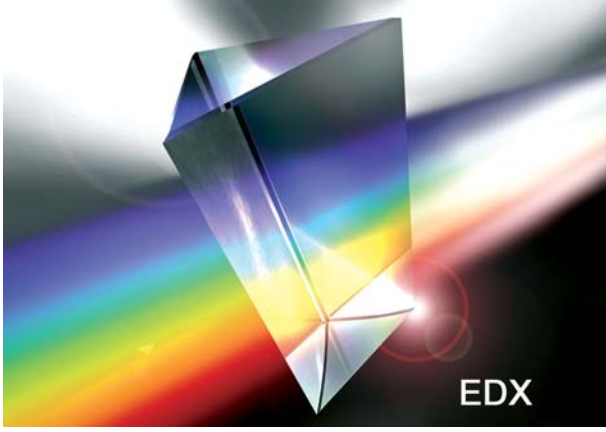
Application Note



Analysis of Ceramics

Description

In this application note, Ceramic powder is analyzed for a wide elemental range. Elements present in a Ceramic are often present in the form of oxides. Various oxides in ceramics are measured in two ways. The first, a standardless approach, uses fundamental parameters to provide elemental content. The second approach, purely empirical, creates a calibration line based on the intensity emitted from each element for a number of standards.

These two methods are compared. It is very clear from the results, that in many cases fundamental parameters can be a very accurate method of analyzing an unknown samples. The ceramic powder, is created from a pre-existing ceramic bulk by means of a sample grinder available from either Kratos or Spex Certiprep.

Semi-Quantitative Analysis

The following are spectrum from a certified ceramic standard, R-603, available from Shimadzu, Japan.

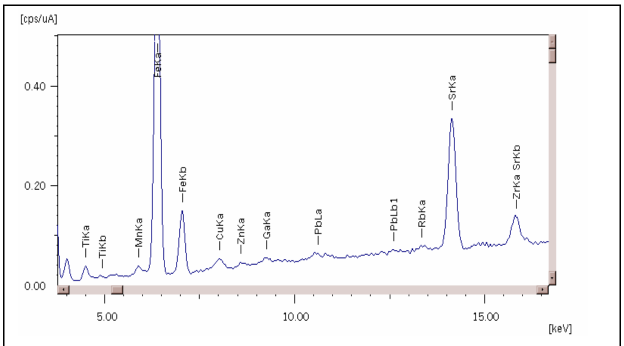


Fig. 1. Spectrum of Oxides in Ceramic Powder

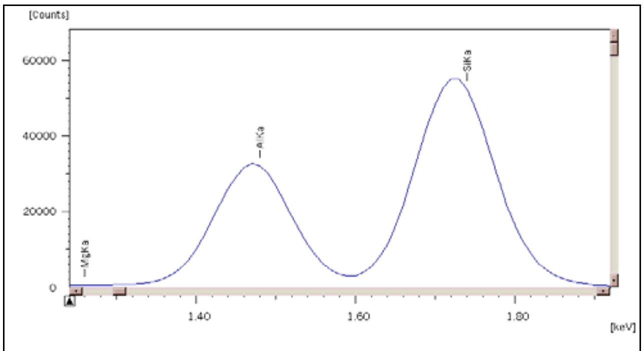


Fig. 2. Al_2O_3, and SiO_2, spectrum in Ceramic Powder

The results in Table 1, shows the ability of Shimadzu's Fundamental Parameter software to quantitatively analyze, an unknown sample, without the use of standards.

	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	Na_2O	MgO	TiO_2
Measured Value(%)	46.6	37.2	1.69	0.60	0.42	0.34	0.31	0.09
Certified Value(%)	46.1	37.0	1.66	0.65	0.40	0.84	0.29	0.08

	SO_3	MnO	CuO	Ga_2O_3	PbO	ZnO	Rb_2O	*Ignition Loss*
Measured Value(%)	0.05	0.03	0.01	0.005	0.005	0.003	0.002	12.60
Certified Value(%)	-----	------	------	------	------	------	------	------

Table1. Comparison of Semi-Quantitative results to Certified Elemental Concentration.

Quantitative Analysis

Although, the Fundamental Parameter method is very accurate in determining elemental concentration, it is often beneficial to use an empirical method. There can be many reasons for using an empirical approach. Most notably, is accuracy. An empirical method almost always provides the user with a higher accuracy. This is often due to physical differences between sample lots, such as particle size.
Many times, analysis, is required to conform to an ISO-900x regulation. In this case, Quality Control (QC) procedures are required to be documented.
Due to the complex nature of the FP method, this is not always possible, and if it is, often it is not straightforward. An empirical method, as used on the EDX-700/800 reports, to the user, a complete set of parameters associated with each Quantitative method. These results are clearly understood by the user and can be used for ISO-900x documentation.
The figures below show examples of empirically created calibration lines that are used for oxides measured in ceramic analysis.

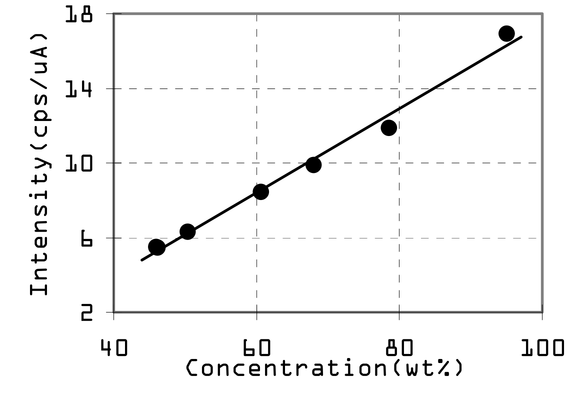


Fig. 3. Calibration Line for SiO_2

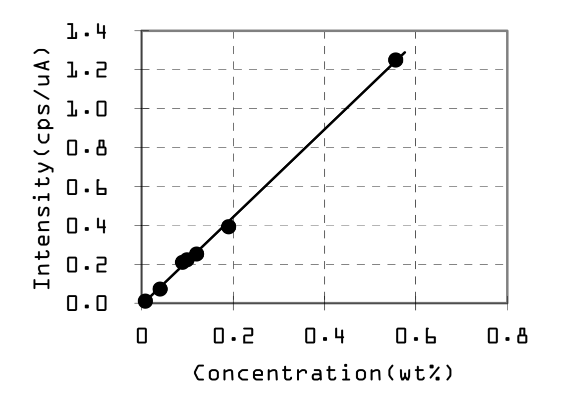


Fig. 4. Calibration Line for TiO_2

Repeatability of Procedure

The samples were measured a number of times, n = 10, to determine the precision of the analysis tool. There are two values associated with a precision measurement. The first, is standard deviation, and is the absolute precision of the instrument. The second value, the coefficient of variation, is the relative precision of the instrument. The results, for R-603 (certified ceramic standard), are shown below in Table 2.

Table 2 Result of Repeatability Test for R-603

Compound	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
Average(%)	47.10	36.89	0.67	0.099	1.62	0.22	0.67	0.41
Standard Deviation(%)	0.061	0.150	0.004	0.004	0.010	0.015	0.091	0.010
Coefficient of Variation(%)	0.13	0.41	0.61	4.0	0.60	7.7	13.7	2.5

Measuring Conditions :

Instrument : EDX-700 X-ray Tube : Rh Filter : None Atmosphere : Vacuum	Power: 15-50 kV at 30 - 250 µA Dead Time 25 % Measurement Diameter 10 mm Measurement Time 40 Seconds