Xenemetrix
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ARUN TECHNOLOGY XENEMETRIX SPECTRO INC. INNOV-X - IOSYSTEMS

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ABSTRACT
A quantitative analysis of seven pressed pellets auto catalysts samples was performed using a Xenemetrix EX-Calibur EDXRF analyzer and regression analysis method to determine the precious metals content (Rh, Pd and Pt).

OBJECTIVE
To analyze automobile catalysts samples on Xenemetrix EX-Calibur and to quantify the content of precious metals using regression analysis.

BACKGROUND
EDXRF is a fast and non-destructive technique that can make the analysis of auto catalysts samples within a few minutes and can be the method of choice for this type of analysis. For the recycling industry a prior knowledge of the precious metals contents will have a large economic impact on the industrial outcome.

EDXRF is an ideal method for a quick and simple elemental analysis for industrial control purposes offering the following advantages: 1.) Fast and minimal sample preparation, 2.) An automated analysis process, 3.) Limited or no exposure to corrosive reagents used by other analytical techniques, 4.) Ease of use for operation by non-technical or non-specialized personnel.

ANALYTICAL CONFIGURATION

Table 1.

Analytical Configuration

Instruments	EX-Calibur EDXRF Spectrometer
Excitation	Rh-Anode X-ray tube, 50W
Detector	High Resolution Si PIN Diode Detector
Analysis Time	300s
Calibration Method	Quantitative Regression Analysis
Modifications	None
Environment	Air
Sample Preparation	Pressed pellets

EXPERIMENTALS

SAMPLE PREPARATION

Seven different powder samples were obtained. Each sample was mixed in a ball mill with wax in a ratio of 9:1. The mixture was pressed in a automatic hydraulic press at 15 tons for 25 seconds. The pressed pellets were analyzed with a Xenemetrix EDXRF benchtop analyzer, model EX-Calibur

CALIBRATION RESULTS

Two calibration procedures were used, one to measure Rh and Pd using a W filter to block the Rh lines coming from the X-ray tube anode and another one using a Rh filter for optimal acquisition of the Pt signal. Typical spectra of the two different acquisitions are show in Figure 4 and 5. The correlation data for the three different elements are presented in Table 2, 3. Correlation plots are shown in Figure 1,2 and 3.
All of the reference samples except 718 contained 24 ppm or less of Pb. Sample 718 contained 15.62% of Pb i.e. a factor of 8000 times more. This sample clearly represents another matrix and as such has to be calibrated with other samples of similar Pb content (high Pb content). Sample 718 was excluded from the calibration curves described in this report due to its outstanding Pb content.

Rh

Table 2: Rh correlation data

Element: Rh		**Correlation: 0.999**
Units: ppm		**Std Dev: 11 ppm**
	Given conc. (ppm)	Measured conc. (ppm)
622	656	663
666	<10	15
828	744	734
830	427	433
948	395	407
949	276	256

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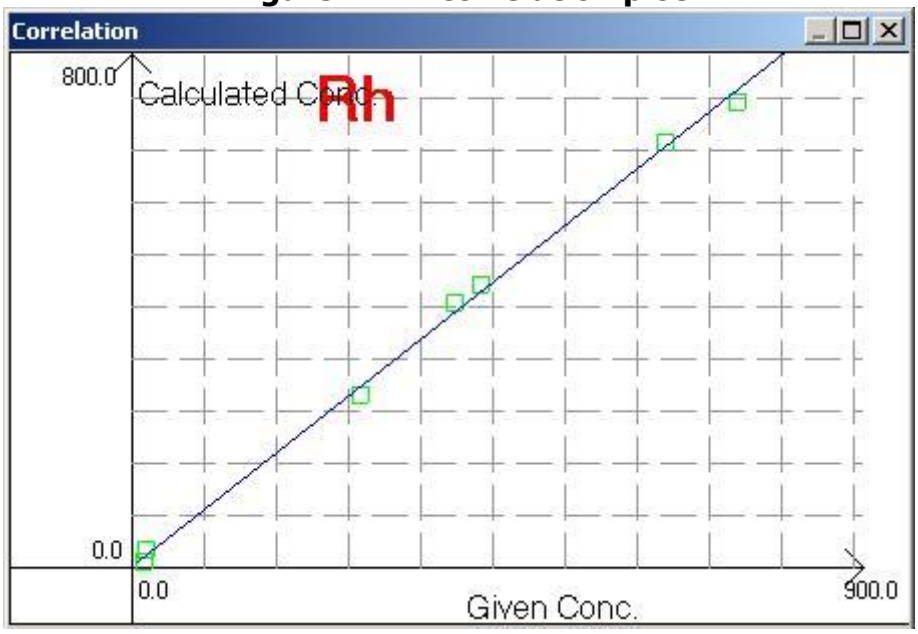
Figure 1: Rh correlation plot



