Kaolin powder

D4 ENDEAVOR

LYNXEYE Detector

Application Report XRD 14

DIFFRAC.DQUANT:
Quartz determination with the Addition Method

The Addition Method implemented in DIFFRAC.DQUANT provides a simple tool for the quantification of impurities in a mixture. This allows minerals producers to exactly specify the quartz content of their products as required by latest EU regulations (REACH).

Quartz as well as other silica (SiO_2) phases (cristobalite, tridymite) may pose an occupational health risks on workers who handle materials such as kaolin, wollastonite or limestone in the quarry and during later processing for the production of cement, ceramics, porcelain and other goods. The amount of siliceous minerals can easily be determined by X-ray powder diffraction (XRD).

Here, we present the quantification of quartz in kaolin, an important industrial minerals resource used in e.g. porcelain, cosmetics, paints, or rubber production. The Addition Method is applied, which

- is well suited for occasional quantification,
- is very simple to perform and does not need drift correction,
- can handle oddly shaped peaks for disordered materials,
- is insensitive to preferred orientation of other components in the mixture,
- delivers the absolute concentration even in the presence of unspecified (e.g. amorphous) material.

The method requires the measurement of several specimens prepared from the same sample with different admixture of the analyte to be quantified (here quartz). One or more background-corrected individual peaks of the analyte, which ideally should not overlap with other peaks, are to be analyzed. DQUANT automatically calculates the original content of quartz and its average from several peaks, and generates a report of the individual concentrations and the resulting mean value.

Four samples were prepared, the original material as received and admixtures with 2, 5 and 10 wt-% quartz. XRD data were collected using a D4 ENDEAVOR diffractometer, equipped with a Cu tube and a LYNXEYE linear silicon strip detector.

Figure 1 shows an overview scan used for phase identification with DIFFRAC.EVA and the ICDD PDF2 database. The major phase is kaolinite. In addition to quartz, mica (biotite/muscovite) and feldspar (orthoclase) are found. Since the major quartz peaks show strong overlap with the other phases, the weaker peaks at about 36.5, 50.1 and 68° were exemplarily selected for the quantitative analysis in DQUANT.

The resulting analysis is shown for the 50.1° peak in Figure 2. The three individual peaks yield concentrations of 1.87, 1.58 and 2.13 wt-% quartz in the original sample, respectively. This results in a mean value of 1.86 wt-% and a standard deviation of 0.27wt-%.
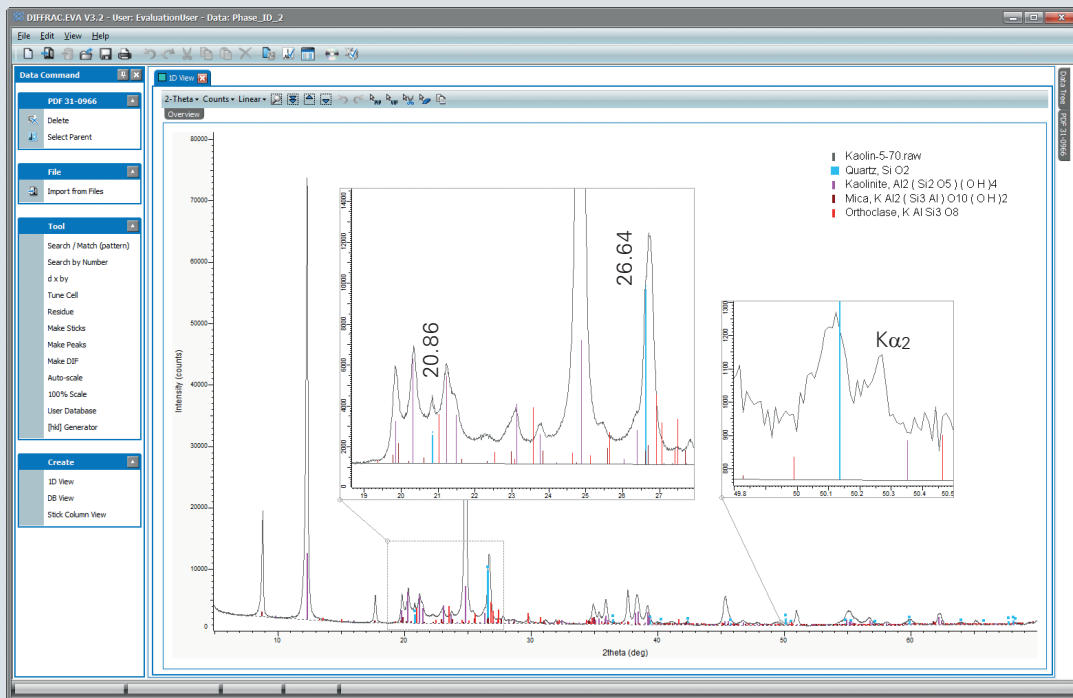
XRD

