AN APPLICATION BRIEF FROM
SPECTRO ANALYTICAL INSTRUMENTS

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When results matter

Advanced ED-XRF Spectroscopy Accelerates Downhole Characterization

Introduction

Accurate characterization of downhole well properties can yield tremendous benefits in assessing well quality, predicting content, geosteering, and much else. Taking advantage of astonishing improvements in portable energy dispersive X-ray fluorescence (ED-XRF) technology, petroleum geology engineers are now able to rapidly characterize important samples in remote areas — with minimal preparation and very high accuracy of analysis.

ED-XRF instruments utilizing this technique have excellent analytical range and precision, as well as low limits of detection. They can provide quick lab-quality analysis on a variety of geological matrices with a single calibration. The utility of such instrumentation comes with practical experience in the field; these instruments can provide crucial information on potential wells, characterize basins, and help determine optimal conditions for pumping. Following are three examples of how advanced ED-XRF can help at the well site.

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New portable instruments such as SPECTROSCOUT offer lab-quality performance in the field.

Replacing or backing up gamma ray-based composition analysis

Gamma ray "logging while drilling" (LWD) tools help determine shale quality and other properties of the material down the bore. (Figure 1) This guides geosteering and helps in characterization of the well by providing information on the rock or sediment in the borehole. LWD tools are typically attached to the drilling rig only a few feet behind the drill bit or lowered down the hole, but under such rigorous conditions they can often break down and fail or lose data. XRF analysis of potassium oxide, thorium, and uranium on drill cuttings deployed topside can match or complement these readings, but typical concentrations of uranium and thorium have traditionally been too low for XRF analysis outside a laboratory environment. The new generation of ED-XRF technology, however, is more compact and more portable, so that excellent limits of detection for these elements can be obtained at-well in near-real time. In the case of downbore tool failure, XRF elemental analysis can be used to backup the gamma ray instrumentation for minimum downtime.

Knowing where to frack — find the sweet spot

In "fracking" (hydraulic fracturing), analysis of trace elements such as vanadium, nickel, molybdenum, and uranium can provide information on organic richness and on the geohistorical environment in which the organics were deposited, which helps determine well quality and content. Analysis of the composition can aid also in determining high brittleness areas sensitive to puncture. Identifying such areas near organically rich volumes will provide for maximum throughput and minimum damage to the well. Analysis of these trace elements helps minimize the number of fractures, optimize well output, and save time and money for efficient operations. ED-XRF technology has excellent detection limits for these elements; modern systems are capable of providing improved excitation that makes detection of many of these trace elements even more precise.

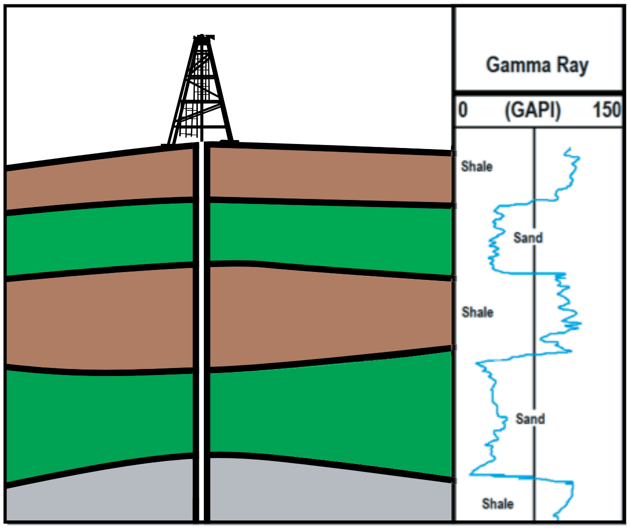


Figure 1. Gamma ray logging measures naturally occurring gamma rays as an indication of the composition of the formation.

