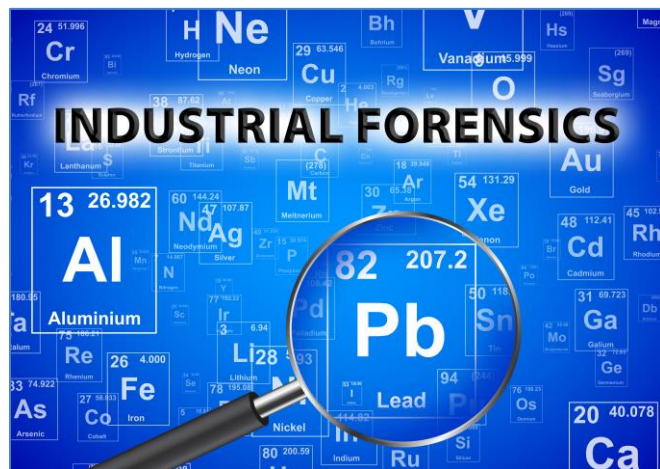


SCOPE

Use of Rigaku NEX DE VS EDXRF analyzer is demonstrated for industrial forensics in the investigation of unknown sample materials.

BACKGROUND

XRF is an analysis tool used for non-destructive analysis in industrial forensics to identify and resolve manufacturing issues or contamination within the production and distributor processes. Analysis using XRF gives the operator a way to determine elemental composition of foreign material in failure analysis and root cause analysis to optimize quality control and testing procedures.



EDXRF is a fast and simple means of obtaining elemental composition of samples investigated in industrial forensics. Samples analyzed are often irregularly shaped, small or available in only small quantity. Rigaku NEX DE VS EDXRF analyzer is an excellent tool equipped with small spot size measurement, camera image and powerful yet simple-to-use software for the investigation and identification of foreign material.

INSTRUMENTATION

Model:	Rigaku NEX DE VS
X-ray tube:	12W 60kV Ag-anode
Primary Filters:	Automatic tube filters for optimum background removal
Detector:	High Throughput SDD 500,000+ cps
Collimators:	10, 3 and 1mm spot size, automatic switching
Camera:	High resolution for sample positioning and automatic capture of sample image
Analysis Time:	Adjustable
Environment:	Air, Helium Purge

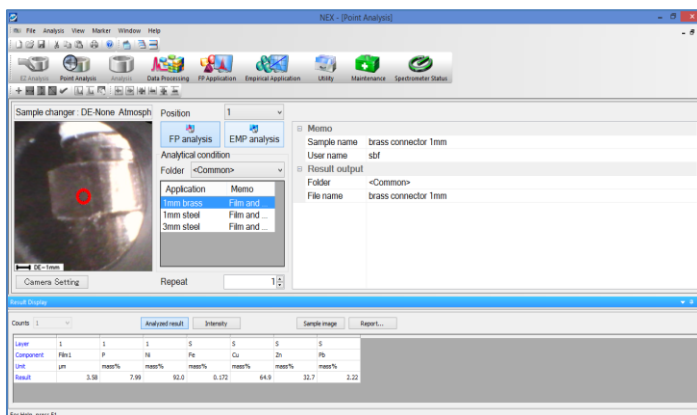


SAMPLE PREPARATION & PRESENTATION

Samples measured for forensics and failure analysis are typically irregular shapes, small pieces, small areas of a large piece, or small amounts of powder.



Large measurement chamber for positioning unusually shaped sample. Autosampler trays available for use with sample cups.



Point Analysis screen for measuring samples requiring small spot analysis and camera image.



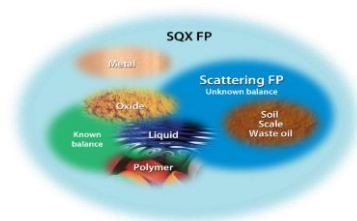
Small spot sample cups are used for pieces of small solid samples or small amount of powder.



Example of very small amount of powder prepared using the film sandwich sample cup method.

