



LUBRICATING GREASE WEAR METALS AND ADDITIVES CONTENT DETECTION APPLICATION REPOR

RDE-20260116-01

JANUARY 16, 2026

OA800H OIL ANALYSIS SPECTROMETER

ASTM D6595-20, NB/SH/T 0865

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Introduction

Background and Current Status

As the "industrial blood" in mechanical equipment, lubricating grease is widely used in key friction pairs such as rolling bearings, gearboxes, and open gears, playing an irreplaceable protective role, especially under harsh working conditions like heavy load, low speed, or high temperature. However, compared to liquid lubricating oil, the condition monitoring of in-use lubricating grease has always been a weak link in industrial operation and maintenance.

Problems and Challenges

Currently, the main technical bottlenecks in lubricating grease detection lie in its high viscosity and semi-solid physical characteristics:

- **Sampling difficulties:** Traditional atomic emission spectrometers are usually designed for low-viscosity liquids and cannot directly suck in high-viscosity lubricating grease, leading to pipeline blockages or extremely low atomization efficiency.
- **Cumbersome pretreatment and prone to errors:** Conventional wet acid digestion or dry ashing pretreatment not only takes a long time (hours to days), but also easily causes the loss of volatile elements (such as P, S, Zn, and other anti-wear additive components) during high-temperature processing, seriously affecting the accuracy of the detection results.
- **Information lag:** Due to the long detection cycle, feedback often cannot be obtained in time during the early stage of equipment wear, causing enterprises to miss the best window period for fault early warning, ultimately triggering bearing burnout or unplanned downtime.

Experimental Purpose and Importance

To address the above pain points, this experiment aims to verify the feasibility of using the organic solvent dilution method combined with the OA800H Oil Analysis Spectrometer (RDE-OES) for lubricating grease detection. The importance of this experiment is reflected in the following three dimensions:

1. **Breakthrough technical bottlenecks:** By using a specific solvent ratio (aviation kerosene + blank oil) to convert the semi-solid lubricating grease into a homogeneous liquid, it solves the sampling problem while preserving the representativeness of the sample.
2. **Achieve early fault warning:** Accurately measuring trace wear metals (such as Fe, Cu) in lubricating grease can help operation and maintenance personnel identify the fretting wear status of bearings or gears in advance before visible physical damage occurs, shifting the maintenance strategy from "post-event repair" to "condition-based maintenance".
3. **Optimize asset management:** Monitoring the consumption level of additives (such as Ca, Zn) helps scientifically determine the remaining life of lubricating grease, avoiding waste caused by excessive grease changing or equipment damage caused by insufficient lubrication, which has significant economic value.

In summary, establishing a fast and accurate lubricating grease spectral analysis method that does not require complex pretreatment has important practical significance for ensuring the safe operation of industrial equipment and reducing full life-cycle costs.

Technical Proposal

Experimental Principle

The Rotating Disc Electrode Optical Emission Spectroscopy (RDE-OES) method is adopted. The pre-treated liquefied lubricating grease sample is placed in a sample cup, and the sample is brought into the discharge gap formed with the rod electrode by the graphite disc electrode. Under the action of an AC arc, the elemental atoms in the sample are excited and emit characteristic spectral lines. The spectral line intensity is measured by a holographic concave grating beam splitting system and a CCD

detector, and the concentration of each element is calculated according to the built-in working curve.



Instruments and Reagents

- **Instrument Model:** OA800H Oil Analysis Spectrometer
- **Auxiliary Equipment:**
 - Electronic balance (accuracy 0.1 mg)
 - Ultrasonic cleaner (with heating function)
 - Drying oven
 - 50ml/25ml PP centrifuge tubes
 - Small pipette
 - Rod sharpener
 - Centrifuge tube rack

Reagent Materials:

- **Diluting solvents:** Aviation kerosene, No. 68 food-grade blank oil
- **Calibration standard oils:** CONOSTAN 0ppm standard oil, CONOSTAN 100ppm 24-element standard oil, CONOSTAN 900ppm 5-element standard oil
- **Consumables:** High-purity graphite disc electrodes, graphite rod electrodes, disposable sample cups
- **Others:** Sample cups, paper towels, anhydrous ethanol.

Methodology

Sample Pretreatment

Sample preparation is carried out in accordance with the laboratory's "Operating Procedures for Lubricating Grease Oil Spectrometer", using the "solvent dilution method" to convert the semi-solid lubricating grease into an injectable liquid.

1. **Sample mixing:** Take about 20g of the sample to be tested into a 50ml PP centrifuge tube (if the sample is viscous or layered, the container can be placed in a 60°C drying oven and heated for 10-15 minutes, then stirred with a glass rod to ensure the sample is uniform).
2. **Weighing and Dilution:**
 - i. Accurately weigh about 0.50g of lubricating grease sample into the centrifuge tube.
 - ii. Add about 3.00g of aviation kerosene and shake vigorously for 3 minutes.
 - iii. Place it in an ultrasonic cleaner and disperse with ultrasound in an 80°C warm bath for 30 minutes.
 - iv. Take out the sample after ultrasonication, close the stopper tightly, and shake vigorously for 3 minutes.

