

Adding Geochemistry to the IRM Toolkit: Acquisition of a Portable X-ray Fluorescence Spectrometer

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Ellery Frahm using the IRM's new portable X-ray fluorescence instrument at an archaeological site in Armenia.

The IRM continues to add to the analytical techniques and instruments available to visiting fellows and other researchers: the latest addition is the non-destructive elemental analysis by portable X-ray fluorescence spectrometry (pXRF). Much of the research at the IRM aims to quantify the composition, concentration, and grain size distribution of Fe-bearing minerals in natural and synthetic samples. Whether we measure the rock magnetic properties of igneous, sedimentary, metamorphic, or even extraterrestrial environments, complementary geochemical information about our specimens can often improve the accuracy of our geophysical interpretations. Recent improvements in the calibration, safety, and sensitivity of pXRF devices has led to a rapid expansion of their use in a number of fields, including sedimentology and stratigraphy (Funk et al., 2004a,b; Lami et al., 2004; Luterbacher et al., 2004; Rohl et al., 2004; Meijers et al., 2016), economic geology (Gazley et al., 2015), soil science (Martin Peinado et al., 2010), environmental remediation (Jamieson et al., 2015), and archaeology and anthropology (Frahm et al., 2014a,b, 2016). In short, there are many innovative ways in which this instrument can serve future visitors to the IRM.

The pXRF is a hand-held instrument the size of a small portable drill that allows one to identify and quantify approximately 50 elements ranging in atomic weight

from Mg to U. Analyses can be conducted in the field, with data being digitally recorded and geo-referenced instantaneously, or in the quiet and coziness of IRM where the pXRF can be conveniently rested on a stand, making measurements more systematic.

Motivation to acquire the pXRF arose from the research of Dr. Ellery Frahm during his postdoctoral work at the IRM. Ellery characterizes obsidian artifacts from archaeological sites and volcanic terranes and has made pXRF a fundamental component of his research (e.g., Frahm et al., 2014a,b, 2016; Adler et al., 2014).

How it Works

When a material is exposed to high-energy (short-wavelength) X-rays, a portion of its constituent atoms may ionize (i.e., one or more orbital electrons are ejected from an atom). Sufficiently energetic X-rays can trigger the ejection of electrons from inner orbitals: when an electron from a higher orbital “falls” into the vacancy left in the inner orbital, it will emit a photon that lies in the X-ray portion of the electromagnetic spectrum and is characteristic of the element involved. These photons can be sorted by either their energies (which is termed energy-dispersive XRF or ED-XRF) or their wavelengths (wavelength-dispersive XRF or WD-XRF) and allow the identification of the elements present. In each

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Visiting Fellow Report

Mudrock fabric using high field magnetic anisotropy

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Rock fabrics are fundamental in earth sciences as they determine numerous physical properties such as seismic anisotropy, fracture development, permeability and fluid circulation, and therefore understanding and characterizing rock fabrics is critical in geosciences.

The anisotropy of magnetic susceptibility (AMS) has been established in the past thirty years as the most versatile, sensitive, and rapid proxy for rock fabrics characterization. Standard measurements of the magnetic anisotropy involve the low-field AMS, which responds to the bulk of the mineralogy of the rock sample, including the paramagnetic, ferromagnetic, and diamagnetic fractions. Separation methods of para- and ferromagnetic anisotropies (K_{para} and K_{ferr}) have progressively gained interest over the last thirty years, including mathematical and instrumental approaches (e.g., Martín-Hernández and Ferré, 2007), and basically they involve the use of either a torque magnetometer or a vibrating sample magnetometer (VSM). The so-called anisotropy of high field magnetic susceptibility (AHFMS, Bilardello and Jackson, 2014) using a Vibrating Sample Magnetometer (VSM) is a very powerful tool to discriminate K_{ferr} from K_{para} tensors. Nevertheless, the method has almost exclusively been used in crystalline, highly magnetic or anisotropic rocks (e.g., granulites- Bilardello and Jackson, 2014; migmatites- Ferré et al., 2004, metagreywackes and slates- Kelso et al., 2002; peridotites- Martín-Hernández and Ferré, 2007). Much less is known as far as the AHFMS in weakly deformed sedimentary rocks with feeble magnetic anisotropy (<5%). Can we also separate the K_{para} from K_{ferr} tensors in these rocks where both bulk susceptibility and degree of anisotropy are much lower?

