Rigaku
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Application of Integrated X-ray Powder Diffraction Software: PDXL

Quantitative analysis of a non-crystalline (amorphous) phase

Amorphous materials have attracted a lot of attentions from many researchers in the materials science field. In a mixture of crystalline and amorphous materials, the amorphous phase is sometimes preferable, but sometimes not. It is very important to know the weight fraction of the amorphous phase in a target material, and the X-ray diffraction technique is one of the best to analyze the amorphous quantity in a sample. Below are described three quantitative analysis methods of an amorphous phase that are available in PDXL.

1. Internal standard method

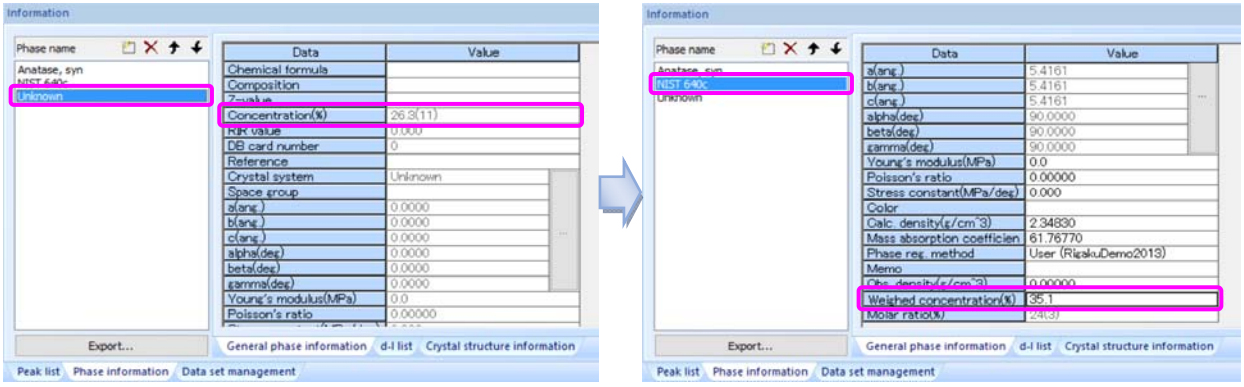
The internal standard method is a conventional one for the quantitative analysis of an amorphous phase. The principle is very simple.

First, you should measure the weight of the sample (*X* mg) and the internal standard material (*Y* mg). Second, add the internal standard to your sample and collect the X-ray diffraction (XRD) data of the mixture. Based on the obtained XRD data, PDXL can determine the weight fraction of each crystalline phase, including the internal standard phase. Now that the weight of the internal standard phase is known, the total weight of the crystalline phases (Phase A, B, C, and internal standard) is calculated (*Z* mg). Finally, PDXL will calculate the weight fraction of the amorphous phase based on the following formula:

$$\text{The weight fraction of the amorphous phase} = (X + Y - Z) / (X + Y)$$

Analysis procedure:

1. Measure the weight of your sample and the internal standard, and mix them.
2. Collect the XRD data of the mixture, and perform phase identification.
3. Enter the ratio of the internal standard to the sample (= $Y/(X+Y)$) in the **Weighed concentration (%)** cell. The weight fraction of the amorphous phase will be shown in the **Concentration (%)** cell of the **Unknown** phase.

