

New and Current USGS Shale Reference Materials for Shale Analysis

by Energy Dispersive X-Ray Fluorescence

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The United States Geological Survey (USGS), in collaboration with industrial partners, has developed five new shale reference materials from major US shale plays in order to support current energy production. These new materials, together with the currently available materials, provide 10 shale references. The set of shale reference materials provides the elemental range as follows for calibrations:

Wt%	Na ₂ O	K ₂ O	MgO	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	MnO	P ₂ O ₅	
Low	0.16	0.34	0.43	.63	1.33	7.58	.99	.01	.05	
High	2.99	4.02	4.44	53.75	21.33	78.87	10.37	.15	.42	

ppm	Pb	Zn	Ti	Cu	Sr					
Low	4.5	58	300	29	75.1					
High	55	419	5100	184	1101.5					

An empirical calibration completed on a benchtop EDXRF system was used to calculate the limitations of accuracy and precision for Fe, Pb, Zn, Ti, Cu and Sr. Using our calibration and normalization approach, we achieved 90+% recoveries for select elements. We will discuss the specific findings of our research in terms of EDXRF data reliability.

Furthermore, the scientific community struggles to determine if handheld EDXRF (pXRF) data can be validated to replace the expense of laboratory analyses. This is important because the continuing application of pXRF has provided real time chemo-stratigraphic information. For example, currently the USGS is evaluating handheld XRF (pXRF) data collected from drill core to assist in finding optimal zones in selected formations when looking for unconventional petroleum resources. To support this effort we are in the process of comparing the empirical parameter (EMP) results mentioned above (from the benchtop unit) to the fundamental parameter (FP) results from a portable EDXRF field unit to evaluate reliability of real time data on our shale reference materials.

Recent journal articles have reported that pXRF data can be accurate and precise for a limited set of elements under “favorable” conditions where the sample has small grain sizes and is relatively homogenous. The development of these calibrations requires numerous standards for each rock type and is still limited by the physical presentation of the sample to the analyzer. The USGS will continue to expand the reference materials available to the public in an effort to assist the analytical community. If you have a suggestion for a new reference material, please contact one of us.