Fabric separation could help to elucidate the deformation mechanism that produces magnetic tectonic fabric in mudrocks: Because phyllosilicates (K_{para}) appear as platy grains whereas magnetite (K_{ferr}) typically as euhedral, it is critical to separately characterize their preferred orientation and degree of alignment, due to their contrasting rheology upon stress. Intrigued by the role of paramagnetic versus ferromagnetic components in these weakly deformed rocks, we aimed at extending the application of AHFMS to mudrocks that according to the standard AMS have embryonic deformation.

The method for subfabric separation based on high-field measurement with a VSM was developed by Ferré

et al. (2000) and Kelso et al. (2002). The principle of VSM analysis is based on the flux change in the pick-up coil system produced by vibrating a sample (rather than by rotation, as in spinner magnetometers). Although primarily designed for the measurement of hysteresis properties, there have been successful attempts to use VSM's to obtain directional hysteresis curves along different orientations of the specimen, hence enabling to the calculation of the high field magnetic anisotropy, which is the purpose of our study.

Some important issues to consider when determining the AHFMS with a VSM include: (1) the thermal equilibrium of the sample with the pole gap space is critical; (2) sample should be centered within the pole gap; (3) cubic specimens seem to produce better results than standard cylinders.

We selected a number of mudrock and fine grain sandstone specimens (1.1 cm³) to measure with the Princeton Measurements Micromag VSM at the IRM. The cubic specimens were carefully well centered within the pole gap- critical for the measurements, and the plastic sample holder was modified by Peat Solheid (IRM) during the visit to reduce the background, diamagnetic noise. The configuration for the measurements was: (1) 1 T maximum field; (2) 0.2 s averaging time for each measurement; (3) 5 mT field increment between measurements; (4) 30° rotation increment between loops. We therefore measured a total of 36 hysteresis curves per specimen (e.g., Bilardello and Jackson, 2014) as opposed to 24 (e.g., Ferré et al., 2004), which largely improve results. Each hysteresis loop (12 per axis) was repeated three times on some pilot samples, although the majority of them was measured once, dramatically reducing the total measuring time to about one hour and a half per specimen. Special care was taken in leaving the specimens within the pole gap for about 20 minutes previous to the measurements, for a uniform temperature (e.g., Kelso et al., 2002).

Figure 1 shows the results for some Eocene mudrocks from the Southern Pyrenean Foreland Basin. Anisotropy of Magnetic Susceptibility was measured on a MFK1-FA, with a field of 200 Am⁻¹, at an operating frequency of 976 Hz. Magnetic ellipsoids are oblate (average $T = 0.66$), and with an average anisotropy degree of 1.044. Principal axes distribution reveals a subvertical K_{min} , which is perpendicular to bedding. K_{max} axes are thought to reflect the intersection between the E-W direction flattening plane and sub-horizontal (<5°) bedding. Such intermediate fabric has been widely observed in the Pyrenean foreland basin and in active accretionary prisms (Parés, 2015). The anisotropy of high magnetic susceptibility (AHFMS) was calculated from the high-field slopes obtained in the Micromag VSM. The degree of the high field anisotropy is 10% ($P_j=1.10$ on average), and the axes distribution is somewhat more intriguing than those of the AMS: Whereas the minimum axes of the AHFMS tend to be sub-vertical and hence around the pole to bedding, the maximum axes are scattered within bedding, almost defining a mean magnetic lineation about a NNE-SSW direction. Comparison of both AMS

and AHFMS axes distribution suggests switching of the maximum and intermediate axes of AHFMS (and of low DC field susceptibility, ADCMS). AHFMS axes permutation relative to those of AMS possibly reflects that the former suffers from incomplete saturation, which can lead to either inverted or misaligned fabrics (e.g., Bilardello and Jackson, 2014). Further investigation will be required to illuminate such axes switching.