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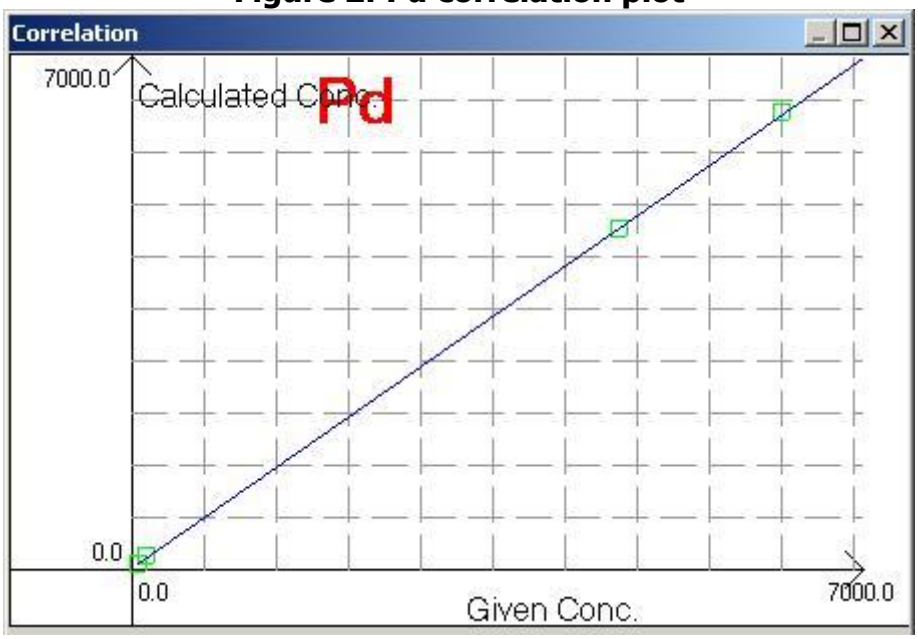
Pd

Table 3: Pd correlation data

Element: Pd	**Correlation: 1.000**	
Units: ppm	**Std Dev: 24 ppm**	
	Given conc. (ppm)	Measured conc. (ppm)
622	15	20
666	6223	6239
828	4647	4625
830	2904	2117
948	90	135
949	19	20

