Methods of Analyzing Clay and Non-Clay Minerals Using X-ray Diffraction

Shatosha Maddix, Stefan Raduha, and Shane Butler
Illinois State Geological Survey, Prairie Research Institute, Champaign, Illinois

The X-ray diffraction (XRD) and clay mineralogy laboratory in the Bedrock Geology and Industrial Minerals Section of the Illinois State Geological Survey has the capacity to provide qualitative and semi-quantitative mineral characterization of whole rock and soil samples. The laboratory offers three types of analysis: bulk powder analysis, <2-µm clay mineral analysis, and <16-µm clay mineral analysis.

Bulk powder analysis of whole rock samples is suitable for quick mineral identification. Samples undergo various size-reduction processes to meet a ~10-µm particle size fraction. Once in powdered form, the sample is packed into a sample tray and analyzed by XRD. The XRD analysis provides semi-quantitative and/or qualitative data for a suite of minerals. Identification of minerals is based on the location and intensity of peaks on the 2θ scale.

The <2-µm clay mineral analysis method is used mainly for identification of clay minerals from Quaternary-age deposits, but can also be used on shales and soils. Samples are immersed in deionized water and disaggregated. The samples are individually stirred in an electronic mixer to isolate the clays in suspension. The particles settle, and the salts that remain in solution are poured off. The samples are stirred again and allowed to settle until only the <2-µm particles are in suspension. Using an eye dropper, the <2-µm clays are placed onto a glass slide, which is then dried, glycolated, and scanned by XRD. This method provides semi-quantitative clay mineral analysis, comparing only the amount of clay minerals in the sample relative to each other.

The <16-µm clay analysis method is used for identification and relative comparison of clays within sandstone samples. In this method, the sample is disaggregated and then rinsed with bleach to remove oil (if in an oil-bearing unit). The sample is immersed in acetic acid to dissolve carbonates. The sample is stirred to bring the clays into suspension for removal. The clays are centrifuged and smeared onto a glass slide. Finally, to properly identify clay minerals, the sample is glycolated, scanned by XRD, heated to 350°C for 1 hour, and rescanned by XRD. This procedure provides two sets of data. The first set provides semi-quantitative clay mineral analysis that compares the amount of clay minerals relative to each other. The second set provides semi-quantitative mineral percentages of the expandable clays in the sample.

Samples prepared by each of the three methods are scanned with a Scintag® XDS2000 X-ray diffractometer using CuKα radiation at 40 kV and 30 mA. The majority of the scans are performed using a continuous scan mode from 2 to 34° 2θ with a 0.05 step size at 2 degrees per minute. The collected data are then analyzed using Jade 9+® software. Semi-quantitative mineral percentages for both clay and non-clay minerals are determined using methods pioneered at the ISGS by Herb Glass and others. To conclude the process, the results are assembled into easy-to-read spreadsheets, and the XRD trace for each sample is put into the form of a .jpg image for our clients.