FUNDAMENTAL PARAMETERS (FP)

Industrial forensics involves measuring samples of unknown material and unknown elemental composition. Semi-quantitative analysis (SQX analysis) is employed to analyze samples for forensics examinations using FP (fundamental parameters) methods to calculate concentration results.



Rigaku RPF-SQX Fundamental Parameters (FP) uses an advanced program that automatically deconvolutes spectral peaks and models the sample matrix using fundamental XRF equations, including unique Scattering FP approach for analyzing lighter materials like powders and polymers that have an unknown balance component that cannot be directly measured.

SAMPLE MODEL SELECTION

Rigaku NEX QuantEZ software includes many application templates for the material categories. Templates include:

- Metals and Alloys
- Powders and Pellets
- Polymers
- Filters and Thin Films
- Water, Oils and Liquids

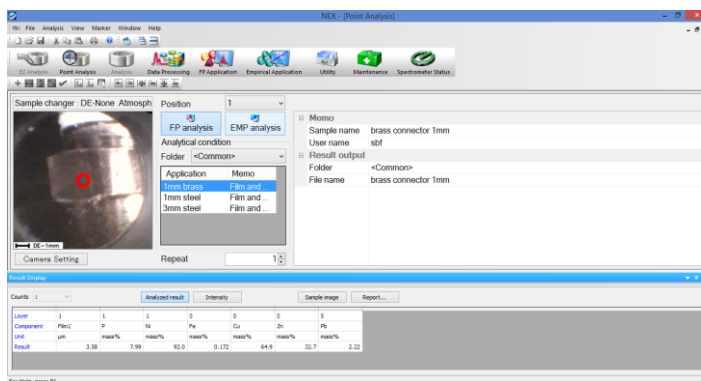
FP models include bulk analysis, analysis of light materials and thin films, and Rigaku Scattering FP.

Templates can be used as is for general screening, while also flexible in design allowing users to change and craft an application to meet specific needs.

Optional Material Identification software for alloy identification can also be used to identify alloy type of unknown metals and foreign matter using the standard ASTM and JIS tables for ferrous, aluminum and copper-based alloys. Custom identification tables can also be made by the user.

SMALL SPOT ANALYSIS

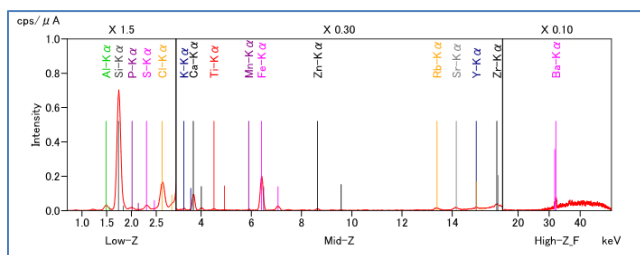
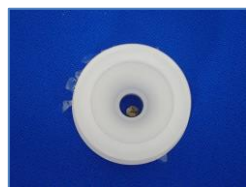
Samples that do not completely cover the measurement aperture require small spot size analysis. NEX DE VS is equipped with 10mm, 3mm, and 1mm automatic switching collimators to focus the analysis on the small spot required to be measured.



EXAMPLES

Chip of foreign material in rice

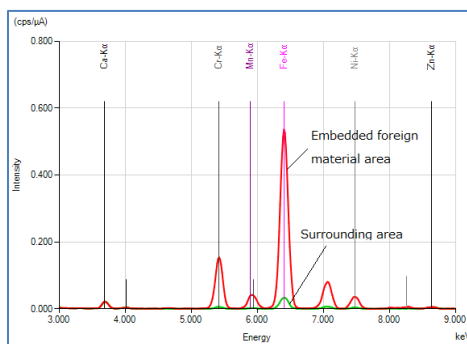
In this example, foreign matter was found in rice. Analysis was made by placing the sample in a small-spot sample cup. Measurement results using small spot point analysis indicated elemental composition consistent with a stone or a pebble, information that is helpful in tracing the origin of the source of contamination.



