



FIRST Newsletter

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The Reynolds Cup for Quantitative Mineralogy

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The [Reynolds Cup](#) is a global, biennial event organized by the Clay Minerals Society with the goal of testing and improving the analytical techniques used in characterizing complex mineral samples. The contest samples generally contain large amounts of clay minerals, which can be challenging to identify and quantify: any analytical technique can be used, but X-ray diffraction (XRD) is by far the most prevalent technique. Samples for the 2020 competition (10th Reynold's Cup) were prepared by the previous winners from the James Hutton Institute and contained 43 mineral specimens in three different mixtures, representing a hydrothermally altered shale, a muddy limestone, and a Martian volcanoclastic sediment/soil.

Bruker's XRD Team in Madison, Wisconsin placed in the top 10 last year, using the [D8 ADVANCE](#) diffractometer with [Dynamic Beam Optimization](#) and [LYNXEYE XE-T](#). These instrument features can help obtain lower background and better sensitivity for trace minerals, low angle diffraction from interstratified clays, and broad amorphous scatter. The winners of the 2020 competition (Qmineral Analysis & Consulting, Belgium) also used a D8 ADVANCE with LYNXEYE XE-T, along with some clever sample preparation methods and quantification methods using DIFFRAC.TOPAS. We asked Qmineral for some insights into their successful approach.

Qmineral is an independent material characterization laboratory specialized in quantitative mineralogical and clay mineralogical analyses by X-ray diffraction. Qmineral also offers a wide range of other analytical services, such as chemical analyses, porosity and permeability measurements, particle size analysis by various techniques, petrography by optical microscopy and electron microscopy, cation exchange capacity measurements, TOC determination, specific surface area and physisorption analyses. Qmineral is active as a service lab or research partner in a wide range of industries and for various applications (see more at www.qmineral.com).

The Reynolds Cup is an excellent opportunity for data validation. Qmineral has participated five times and has evolved from the 10th place in 2010, to the 5th place in 2012 and 2014, to the 1st place in 2016 and again in 2020. In 2018, Qmineral organized the Reynolds Cup event.

Our approach in the 2020 RC edition consisted of performing XRD measurements on random-oriented powders and oriented clay specimens measured in air-dry and glycolated conditions. An appropriate sample preparation, the correct use of instrument and software and correct identification are the key to success in quantitative X-ray diffraction.

The powders were prepared by micronizing a representative part of each sample and ensuring



Congratulations to the Qmineral team for their second Reynolds's cup win and for their exceptional analyses! Well done!

optimal random orientation when filling the measurement holder. Measurements were performed using our Bruker D8 ADVANCE equipped with a LYNXEYE XE-T detector. Identification of the phases was performed using DIFFRAC.EVA software and the subsequent quantification using TOPAS software (Bruker AXS). A second part of each sample was used to isolate a fine fraction in which clay minerals are concentrated. Slides with oriented clay aggregates were measured in air-dry and glycolated conditions and finally heated and remeasured. These measurements allowed to identify all clay minerals present in the samples.

The QXRD results were complemented by chemical analyses. The chemical data were however not very helpful as the mixtures were composed of many phases with a strongly however not very helpful as the mixtures were composed of many phases with a strongly variable and overlapping chemical compositions. Hence, we trusted our data obtained by QXRD.

We are very proud to win the Reynolds Cup for the second time, after our victory in 2016, as this proves the consistent accuracy and precision of Qmineral's mineralogical data.