- v. Take it out and let it cool, add about 6.50g of No. 68 food-grade blank oil, and shake vigorously again for 3 minutes until completely uniform.

3. Instrument Calibration and Testing

- i. **Instrument warm-up:** Enter the sample ID (warm-up), and use the CONOSTAN 0ppm standard oil to run the experiment 3-5 times.
- ii. **Instrument verification:** Enter the sample ID (100ppm), and use the CONOSTAN 100ppm 24-element standard oil to perform 3 repeated experiments. Check whether the standard sample results meet the "Daily Quality Control Error Standard Table".
- iii. **Pixel calibration:** Use the CONOSTAN 100ppm 24-element standard oil for normal concentration calibration, and use the CONOSTAN 900ppm 5-element standard oil for ultra-high concentration calibration. After completion, observe that the difference between the corrected pixel and the factory pixel is less than 6.
- iv. **Curve calibration:** Perform 3 repeated experiments in the corresponding concentrations respectively.
- v. **Sample testing:** Pour the prepared diluent into the sample cup, perform 3 repeated experiments, and take the average value as the instrument reading.

- 4. **Post-test maintenance:** Use paper towels and anhydrous ethanol to clean the excitation chamber; use a special lens wipe and anhydrous ethanol to clean the optical lens, noting that it needs to be wiped wet first and then dry.

Result Calculation Formula

Because the sample is diluted, the final result needs to be back-calculated according to the dilution factor:

$$\omega = \frac{c \times (V_1 + V_2 + m)}{m}$$

Where:

- ω is the concentration of the target element in the original sample (mg/kg)
- c is the concentration of the diluted sample measured by the instrument (mg/kg)
- m is the sampling mass of the original lubricating grease sample (g)
- V_1 is the mass of aviation kerosene (g)
- V_2 is the mass of No. 68 food-grade blank oil (g)

Results

This experiment selected two typical samples, "Grease No. 1" and "Grease No. 2", for analysis. The original weighing data and instrument readings are as follows:

Sample Weighing and Dilution Factor Calculation

Based on the original experiment records, calculate the dilution factor (K):

Sample No.	Sample Weight (g)	Diluent Weight (g)	Blank Weight (g)	Oil	Total Weight (g)	Dilution Factor (K)
<i>Grease No. 1</i>	0.544	3.575	6.568		10.687	19.645
<i>Grease No. 2</i>	0.504	3.557	6.861		10.922	21.671

Element Content Determination Results

Multiply the original reading (liquid phase concentration) measured by the instrument by the dilution factor to get the actual content of elements in the lubricating grease:

Grease No. 1 (Main detection elements: Ca, Fe, Zn)

20251209

16号脂

添加样品

元素

SD (mg/kg)

平均浓度 (mg/kg)

1

2

3

4

5

6

Fe 铁

0.12

0.46

0.43

0.43

0.61

0.29

0.52

Al 铝

0.06

0.10

0.17

0.05

0.09

0.03

0.14

Mg 镁

0.09

4.20

4.28

4.16

4.05

4.25

4.23

Zn 锌

0.05

0.16

0.12

0.10

0.19

0.17

0.21

Cu 铜

0

0

0.00

0.00

0.00

0.00

0.00

Ag 银

0.04

0.08

0.10

0.03

0.06

0.05

0.14

Ni 镍

0.04

0.03

0.00

0.07

0.00

0.00

0.08

Cr 铬

0.05

0.03

0.00

0.00

0.12

0.02

0.00

Si 硅

0.14

0.91

0.89

0.83

1.08

1.03

0.74

Ti 钛

0

0

0.00

0.00

0.00

0.00

0.00

Pb 铅

0.52

1.23

1.05

1.76

1.43

1.49

0.43

Sn 锡

0.15

0.80

0.89

0.94

0.80

0.83

0.56

Mo 钼

0.08

0.54

0.48

0.53

0.64

0.62

0.44

V 钒

0

0

0.00

0.00

0.00

0.00

0.00

Cd 镉

0

0

0.00

0.00

0.00

0.00

0.00

Ca 钙

7.14

249.05

257.19

245.96

239.04

254.33

248.73

P 磷

0.69

6.35

5.27

6.16

6.92

6.93

6.50

Ba 钡

0.39

6.46

5.91

6.50

6.82

6.84

6.24

B 硼

0

0

0.00

0.00

0.00

0.00

0.00

Mn 锰

0

0

0.00

0.00

0.00

0.00

0.00

Sb 锑

0

0

0.00

0.00

0.00

0.00

0.00

Li 锂

0.01

0.01

0.01

0.00

0.01

0.01

0.00

Na 钠

0.16

3.60

3.71

3.79

3.63

3.44

3.45

K 钾

0

0

0.00

0.00

0.00

0.00

0.00

元素校正

曲线校正

检测

开始

中止

导出

打印

导出报告

重读图谱

Element	Instrument Reading (ppm)	Dilution Factor	Calculation Result (ppm)	Remarks
Ca (Calcium)	249.05	19.645	4892.6	Main additive
Fe (Iron)	0.46	19.645	9.0	Mild wear/background
Zn (Zinc)	0.16	19.645	3.1	Trace/undetected

