X-Ray Fluorescence Spectroscopy Workflow

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About XRF

XRF is an elemental (or chemical) analysis technique in which the painted surface of the artifact is hit with an X-ray beam. The beam causes electronic transitions within the atoms of painted areas on the artifact, a phenomenon that can be measured and recorded as a spectrum. The spectrum is used to determine which elements are present in the paint (Figure 1).

Equipment

- Our laboratory's spectrometer is a Bruker Tracer 5g handheld XRF unit, and the following workflow is written for this spectrometer
    - Note: Similar handheld instruments include the Thermo Niton series and the Olympus Vanta—all can be made with acquisition parameters that are suitable for pigment analysis
- Oben tripod with Harris adapter—although the Tracer is designed to be handheld, we opted to attach it to a tripod to distance ourselves from the instrument while conducting the survey (see "A Note on Safety" below)
- Software interface for remote instrument operation—we prefer ARTAX software to other Bruker programs
- Dosimeter for each user

Helpful Hints

- Consider purchasing an XRF stand, platform, and lid if you plan to analyze small (coin-size) objects and a backing plate if you want to analyze materials on textiles

A Note on Safety

The workflows presented here are optimized for the equipment and processing techniques used at the Kelsey Museum. They are NOT a substitute for radiation and instrument safety training and monitoring, and users should closely follow the specific safety instructions provided with their XRF unit purchase or rental.

- XRF units produce X-rays that can cause biological radiation damage—it is important to follow the manufacturer's safety parameters and to monitor your exposure to radiation while using the instrument
- Review the Bruker X-Ray Radiation Safety Overview (or the safety document provided with your XRF unit—ask the manufacturer for one if it is not provided)
- User should wear a dosimeter badge and rings while instrument is on—these detect how much radiation is being produced in your workspace while using the XRF unit
- For the Bruker Tracer 5g:

- o We recommend operating the instrument remotely if conducting a technical survey or using the instrument over an extended period to reduce cumulative exposure to biological radiation
- o Maintain two feet of distance from the instrument when the X-ray beam is on
- o If a distance of two feet is not possible, position yourself away from the beam (beam window is on the nose of the instrument, and the beam itself bends slightly upward as it leaves the instrument)
- o If operating with "Backscatter Check" disabled, the beam will turn on if the trigger is engaged, **even if the nose is not in contact with anything**

Start-Up

Software Interfaces

- ● Bruker offers different programs that allow for remote use of the instrument, including Remote Control, Instrument Toolbox, and ARTAX software—we've opted to use ARTAX because we're more familiar with this program than the others.
- ● You may need administrator access to install and access the software on your computer—contact your institution's IT department if needed

Powering Up the Instrument

1. Charge the battery pack (press PUSH to check power levels) and insert the battery by open the instrument at the base of the handle OR plug the instrument into the AC adapter and plug the adapter into a power source.
2. Press the power button to the upper left of the instrument's screen.
3. Using the instrument's screen, enter the security code provided by the manufacturer (or the person training you) to log in and unlock the instrument.

Connecting the Instrument to Your Computer

- ● When connecting the instrument to your computer for the first time:
  1. Plug the USB cable into the mini-USB port on the instrument and into any of the USB ports on your computer. Turn on the XRF unit. The instrument's device network should appear at the top of the list of networks detected by your computer—one of them will be your XRF unit.
  2. If the instrument's network is not showing up, it may be necessary to adjust your network settings.
     a. Note: At the Kelsey, we needed to change the instrument's network profile from Public to **Private**. Your computer's firewall settings might also be an issue. Contact your institution's IT department if you encounter more issues connecting—after some troubleshooting, our IT team got the instrument up and running.
  3. Log in to the instrument's screen with the security code provided by the manufacturer (or the person training you) and click OK. You can also interface

with the instrument's control panel via the Bruker Remote Control software (select "Connect" under "Device").

      a. Pull XRF unit's trigger once to advance to the instrument's control screen (this will not turn on the beam).

      b. Click the "Utilities" button → Settings → Connections → Configure Connections.

      c. In the second tab along the top, check the box next to "Enable USB."

      d. Click "Back." Make sure the "Enable USB" box is selected on that screen as well.

4. Open ARTAX and select "Connect" under "Device." The instrument's serial number should appear in the drop-down window. Select it and click "Connect." If the connection is good, a green band will appear above the spectrum graph.

- Connecting via Wireless—recommended, as this will allow greater distance between you and the instrument. *Note: At the Kelsey, we needed an external Wireless router to connect our computers to the instrument; typically, this is only needed in unstable Wi-Fi situations, such as remote fieldwork. But it goes to show how finicky connecting to the instrument over Wi-Fi can be.*

1. Click on the Wireless icon at the bottom right of your taskbar. A drop-down menu will appear near the top of your network list, and this will show your Wireless options.

2. Log in to the instrument's screen with the security code provided by the manufacturer (or the person training you) and click OK. Proceed to the screen with the "Connections" tabs (see steps 3. a., b., above).

      a. Deselect USB connection by unchecking the "Enable USB" box on the middle tab.

      b. Under the "Wireless" tab to the right, click the "Enable Wireless" box. A list of wireless networks will appear in the menu below. Connect the instrument to the Wireless network your computer is connected to.

      c. Click "Back." Check that the "Enable Wireless" box is selected.

3. Open ARTAX and select "Connect" under "Device." The instrument's serial number should appear in the drop-down window. Select it and click "Connect." If the connection is good, a green band will appear at above the spectrum graph.