Figure 2: Pd correlation plot

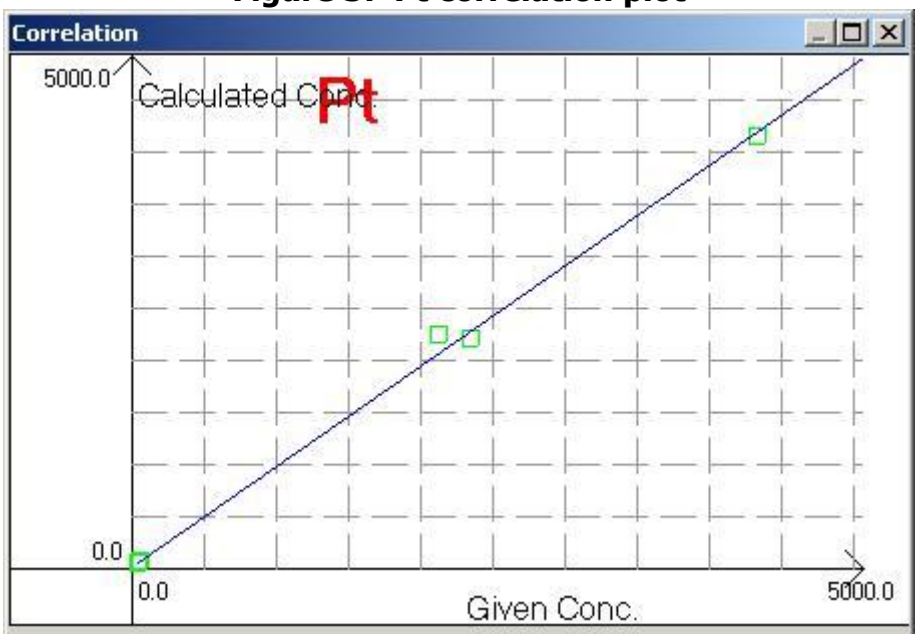


Pt

Table 4: Pt correlation data

Element: Pt		**Correlation: 0.998**
Units: ppm		**Std Dev: 89 ppm**
	Given conc. (ppm)	Measured conc. (ppm)
622	4275	4220
666	13	55
828	6	0
830	14	16
948	2305	2145
949	2073	2169

Figure 3: Pt correlation plot



SPECTRA

Figure 4: Spectrum of sample 828 with acquired with a W filter

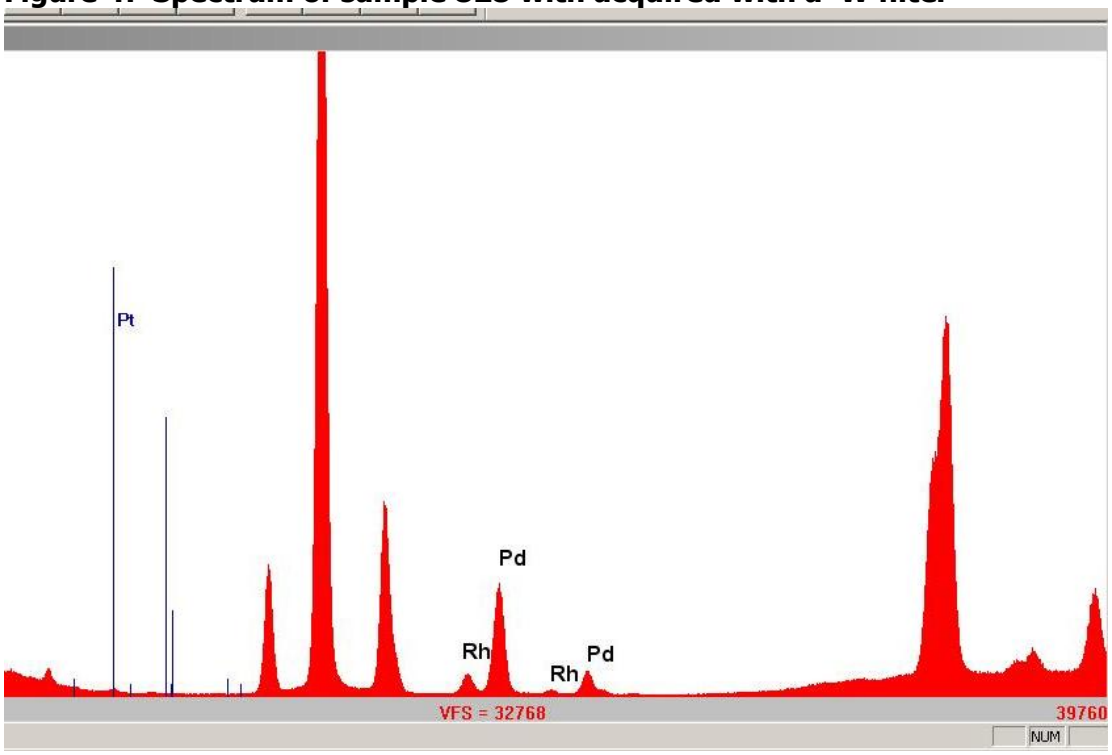
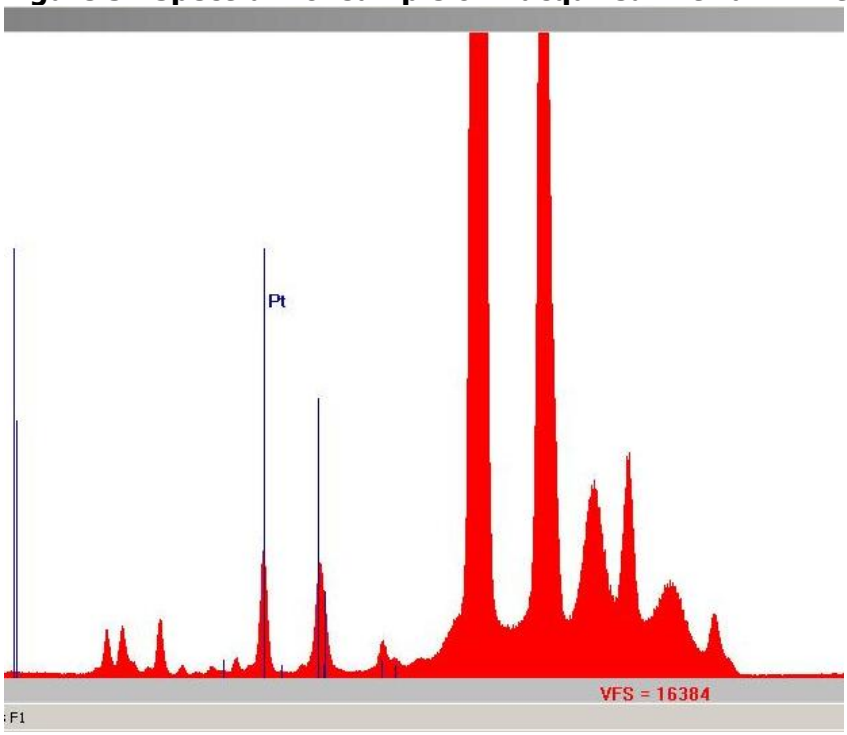


