APPLICATION REPORT

Rigaku

XRF 173

ZSX Primus SERIES

ANALYSES OF TRACE LEAD IN TITANIUM DIOXIDE POWDER

INTRODUCTION

In recent years, there is a growing demand for environment-friendly products and materials in various industrial fields. Especially, taking account of the influence of trace heavy metals on the environment, many regulations have been established to lower the limits of these components drastically from conventional ones.

The X-ray fluorescence analysis is widely used in process control, quality control and other fields because it can make qualitative and quantitative analyses quickly and non-destructively. Furthermore, with advances in the fundamental parameter method (FP method hereafter), the SQX analysis program to calculate semiquantitative values from qualitative analysis results without using standard samples is also widely used and higher accuracy is required.

We made analyses of trace Pb in TiO_2 powder, which is pigment widely used for cosmetics etc., and obtained satisfactory results. Details are described below:

1. SAMPLE PREPARATION AND INSTRUMENT

1.1 INSTRUMENT

X-ray fluorescence spectrometer ZSX Primus Series

1.2 SAMPLE PREPARATION

Sample was pressed at 200kN using an Al ring to form a pellet.

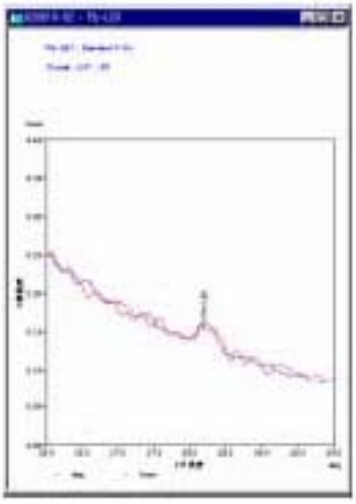
1.3 MEASURING CONDITIONS

Element	F-Mg	Al, Si	P, S	Cl	K, Ca	Pb	Ti-U
X-Ray Tube	End-window Rh tube						
kV-mA	30-100	30-100	30-100	30-100	40-75	50-60	50-60
Filter	Out						
Diameter	30mm						
Slit	S4	S4	S4	S2	S4	S4	S4
Analyzing Crystal	RX25	PET	Ge	Ge	LiF200	LiF200	LiF200
Detector	F-PC	F-PC	F-PC	F-PC	F-PC	SC	SC
PHA	Differential						
X-Ray Path	Vacuum						

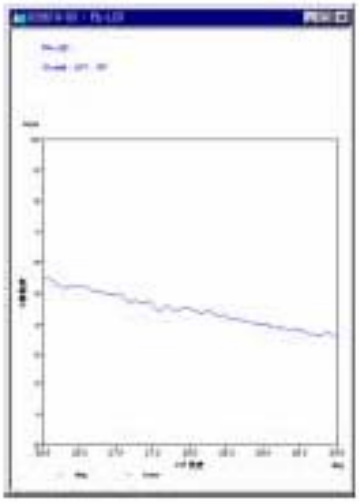
Fixed angle measurement mode: Measurements at peak angle for 10 seconds and at background angles for 10 seconds

2. MEASUREMENT RESULTS

2-1 EFFECT OF COPPER FILTER



a. With Cu Filter



b. Without Primary Filter

When analyzing Pb, the Pb-Lβ1 line is generally used as the analysis line. It is known that a small peak of this spectrum line is difficult to detect using ordinary measuring conditions because it appears in an angle range with high background intensities (see Fig.b on the left).

When it is measured using a primary X-ray filter (Cu), background intensities caused by scattered X-rays lower and the Pb-Lβ1 line can be identified clearly (see Fig.a).

In the analyses described below, the Pb-Lβ1 line was measured using a primary X-ray filter. The fixed angle measurement mode was used to obtain X-ray intensities with smaller statistical errors of counting.

2-2 SQX ANALYSIS RESULTS

Shown below are results of SQX analyses of Pb using the fixed angle measurement mode. Satisfactory results are obtained for the samples in Group A, which contain approximately 10 ppm Pb.

Table 1 SQX Analysis Results (mass%)

	A1	A2	B
Na_2O	0.29	0.29	0.29
MgO	0.086	0.089	0.089
Al_2O_3	-	-	0.029
SiO_2	0.058	0.056	0.061
SO_3	0.052	0.054	0.052
Cl	0.20	0.20	0.30
CaO	0.10	0.10	0.10
TiO_2	99	99	99
Fe_2O_3	0.018	0.018	0.018
ZrO_2	0.022	0.022	0.021
Nb_2O_5	0.18	0.18	0.19
PbO	0.0011	0.0009	0.0020

-: Not detected

However, care must be taken about the propagation of the measurement errors of other components because in the SQX analysis a calculation is made for all detected components.

3. CONCLUSION

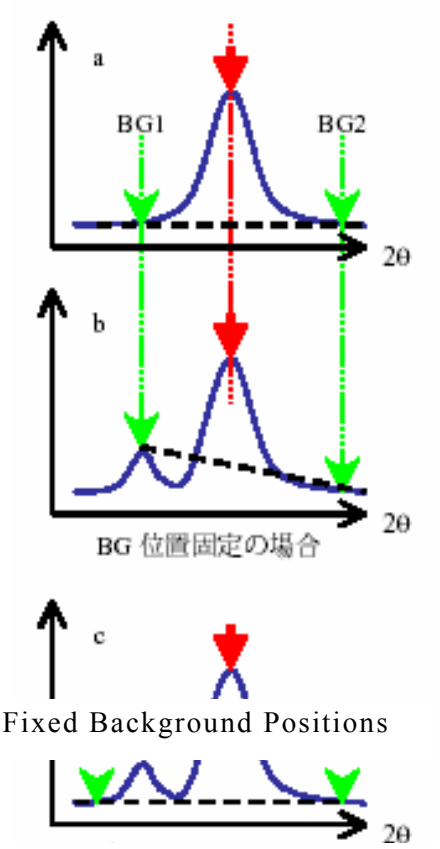
By using the primary X-ray filter and the fixed angle measurement mode, satisfactory SQX analysis values were obtained even for trace components difficult to detect using the qualitative analysis. We think that the SQX analysis, which does not need standard samples, is a method suitable for analyses of trace heavy metals.

Reference Material: Background Measurement Using Fixed Angle Measurement Mode

In the ordinary fixed angle measurement, background positions must be specified beforehand. When BG1 and BG2 are specified as background positions, in the case of Fig.a an accurate net intensity can be obtained because background processing is performed correctly. However, in the case of Fig.b background processing becomes unsuitable and an accurate net intensity cannot be obtained because another spectrum overlap with BG1.

In the fixed angle measurement mode, background measurement positions need not be specified beforehand. To cope with the above-mentioned defect, the program automatically determines the suitable background positions BG1 and BG2 and carries out counting during a predetermined measurement time, referring to a qualitative analysis chart that has been prepared in advance (Fig.c).

Therefore, suitable background processing can be done for samples of any matrices.



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