Quantitative phase analysis of clay mineral assemblages: Methodology and accuracy

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Since 2002 The Clay Minerals Society has conducted biennial quantitative mineralogy round robins. The Reynolds Cup competition, as it is called, is named after Bob Reynolds for his pioneering work in quantitative clay mineralogy and exceptional contributions to clay science. Established in 2000, the first contest was run in 2002 with 40 sets of 3 samples prepared and composed of mixtures of purified, natural and synthetic minerals commonly found in clay bearing rocks and soils that represent realistic mineral assemblages.

2012 marked the 6th Reynolds Cup quantitative mineralogy competition where a total of 74 sets of three samples were distributed to participants from 25 countries. Samples were made available to individuals in commercial, industrial, government, or academic laboratories. Any method or a combination of methods could be used to obtain the most accurate quantitative phase analysis. X-ray diffraction was dominantly the method of choice for quantifying the mineralogy of the sample mixtures, however, a multitude of other techniques were also used to assist with phase identification and quantification.

A summary of the 6 contests between 2002 and 2012 are given in Table 1. The number of participants, and more importantly, the return rate has steadily increased since 2002 despite the highly complex mineral assemblages prepared. The accuracy of a particular quantification is judged by calculating a "bias" for each phase in an assemblage. Determining exactly the true amount of a phase in the assemblage would give a bias of zero. Average biases per mineral phase for the winners of the first 4 contests between 2002 and 2008 remained relatively constant at a little under 1%. Since 2010 the contest was assessed in greater detail with the clay minerals in particular being judged to a higher level. For example, dioctahedral 2:1 clay minerals (muscovite, illite, illite-smectite and smectite) and trioctahedral 2:1 clays minerals (saponite, biotite and vermiculite) were grouped together prior to 2010 but were assessed separately in 2010 and 2012. As a result of this closer scrutiny the average bias per mineral phase for the winners of the 2010 and 2012 contests had doubled to ~2%.

Generally the higher placed participants correctly identified all or most of the mineral phases present. Conversely the worst performers failed to identify or misidentified phases. Several contestants reported a long list of minor exotic phases, likely reported by automated search/match programs but mineralogically not possible. Not surprisingly, clay minerals provided the greatest source of error.

Plaques are presented to the top 3 finishers and the perpetual Reynolds Cup Champion's trophy is presented to the winner during the annual meeting of the society. Winners of each contest are also given the opportunity to chair the organizing committee and prepare the sample mixtures for the following Reynolds Cup contest to be run in 2 years time.

Table 1. Summary of the 6 Reynolds Cup round robin contests 2002 to 2012

Year	*Number of participants*	*Returned results*	*Return rate (%)*	*No. of mineral phases*	*1st place total bias (%)*	*1st place average bias per mineral (%)*	*Average total bias for all participants (%)*
2002	40	15	37.5	36	32.4	0.9	90.5
2004	60	35	58.3	34	14.0	0.4	102.4
2006	64	37	57.8	42	33.8	0.8	138.1
2008	53	42	79.2	35	35.2	1.0	162.3
2010	76	63	82.9	42	87.8	2.1*	269.2
2012	74	62	83.8	40	72.0	1.8*	213.4

* the method for assessing the clay biases were changed for the 2010 and 2012 contests