

XRD Analysis

Whole Rock and Clay Fraction Methodology

The formation material is ground to a coarse powder (approximately <300 microns). A portion of the homogenized sample is reserved for the clay separation procedure; the rest is wet milled in a McCrone XRD mill for 13 minutes to ensure a narrow grain size distribution for proper phase quantification. Each sample is measured in the Bruker D8 Advance ECO instrument for 60 minutes to detect minor peaks, further improving the accuracy of quantitative analysis.

The clay fraction separation is performed by combining the coarse material with DI water and sonicating for 7 minutes to guarantee clay disaggregation from the framework minerals avoiding clay exfoliation. The sonicated solution is centrifugated for 3 minutes to extract the desired clay size fraction. The extracted fraction is prepared following the smear mount filter method to ensure the proper orientation of clay particles onto a glass slide. The air-dried clay samples are then measured using the Bruker D4 Diffractometer for 25 minutes. The clay samples are treated with ethylene glycol in a glycol desiccator for 24 hours and remeasured to confirm the presence of swelling components in the clay structures. Lastly, the samples are heated in a furnace for 1hr at 400oC to identify heat-sensitive phases and measured by the iterated convention above.

We use the Reference Intensity Ratio (RIR) method to quantify the whole rock mineralogy of random powders using Al₂O₃ as the internal standard. This method determines the weight percentage of each mineral present from the intensity ratio of selected peaks of the mineral to those of the internal standard, corrected by a constant of proportionality determined from mixtures of pure minerals and the used standard. For minerals in amounts greater than 15%, the precision and accuracy of the determination are to be +/- 10% relative to the true values.

Premier XRD interpretation and quantification workflow relies on a state-of-the-science RIR procedure, allowing for fast and consistent results. This allows us to provide:

- An accurate identification of mineral phases aided by the Bruker EVA DIFFRAC SUITE software and the most recent mineral database available on the market (PDF4+).
- Detailed clay mineral speciation and %expandability determined using the clay separation/glycolation/heat treatment method from oriented samples.
- An accurate quantification of total clay vs. non-clay minerals.

Accurate and reliable mineral quantification is critical for reservoir characterization used in geological models, and we aim for accuracy.

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