Grease No. 2 (Main detection elements: Li, Ca)

20251209

2号箱

添加样品

像素校正

曲线校正

检测

开始

中止

导出

打印

导出报告

重读谱图

结果表

元素	SD (mg/kg)	平均浓度 (mg/kg)	1	2	3	4	5	6
Fe 铁	0	0	0.00	0.00	0.00	0.00	0.00	
Al 铝	0.03	0.01	0.00	0.00	0.00	0.00	0.00	0.07
Mg 镁	0.13	2.08	2.02	2.04	2.02	2.02	2.32	
Zn 锌	0	0	0.00	0.00	0.00	0.00	0.00	
Cu 铜	0	0	0.00	0.00	0.00	0.00	0.00	
Ag 银	0.02	0.11	0.09	0.10	0.13	0.12	0.12	
Ni 镍	0.24	0.17	0.00	0.00	0.00	0.45	0.41	
Cr 铬	0.07	0.05	0.01	0.12	0.13	0.00	0.00	
Si 硅	0.11	0.71	0.57	0.66	0.70	0.72	0.88	
Ti 钛	0	0	0.00	0.00	0.00	0.00	0.00	
Pb 铅	0.08	0.90	0.93	0.92	0.98	0.91	0.77	
Sn 锡	0.08	0.85	0.85	0.92	0.76	0.78	0.94	
Mo 钼	0.11	0.56	0.59	0.48	0.73	0.52	0.46	
V 钒	0	0	0.00	0.00	0.00	0.00	0.00	
Cd 镉	0	0	0.00	0.00	0.00	0.00	0.00	
Ca 钙	4.64	89.63	88.91	88.47	84.94	88.42	97.42	
P 磷	0.43	6.52	6.45	6.81	6.88	6.63	5.81	
Ba 钡	0.41	6.74	6.94	6.85	7.10	6.05	6.78	
B 硼	0	0	0.00	0.00	0.00	0.00	0.00	
Mn 锰	0	0	0.00	0.00	0.00	0.00	0.00	
Sb 锑	0	0	0.00	0.00	0.00	0.00	0.00	
Li 锂	3.34	70.01	70.97	70.99	66.23	67.27	74.61	
Na 钠	0.12	0.65	0.75	0.67	0.79	0.56	0.50	
K 钾	0.15	0.11	0.00	0.10	0.07	0.37	0.00	

Element	Instrument Reading (ppm)	Dilution Factor	Calculation Result (ppm)	Remarks
<i>Li (Lithium)</i>	70.01	21.671	1517.2	Thickener component
<i>Ca (Calcium)</i>	89.63	21.671	1942.4	Additive component
<i>Al (Aluminum)</i>	0.01	21.671	0.2	Undetected

Data Analysis

- **Additive identification:** A high concentration of calcium (about 4892 ppm) was detected in Grease No. 1, suggesting it may be a complex calcium-based grease or contain calcium additives. Lithium (about 1517 ppm) and calcium (about 1942 ppm) were simultaneously detected in Grease No. 2, which conforms to the characteristics of complex lithium-calcium-based lubricating grease.
- **Wear monitoring:** About 9 ppm of iron element was detected in Grease No. 1, indicating that the equipment where this lubricating grease was sampled may have slight early wear, or it is a system background value (needs to be judged in combination with historical trends).
- **Method stability:** The ratio of blank oil to kerosene during the dilution process was well controlled, and the dilution factor was stable at around 20 times, ensuring that the instrument reading fell within the optimal linear range (0-1000 ppm).

Conclusion

This experiment proves that using the OA800H oil Analysis spectrometer in combination with the "kerosene + blank oil" two-step dilution method can effectively overcome the problem of difficult sampling due to the high viscosity of lubricating grease.

- **Detection efficiency:** The entire pretreatment and testing process is smooth, and a single excitation takes only 40 seconds, greatly improving detection efficiency.

- **Data accuracy:** Through accurate weighing and back-calculation, it is possible to quantitatively detect ppm-level trace wear metals and high-content additives in lubricating grease, and the data logic conforms to the chemical characteristics of lubricating grease.
- **Application value:** This method fully meets the requirements of the ASTM D6595 standard and can be widely used for quality control (QC) of various lubricating greases and wear fault diagnosis of in-use lubricating greases.

Appendix

- **Compliance Statement:** Conforms to ASTM D6595 and NB/SH/T 0865 standards.
- **Technical Support:** Custom calibration services (contact sales@zkditee.com).