Difference between quantitative results using the WPPF method and the RIR method

Introduction

In quantitative analysis using X-ray diffractometry, different quantification methods are used depending on factors such as the state of the sample and concentration of the measured components. The method using calibration curves is complicated by the need to procure standard samples, or prepare and measure samples, and thus, at present, there is a switch toward analysis using the WPPF (Whole Powder Pattern Fitting) method and the RIR (Reference Intensity Ratio) method. In the WPPF method, profile fitting is performed over a comparatively broad angular range, based on information about the crystal system and lattice constants. The RIR method uses RIR values listed in a database and integrated intensity of the maximum intensity curves. Both methods enable easy calculation of quantitative values by using dedicated analysis software.

Measurements and results

Samples 1 and 2, each comprised of four components, were measured under conditions where the maximum intensity peak of the X-ray diffraction pattern exceeded 10,000 counts. In the quantification results for Sample 1, there was a good match between sample preparation values and quantification values with both the WPPF method and RIR method, as shown in Fig. 1. However, the results of analysis using the WPPF method yielded values closer to the sample preparation values than the results of the RIR method. On the other hand, it was found that, in the quantification results for Sample 2, the quantification values of the RIR method differed greatly from the sample preparation values, as shown in Fig. 2. The primary cause is that c-axis of the $CaCO_3$ (calcite) contained in Sample 2 is longer than the a- and b-axis, and a preferred orientation was caused during sample filling. Therefore, the reflection of the maximum intensity peak (104) was strongly detected, and the quantification value of calcite was overestimated. With the WPPF method, the effects of orientation can be corrected with an orientation function, and thus results matching well with sample preparation values were obtained for all components.

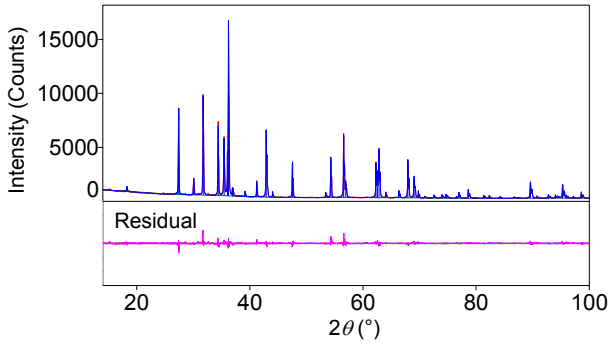

Fig. 1: Results of WPPF analysis of Sample 1

Table 1: Components of measurement sample 1 and the results of quantification using the WPPF and RIR

Components	Amounts of preparation	WPPF	RIR
ZnO	35.8	35.11(10)	34.4(6)
MgO	26.3	25.14(17)	29.8(8)
TiO_2	24.7	25.18(14)	21.6(4)
Fe_3O_4	13.2	14.57(10)	14.2(2)

RIR

ZnO : 5.57 MgO : 3.04 TiO_2 : 3.44 Fe_3O_4 : 5.14

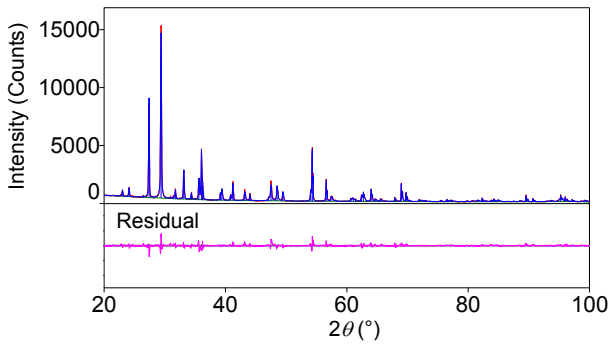

Fig. 2: Results of WPPF analysis of Sample 2

Table 2: Components of measurement sample 2 and the results of quantification using the WPPF and RIR

Components	Amounts of preparation	WPPF	RIR
$CaCO_3$	**43.7**	**43.8(9)**	**62.0(6)**
TiO_2	37.3	37.6(6)	25.3(3)
Fe_2O_3	14.7	13.9(3)	9.79(16)
ZnO	4.3	4.74(18)	2.86(13)

RIR

$CaCO_3$: 3.24 TiO_2 : 3.44 Fe_2O_3 : 3.27 ZnO : 5.57

Recommended equipment and software

- Benchtop X-Ray Diffractometer MiniFlex300/600
- Theta-Theta Type X-ray Diffractometer Ultima IV
- Automated Multipurpose X-ray Diffractometer SmartLab
- high-speed 1D detector D/teX Ultra
- Integrated X-ray Powder Diffraction Software PDXL