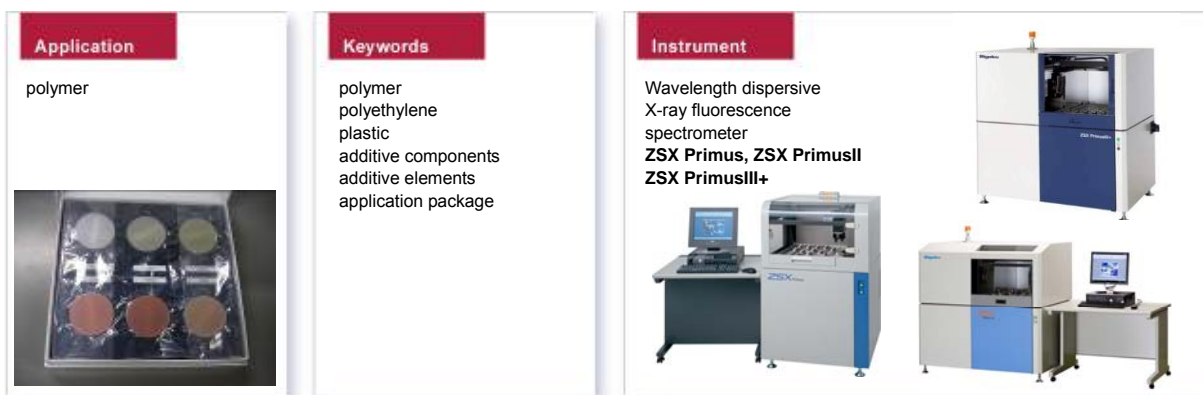


Analysis of Additive Elements in Polymers Using a ZSX Primus Series/Application Package



Introduction

This application note introduces an Application Package for quantitative analysis of additive elements in polymers. The Application Package includes calibration standards and drift correction samples required for the quantitative analysis, the step-by-step instruction manual, etc. This Polymer Application Package makes it easy to set up the quantitative analysis application by the use of polyethylene disk specimens as calibration standards.

Instrument

The Polymer Application Package is applicable to Rigaku sequential wavelength-dispersive XRF spectrometer, ZSX Primus, ZSX PrimusII and ZSX PrimusIII+. For Mg, Na and F analysis, an optional high-sensitivity crystal RX35 is required. In addition, an optional Ge crystal is needed for the ZSX PrimusIII+. The analysis area is 30 mm in diameter.

Analysis component and calibration range

Table 1 lists the analysis components (elements) and their calibration ranges of this Application Package.

Contents of this Application Package

The measurement conditions shown in Table 2, standard values of six set-up standard samples, various correction coefficients and conditions of two drift cor-

rection samples are automatically set by running the installation CD. After the installation, the instruction manual provides simple, step-by-step instructions on how to set up a quantitative application, which enables less-experienced users to generate accurate calibration curves. Figure 1 shows the outline of this Application Package.

Table 1 Analysis element and calibration range

Analysis element	Calibration range (ppm)
Zn	(1) – 206
Fe	(1) – 105
Cr	(1) – 96
Ti	(1) – 81
Ca	3 – 223
(Cl: reference)	(13) – (218)
S	(1) – 125
P	1 – 85
Si	(1) – 808
Al	(1) – 465
Mg	(1) – 796
Na	(1) – 311
(F: reference)	(35) – (201)

Numbers in parentheses denote reference values.

Table 2 Measurement condition

Element	Zn	Fe	Cr	Ti	Ca	Cl	S	P	Si	Al	Mg	Na	F	
Line	K α													
kV-mA	40 - 50													
Primary beam filter	Al125			Al25			Out							
Slit	S4					S2		S4						
Analyzing crystal	LiF(200)					Ge			PET		RX35			
Detector	SC			F-PC										
Counting	Peak(s)	20	20	20	20	20	20	20	20	20	20	20	20	100
time	BG (s)	20	10x2	10x2	20	20	20	20	10x2	10x2	10x2	20	10x2	100