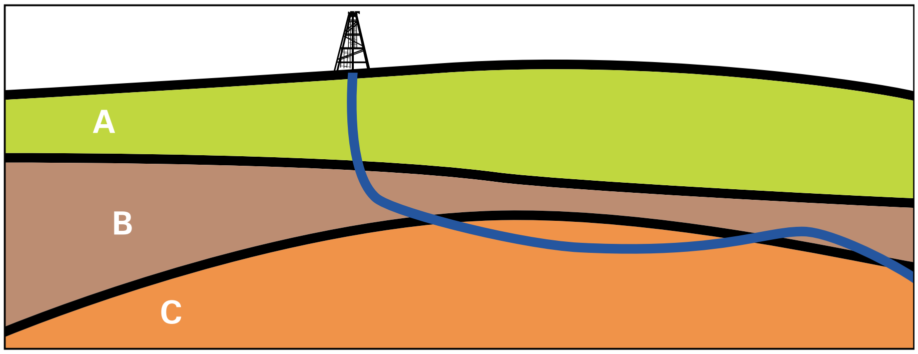


Figure 2. Geosteering to stay in formation. SPECTRO ED-XRF instruments for downhole applications

Analyzing the stratigraphy of the drill cutting

Elemental XRF analysis of drill cuttings allows one to characterize stratigraphy as the drill cuts through or is maintained through a formation. This is especially important for geosteering the drill (Figure 2) to keep it within formation or guide it to more interesting and profitable formations. With various formations being drilled through, it is vital to know where you are. In Figure 2, drilling has started through formation A, then broken into B and briefly into C. Formation C may be a crucial one to follow, but with lateral drilling, the drill has broken out of formation C and returned into B; steering back can be time-consuming and difficult.

Analysis of major elements such as magnesium, aluminum, silicon, calcium, titanium, and iron, in addition to the trace elements, (i.e. Ni, V, Mo, Th, U...) can be done rapidly and easily with ED-XRF. Formations may change geochemistry dramatically. Geological samples, for example, often contain a wide range of elements at vastly differing concentrations, from major components to traces. As such the analytical signal from the same concentration of a given element could be different in different matrices – for example in acidic, intermediate or igneous rocks. Modern ED-XRF analysis is sensitive enough to provide the corrections needed for an accurate analysis of the base formation.

Analytical performance

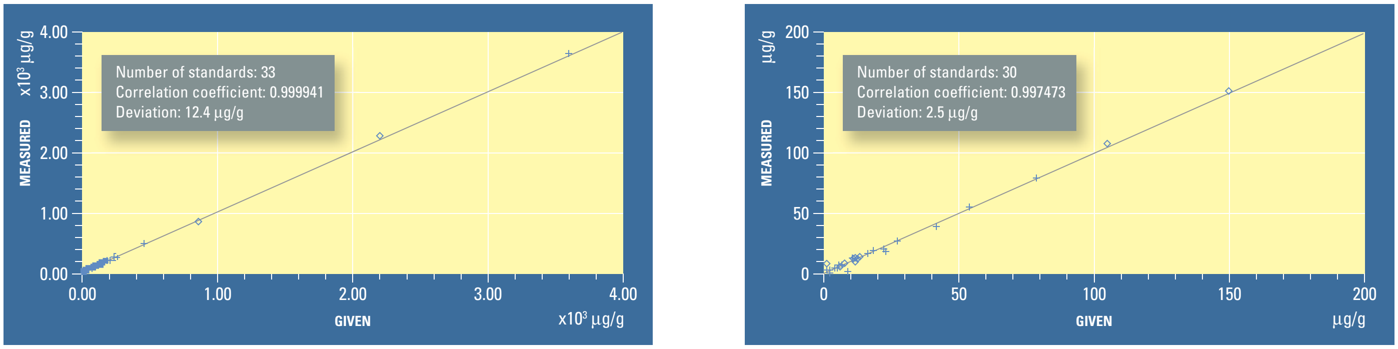
Realizing the benefits of ED-XRF technology requires compensating for “matrix effects,” which are the influences of other elements of the sample matrix. Matrix effects can interfere with test results when the instrument attempts to read signals from target analytes. The fluorescent signal for 10 parts per million of thorium in shale, for example, would be much different than the corresponding signal in limestone; accurate analysis of such different responses would typically require separate calibrations. By measuring the influence of the matrix on X-rays in a sample directly through advanced scattering measurements, however, modern spectroscopic techniques can now characterize samples containing multiple elements with only one calibration. This results in faster, more accurate downhole information, and dramatically reduces costs. (See Figure 3.)

