

# APPLICATION REPORT

XRF 163

## Analysis of Ni, V, Fe, S in Heavy Oil by ZSXmini

### Introduction

This report is to introduce the analysis of Ni/V in heavy oil by Table Top WDXRF model **ZSXmini**.

It is said that the analysis of component Ni/V in petroleum products or byproducts is rather difficult. The reason is that, as they are easy to scatter when dry ashing is made because that the shape of Ni/V in heavy oil is an organic family, analysis result shows always lower than true value. Therefore, wet ashing is recommended, but it takes long time awfully.

**ZSXmini** will solve these problems easily. Moreover, analysis of S, Fe, Cl, etc. can be added according to user's request, and highly reliable analysis can be carried out in a short time.

### 1. Equipment and measurement conditions

#### 1.1 Equipment

Table top type WDXRF model **ZSXmini**

#### 1.2 Measurement conditions

Element	Ni	V	Fe	S
Spectrum	Ka	Ka	Ka	Ka
X-ray tube	50 W, End window, Pd target			
X-ray path	Helium			
KV - mA	40 – 1.2			
Analyzing crystal	LiF200	LiF200	LiF200	PET
Detector	S-PC	S-PC	S-PC	F-PC
Slit	Standard resolution			
Peak measurement time (sec)	100	150	100	60
BG measurement time (sec)	50 x 2	75 x 2	50 x 2	30 x 2
Sample and its amount	Heavy oil, 4 ml			

### 1.3 Standard sample

Standard samples for making calibration curve were made by dilution of CONOSTAN standard liquid and additives.

Table-1. Standard samples for calibration curve and concentration (in ppm)

Sample	Ni	V	Fe	S
1	0	0	0	0
2	0	0	0	1808
3	99.6	100	50	2437
4	50.4	49.8	9.6	2188
5	24.5	150	25.2	2277
6	74.6	24.6	75.3	2146
7	10.3	200	99.9	2674

## 2. Calibration curves

Calibration curves are shown in Fig.-1 ~ Fig.-4, and accuracy and detection limit calculated from the calibration curves are shown in table-2.

Besides above elements reported, catalyst elements like Si, Al, etc. may be mingled into heavy oil. For these elements analysis, calibration with matrix correction will be required for accurate analysis.