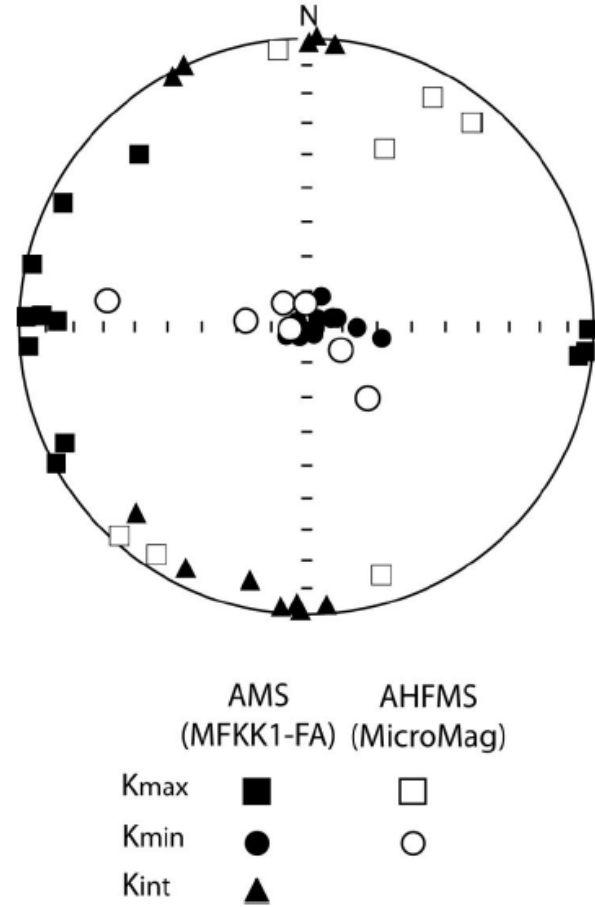


Figure 1. Figure 1: Magnetic fabrics measured for some Eocene mudrocks from the Pyrenean Foreland Basin. Closed symbols show the principal susceptibility axes obtained from the measurement of the low-field anisotropy of magnetic susceptibility (AMS). Open symbols are the axes (only Kmax and Kmin are represented) of the anisotropy of high field magnetic susceptibility (AHFMS)

Mike Jackson was very kind to add few modifications in “real time” to the “Princeton Ferret Software” to facilitate data evaluation and quality assessment, including the possibility to visualize the induced magnetization versus orientation (Fig. 2). The variation of magnetization as a function of orientation should have a π response, as every 7th hysteresis cycle is measured along the same axis. On the other hand, apparent anisotropy due to the shape effect of the measured cubes (e.g., Kelso et al., 2002) would have a $\pi/2$ term (i.e., along the corners), which is not observed in our samples. This result indicates that the sample shape effects are minimal and that the directional variability in magnetization mostly responds to the anisotropy of the sample.

Our pilot study showed that although much weaker than in crystalline rocks, the high field magnetic suscep-

tibility anisotropy can be obtained with the Micromag VSM in mudrocks and sandstones, although several aspects do need further research: (i) Comparing cylindrical versus cubic samples, (ii) reducing the mass of the holders, (iii) changing pole spacing to gain field uniformity within the pole gap of the magnetometer, (iv) origin of axes permutation.

