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XRF1023 - Pt, Rh and Pd analysis of used automobile catalyst by WDXRF

Introduction

The number of automobiles in developing nations has increased dramatically in recent years, which has caused serious air pollution in those countries. In developed nations, tight control on exhaust emission is already being enforced while emission control standards in developing countries are still lax. In the future, emission control standards must become tighter throughout the world.

In general, a three-way catalyst is used for the exhaust of automobiles, where hydrocarbons (HC), carbon monoxide (CO) and nitrogen oxides (NO_x), the harmful compounds in exhaust, are removed with a catalytic converter using Pt (platinum), Rh (rhodium) and Pd (palladium). Since these precious metals exist only in trace amounts in deposits around the world, catalytic converters are collected from discarded automobiles so that the precious metals can be extracted and recycled.

This application note introduces XRF analysis of Pt, Rh and Pd in used automobile catalyst.

Instrument

The ZSX Primus IV and ZSX Primus III NEXT are tube-above sequential wavelength-dispersive X-ray fluorescence (WDXRF) spectrometers, optimized for routine analysis of powder samples.

The tube-above optics, changeable vacuum speed, powder trap and pre-evacuation chamber of the ZSX Primus IV and ZSX Primus III NEXT enable secure analysis of powder samples and infrequent maintenance by preventing pressed powder pellets from breaking and falling into the optics and by protecting the vacuum pumps and magnetic valves from fine particles scattered from samples.

Standard sample and sample preparation

House standard samples of used automobile catalyst were used for calibration.

Powders of the standard samples were ground with a vibration mill using a tungsten-carbide container. The ground powders, pre-dried at 110 °C, were pressed into iron cups (I.D. 40 mm) at 100 kN.

Measurement

Measurements were performed on the ZSX Primus IV with a 4 kW Rh-target X-ray tube. Measurement conditions are shown in Table 1.

Table 1: Measurement conditions

| | | | | |
|------------------------|-------------------|-----------|-----------|------------|
| Path atmosphere | Vacuum | | | |
| Analysis area | 30 mm in diameter | | | |
| Element | Pt | Rh | Pd | Rh |
| Line | Lβ1 | Kα | Kα | Kα-Compton |
| Tube condition (kV-mA) | 50-60 | 40-75 | 50-60 | 50-60 |
| Primary beam filter | Ni40 | Ni400 | Ni400 | Out |
| Slit | S2 | S2 | S2 | S2 |
| Analyzing crystal | LiF(200) | LiF(200) | LiF(200) | LiF(200) |
| Detector | SC | SC | SC | SC |
| Counting time Peak (s) | 100 | 100 | 100 | 20 |
| Counting time BG (s) | 100 | 50 x2 | 50 x2 | |

Scatter lines of Rh, the target material of the X-ray tube mounted on the ZSX Primus IV, interfere with Rh and Pd element lines. The “Ni400” primary beam filter in the standard configuration eliminates these scatter lines and enables analysis of trace amounts of Rh and Pd.

Calibration

In order to demonstrate the effectiveness of the Compton scattering ratio method, where Compton scatter is used as an internal standard, for this application calibration curves with and without the Compton scattering ratio for each element are shown in Figure 1. For Pt, the Zr-Kα intensity was used to correct for the absorption of Pt-Lβ1 by Zr. The calibration results are summarized in Table 2.

The accuracy of calibration is calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-m}}$$

C_i : calculated value of standard sample

\hat{C}_i : reference value of standard sample

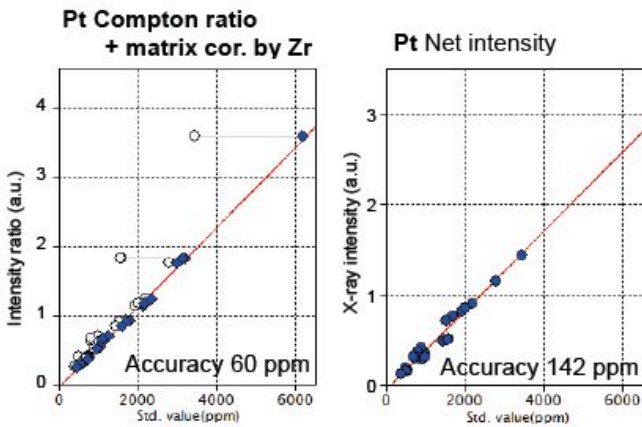
n : number of standard samples.

m: degree of freedom (2 for linear fitting)

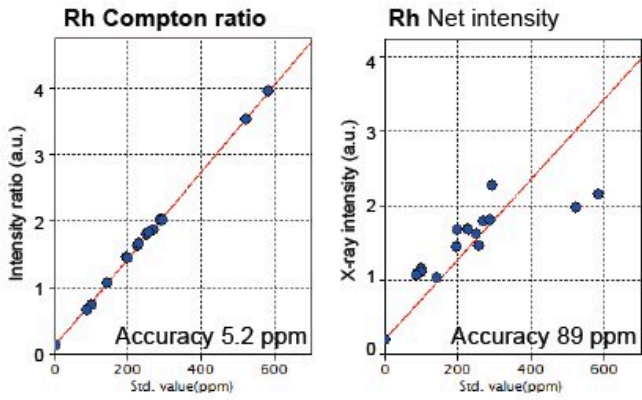
Table 2: Calibration summary (unit: ppm)

| Element | Calibration range | Calibration accuracy | |
|---------|-------------------|---|-------------------------------|
| | | Compton scattering ratio method applied | No correction (net intensity) |
| Pt | 374 - 2792 | 60 ¹⁾ | 142 |
| Rh | 0 - 583 | 5.2 | 89 |
| Pd | 0 - 5963 | 33 | 275 |

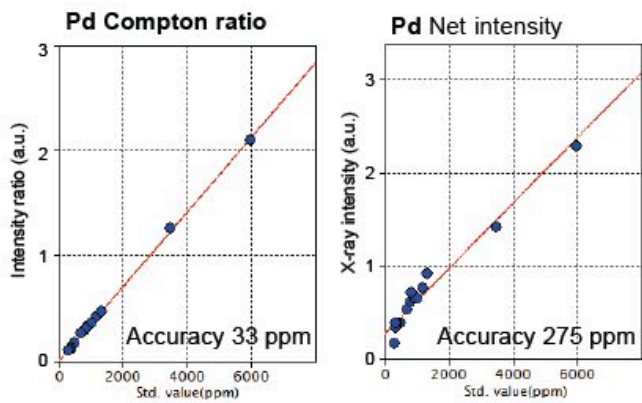
1) Absorption correction by Zr was also applied



(1) Pt calibration curves



(2) Rh calibration curves



(3) Pd calibration curves

Figure 1: Calibration curves with and without the Compton scattering ratio method - (1) Pt (2) Rh (3) Pd. For Pt, absorption correction by Zr is also applied.

Conclusions

This application note demonstrates that Pt, Rh and Pd can be quantified reliably at trace level concentrations in used automobile catalyst using the pressed powder method and Rigaku ZSX Primus series spectrometers.

Related products



ZSX Primus IV

High power, tube above, sequential WDXRF spectrometer with new ZSX Guidance expert system software