K/T GeoServices, Inc.
XRD3 - Bulk and Clay (<4 micron) XRD Analysis
Sample Preparation and Analytical Procedures

Sample Preparation

Samples submitted for bulk and clay mineral XRD analyses are cleaned of obvious contaminants and disaggregated in a mortar and pestle. A split of the sample is then transferred to distilled water and pulverized using a McCrone micronizing mill. The resultant powder is dried, disaggregated, and loaded into a metal sample holder to produce random bulk mounts. A separate split of each sample is dispersed in distilled water using a sonic probe. The suspensions are size fractionated with a centrifuge to isolate <4 micron particles (<4 micron equivalent spherical diameter). The <4 micron suspensions are vacuum deposited on nylon membrane filters to produce oriented clay mineral mounts.

Analytical Procedures

X-ray Diffraction (XRD) analyses of the samples are performed using a Siemens D500 automated powder diffractometer equipped with a copper X-ray source (40kV, 30mA) and a scintillation X-ray detector. The bulk powder samples are analyzed over an angular range of five to sixty degrees two theta at a scan rate of one degree per minute. The oriented clay mounts are analyzed over an angular range of two to thirty six degrees two theta at a scan rate of one degree per minute. Data are first collected on the air dried clay mounts. Next, the clay mounts are exposed to ethylene glycol vapor for a minimum of 12 hours and data are collected again.

XRD patterns from air-dried and glycol-solvated clay-fraction samples are compared and qualitatively analyzed to determine the types of clays present in the samples. Determinations of mixed-layer clay ordering and expandability are done by comparing experimental diffraction data from the glycol-solvated clay mounts with simulated one dimensional diffraction profiles generated using the program NEWMOD written by R. C. Reynolds.

Semiquantitative determinations of bulk powder mineral amounts are done using Jade Software (Materials Data, Inc.) with the Whole Pattern Fitting option. All quantitative data (including clay mineral amounts) come from the bulk powder pattern. This is done by using Whole Pattern Fitting (WPF) and Rietveld refinement methods on the observed data. A diffraction model is fit to the measured pattern by non-linear least-square optimization in which certain parameters are varied to improve the fit of the model to the observed data. Modeling parameters include background, profile parameters, and lattice constants. For Rietveld refinement, a complete physics simulation is generally used in which crystal structures of the phases are required. Since the physics of scattering is well known, this method can be very exact and even allow adjustment of atomic coordinates, occupancies, and thermal parameters.

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Discussion of Terminology and Limitations

Weight percentage data from X-ray diffraction methods are considered semi-quantitative; there are many factors affecting the results.

XRD methods can quantify crystalline material only. Non-crystalline material in large concentrations can be detected but not quantified. Therefore, any non-crystalline material is not included in the accompanying results.

Detection limits for XRD are on the order of <1 to 5 weight percent. Detection limits differ for each mineral species.

Mineral standards used to determine calibration factors are often different from the actual minerals analyzed. Minerals such as feldspars that undergo solid solution are especially problematic. Clay minerals are problematic for this same reason. Clay minerals also have a wide range of crystallinities (poorly crystallized to well crystallized) which may compound this problem.

With this method the data always sums to 100%. This means that the percentages reported for each mineral are dependent upon the percentages reported for the other minerals. If one mineral is underestimated the others will be overestimated. Also, if one or more minerals are present but not detected then the percentages of the minerals that are detected will be overestimated.

Any or all of the above factors may affect the estimated weight percentages.

For this analytical method, the clay fraction is defined as the <4 micron (Equivalent Spherical Diameter) fraction of the sample. Clay fraction does not mean clay minerals (phyllosilicates) only, it is a size term and as such this size fraction can and almost always does include non-clay minerals (quartz, plagioclase, etc.). This size fraction is used because it typically contains abundant clay minerals.