

EDXRF APPLICATION NOTE

Pt, Pd, Rh IN RECYCLED AUTOMOTIVE CATALYSTS

AUTO CATS-PAK

#1943

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 as the Rigaku Auto Cats-Pak.

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 QC⁺ gives the user flexible FP templates for crafting specific methods and Matching Libraries for optimum accuracy of specific materials. The versatile software of the NEX QC⁺ also allows for semi-quant screening of totally unknown samples without any reference material in a library.

INSTRUMENT CONFIGURATION

| | |
|-----------------------------|------------------------------------|
| Model: | Rigaku NEX QC ⁺ QuantEZ |
| Excitation: | Direct with Filters |
| X-ray tube: | 4W Ag-anode |
| Detector: | High-performance SDD |
| Atmosphere: | Air |
| Sample Film: | 4 µm polypropylene |
| Sample cup: | 32 mm cup |
| Sample Tray: | 6 position Automatic Sample Tray |
| Compaction: | Manual Compaction Press |
| Total Analysis Time: | 300 Sec |



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 Compaction Press at 250 inch-pounds of torque.



Dry Powder Sample



XRF Sample Cup



Manual Compaction Press

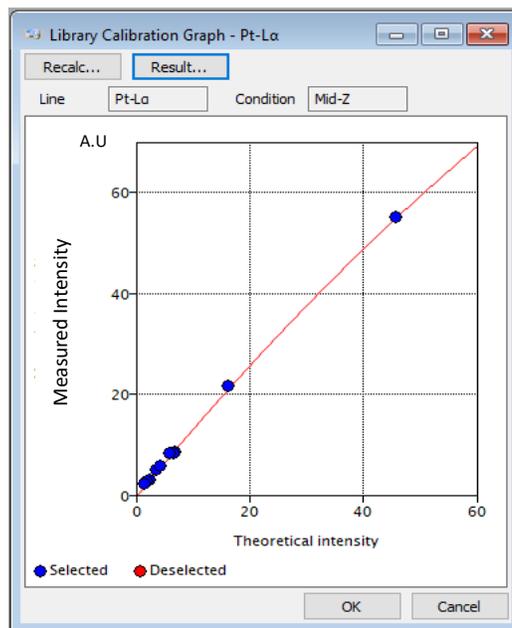
FUNDAMENTAL PARAMETERS and MATCHING LIBRARY

For screening purposes Fundamental Parameters (FP) is used which calculates concentration based on X-ray 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 and match the XRF to the referee values, either fire assay or ICP.

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 or families. 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.

| Element | Concentration Range (ppm) |
|---------|---------------------------|
| Pt | 400-6230 |
| Pd | 260-6445 |
| Rh | 0-365 |



Typical Factory Matching Library for Pt matching XRF intensities to fire assay values

RESULTS - Certified Reference Material ERM EB504

The European reference standard ERM EB504E was measured as a check sample, not part of the factory starter Matching Library. Typical recovery is <10% relative in accordance with potential error in ICP or fire assay values.

| Sample ID: ERM-EB504E | | Units: ppm | | | |
|-----------------------|-----------------|----------------------------|-------------|-------------|-----|
| Element | Certified Value | NEX QC ⁺ Result | % Deviation | Stat. Error | LLD |
| Pt | 1777 | 1742 | 2.0% | 6.0 | 6.2 |
| Pd | 279 | 285 | 2.2% | 1.4 | 0.8 |
| Rh | 338 | 341 | 1.0% | 1.6 | 1.1 |

DISCUSSION

The Rigaku Auto Cats-Pak includes the factory starter Matching Library for general, mixed and homogenized auto cats and also includes a small supply of the ERM certified check standard.

To meet various analytical needs at various sites, the Rigaku QuantEZ software is versatile and simple to use. The starter Matching Library can be augmented with additional standards from the site for mixed, homogenized cats, and the Matching Library software allows the user to easily build specific methods by grouping cats into families, for example a family for European cats, another for Asian cats, or a separate method for US cats. Families of auto cats are typically determined by the other ceramic and metal elements in the powder, such as a specific family for silicon carbide cats.

The versatile software also allows for empirical best-fit calibration using a large set of accurately assayed material, giving the site several ways to craft methodology to meet the specific analytical needs of the facility.

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 QC⁺ QuantEZ FP software allows for more versatile expansion or design of methods and libraries, as well as standardless analysis capabilities for screening. Thus, the Rigaku NEX QC⁺ QuantEZ with its simple yet advanced software, provides an easy-to-use and valuable tool for the catalytic converter recycling industry.