Component	Content (mass%)
SiO ₂	74.9
CaO	9.25
Al ₂ O ₃	8.80
Fe ₂ O ₃	1.35

Metal shard foreign material

In this example, a metal shard is examined, prepared using the film sandwich sample cup method and small spot analysis. Results of the measurement of the shard compared to the surrounding area where it was found indicated an Fe-based metal alloy. Material Identification software verified the shard was a piece of stainless steel, and important clue in foreign matter investigation of a product or failure analysis of machinery.

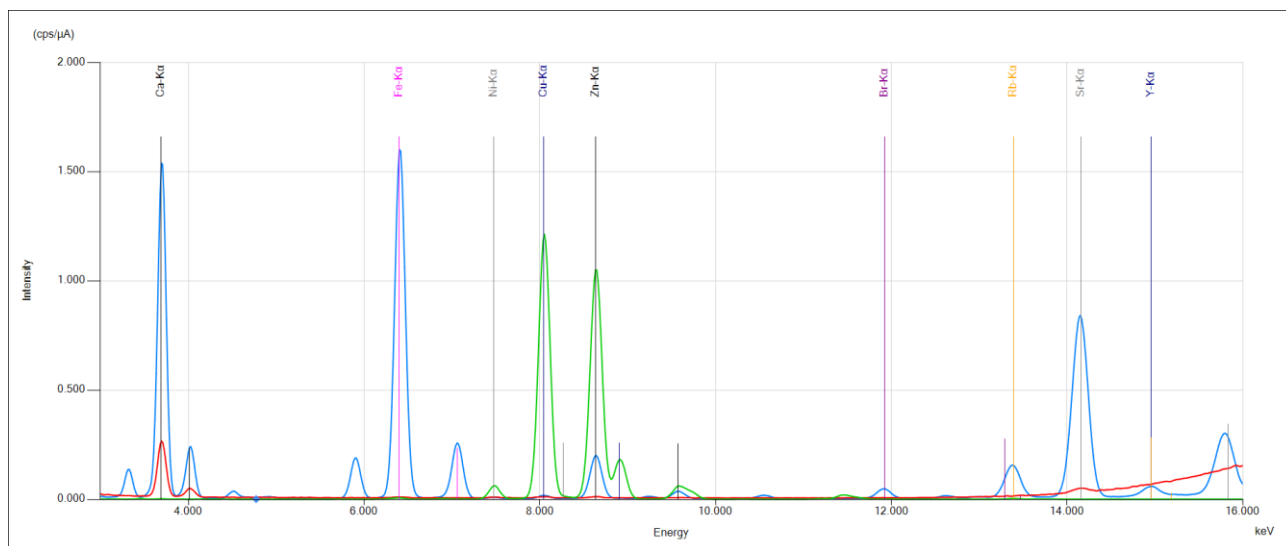


Material Identification Result				
Standard	[All]			
Category	[All]			
No.	Matching	Standard	Category	Material type
1	98.32	JIS	for special purposes	SUS 304 J 1
2	98.10	JIS	for special purposes	SUS 301
3	98.10	JIS	for special purposes	SUS 301 L
4	97.94	JIS	for special purposes	SUS 301 J 1

Component	Content (mass%)
Fe	71.2
Cr	16.9
Ni	6.92
Mn	2.52

QUALITATIVE SPECTRA ANALYSIS

Spectra can be easily overlapped for comparison purposes, manufacturing forensics and failure analysis testing. Zoom controls and KLM markers identify element peaks for qualitative analysis.



CONCLUSION

Innovative design and simple, intuitive software makes the NEX DE VS an ideal EDXRF analyzer for use in industrial forensics. Fundamental Parameter methodology with small spot measurement capability to analyze irregularly shaped samples give the operator valuable tools in the investigation of identification of foreign material of unknown composition.

Reference

Foreign material analysis using energy dispersive x-ray fluorescence spectrometers, Rigaku Journal, Vol. 35 No. 1, Winter 2019