Application Note EDXRF1033

View on rigaku.com

EDXRF1033 - Chlorine & RoHS Elements In Polyethylene

Scope

This Application Note shows performance for the elemental analysis of Cl and elements regulated by RoHS in plastics. Analysis is shown for polyethylene. Empirical calibration summary and detection limits are shown and instrument repeatability is demonstrated.

Background

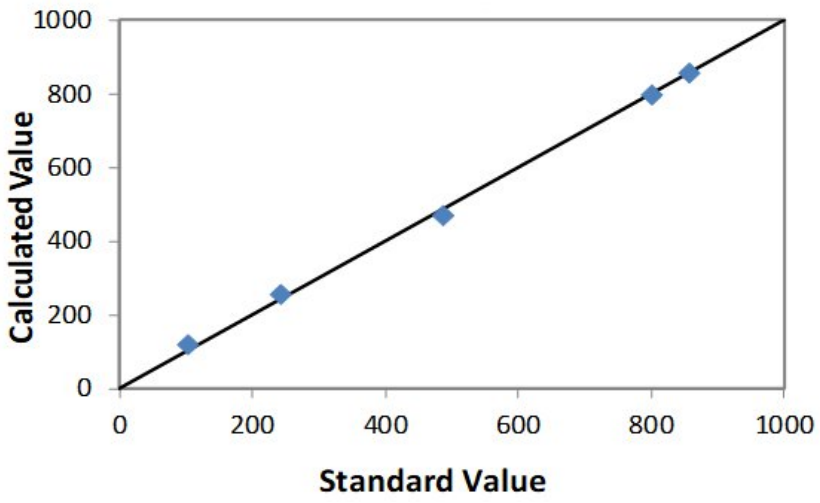
The Restriction on Hazardous Substances initiative (RoHS) has been in force for several years. RoHS limits the allowable amounts of various toxic elements in plastics and consumer goods. The latest extension to the RoHS regulatory guidelines is the measurement and control of Total Halogens, with particular emphasis on the chlorine content. EDXRF is an accepted analysis technique for the screening and quantification of the hazardous element according to RoHS norms. To meet the industry challenge, Rigaku offers the NEX CG EDXRF analyzer using indirect excitation and polarization, giving QA/QC processes the means for fast and simple screening and analysis of materials that must conform to RoHS and similar directives.

Model: NEX CG

Cl Calibration

Six reference standards were used for empirical calibration of Cl. The samples also contained unassayed amounts of the other elements. These elements were also measured and used to enable alpha corrections, which compensate for variations in X-ray matrix effects within the sample.

Element: Cl **Units: mg/kg**		
Sample I.D.	**Standard value**	**Calculated value**
EC680k	102	122
CLPE-2018	243	259
CLPE-3018	486	472
EC681k	800	800
CLPE-4018	856	856



Correlation plot Cl

Cl Repeatability

To demonstrate instrument precision, ten repeat analyses were performed with samples in static position.

Element: Cl **Units: mg/kg**				
Sample ID	**Standard value**	**Average value***	**Std dev**	**% Relative**
EC680k	102	121	1	0.8
CLPE-2018	243	261	1	0.4
CLPE-3018	486	479	3	0.6
EC681k	800	799	7	0.8
CLPE-4018	856	851	4	0.5

* Average value reflects the calculated value from the calibration.

Multi-element calibrations

Three of the certified standards were assayed for As, Br, Cd, Cl, Cr, Hg, Pb, S, Sb, Ba, and Se. A calibration was created for each element. Depending on the combination of elements in each standard, 1-, 2- or 3-point calibration curves were established for the particular element series. All samples were measured against the calibrations to ensure effective recovery.

Multi-element repeatability

To demonstrate instrument precision, ten repeat analyses were performed with samples in static position.