The excellent performance of ED-XRF for petrogeology is apparent in multi-element applications. Table 1 presents analytical results for AGV-2 (andesite) in comparison with certified values.

SPECTRO ED-XRF instruments for downhole applications

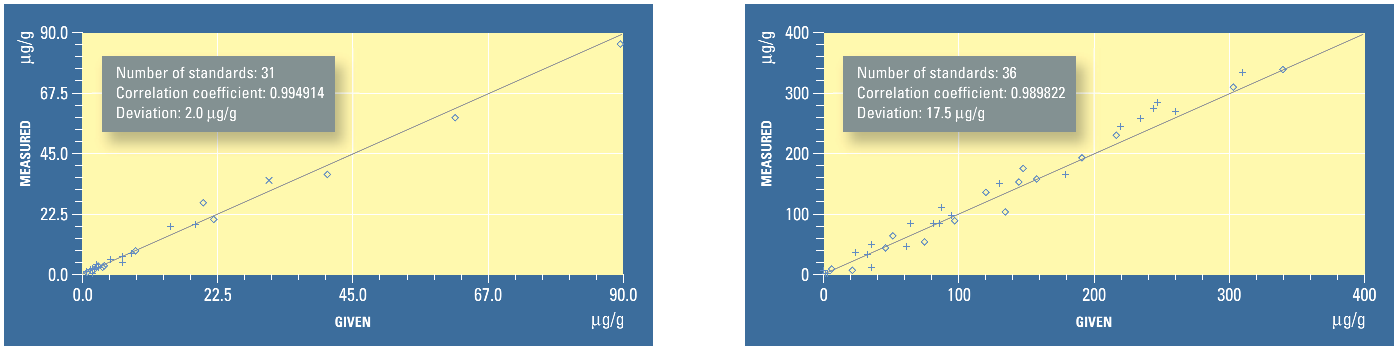
By measuring the influence of the matrix on X-rays in a sample directly through advanced analysis techniques, Spectro ED-XRF instruments — XEPOS and SPECTROSCOUT — are able to analyze a wide variety of geological materials under the same calibration, with excellent linearity from very low to percent-level concentrations for elements from sodium to uranium. (See Figure 4.) Optimizing measurement conditions, SPECTRO XEPOS and SPECTROSCOUT analyzers achieve excellent detection limits. Single-digit parts per million limits of detection are present for the majority of transition elements, with sub-parts per million limits of detection for some elements such as thorium and uranium. For even better limits of detection and precision in analyzing samples containing high atomic number elements such as the first-row lanthanide series, the SPECTRO XEPOS HE comes with an optimized 60 kV excitation condition yielding improved excitation,

Figure 3. Characterization of target analytes in a typical downhole matrix with only one calibration



Correlation Rb

Correlation Th



Correlation U

Correlation V

and a wide-area silicon drift detector for improved detection and high precision statistics.

