



APPLICATION REPORT ON ANALYSIS OF CRUDE OIL AND RESIDUAL FUEL OIL SAMPLES

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INSTRUMENT MODEL: DITEE SWORD 510

REFERENCE STANDARD: IP501-2005

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Introduction

Background and Challenges

The content of metal and non-metal elements in crude oil and residual fuel oil is a key indicator for evaluating oil quality, optimizing catalytic cracking processes, and protecting refining equipment. For example:

- The presence of Vanadium (V), Nickel (Ni), and Sodium (Na) can lead to catalyst poisoning.
- Iron (Fe) easily deposits on the catalyst surface, leading to reduced catalyst activity; the presence of iron itself is also an indicator of equipment corrosion.
- Aluminum (Al) in residual oil easily forms hard particles, causing wear on components such as engine cylinders.
- Silicon (Si) forms silicon dioxide during combustion, which easily covers sensors, spark plug electrodes, and other positions, causing engine failure; the formed particles also easily scratch the combustion chamber, intake ports, pistons, and other components, affecting normal engine operation and the lifespan of mechanical parts.

For such heavy oil products, direct organic injection methods are often limited by high sample viscosity, difficulty in completely dissolving with solvents, particulate deposition, and a lack of matching standards, making it difficult to ensure the accuracy of test results. Therefore, this scheme adopts the internationally recognized arbitration method—the IP 501 Standard (Ashing-Alkali Fusion Method). This method completely converts complex organic oil samples into clear inorganic aqueous solutions through high-temperature ashing and borate fusion. Although this process completely eliminates the influence of the organic matrix on viscosity and spectral background, it also introduces high concentrations of flux components, making the final test liquid a high-salt solution (high Total Dissolved

Solids/TDS), which places extremely high demands on the salt tolerance of the spectrometer's injection system and the load capacity of the plasma.



DITEE SWORD 510 Solution

This application employs the DITEE SWORD 510 Full-Spectrum Direct Reading Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES), independently developed by Guangdong DITEE Scientific & Technical Co., Ltd. With its powerful and stable solid-state RF generator and design resistant to high-salt injection, it is perfectly adapted to the high-salt matrix analysis requirements of the ashing-alkali fusion method:

- **Excellent Matrix Tolerance:** Capable of handling high-salt and complex organic matrices with ease, ensuring the process does not extinguish the plasma or cause carbon buildup.
- **High Sensitivity and Accuracy:** Uses a scientific-grade large-format CCD detector to complete full-spectrum simultaneous detection in a single exposure, ensuring precise quantification of trace elements (such as V, Ni).

- **Compliance with International Standards:** This method completely references standard IP 501-2005, utilizing ashing-alkali fusion pre-treatment combined with SWORD 510 for determination, providing a comprehensive quality control solution for petrochemical laboratories from raw materials to final products.

Technical Proposal

Main Laboratory Equipment and Consumables

- **Instrument Model:** DITEE SWORD 510 Inductively Coupled Plasma Optical Emission Spectrometer.
- **Auxiliary Equipment:**
 - Analytical Balance: Precision 0.0001g
 - Muffle Furnace: Equipped with dual temperature zones or programmable temperature control, capable of accurately maintaining constant temperatures at 525 ± 25 °C and 925 ± 25 °C.
 - Platinum Crucible: 100 mL (Cleaning requires treatment with molten potassium bisulfate to eliminate sodium residues).
 - Glassware: Beakers (100mL, 200mL), funnels, volumetric flasks (50mL, 100mL). All glassware must be cleaned with 10% nitric acid before use.
 - Pipettes: 1000 μ L
 - Electric Stove

Reagents and Standard Solutions

To ensure measurement accuracy, all reagents used are of high-grade purity or sufficient purity, and water is Grade 1 water as defined by ISO 3696.