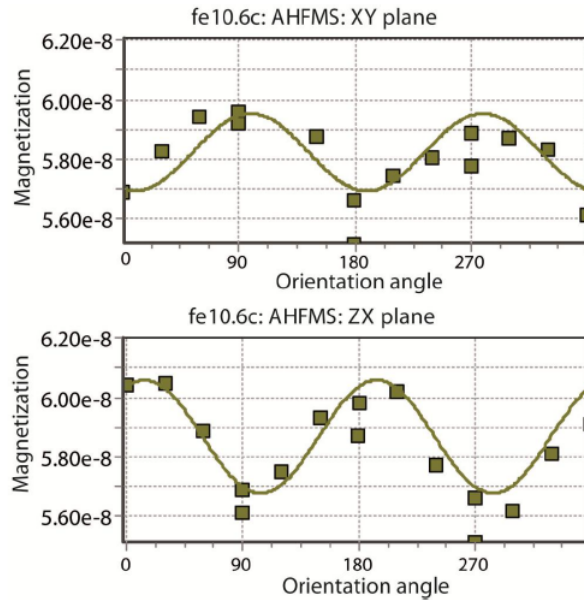


Figure 2. Example of the variation of magnetic moment with sample orientation (XY and ZX planes). Notice the π response of the magnetization (solid lines).

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Current Articles

A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most are taken from ISI Web of Knowledge, after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

Archeomagnetism

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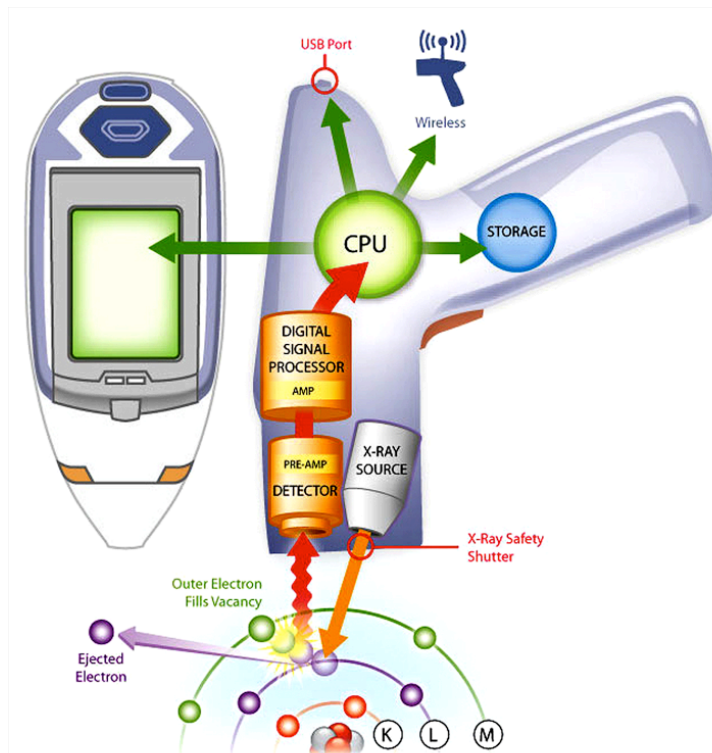
case, the number of characteristic X-ray photons reveals the concentration of each element. Because this process involves inner-shell orbital electrons, XRF is suitable for identifying elements only, not isotopes or chemical bonds. The IRM's new instrument is an ED-pXRF with rapid measurements (30-60 s) that are entirely non-destructive, so no material is lost or damaged during the course of an analysis.