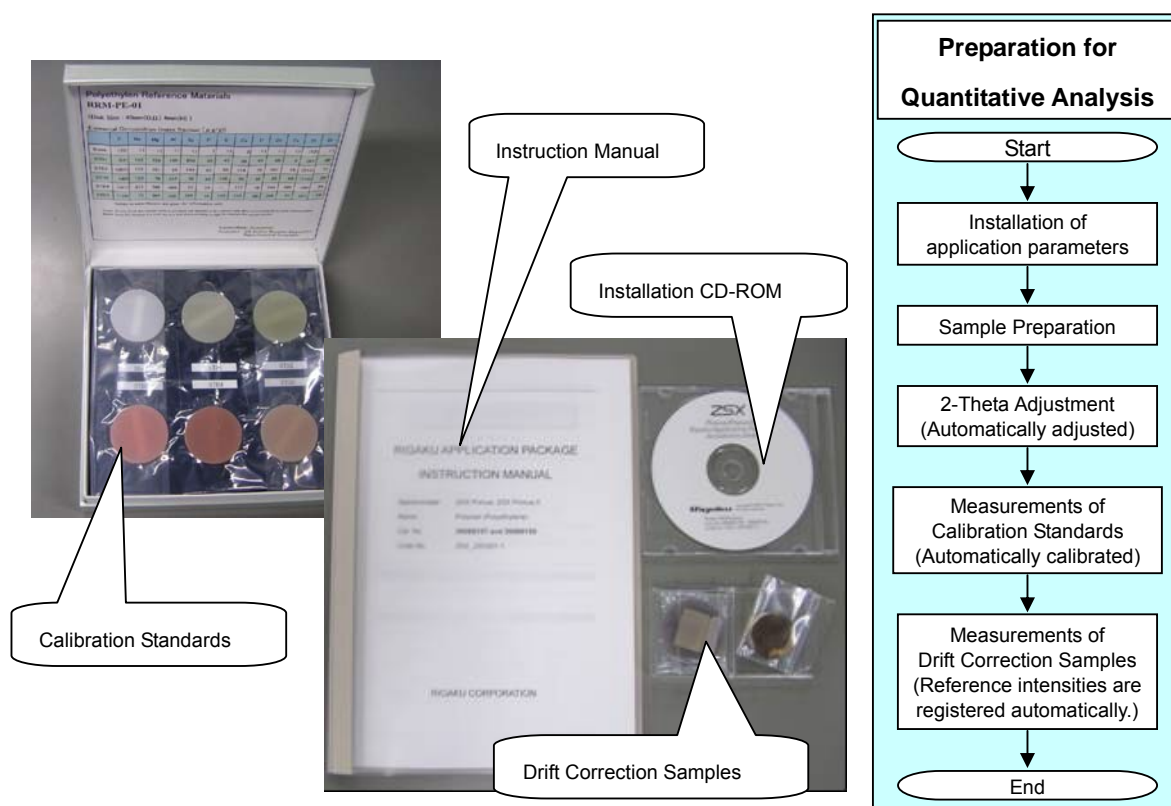


Figure 1 Outline of Polymer Application Package

Sample preparation

Before measurement, the analysis surface of each sample is polished using sand paper and then cleaned with ethanol.

Calibration

Calibration curves and their accuracies for typical elements are shown in Figure 2.

The accuracy of calibration is calculated by the following formula,

$$\text{Accuracy} = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n - m}}$$

C_i : calculated value of standard sample

\hat{C}_i : reference value of standard sample

n : number of standard samples

m : degree of freedom .