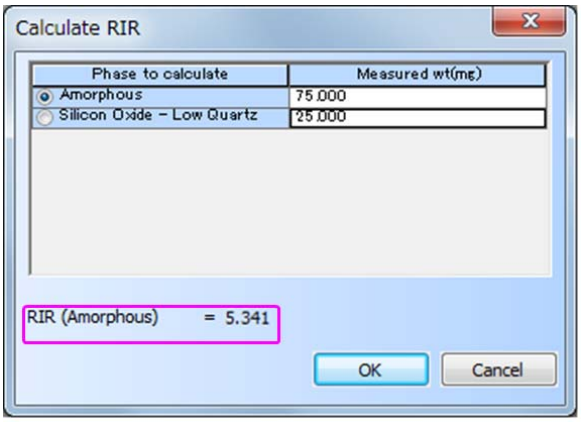


2. RIR method

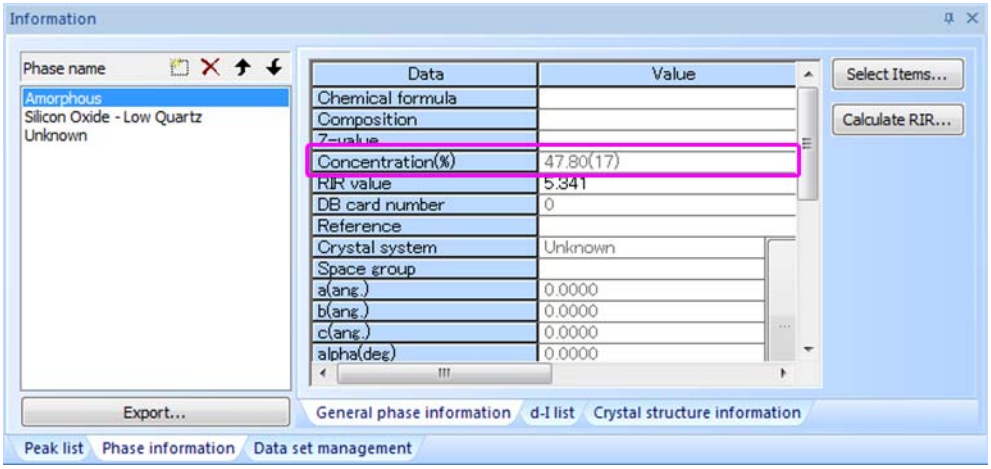
The RIR (Reference Intensity Ratio) method is similar to the PONKCS (Partial Or No Known Crystal Structure) method. If you can obtain a known sample containing the target amorphous phase whose weight fraction is also known, the RIR method is applicable. First, you should collect the XRD data of the sample. PDXL will calculate the RIR value of the target amorphous phase based on the XRD data. Once you obtain the RIR value, you can handle the target amorphous phase in your sample as if it were one of the general crystalline phases.

Analysis procedure:

1. Prepare the reference sample, in which the weight fraction of the amorphous phase is known.
2. Collect the XRD data, do the Rietveld analysis. Refine the amorphous phase halo also.
3. Enter the measured weight of each phase to get the RIR value of the amorphous phase.



4. Collect the XRD data of your sample, and do the phase identification. Rietveld analysis will provide more precise weight fraction of the amorphous phase.



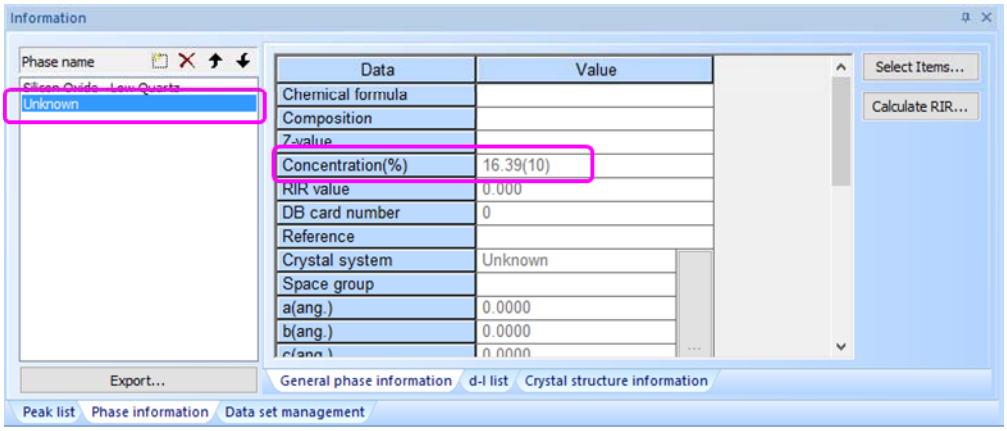
See Section 3.3 of the *PDXL Rietveld Analysis User Manual* for more details.

3. External standard method (Quantification standard method)

The external standard method evaluates the weight fraction of an amorphous phase based on the assumption that the incident X-ray intensity is the same regardless of the sample. First, you should collect the XRD data of an external standard sample with the %crystallinity = 100%. When you analyze the XRD data of the external standard sample using PDXL, a quantification standard project file will be created. When you perform the quantitative analysis of your sample containing an amorphous phase, you shouldn't assign an amorphous phase to a halo. Based on the difference between the total intensity of the diffraction peaks, PDXL will calculate the weight fraction of the amorphous phase.

Analysis procedure:

1. Collect the XRD data of a reference sample such as corundum (of which %crystallinity is 100).
2. Do the Rietveld analysis and set the project file as the quantification standard.
3. Collect the XRD data of your sample, and do the Rietveld analysis of the obtained data.
4. The weight fraction of the amorphous phase will be displayed in the **Concentration(%)** cell of the **Unknown** phase.



See Section 3.7 of the *PDXL Rietveld Analysis User Manual* for more details.

4. Comparison

Each method has advantages and disadvantages. The following table summarizes the differences among the three methods. We hope at least one of the methods is applicable to the quantitative analysis of your samples.

Method	Advantage	Disadvantage/Prerequisite
Internal standard	Any sample can be analyzed.	Your sample will be contaminated with an internal standard material.
RIR	Once RIR is determined, the target amorphous phase will easily be analyzed from then onwards.	Need to prepare a sample in which each phase (including the amorphous phase) is known in terms of material and quantity.
External standard	Any sample can be analyzed.	Need to prepare a sample with the %crystallinity = 100%.