Figure 5: Spectrum of sample 622 acquired with a Rh filter



STATIC PRECISION DATA

Static precision studies were performed on two of the pressed pellets samples by running the two different acquisitions ten consecutive times per same sample without moving the sample in between the runs. Statistical analysis of the data show the average measured concentration in ppm of the ten runs together with standard deviation in ppm (one sigma) and relative standard deviation in % (one sigma). The results for pressed pellet sample 622 and 948 are presented in Tables 5 and 6.

Table 5: Repeatability of analysis on pressed pellet sample 622

Element	Given conc. (ppm)	Measured average conc. (ppm)	Standard Deviation (ppm)	% RSD
Rh*	656	675	30	4.4
Pd*	15	20	0	NA
Pt**	4275	4204	42	1.0

*measured with W filter
**measured with Rh filter

Table 6: Repeatability of analysis on pressed pellet sample 948

Element	Given conc. (ppm)	Calculated conc. (ppm)	Standard Deviation (ppm)	% RSD
Rh*	395	353	29	8.3
Pd*	90	116	8	7.2
Pt**	2305	2232	22	1.0

*measured with W filter
**measured with Rh filter

DISCUSSION

The Xenemetrix EDXRF analyzer, model EX-Calibur has the great advantage of a high resolution Si PIN diode thermoelectrically cooled detector, coupled with a highly flux efficient Rh anode end window X-ray tube which is especially designed to give accurate and highly sensitive data for Rh, Pd and Pt in auto catalysts. EX-Calibur provides an excellent tool to quickly scan and determine the content of the precious metals in these auto catalysts. In the case there is an outstanding high Pb content in the samples special calibration curves should be done using several reference samples with high Pb content.

CONCLUSION

This auto catalysts application is a very good example of how fast and reliable EDXRF quantitative analysis can be used to determine the content of precious metals in auto catalysts.

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The data presented herein is hereby warranted to be generated from standard Xenemetrix production instruments. Under qualified operation — which includes sample preparation, instrument set up, and other variable parameters affecting performance — any instrument manufactured by Xenemetrix as specified by model type, including options, is expected to meet the performance stated above.

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