

Analysis of Ni and V in Gas Oil

ARL ADVANT'X Series with IntelliPower™ Sequential X-Ray Fluorescence Spectrometer

- ARL ADVANT'X - 3600W
- Oil
- X-Ray Fluorescence
- XRF

Introduction

Norms for the petrochemical industry demand that the sum of Ni and V contents are below 0.5 ppm in gas oil (diesel).

Instrument

The Thermo Scientific ARL ADVANT'X Series spectrometer has been used for this test. The instrument is equipped with a Rh anode end-window X-ray tube of 3.6 kW maximum power.



For liquid analysis, a helium atmosphere is required. Thanks to an innovative shutter, the changeover from vacuum to helium atmosphere is achieved in less than 2 min.

The instrument is driven by WinXRF, a Windows® based software with several attractive features such as on-line SPC, networking capabilities and remote diagnostics.

Samples and methods



A series of 4 gas oil secondary standard samples were chosen for the analysis. The sample is poured directly into a special liquid cell which is fitted with a 6 micron thick polypropylene film (10 grams of liquid). The liquid cell is placed into a metallic sample holder which will be loaded into the XRF spectrometer. A helium gas environment is used to analyze all samples.

Results and discussion

Table 1 shows the analytical conditions used, while Table 2 demonstrates the good sensitivity of the method and the limits of detection that have been achieved.

Figures 1 and 2 show the calibration lines obtained without the need for any matrix correction. The SEE of 0.020 ppm for vanadium and 0.025 ppm for nickel confirms the excellent sensitivity obtained.

ELEMENT/ LINE	CRYSTAL	DETECTOR	ANALYSIS TIME
V / K α	LIF200	FPC	150 sec.
Ni / K α	LIF200	SC	150 sec.

Table 1: Analytical conditions

ELEMENT/ LINE	SENSITIVITY (KCPS/%)	BEC (%)	LOD (PPB)
V	973	0.00095	76
Ni	5954	0.00071	28

Table 2: Performance and limits of detection

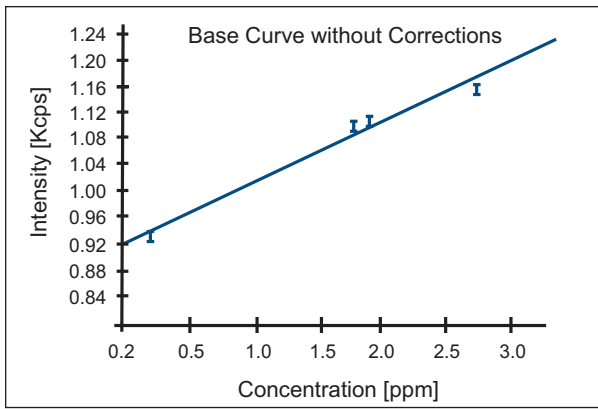


Figure 1: Calibration for Vanadium in Gas Oil
SEE = 0.020 ppm

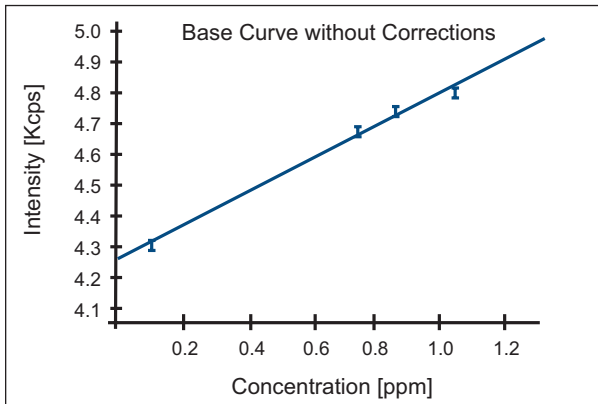


Figure 2: Calibration for Nickel in Gas Oil
SEE = 0.025 ppm

Repeatability at ppm level

The repeatability of nickel analysis at the ppm level is demonstrated in Table 3 where 4 different liquid cells were filled with gas oil from the same sample and introduced into the spectrometer at 3 min intervals. A counting time of 120 seconds per run was used.

CELL NR.	Ni CONCENTRATION
1	1.6 ppm
2	1.6 ppm
3	1.7 ppm
4	1.7 ppm
Avge	1.7 ppm
SD	0.06 ppm
RSD	3.1 %

Table 3: Repeatability of Ni

Conclusion

The very high sensitivity and low limits of detection achieved with the ARL ADVANT[™]X X-ray spectrometer allow the analysis of Ni and V below ppm levels in gas oil with good precision thereby ensuring that the sum of their concentrations can be controlled below 0.5 ppm.

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