verifies than the Mini-X has been safely mounted. If either interlock switch is open, there is no power for the HVPS, so X-rays cannot be produced.

2) If the interlock is opened, the power input to the control logic generates a RESET signal, which resets the DACs used to control the HV and current. The DACs go to zero volts when reset and

latch the zero voltage. The ENABLE pin is also latched off. The Mini-X requires a command be sent over the USB interface to set these to a non-zero voltage.

This is important because this prevents the HVPS from turning back on as soon as power is restored. When the interlock is restored, power is restored to the HV supply but the control inputs are still at zero volts. A command must be sent after the interlock is restored to produce X-rays. This ensures a redundant sequence for turning on X-rays: a hardware switch must be closed and a software command must be issued.

The figure also shows how the alarm functions. When the controller brings the control line high, it activates a 1.6 Hz clock. This modulates the speaker, which produces a signal of approximately 5 kHz. The 1.6 Hz is also input to the FET, Q1, which is attached to the warning light and makes it flash.