HH-XRF

Analysis of Metals in Catalytic Converters for Recycling, Recovery, and Reuse

Application Note # XRF 472

Introduction

An important emission control component of a gasoline engine is the auto catalytic converter (ACC). Installed in the exhaust system, it converts NO_X, hydrocarbons, and carbon monoxide into carbon dioxide and water to comply with exhaust emission regulations.

ACCs typically have a stainless-steel housing containing a monolithic catalyst support made of ceramic and synthetic cordierite in a honeycomb structure. The catalysts used are a mix of precious metals such as platinum (Pt), palladium (Pd), and rhodium (Rh). Catalyst metals are suspended in a washcoat, usually aluminum oxide, titanium oxide, or silicon oxide, to help disperse them over the monolith's large surface area.

Catalytic converters which are no longer useful are recycled. The precious metals recovered are sold to use for other applications. Therefore, it is critical to know the composition of the precious metals recovered from the ACCs.

This lab report describes the analysis of catalytic converter samples using Bruker's Catalytic Converter Calibration with an emphasis on the Platinum Group Metals (PGM), rhodium, palladium, and platinum.

Instrumentation

XRF analysis is an excellent choice for catalytic converter materials analysis. It is fast, easy, and economical especially when compared to other lab methods.

The CTX portable XRF analyzer is ideal for this application because it is easy to use in a production environment or in a laboratory. The CTX is rugged and well-sealed for use in harsh conditions and there are no active cooling filters which can clog in dusty recycling facilities.

The CTX has a very small footprint which is perfect when space is limited and the front facing touchscreen provides easy operational and results access. This self contained spectrometer does not require an external PC for measurements. It provides built-in safety interlocks, easy access trigger, and touch screen for operation and results viewing.

If sorting of incoming metals and alloys at a recycling facility is also required, the S1 TITAN handheld XRF analyzer with a desktop stand would be flexible enough to cover all applications. Not only does it enable „point-and-shoot" measurements, but with the optional desk or bench top stand and PC, it also easily measures small samples and prepared powder catalytic converter samples.

Bruker's portable and handheld XRF analyzers can be preloaded with a comprehensive catalytic converter calibration which includes Rh, Pd, and Pt metals as well as at least 19 other elements at applicable concentration ranges. That means the CTX and S1 TITAN analyzers are ready to measure ACC samples and provide results right out of the box.

Figure 1
Auto catalytic converter stainless steel housing cut open to reveal the ceramic honeycomb structure containing precious metals.

Sample preparation

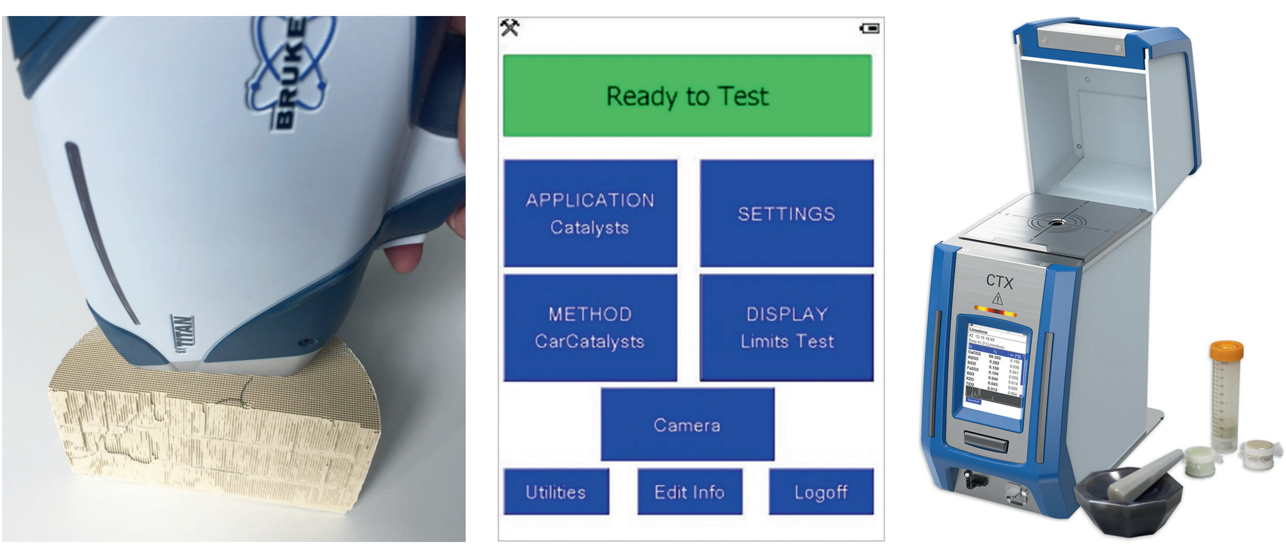
To recycle an ACC, the stainless-steel housing is cut open (Fig. 1) and the ceramic honeycomb structure is removed to recover the precious metals. For the most accurate and precise measurement results, the honeycomb cores are ground into a homogenous powder and placed in a sample cup or sample bag to analyze for their full elemental composition.

