

WDXRF ultra low sulfur analyzer

Micro-Z ULS

Excellent performance for ultra-low sulfur analysis in petroleum



1. Introduction

The newly released wavelength-dispersive X-ray fluorescence sulfur analyzer, Micro-Z ULS (Ultra Low Sulfur) meets the current needs for the analysis of sulfur in petroleum.

The sulfur content in gasoline needs to be controlled in order to reduce air pollution, lengthen the lifetime of automobile catalysts, and improve engine reliability. Many countries and regions have established regulations for gasoline sulfur (S) content in line with Euro 5⁽¹⁾ (less than 10 ppm).

For compliance verification, X-ray fluorescence (XRF) spectrometry is the definitive analysis tool for use at distribution terminals and refineries, as well as mobile or stationary testing laboratories.

In recent years, there has been an increasing need for an instrument which does not require the use of helium gas for situations where acquisition or delivery of helium to the analysis site is difficult.

The Micro-Z ULS was designed to meet these trends

- ◆Optimized optics for sulfur analysis
- ◆No need for cooling water
- ◆Plug-in power supply system
- ◆Simple and easy operation for daily analysis
- ◆Helium gas is not required
- ◆Safety method without combustion
- ◆Meets the requirement of ASTM D2622⁽²⁾, ISO20884⁽³⁾ and JIS K2541-7⁽⁴⁾

2. Specifications

The Micro-Z ULS is a benchtop analyzer which is equipped with an air-cooled 40 W Cr-target X-ray tube

and a doubly curved RX-9 analyzing crystal optimized for low concentration sulfur. The analyzer is operated at 100–120V AC 15A or 200–240V AC 10A, which can be supplied by a wall outlet. The atmosphere of the optical path is vacuum so helium gas is not required and the detector is a sealed proportional counter (S-PC) which does not need any detecting gas supply.

The Micro-Z ULS has a built-in operation panel and analysis results are displayed on the built-in LCD panel. If necessary, a thermal dot printer or personal computer can be connected and the analysis results can be automatically printed or exported to the computer.

To measure a sample, a simple and inexpensive plastic sample cell as shown in Fig. 1 is used.



Fig. 1. Sample cell for Micro-Z ULS.

About 4 mL of liquid sample is poured into the cell and covered with a sample film.

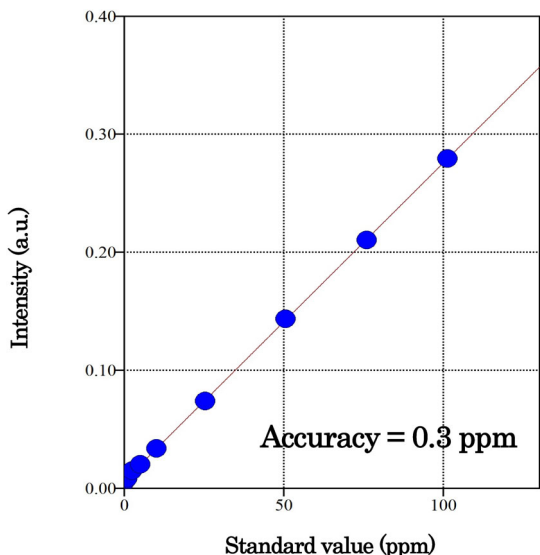


Fig. 2. Calibration curve of gasoline for S from 0 to 100 ppm.

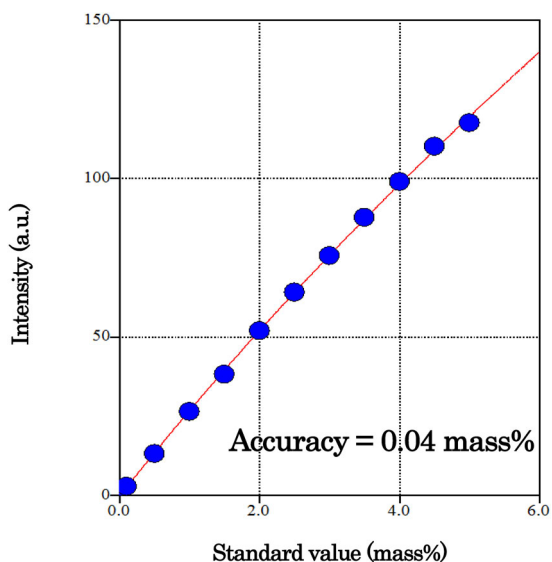


Fig. 3. Calibration curve of crude oil for S from 0 to 5 mass%.

3. Analysis example

Figures 2 and 3 show the calibration curves created by the Micro-Z ULS and exported to a personal computer. Accuracy of the calibration curves are 0.3 ppm for the lower concentration and 0.04 mass% for the higher concentration. The standard deviation (σ) calculated from the repeat measurement for the blank sample is 0.1 ppm and the LLD defined as 3σ of the blank sample is 0.3 ppm. Table 1 shows the results of the repeatability test according to ASTM D2622-10. It shows that Micro-Z ULS is compliant with the standard.

4. Summary

The Micro-Z ULS is designed to enable simple, easy and high-performance operation for daily analysis with low running cost. It has a wide analysis range from ppm to mass% level of sulfur. The LLD of the Micro-Z ULS is 0.3 ppm for S, which means it can accurately analyze ultra low sulfur petroleum. The Micro-Z ULS has

Table 1. Repeatability test result for Micro-Z ULS. (qualification test for ASTM D2622-10)

(a) Diesel fuel			(b) Gasoline		
Run #	Average (ppm)	Difference (ppm)	Run #	Average (ppm)	Difference (ppm)
1	8.0	0.3	1	11.8	0.8
2	8.2	0.1	2	11.7	0.7
3	8.1	0.3	3	12.1	0.1
4	8.0	0.3	4	11.8	0.8
5	8.2	0.1	5	11.5	0.1
6	8.1	0.3	6	11.5	0.1
7	8.1	0.3	7	11.8	0.4
8	8.2	0.1	8	12.1	0.4
9	8.1	0.0	9	12.1	0.4
10	8.1	0.1	10	11.7	0.4
11	8.2	0.5	11	11.7	0.4
12	8.5	0.0	12	11.9	0.2
13	8.6	0.2	13	11.7	0.1
14	8.7	0.0	14	12.0	0.6
15	8.4	0.7	15	12.1	0.4
16	8.3	0.6	16	11.6	0.6
17	8.3	0.5	17	11.5	0.3
18	8.0	0.2	18	11.6	0.0
19	8.2	0.7	19	11.6	0.1
20	8.3	0.5	20	11.8	0.4
Avg.	8.2		Avg.	11.8	
Maximum		0.7	Maximum		0.8
<i>r</i> (repeatability) defined by ASTM D2622		0.8	<i>r</i> (repeatability) defined by ASTM D2622		1.1

excellent repeatability, which meets regulations such as ASTM D2622, ISO20884 and JIS K2541-7.

References

- (1) DIRECTIVE 2009/30/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 23 April 2009, amending Directive 98/70/EC as regards the specification of petrol, diesel and gas-oil and introducing a mechanism to monitor and reduce greenhouse gas emissions and amending Council Directive 1999/32/EC as regards the specification of fuel used by inland waterway vessels and repealing Directive 93/12/EEC.
- (2) ASTM D2622-10, Standard Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry.
- (3) ISO20884:2011(E), Petroleum products—Determination of sulfur content of automotive fuels—Wavelength-dispersive X-ray fluorescence spectrometry.
- (4) JIS K2541-7:2003, Crude oil and petroleum products—Determination of sulfur content Part 7: Wavelength-dispersive X-ray fluorescence method.