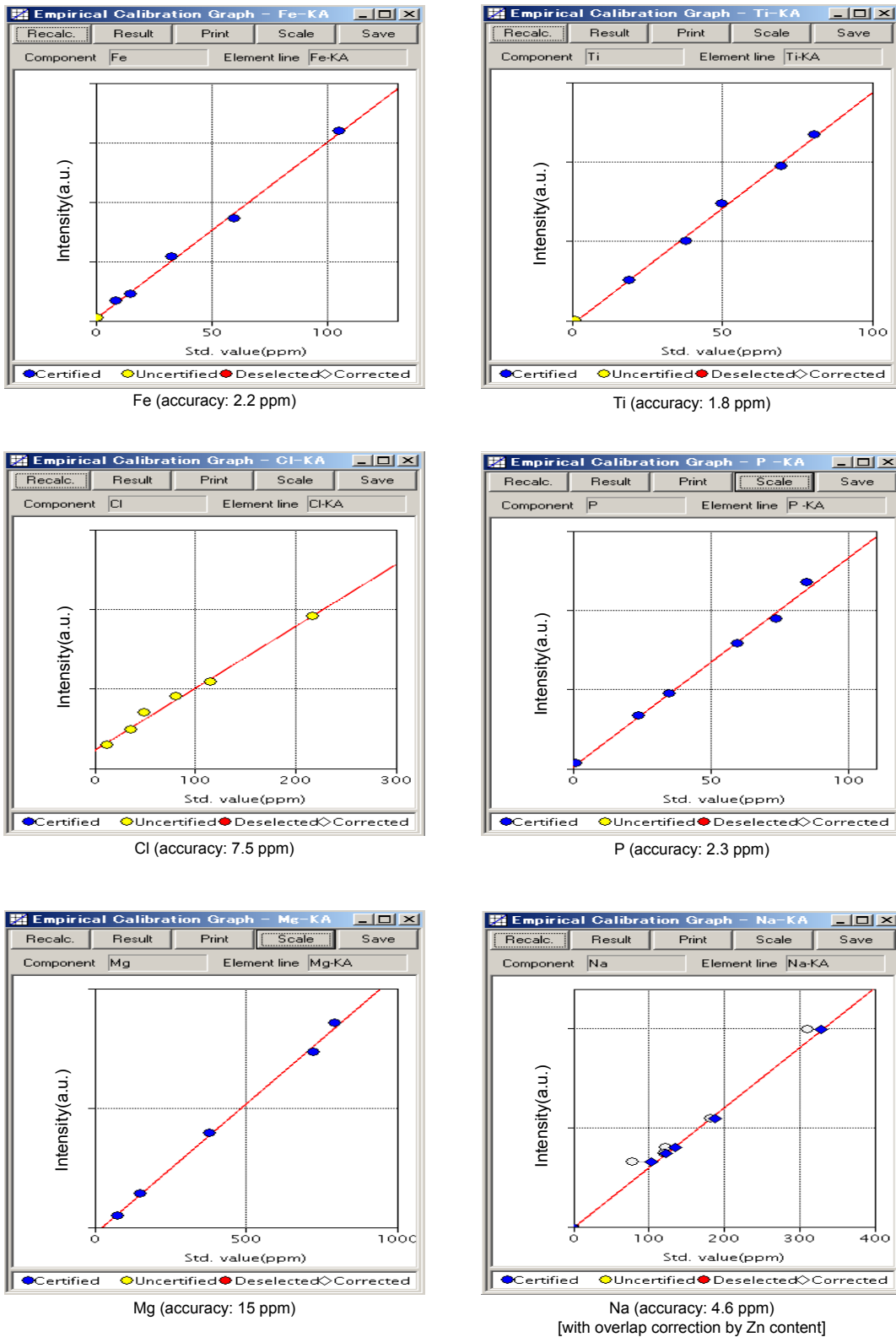


Figure 2 Calibration curves and their accuracies for typical elements

Repeatability test

Table 3 shows the test results of 5-time measurements with sample preparation on the ZSX PrimusII.

Table 3 Repeatability test result (unit: ppm)

Element	Zn	Fe	Cr	Ti	Ca	Cl	S
Calibration range	(1) – 206	(1) – 105	(1) – 96	(1) – 81	3 – 223	(13) – (218)	(1) – 125
Conc. (1)	28	60	20	38	58	(115)	106
Std. dev.	0.2	0.9	0.5	0.4	0.4	(1.5)	1.0
Conc. (2)	206	33	54	50	223	(37)	125
Std. dev.	0.5	0.5	0.5	0.4	0.6	(1.5)	1.1
Element	P	Si	Al	Mg	Na	F	
Calibration range	1 – 85	(1) – 808	(1) – 465	(1) – 796	(1) – 311	(35) – (201)	
Conc. (1)	85	42	337	76	120	(42)	
Std. dev.	0.8	2.9	2.0	2.0	4.5	(9)	
Conc. (2)	74	305	225	380	78	(139)	
Std. dev.	1.3	7.2	1.8	3.2	3.8	(14)	

Numbers in parentheses are reference values.

Conclusion

Polymer Application Package, introduced in this application note, makes it possible to easily set up a calibration for additive elements in polyethylene-base samples. This Application Package is effective for analysis of polymers similar in composition to polyethylene.



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