Sample measurements

Direct measurement of the ceramic honeycomb core is possible with the S1 TITAN handheld spectrometer; however, it only provides elemental identification of the surface. The grounded honeycomb samples were measured with the CTX portable XRF analyzer (Fig. 2).

Everyday operation consists of three simple steps. The sample is placed in position on the analyzer, the catalyst application is selected,

Figure 2
Left: Direct measurement of the ceramic AAC honeycomb with the S1 TITAN handheld XRF spectrometer. Middle: Display of software user interface. Right: Measurement of grounded honeycomb core powder in a sample cup of the CTX portable XRF spectrometer.



and the start trigger is pressed. Measurement results begin appearing on the screen immediately. The measurement precision can be tested with repeated measurements using a known catalytic converter powder sample. Homogeneity of the powder can be tested by performing multiple measurements. To take into account possible sample variability, it is also important to take multiple samples from the material batch and use the averaging feature.

Bruker's CTX portable and S1 TITAN handheld XRF analyzers can both be equipped with the catalytic converter calibration providing an economical way to successfully measure catalysts.

S1 TITAN and CTX catalytic converter calibration

The latest S1 TITAN and CTX catalytic converter calibration is optimized for analysis of Rh, Pd, and Pt packed in a sample cup. However, it is possible to measure ground powder through a sample bag, but with some reduced accuracy.

This calibration can also be used for direct measurement of a ceramic core honeycomb, but the result does only represent its surface and not the overall composition of the honeycomb structure:

- Surface measurements can indicate the presence of precious metals, but not the actual average concentrations.
- The honeycomb surface does not represent the overall composition of the monolith and measurement results do not reflect the average composition.
- Surface measurement typically ends up providing very little or no actual value.

Reproducibility measurements of Rh, Pd, and Pt with Bruker's Catalytic Converter Calibration.

Meas. #	Rh (ppm)	Pd (ppm)	Pt (ppm)
1	236	3781	407
2	219	3800	329
3	210	3745	384
4	214	3722	376
5	220	3756	417
6	230	3802	415
7	207	3711	386
8	225	3781	393
9	228	3791	378
10	210	3792	395
11	224	3785	366
12	221	3768	396
13	229	3772	409
14	225	3813	388
15	220	3780	400
16	235	3832	407
Average	222	3777	407
Std ev	8.2	28.9	14.6
Rel Std eV	3.7 %	0.8 %	3.7 %

The calibration enables analyses of Rh, Pd, and Pt (Fig. 3) as well as 19 other elements which improve the analysis reliability to account for all common matrix effects and overlaps. For example, the presence of selenium (Se) and tantalum (Ta) sometimes falsely increase the Pt values because they overlap with Pt peaks. These overlaps are compensated for in order to prevent costly mistakes with Se or Ta spiked catalyst powders.

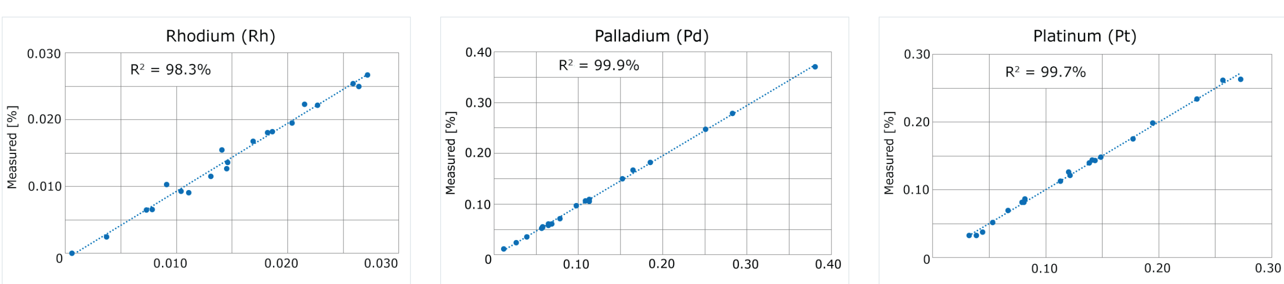


Figure 3
Typical precious metal accuracies for Bruker's Catalytic Converter Calibration for well characterized powder ACC samples.

Results

Data in the table show excellent repeatability measurements using Bruker's Catalytic Converter Calibration for catalytic converter powdered samples with a relative standard deviation of 3.7% for Rh, 0.8% for Pd, and 3.7% for Pt. The calibrations in Fig. 3 show excellent correlation of PXRF measurements to expected results for a set of powdered reference materials with an R2 value for Rh of 0.983, Pd of 0.9991, and Pt of 0.997.

Conclusion

XRF analysis is a key component in the recycling process of catalytic converters and particulate filters. In addition to precious metals, XRF analysis provides a full chemical composition of the recycled materials. This technology supports both specialized catalytic converter recyclers and precious metal refineries for precious metal analysis and measurement of other elements to optimize PGM recovery and refining.

Bruker's portable and handheld XRF analyzers can provide very consistent measurement results for well-prepared samples using the catalytic converter calibration. The most accurate and precise quantitative results can only be obtained on finely ground and mixed catalytic converter powders. The correlation with reference samples and other well-prepared and characterized samples is typically over 99%. Correlation can be lower if samples are not fully homogeneous.

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