- **Acid Reagents:**
 - Nitric acid: $\rho(\text{HNO}_3) = 1.42$ g/mL
 - Hydrochloric acid: $\rho(\text{HCl}) = 1.19$ g/mL

- **Fluxes:**
 - Lithium Tetraborate.
 - Lithium Fluoride.
 - Mixed Flux: A mixture of 90% (mass fraction) Lithium Tetraborate and 10% (mass fraction) Lithium Fluoride.
- **Tartaric Acid-Hydrochloric Acid Solution:** Weigh 5g of tartaric acid and dissolve it in approximately 500 mL of water acidified with 40 mL of 36% (m/m) hydrochloric acid, then dilute with water to 1000 mL.
- **Standard Stock Solution:** Certified reference solutions containing Aluminum, Silicon, Vanadium, Nickel, Iron, Sodium, Calcium, Zinc, and Phosphorus at 1000 mg/kg (purchased from the National Center for Standard Materials).

Methodology

Sample Pre-treatment

Samples are treated using dry ashing combined with alkali fusion. The specific steps are as follows:

1. **Sample Homogenization:** Heat the sample in an oven at 50-60°C until it reaches a fluid state, then use a high-speed mixer to stir for about 5 minutes. This step is crucial to ensure the representativeness of the results.
2. **Weighing and Ashing:** Weigh 20-50 g of sample (appropriate to obtain about 550 mg of ash) into a platinum dish. Slowly heat until combustion occurs to remove most combustible matter. Subsequently, move it into a muffle furnace preheated to 525°C $\pm$ 25°C until the carbon is completely removed (usually requires overnight).
3. **Alkali Fusion:** After cooling, add 0.4 g of mixed flux, move to a muffle furnace at 925°C $\pm$ 25°C, and fuse for 5 minutes; remove and swirl to ensure full contact between the flux and the ash, then fuse for another 10 minutes.

4. **Dissolution and Volume Setting:** After the melt cools, add 50 mL of tartaric acid-hydrochloric acid solution, warm on a hot plate to dissolve (do not boil), cool, and transfer to a 100 mL volumetric flask to make up to volume.

Standard Curve Preparation

Pipette 0, 100 μL , 200 μL , and 500 μL of the mixed element standard stock solution (1000 $\mu\text{g/mL}$) into four 100 mL volumetric flasks, respectively, and dilute directly to the mark with the diluent. The standard series concentrations are as follows:

- **Std 0:** 0 mg/L
- **Std 1:** 1.0 mg/L
- **Std 2:** 2.0 mg/L
- **Std 3:** 5.0 mg/L

Instrument Operating Parameters

The DITEE SWORD 510 instrument parameters are set as follows to ensure optimal excitation efficiency and signal-to-noise ratio:

Instrument Operating Parameter	Setting Value
RF Power / W	1150W
Plasma Gas Flow / $\text{L}\cdot\text{min}^{-1}$	16
Carrier Gas Flow / $\text{L}\cdot\text{min}^{-1}$	0.5
Auxiliary Gas Flow / $\text{L}\cdot\text{min}^{-1}$	0.99
Peristaltic Pump Speed /rpm	30
Flush Time /s	40

Test Wavelength Selection

Based on the optical system characteristics of the DITEE SWORD 510, interference-free characteristic spectral lines were selected for analysis:

NO.	Element	Test Wavelength (nm)
1	Phosphorus (P)	213.620
2	Zinc (Zn)	213.856
3	Nickel (Ni)	231.604
4	Iron (Fe)	259.940
5	Silicon (Si)	251.611
6	Vanadium (V)	309.311
7	Sodium (Na)	589.595
8	Calcium (Ca)	317.933
9	Aluminum (Al)	396.153

Data and Results

Summary of Test Data

The treated samples were measured using the DITEE SWORD 510, and the results are shown in the table below. The data indicates that the instrument possesses good detection capabilities for various elements in crude oil and fuel oil.

Test Item	Element	Result (mg/kg)
Elemental Analysis	Phosphorus (P)	0.8
	Zinc (Zn)	1.1
	Nickel (Ni)	12.0
	Iron (Fe)	110.8
	Silicon (Si)	90.2
	Vanadium (V)	5.6
	Sodium (Na)	21.4
	Calcium (Ca)	36.5

Analysis and Discussion of Results

This analysis conducted a comprehensive test for key harmful elements in crude oil and residual fuel oil. The data reveals the specific characteristics of the samples and the excellent performance of the instrument:

– **Verification of Wide Dynamic Range: Synchronous Determination from Trace to Major Levels**

The test results show a vast span in the concentration of elements in the sample.

