

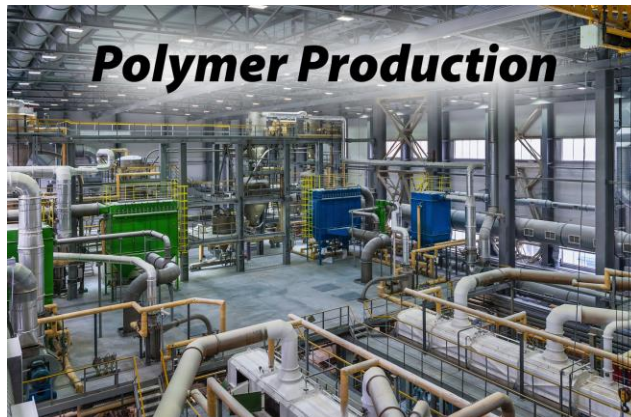
SCOPE

The measurement of trace levels of Al, Si, P, Cl, Ti and Ca in polypropylene is demonstrated.

BACKGROUND

In the production of polymers such as polypropylene (PP), polyethylene (PE) and polyethylene terephthalate (PET, PETE) various additives and stabilizers are added, as well as Si (as SiO₂) as an anti-block. Trace levels of catalyst residues may also be present. To ensure proper quality control during the production process test samples are made and the trace elements are measured. To

meet this industry need Applied Rigaku Technologies offers a full line of benchtop EDXRF analyzers. With simple, intuitive software interface and powerful, innovative design, Rigaku EDXRF systems make an excellent tool for quality control check at several places along the polymer production process.



INSTRUMENTATION

Model:	NEX QC ⁺ QuantEZ
X-ray tube:	4 W Ag-anode
Detector:	SDD
Sample Type:	Solid, homogeneous pucks
Film:	4um polypropylene
Environment:	Helium
Analysis Time:	600 seconds total
Optional:	6-position 32mm or 5-position 40mm automatic sample changer

 **NEX QC⁺**
QUANTEZ



Total analysis time: 600 sec (300 sec for Al, Si, P, Cl and 300 sec for Ca, Ti)

SAMPLE PREPARATION

Test coupons are prepared as solid, homogeneous circular pucks of consistent dimension. Samples are placed directly in the analysis chamber for measurement.

CALIBRATION

Empirical calibration is used. Because all elements are at trace levels, X-ray absorption/enhancement effects are negligible and can be ignored for process quality control. Each calibration standard can include all elements dispersed independently, or series of single element standards can be used to make calibrations. In this demonstration series of single element standards were used, with the calibration results summarized here.

Element	Number of Standards	Concentration Range	RMS Deviation	R ² Confidence
Al	3	76 – 230 ppm	11.4	0.99508
Si	5	0.018 – 0.097 mass%	0.0030	0.99649
P	4	36 – 92 ppm	1.3	0.99907
Cl	5	24 – 103 ppm	5.5	0.99100
Ca	4	38 – 188 ppm	0.8	0.99995
Ti	2	3.7 – 9.2 ppm	N/A	N/A

REPEATABILITY (Precision)

Typical precision is shown here. Ten repeat analyses of each middle concentration sample were performed with the sample in static position using a total analysis time of 600 sec per analysis.

Element	Units	Assay Value	Average Value	Std Dev	% Relative
Al	ppm	109	114	11	10%
Si	mass%	0.0597	0.0584	0.0010	1.7%
P	ppm	51	55.2	2.4	4.7%
Cl	ppm	54	52.0	0.7	1.3%
Ca	ppm	74	73.8	1.1	1.5%
Ti	ppm	3.7	3.8	0.3	8.1%

DETECTION LIMITS

The empirical method was used to determine detection limits. In the empirical method, ten repeat analyses of a blank sample are taken with the sample in static position, and the standard deviation is determined. The Lower Limit of Detection (LLD) is then defined as three times the standard deviation. Typical LLD are shown here. Detection limits depend on several factors including analysis time, overall composition of the limestone and calibration concentration ranges.

Typical LLD 600 sec Total Analysis Time

Element	LLD	Condition Count Time
Al	26 ppm	300 sec
Si	0.0015 mass%	
P	5.7 ppm	
Cl	2.0 ppm	
Ca	1.5 ppm	300 sec
Ti	1.2 ppm	

CONCLUSION

EDXRF provides a rapid, non-destructive means of semi-quantitative measurement for screening, identification, as well as elemental quantification of metals, solids, powder, pellets, thin films and liquids. The NEX QC⁺ QuantEZ powerful yet simple and intuitive software gives the quality control lab and at-line technicians a fast means of making QC checks along the production process line in the manufacturing of polypropylene and other similar polymers.