Rigaku

SCOPE

The measurement of Pt, Rh and Pd in recycled automotive catalytic converters is demonstrated using Fundamental Parameters (FP) approach and pre-installed starter Matching Library.

BACKGROUND

Used catalytic converters are collected and recycled in order to reclaim the precious metals Pt, Rh and Pd. Typically, the entire honeycomb core is removed and ground into a powder. Once in powder form, the precious metal catalysts can be extracted, or the powder can be sold to a refiner.

The honeycomb core, also called the monolith, is typically ceramic, containing combinations of the elements Al, Si, Fe, Zn, Sr, Zr, Ba, La and Ce. Some cores can be made of a stainless steel honeycomb and so would contain higher Fe and other metals. Catalyst material may also be high in Pb content, in regions where leaded gasoline is still used. Because all of these elements can occur at various levels, Rigaku's software offers quick direct analysis and easy use for crafting individual methods, as desired.

NEX CG gives the user expanded capabilities, including higher sample batch throughput using 15-position autosampler, as well as more powerful and flexible FP method for crafting specific methods and Matching Libraries for optimum accuracy of specific materials. The versatile software of the NEX CG also allows for screening of totally unknown samples without any reference material in a library.

INSTRUMENTATION

Model:	Rigaku NEX CG
X-ray tube:	50 W Pd-anode
Excitation:	Indirect with polarization
Detector:	High performance SDD
Analysis Time:	200 seconds
Autosampler:	15-position
Environment:	Air
Options:	Manual Sample Press
	9-position Sample Spinner Tray

SAMPLE PREPARATION

Samples are ground and measured as dry, homogeneous powder. It is not necessary to make hydraulically pressed pellets. A ball mill or mixer mill may be used to take the material down to a powder. For analysis, about 5g of powder is poured into an XRF sample cup. To ensure consistent compaction from sample to sample, it is recommended that the samples be compacted with a Manual Sample Press at 250 inch-pounds of torque.

Dry Powder Sample

XRF Sample Cup

Manual Sample Press

FUNDAMENTAL PARAMETERS and MATCHING LIBRARY

For screening purposes Fundamental Parameters (FP) is used which calculates concentration based on theory. A factory installed Matching Library for mixed, homogenized materials is included with the analyzer, using twelve samples that have been fire assayed, including two certified NIST standards. FP uses the assayed values and measured information to more closely match theoretical intensities to measured intensities, thus providing a more accurate model of the real matrix.

The software is simple and intuitive, allowing the user to expand on the starter Matching Library, or design new Matching Libraries to suite various specific converter materials. Empirical calibrations can also be crafted in order to ensure the optimum accuracy for a specific material type.

The factory starter Matching Library covers the following ranges for Pt, Pd and Rh
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Element	Concentration Range (ppm)
Pt	400-6230
Pd	260-6445
Rh	0-365

TYPICAL RESULTS

To demonstrate the results using the starter Matching Library, two unknown field samples that had associated fire assay numbers were measured that were not included in the Matching Library.

Sample 9	Units: ppm		
Element	Fire Assay Value	NEX CG Result	% Relative Difference
Pt	392	360	8.2%
Pd	509	553	8.6%
Rh	94	94	1.1%

Sample 12	Units: ppm		
Element	Fire Assay Value	NEX CG Result	% Relative Difference
Pt	1336	1297	2.9%
Pd	922	964	4.6%
Rh	331	330	0.3%

In general, when using a library of similar materials typical accuracy is approximately 10-15% relative or better. Accuracy can be improved by augmenting the Matching Library with additional samples and using longer analysis count times. Highest accuracy is achieved by building separate methods for each specific material. The simple and intuitive software of the NEX CG allows the user to easily create new Matching Libraries and methods. The versatility of the NEX CG software also allows for screening totally unknown samples without the use of reference materials and libraries. This standardless approach typically gives accuracy approximately 20% relative.

RECOVERY & PRECISION

The European reference standard was selected to demonstrate repeatability (precision) and is considered a check sample, not part of the factory Matching Library. ERM-EB504E was measured in static position for ten repeat measurements. Typical performance results are shown below.

Sample ID: ERM-EB504E	Units: ppm		
Component	Certified Value	NEX CG Average Value	Standard Deviation
Pt	1777	1780	53
Pd	279	302	9
Rh	338	363	9

CONCLUSION

Determination of the precious metals content of recovered catalytic converter cores is critical to accessing the valuations needed in this specialized business within the automotive recycling stream. The NEX CG software allows for more versatile design of methods and libraries, as well as standardless analysis capabilities for screening. Thus, the Rigaku NEX CG and simple yet advanced software, provides an easy-to-use and valuable tool for the catalytic converter recycling industry.