- **Trace Element Detection:** The contents of Phosphorus (P) and Zinc (Zn) are low, at 0.8 mg/kg and 1.1 mg/kg respectively. Despite the complex oil matrix background, the DITEE SWORD 510 was able to accurately capture sub-ppm level signals, thanks to the high quantum efficiency and low noise characteristics of its scientific-grade large-format CCD detector, proving the instrument's superior detection limit advantages.
- **High Content Element Detection:** The Iron (Fe) content is as high as 110.8 mg/kg, typically originating from corrosion in pipelines or storage tanks. The SWORD 510 completed precise quantification spanning three orders of magnitude (from 0.8 mg/kg to 110.8 mg/kg) in a single exposure without the need for secondary dilution of high-concentration elements. This fully validates the wide linear dynamic range and signal processing capability provided by its all-digital solid-state RF generator.

– **Precise Monitoring of Key Contaminants: Catalyst Fines (Al + Si)**

Aluminum (Al) and Silicon (Si) are the main components of catalyst powder residue from the catalytic cracking process. Their extreme hardness easily causes severe wear on engines.

- Aluminum (36.5 mg/kg) and Silicon (90.2 mg/kg) are key indicators characterizing Catalyst Fines residue in fuel oil. This analysis adopted the IP 501 alkali fusion method, thoroughly digesting the refractory silicon-aluminum oxide particles in the

sample and effectively avoiding the incomplete extraction issues that may occur with conventional acid digestion methods. The test results demonstrate that the DITEE SWORD 510 can accurately determine silicon and aluminum content in high-salt matrices, providing real and reliable data support for assessing fuel wear performance.

– **Effective Identification of Catalyst Poisons (V, Ni)**

Vanadium (V) and Nickel (Ni) are key elements that cause poisoning of refining catalysts.

- Nickel (12.0 mg/kg) and Vanadium (5.6 mg/kg) were measured in this test. The DITEE SWORD 510's Echelle grating and prism cross-dispersion optical system provided superior optical resolution (pixel resolution <0.007nm @ 200nm), effectively eliminating complex organic spectral interferences (such as overlapping iron and carbon lines) in the oil, ensuring accurate readings for these two key poisonous elements, and providing precise data support for the adjustment of refining process parameters.

Conclusion and Value

Comprehensive analysis indicates that the DITEE SWORD 510 can effortlessly handle the analytical challenges of "complex matrices, large elemental differences, and high detection limit requirements" in crude oil and residual fuel oil samples. The test data is authentic and accurate, and the proportional relationships between elements conform to oil characteristics, fully demonstrating the professional standard of this instrument in petrochemical applications.

Appendix

Sample Pre-treatment Configuration Plan

NO.	Name	Specification	Quantity
1	Electronic Balance	Ten-thousandth (0.1mg)	1 Unit
2	Platinum Crucible	100ml	2 Units
3	Fume Hood	1.5m	1 Unit
4	Muffle Furnace	Above 1000°C	1 Unit
5	Electric Stove		1 Unit
6	Ultrapure Water Machine	Resistivity 18.25	1 Unit
7	3ml Pasteur Pipette	3ml	1 Pack
8	Quantitative Filter Paper	11cm	2 Packs
9	Funnel	Glass	10 Units
10	50ml Volumetric Flask	50ml	20 Units
11	100ml Volumetric Flask	100ml	10 Units
12	100ml Beaker	100ml	10 Units
13	250ml Beaker	200ml	5 Units
14	1000ul Pipette (with tips)	1000ul	1 Set
15	Medicine Spoon	Stainless Steel	5 Units
16	Lithium Tetraborate	High-Grade Pure	1 Bottle
17	Lithium Fluoride	High-Grade Pure	1 Bottle
18	Tartaric Acid	High-Grade Pure	1 Bottle
19	Hydrochloric Acid	High-Grade Pure	5 Bottles
20	Standard Solution for Elements to be Tested	50ml, 1000ug/ml	1 Bottle Each