Element: As **Units: mg/kg**	

Sample ID	Std value	Avg value*	Std dev	% Relative
EC-680k	4.1	4.0	0.1	2.9
EC-681k	29.1	30.1	0.2	0.6
EN 71-3	50.0	50.5	0.2	0.3

Element: Cr
Units: mg/kg

Sample ID	Std value	Avg value*	Std dev	% Relative
EC-680k	20.2	20.4	0.4	1.8
EC-681k	100.0	92.9	0.7	0.7
EN 71-3	102.0	102.0	0.4	0.3

Element: Cd
Units: mg/kg

Sample ID	Std value	Avg value*	Std dev	% Relative
EC-680k	19.6	17.3	1.0	5.9
EC-681k	137.0	148.1	1.8	1.2
EN 71-3	290.0	297.7	4.2	1.4

Element: Pb
Units: mg/kg

Sample ID	Std value	Avg value*	Std dev	% Relative
EC-680k	13.6	15.9	0.4	2.23
EC-681k	98.0	94.9	0.6	0.6
EN 71-3	151.0	156.2	0.5	0.3

Element: Br
Units: mg/kg

Sample ID	Std value	Avg value*	Std dev	% Relative
EC-680k	96.0	95.1	0.4	0.5

EC-681k	770.0	779.0	0.9	0.1

Element: Hg **Units: mg/kg**				
Sample ID	**Std value**	**Avg value***	**Std dev**	**% Relative**
EC-680k	4.6	3.5	0.3	7.4
EC-681k	23.7	26.1	0.9	3.3
EN 71-3	101.0	102.5	0.6	0.6

Element: Ba **Units: mg/kg**				
Sample ID	**Std value**	**Avg value***	**Std dev**	**% Relative**
EN 71-3	707.0	714.6	5.3	0.7

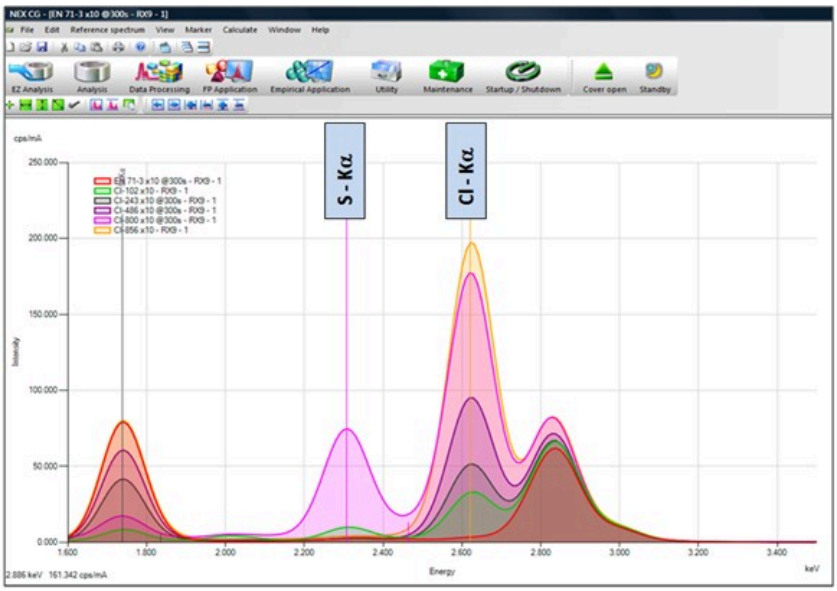
Element: Sb **Units: mg/kg**				
Sample ID	**Std value**	**Avg value***	**Std dev**	**% Relative**
EC-680k	10.1	9.4	1.3	14.0
EC-681k	99.0	103.4	2.3	2.2
EN 71-3	96.0	122.6	2.2	1.8

Element: S **Units: mg/kg**				
Sample ID	**Std value**	**Avg value***	**Std dev**	**% Relative**
EC-680k	76.0	76.4	0.7	0.9
EC-681k	630.0	629.3	1.3	0.2

Element: Se **Units: mg/kg**				
Sample ID	**Std value**	**Avg value***	**Std dev**	**% Relative**
EN 71-3	204.0	204.5	0.5	0.2

Discussion

Review of spectral information shows that the Cl Kα peak is isolated and does not have any significant overlaps within the samples. This allows for accurate and repeatable results throughout the analysis range. An overlay of the spectrum from each sample is provided below:



Conclusion

The Rigaku NEX CG combines indirect excitation with secondary targets, polarization targets and a high performance SDD detector to yield the optimum performance in EDXRF instrumentation. The results shown here indicate the NEX CG is an excellent tool for the measurement of Cl and the RoHS elements in polyethylene. Similar performance can be expected in other non-PVC plastics. The NEX CG is wells-suited for QA checks of incoming material and QC of process control, as well as R&D of plastics formulations.