

K/T GeoServices, Inc.
XRD1 - Bulk Only XRD Analysis
Sample Preparation and Analytical Procedures
(Copyright 2019 K/T GeoServices, Inc.)

Sample Preparation

Samples submitted for bulk 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 packed into a metal sample holder to produce random bulk 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.

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 an estimate of 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.

K/T GeoServices, Inc.
XRD1 - Bulk Only XRD Analysis
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.