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Figure 1: Phase identification of kaolin with DIFFRAC.EVA. The left inset indicates peak overlap of the strongest quartz peaks at 20.86 and 26.64°, making them unsuitable for quantification. The right inset shows a weak but non-overlapping peak of quartz at higher angle used for quantification in DQUANT.
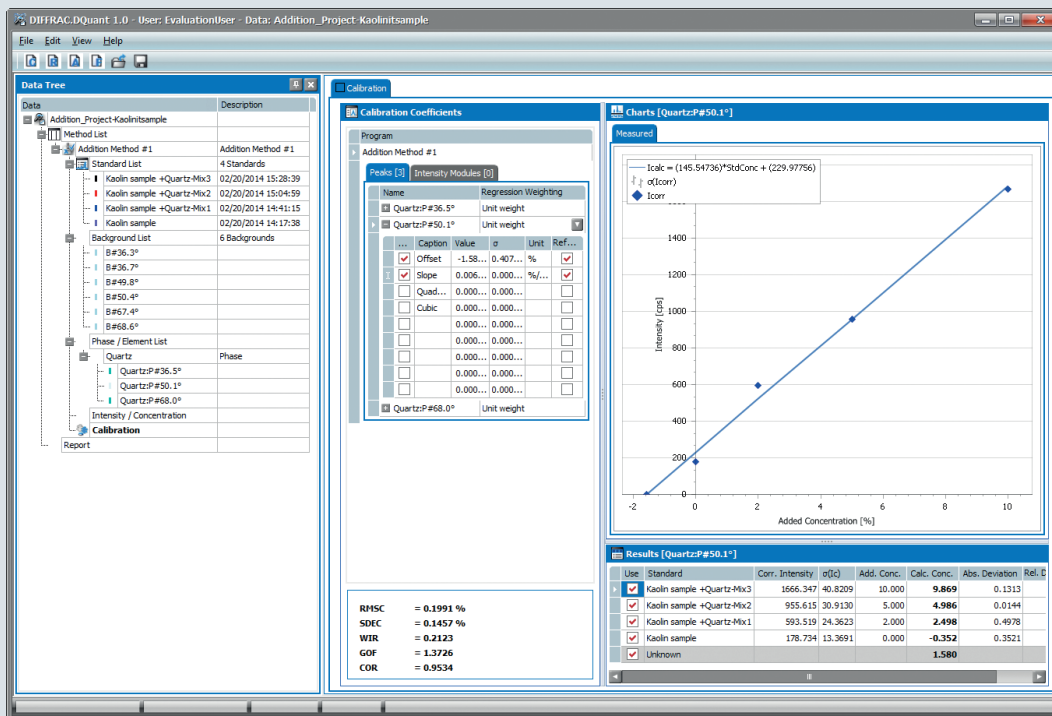

Figure 2: Calibration view of DIFFRAC.DQUANT for the Addition Method.

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