Instrument Settings

1. In ARTAX, go to the "Measurement" tab and select "Method."

      a. At the Kelsey, we have used the following instrument settings for pigment characterization: 40 kV voltage, 5.50 μA current, 30 second run time, and no filter.

            i. If checking for aluminum, consider dropping the voltage and switching to the instrument's Ca filter (or insert Teflon filter manually). Note: You may need to raise the current if using a filter.

    ii.   If your substrate is wood or paper, consider raising your current to ~13 µA.

    iii.   Settings can be titled and saved in ARTAX for quick access.

    iv.   It may be necessary to turn off the "Backscatter Check" on the instrument, especially if you are looking at textiles or objects with irregular surfaces.

2. Select all options under "Corrections" tab. The "Parameter" tab will show your metadata.

Acquire, Document, and Interpret Spectra

1. Connect the XRF unit to the tripod using the Harris adapter—tilt adapter into insert on the tripod's ball head and push down until prong snaps in, then push prong further in to tighten. Position instrument over the sample area using tripod, and slowly lower the beam window onto the surface of the object—it should be completely flush with the surface (or as close to flush as possible). Use "Camera" tool on the instrument's screen to position the beam over small color traces.

    a.   Make sure to capture a spectrum from an unpainted area as well to get a sense of what other elements might be on the surface.

    b.   Ideally, try to capture more than one spectrum per color area—this will increase the accuracy of your results.

2. Open the documentation photo of the object in Photoshop. Each time you acquire a spectrum, mark the sample's location on the documentation photo of the object. Use text in a highly visible color to label each sample location with a letter: a, b, c, etc. When sample acquisition is complete, you will save this image.

3. Open a Word or PowerPoint document and change orientation to landscape view. This is where you will copy and paste images of your spectra; you will also save this document once sample acquisition is complete.

4. Click "Play" button on the XRF unit to acquire a spectrum. Radiation and Beam icons will appear when the instrument is firing.
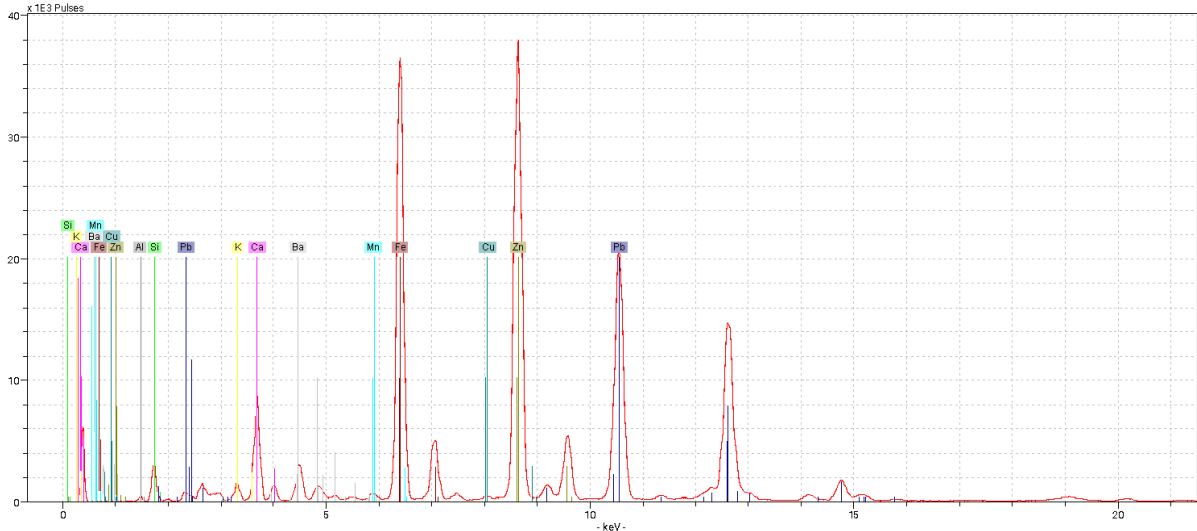
Figure 1. Sample XRF spectrum generated using the Bruker ARTAX program

5. Use Magnifying Loop icon to select a specific area of the spectrum to analyze. Click on the upper left corner of the spectrum (inside the y-axis) and drag the box down to the right to ~ 20 keV. Increase peak heights by clicking to the left of the y-axis and dragging up.
6. Select Peak Bar icon for periodic table. Right click on unknown peaks to identify element present. Click on elements in periodic table to label peaks.
7. If desired, check your results using the Deconvolution Spectrum tool (to the right of the Peak Bar icon).
   a. Note: Elements **Ar, Ni, Ra, Rh, and Mo** are part of the machine itself and not from the object. We opt not to label them for this reason.

XRF Data Management
1. Under File, select "Save Spectrum As…" and save the file with the naming convention and in the file structure you have established for your project.
   a. When naming files, we use the object's accession number, the letter of the sample area, and a brief description of the sample area. For example, a file name might be "0000.00.3601_a_spot of red on hair."
2. Under Export, select "Spectrum" and save Text file in the same folder.
3. Under Export, selection "Copy Graphic." Paste spectrum into Word or PowerPoint document and write in a caption using the naming convention you have established (for the Kelsey, this is the same as our spectrum file name). Save the Word doc using the naming convention for your project.
4. Return to the documentation photograph of the object, which is now marked with your sample areas. Add an overall text label that reads "XRF sample areas a-x." Merge the image's layers and save this file using the naming format for your project. We use the object's accession number and the text "XRF Sample areas."