XRD: Quantify Phases and the Missing Fraction

RUIKANG Li (Neo)

Hongkong Kannry Industrial Co., Ltd. | www.kannrybio.com

Abstract. Check the normalization basis before using a Rietveld phase percentage. This note distinguishes crystalline phase proportions from whole-sample composition and explains the role of standards, detection capability and amorphous content. A good fit alone does not establish quantitative accuracy.

Keywords: XRD; Rietveld; quantitative phase analysis; amorphous content

1. State what sums to one hundred

Quantitative phase results can be normalized to the crystalline phases included in the model. Unmodeled or amorphous material can therefore be absent from the reported total. Internal-standard analysis can help assess a noncrystalline fraction, subject to the method and sample limitations [1].

Original example: add 0.200 g certified crystalline standard to 0.800 g unknown. Its actual fraction in the spiked mixture is s = 0.20. If the refinement reports standard fraction r = 0.25 among modeled crystalline material, total crystalline fraction of the spiked mixture is s/r = 0.80.

The apparent missing fraction is 0.20 of the spiked mixture. Relative to the original 0.80 g sample, it is 0.20/0.80 = 25%. This inference assumes a fully crystalline standard, correct masses, no reaction, a valid model and no other unaccounted crystalline phases.

2. Strengthen a phase conclusion

2.1 Check the residual pattern. Examine unexplained peaks and systematic misfit rather than only the reported agreement statistic. Compare against a starting-material pattern and plausible process products. Do not force every feature into the expected phase merely to produce a clean percentage table.

2.2 Test a known mixture. When phase fraction is a release-critical property, assess accuracy on relevant mixtures or reference materials across the intended range. The uncertainty near a low concentration may be much larger than the number of decimal places displayed by the software.

2.3 Prepare replicate specimens. Use independent sample preparation to examine orientation, grinding and sampling effects. Confirm that the preparation does not transform a sensitive material. Report the method, phase list and normalization basis with the result so another laboratory can interpret the numbers.

3. Development decision

Use a phase percentage as a quantitative claim only on its stated basis. If the unaccounted fraction is large or the pattern contains unexplained features, resolve them before labeling the powder phase-pure.

Table 1. Development comparison and interpretation controls.

Reported quantity	Question to ask	Interpretation risk
Crystalline phase wt%	Which phases were modeled?	Excluded phases distort the total
Minor phase absent	What is the applicable detection limit?	Not detected is not zero
Apparent amorphous fraction	Was a valid standard used?	Model errors can enter the balance
Peak-width change	Were instrument effects controlled?	Not a direct particle-size result

Scope. The 25% example demonstrates an ideal mass balance. It is not a validated uncertainty calculation or a detection limit for a particular calcium phosphate.

References

1. León-Reina L, García-Maté M, Álvarez-Pinazo G, Santacruz I, Vallcorba O, De la Torre AG, et al. Accuracy in Rietveld quantitative phase analysis: a comparative study of strictly monochromatic Mo and Cu radiations. J Appl Crystallogr. 2016;49(3):722-735. doi:10.1107/s1600576716003873.

Prepared by: RUIKANG Li (Neo)

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