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Sample Collection Procedure for Determining Precious Metal Content in Metal Mesh (Non-Ceramic) Equipped Catalytic Converters

Catalytic converters, utilized to treat on-road vehicle engine exhaust, typically consist of a ceramic honeycombed monolith substrate bearing a washcoat material that contains precious metal catalysts. Sample collection procedures for catalyst content in these "automobile-type" converters are well established. This application note is largely based on the EPA publication, *Laboratory Analysis of Catalytic Converters Leads to Better Enforcement Results,* by Smith, Suggs and Isin.[1] It describes a precious metal sample collection technique employed when metal-mesh and other similar non-ceramic based substrates are encountered. This type of exhaust converter is most often used in motorcycle, recreational vehicle or small engine applications.

Background

Regulation History: The United States (U.S.) Environmental Protection Agency (EPA) engine emission standards have been in effect in various forms since 1968. Today, these standards apply to nearly all of the vehicles and engines sold in the United States, whether they are manufactured domestically or abroad. Recently, reduced exhaust emission requirements for new recreational vehicles and small (< 25 H.P.) spark-ignition engines have been implemented.

Key Component: Catalytic converters have continued to play an essential role in air-quality improvement efforts since their introduction in the mid-1970s as a mandatory component of on-road vehicle exhaust systems. They are required equipment for the exhaust treatment of most combustion engines throughout the U.S. and many other countries.

Enabling Chemistry: The catalyst itself is most often a combination of one or more precious metals. Platinum (Pt), Palladium (Pd) and Rhodium (Rh) are widely used in contemporary catalytic converters. Due to limited quantities and a large demand, the cost for these metals remains high.

Regulatory Challenge: Global economic factors have caused large numbers of the recently regulated recreational and small spark ignition engines imported by the U.S. All too often, these engines are found to be in violation of EPA standards due to poor catalytic converter quality.

Enforcement: EPA looks at all combustion engine products and all manufacturers. Engine inspections occur at retail outlets, as well as upon importation into the U.S. As the use of catalytic converters expands, the need to promptly determine converter quality in a diverse population of engines has never been greater.

Introduction

Non-ceramic substrate equipped catalytic converters present a unique challenge because the washcoat containing the precious metal is securely bound to an inaccessible metal mesh contained in a metal sleeve (Figure 1). The converters are small in size, compared to automobile varieties, and usually installed as an integrated part of the engine's muffler. After removing the unit from the muffler assembly, the intact converter is sent to laboratories such as EPA's National Enforcement Investigations Center (NEIC) for analysis. In enforcement cases, chain-of-custody procedures are employed.

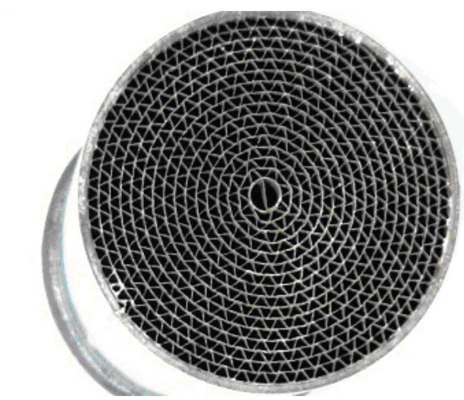

Figure 1: End view of a metal mesh substrate catalytic converter.
Photograph courtesy of EPA

Sample Collection

Washcoat sample collection begins with the disassembly of the converter body and occurs in conjunction with collection of various data including physical measurements, photographs, and weights that are required to determine the specifications for the catalytic converters being examined. The encasing metal sleeve is cut away to enable the metal mesh to be manually unwound.

In some cases, the fabrication of the substrate prevents the mesh from unwinding. This type of sample requires additional effort to disassemble. In all cases, a great deal of care is used ensure a minimal loss of washcoat.

Removal of washcoat material is not easily achieved. The main constituents are porous base-metal oxides, such as aluminium, cerium and zirconium. The washcoat compound serves as a carrier for application of the catalyst onto the substrate during converter construction and as a rugged high surface area support medium once the converter is put into service. Direct exhaust gas/catalyst contact is critical to converter functionality, thus the washcoat is designed to strongly adhere to the metal mesh substrate. Varying degrees of force are always required, but simply unwinding the mesh may remove a significant amount of washcoat material. In cases where the fabrication of the substrate prevents mesh unwinding, additional force is required.

To disassemble more challenging support structures, various methods of mechanical force are utilized. The metal mesh surface area must be exposed so that washcoat materials can be scraped from the mesh surface. Various tools, including mallet, chisel, awl and metal bristle brush are frequently employed. The washcoat presents itself as a powder when it is separated from the mesh.

This activity often results in the mesh being reduced to small metal pieces. Both washcoat and metal pieces are typically collected in large weigh boats positioned beneath the samples. Metallic fragments are then removed from the recovered materials. Larger pieces may be separated individually, while a magnet may be used for removal of small metal debris (Figure 2).

Figure 2: Removal of metallic debris using a magnet.
Photograph courtesy of EPA

Test Specimen Preparation

Once sample collection and cleaning is accomplished, the ability to evaluate a test specimen for precious metal content using X-ray fluorescence (XRF) methodologies is relatively easy. Because powder matrices may be readily analyzed, there in no need for further sample treatment. Testing is also non-destructive, which preserves the quantity and quality of the collected sample material and eases sample retain and chain of custody procedures.

Figure 3: Recovered washcoat in 31 mm diameter sample Cell;
Typical test specimen depth – 2-3 mm

Testing

Analysis is accomplished by placing the test specimen into a standard 31 mm diameter X-ray sample cell to a depth of 2-3 mm. This small test specimen volume requirement is important since the amounts of recovered washcoat are often small (less than 5 g). Mylar film (2-6 μm) is used to provide the sample cup seal. Once the cup is sealed (Figure 3), it is placed in the analyzer sample compartment and analysis begins. For more information regarding precious metal test methods, see HORIBA application note AN208.

Conclusion

Although laborious, the sample collection procedure described enables the determination of precious metal (catalyst) content in washcoat materials employed in metal mesh (non-ceramic) substrate catalytic converters. Recovered materials may then be readily analyzed by XRF testing methods.

Bibliography

[1]Smith, D., Suggs, J., and Isin, A., *Laboratory Analysis of Catalytic Converters Leads to Better Enforcement Results*, presented at the 9th International Conference on Environmental Compliance and Enforcement, Canada, June 20-24, 2011, http://inece.org/conference/9/papers/SmithSuggs_USEPA_Final.pdf.

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