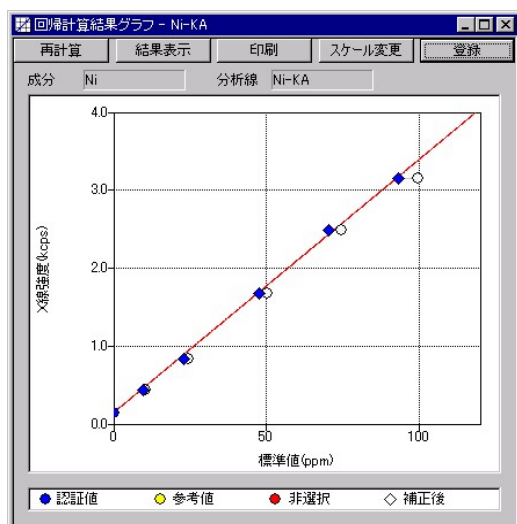


Fig.-1 Cal. curve for Ni

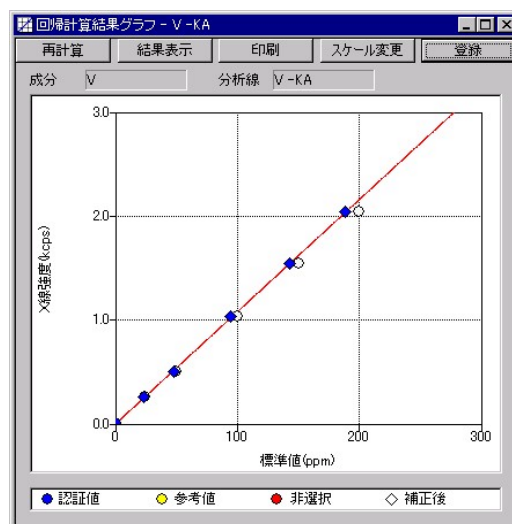


Fig.-2 Cal. curve for V

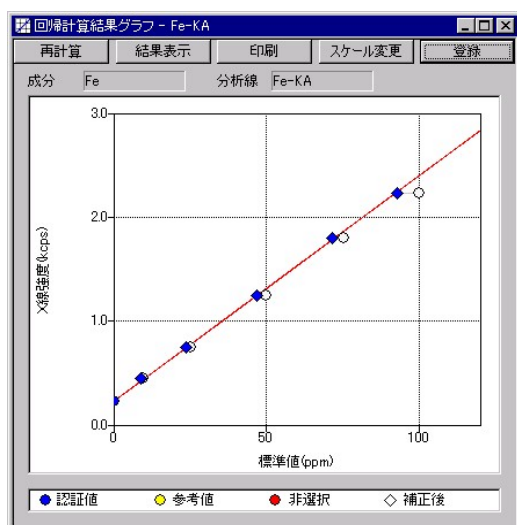


Fig.-3 Cal. curve for Fe

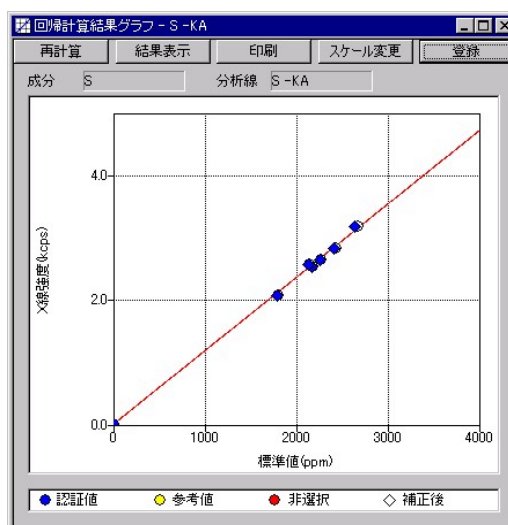


Fig.-4 Cal. curve for S

Table-2 Accuracy and Detection limit (in ppm)

	Ni	V	Fe	S
Concentration range	0 ~ 90	0 ~ 190	0 ~ 90	0 ~ 2600
Accuracy	1.2	0.4	0.9	35.8
Detection limit	1.0	1.3	1.3	3.9

### 3. Reproducibility in laboratory (Refilling dynamic precision)

Heavy oil standard samples for Ni/V made by The Japan Petroleum Institute were put into 10 sample holders and measured. Reproducibility in laboratory was calculated from the results as below.

Table-3 Reproducibility in laboratory (Refilling dynamic precision) (In ppm)

	Ni	V	Fe	S
1	19.8	51.8	10.2	31830
2	22.1	51.8	10.3	31500
3	20.2	49.8	9.1	32250
4	18.3	49.4	10.1	31530
5	20.2	51.2	10.2	31850
6	20.7	51.2	8.8	31730
7	20.3	50.8	7.4	32330
8	20.3	50.0	9.5	31410
9	19.9	50.5	7.5	31840
10	20.1	51.6	7.7	31030
Average	20.2	50.8	9.1	31730
S.D. (Sigma)	0.9279	0.8595	1.1793	389.10
C.V. (%)	4.60	1.69	12.99	1.23
Certified value	22	52	-	-

#### 4. Analysis example

Standard samples made by The Japan Petroleum Institute were measured as unknown samples. Two times repeat measurement was made and result shows good matching with true value from low to high concentration range as below.

Table-4 Analysis result of Ni and V (in ppm)

Sample name	S0264		S0265		S 265*	
	Ni	V	Ni	V	Ni	V
1 <sup>st</sup> trial	1.1	8.9	21.1	51.7	36.4	106.9
2 <sup>nd</sup> trial	2.6	8.7	19.9	50.6	35.8	109.2
Average	1.9	8.8	20.5	51.2	36.1	108.1
Certified value	2.6	8.8	22	52	37	107

Note \* : Sample #S 265 is currently not available.

## 5. Summary

It is not too much to say that XRF analysis does not require sample preparation.

Therefore, quick and reliable analysis can be realized by this method.

**ZSXmini** is offered to analysts who is bothered by sample preparation, who expects one rank higher analytical performance, who wants analysis of Ni/V along with other elements.

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