Nonstandard use of powder diffraction methods for qualitative phase analysis

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Interpretation of the powder diffractogram can be a simple or complex operation depending on the number and structural complexity of the phases that make up the sample and the amount and nature of the information required. A relatively simple way of interpreting the diffractogram is to compare it with the diffractogram of the reference material. To do this, it is generally required to calculate the interplanar distances belonging to the different diffraction lines, and less often it is necessary to determine the diffraction indices.

Qualitative phase analysis provides elementary chemical information about a substance just like many other analytical methods. However, what distinguishes powder diffraction methods from them is their unique ability to characterize crystalline phases. It is based on the following principles:

• Each crystalline substance is characterized by a unique diffraction pattern

• Each substance in the mixture produces its diffractogram independently of the others

• The identification of the crystalline phase is based on its chemical structure

Other advantages of powder diffraction analysis for ”chemical analysis” are its non-destructive nature, only a small amount of the substance needed for analysis and the possibility to develop it for semi-quantitative analysis.

Standard qualitative phase analysis is performed by comparing the diffractogram of an unknown sample with a set of reference powder diffractograms. The same principle applies to crystalline mixtures, in which the diffractogram of the identified component is ”subtracted” from the diffraction pattern of the mixture and the identification process continues with the remaining diffraction lines until all lines are completely assigned. The key role here is the existence (availability) of a sufficiently large, well-organized library (database) of reference powder diffractograms, continuously updated with high-quality diffraction data. The ICDD PDF powder database currently meets these criteria.

Among the non-standard procedures of qualitative phase analysis, we can include:

Crystallographic analysis focused on the processing of basic experimental data, from which it allows to obtain basic crystallographic information about the investigated substance. These include interplanar distances d_{hkl} of individual lattice planes, diffraction indices h, k, l, dimensions of the basic parallelepiped (lattice parameters a, b, c, α, β, γ), type of crystal lattice and diffraction intensities.

Le Bail analysis, which is a method of refining the crystal structure by comparing the whole digitized experimental powder pattern with the digitized calculated powder diffraction pattern.

A major benefit of non-standard qualitative phase analysis procedures is that, in addition to the possibility of identifying a known crystalline phase, they provide additional information on the investigated substance. These can often be used to correlate with some of the physical properties that can be used in technological and research practice.

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