Features

Specifically, the IRM instrument is a Thermo Scientific Niton XL3t 950 GOLDD+ (Geometrically Optimized Large-Area Drift Detector) ED-XRF analyzer. The instrument is portable, but it is set up primarily for benchtop operation for IRM visitors. It has a miniaturized 50-kV, 2-W, silver-anode X-ray tube as an excitation source (not the radioactive isotopes found in many older models). The X-ray detector is the latest generation of silicon drift detector (SDD), which is up to ten times faster than older Si PN-diode detectors and up to three times faster than first-generation SDDs. The circular analytical window is 10 mm across, and the incident X-ray beam is about 8 mm in diameter. A built-in collimator can, if one chooses, limit the beam diameter to 3 mm, thereby reducing the analyzed area on a specimen from 50 to 7 mm² (an area 85% smaller); however, collimation should be used judiciously, as the number of incident X-rays is reduced commensurately, yielding higher measurement uncertainties and detection limits. A camera inside the instrument can aid in positioning small specimens in the collimated X-ray beam. While the pXRF is notably smaller than the laboratory-based XRF instruments that many of us are familiar with, equally important is the development and refinement of the instrument's data correction algorithms that turn X-ray intensities into element concentrations.

Modes of Operation

Our instrument has two principal analytical modes: Soils and Mining. The "Mining" mode was initially developed for exploration geologists to measure elements of interest to the mining industry in silicate rocks, whereas the "Soils" mode was originally intended for conducting environmental surveys of soil contaminants, where elements of interest might fall between their natural abundances in Earth's crust and the much higher concentrations encountered at contaminated sites. Thus, these modes differ in several important ways. For example, the pre-set list of measured elements differs between modes. The "Soils" mode measures the concentrations of 33 elements: Ag, As, Au, Ba, Ca, Cd, Co, Cr, Cs, Cu, Fe, Hg, K, Mn, Mo, Ni, Pb, Pd, Rb, S, Sb, Sc, Se, Sn, Sr, Te, Ti, Th, U, V, W, Zn, and Zr. The "Mining" mode measures 42 elements: Ag, Al, As, Au, Ba, Bi, Ca, Cd, Ce, Cl, Co, Cr, Cu, Fe, Hf, K, La, Mg, Mn, Mo, Nb, Nd, Ni, P, Pb, Pd, Pr, Rb, Re, S, Sb, Se, Si, Sn, Sr, Ta, Ti, V, W, Y, Zn, and Zr. Earlier XRF instruments required helium purging or vacuum pumps to analyze light elements ($Z < 17$), but this is no longer necessary with the "Min-



Schematic diagram of the p-XRF from Thermo-Scientific.

ing" mode and the latest generation of SDD technology. Different sets of elements are measured using a sequence of X-ray "filters" (up to four in "Mining" mode, up to three in "Soils" mode) that improve sensitivity for select portions of the energy spectrum. The instrument automatically changes these built-in filters during an analysis so that the low, middle, and high end of a specimen's X-ray spectrum is sequentially enhanced.

Conversion

Another difference between these two analytical modes involves the algorithms used to convert raw X-ray intensity measurements into element concentrations. The relationship between an element's characteristic X-ray emission intensity and its concentration within a material is not straightforward. Various phenomena, including the excitation of lighter elements by characteristic X-rays from heavier elements, occur within a material, altering the measured intensities. The two operational modes use different correction approaches to convert X-ray counts into first approximations of the elements' concentrations. The "Soils" mode uses a simpler "Compton normalization" approach. The lower the mean atomic number of a material, the greater the intensity of the incoherently scattered X-rays (the Compton peak). This relationship also applies, albeit to a lesser degree, to the coherently scattered X-rays (the Raleigh peak). The ratio of the two peaks (Compton/Raleigh) is proportional to a specimen's mean atomic number and, in turn, its average composition. Additionally, this correction approach uses the Compton and Raleigh peaks as a means to compensate for a specimen's morphology, texture, density, and other effects. In contrast, the "Mining" mode applies the more calculation-intensive "fundamental parameters

(FP)” approach. FP correction involves iterative corrections to the measured X-rays using a mathematical description of instrument conditions (e.g., X-ray tube emissions, detector efficiency) and instrument-independent parameters for each element (e.g., X-ray fluorescence intensities, absorption coefficients, absorption edges). These parameters are brought together in an algorithm that solves a range of nonlinear equations describing the relationships between measured X-ray intensities and accurate element concentrations.