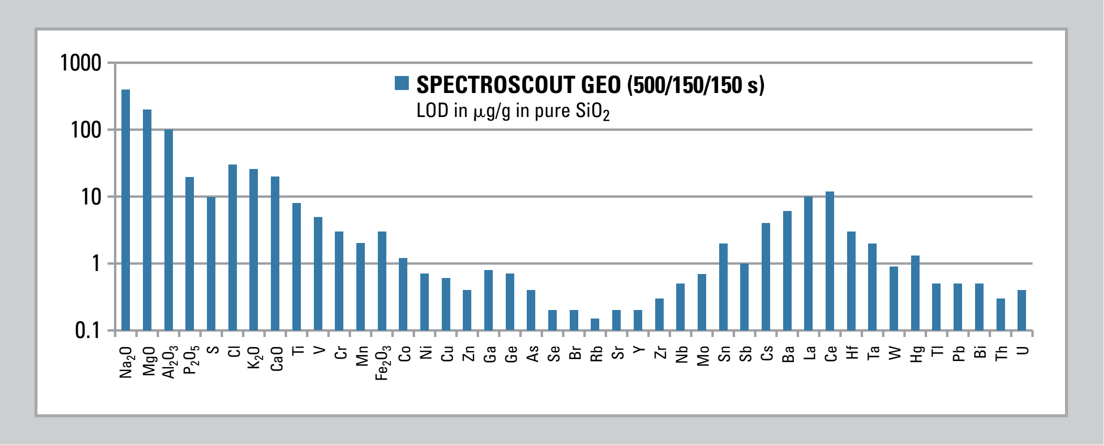
With such excellent analytical range, highly precise analysis, and low limits of detection, SPECTRO XEPOS and SPECTROSCOUT analyzers can supply quick, lab-quality analyses on a variety of geological matrices with a single calibration. The utility of such instrumentation comes with practical experience in the field; these instruments can provide crucial information on potential wells, basin characterization, and optimal pumping conditions.

Table 1: Analytical results for AGV-2 in comparison with certified values including counting statistical error

Element Oxide	Measured			Certified	Unit
Na_2O	3.9	±	0.2	4.19	%
MgO	1.69	±	0.04	1.79	%
Al_2O_3	14.9	±	0.1	16.91	%
SiO_2	58.8	±	0.1	59.3	%
P_2O_5	0.470	±	0.006	0.48	%
K_2O	2.74	±	0.02	2.88	%
CaO	4.97	±	0.03	5.2	%
Ti	6,240	±	60	6,294	µg/g
V	120	±	14	120	µg/g
Cr	18	±	2	17	µg/g
MnO	963	±	12	994.2	µg/g
Fe_2O_3	6.39	±	0.01	6.69	%
Co		<	20	16	µg/g
Ni	21	±	1	19	µg/g
Cu	52	±	2	53	µg/g
Zn	81	±	1	86	µg/g
Ga	18	±	1	20	µg/g

Element Oxide	Measured			Certified	Unit
Rb	67	±	0.6	68.6	µg/g
Sr	652	±	1	658	µg/g
Y	21.6	±	0.4	20	µg/g
Zr	222	±	1	230	µg/g
Nb	12	±	0.4	15	µg/g
Sn	5.9	±	0.6	2.3	µg/g
Sb		<	1.1	0.6	µg/g
Cs		<	5.3	1.2	µg/g
Ba	1,082	±	8	1,140	µg/g
La	24	±	9	38	µg/g
Ce	44	±	7	68	µg/g
Hf	3.5	±	0.6	5.1	µg/g
Ta	8	±	2	0.9	µg/g
Tl		<	0.7	0.3	µg/g
Pb	15	±	1	13	µg/g
Th	6.7	±	0.4	6.1	µg/g
U	2.7	±	0.8	1.9	µg/g

Figure 4: Limits of detection (in ppm) for various elements using SPECTROSCOUT



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Scoping out a portable ED-XRF system

Although various portable or small ED-XRF spectrometers may implement the same technology, there will be vast differences in the performance, ease of use, and instrument suitability for the at-line application. Here are some of the factors to consider when specifying and comparing ED-XRF spectrometers:

- **Performance.** ED-XRF instruments used in the field must have dependable accuracy, repeatability, and sensitivity across a wide range of detection levels enabling precise analysis from high percentage amounts to trace element concentrations.

- **Ease of operation.** Consider who will be using this instrument as ease of use translates into greater productivity and lower training costs. Look for an intuitive interface, simplified software that interacts with a standard PC, and comes with predefined application packages and calibration tools.

- **Cost.** A portable ED-XRF instrument can cost as little as half that of a dedicated laboratory instrument. That's still a pretty solid investment. Be sure the system is designed for the application and is rugged enough to withstand operation in the field.

- **Portability, compactness and integral power supply.** Weight, design and transportability are all key factors in making the most of measurements in the field. If access to a power outlet might be a problem, be sure to specify an instrument that runs on batteries.

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