Calibration

Both correction approaches also use responses to standards or reference materials as a means to calibrate the data and return quantitative results. The factory-set “internal” calibrations are intended to remain stable over long periods and accurate over a range of materials and elemental concentrations. For example, for environmental surveying, the calibration must account for materials with element concentrations which fall at the two extreme ends of the spectrum: loess deposits in which cadmium occurs near or below the detection limit may constitute one extreme, whereas contaminated river sediments that possess much higher concentrations may lie at the opposite end. The intention is that these “internal” calibrations can be used in various tasks and acquire suitable data. Applications such as obsidian characterization, in which trace elements and the overall compositions fall within narrow ranges, tend to reveal the limitations of factory-set calibrations. In such applications, analyses of geochemical standards or other reference materials are commonly needed to “fine tune” the factory-set calibrations using linear regression in order to yield the most accurate measurements. The IRM maintains an impressive suite of geochemical standards for granular and solid materials to aid with most geological applications.

Specimen Limitations

XRF is a largely surficial analytical technique, but it is important to note that X-rays from different elements escape a specimen from different depths. As a general rule, the greater the atomic number, the greater the depth of X-ray penetration in a specimen. The characteristic X-rays emitted from, for example, iron (6.4 keV) escape from the top few micrometers of the specimen surface, whereas zirconium X-rays (15.8 keV) can escape from a few hundred micrometers deep. Energetic barium X-rays escape from a centimeter or more deep, depending on the material. In thick, homogenous specimens, this difference in measurement depth has little effect. If specimens are thin, however, it can affect the spectrum in various ways, depending on the specimen thickness and elements of interest. Hence, for the best data correction, a specimen should be sufficiently thick to completely absorb the incident X-ray beam. For many rocks and minerals, only a few millimeters of material is sufficiently thick. For a light-element matrix such as, for example, coal, a few centimeters of material is needed. Additionally, because characteristic X-rays from light elements have

lower energies, they are more easily reabsorbed within the specimen and, in turn, are more sensitive to irregular specimen morphology, surface contamination, and other effects. Consequently, analyzing for Zr, Sr, and Rb in a specimen of obsidian that is 1-cm thick and has a clean, flat surface will yield ideal results. As a specimen becomes smaller, thinner, or less homogeneous and/or the elements of interest become lighter, an understanding of the X-ray interactions becomes more important.

Detection Limits

The minimum detection limits also vary by element, concentration, material, and measurement time. For favorable elements like Zr, Sr, and Rb, detection limits circa 1-2 ppm in a silicate matrix are often attainable in only 30 seconds. Detection limits for other elements, especially lighter ones, will be higher and/or take longer.

Safety and User Friendliness

For the safety of visiting researchers, the instrument is mounted in a test stand that allows hands-free measurements. The pXRF is fully shielded to protect operator from primary or scattered radiation, and a computer operates the instrument remotely for additional safety.

Our particular test stand is optimized for one-inch round drill cores, similarly sized cups are available for finely powdered materials, or smaller specimens. Larger specimens can either be accommodated by the stand or measured with the instrument in handheld mode.

The p-XRF and the IRM Database

All measurements are downloaded from the instrument via the computer and exported to an Excel file and will ultimately be stored in the IRM Database along with other data generated by a user. The goal is for users to be able to generate bivariate plots with combinations of element concentrations or ratios and magnetic properties, or composition as a function of stratigraphy. For the time being, however, users will be able to export a spreadsheet and plot their data using their favorite software.

Having non-destructive XRF available at the IRM will allow future visitors to correlate magnetic and elemental data from the very same specimens, double-check that specimens originated from the same geological facies or stratum, collect independent data for petrological identifications, and a variety of other yet-to-be-discovered applications. We hope that this instrument will contribute to rock magnetic research across a variety of fields. For further details on XRF analysis, interested readers are referred to Beckhoff et al. (2006).

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