

# Micro-Z ULS

Wavelength dispersive X-ray fluorescence

WDXRF ultra-low sulfur analyzer for ULSD and Tier 3 gasoline



Sulfur

S

16

Atomic Weight = 32.07

ASTM D2622-10

ISO 20884

JIS K12541-7



**Rigaku**

POWERING NEW PERSPECTIVES

# Micro-Z ULS



## Ultra-low sulfur analyzer delivers performance with Rigaku durability

### Proven design and analytical functionality

Designed for ultra-low level sulfur analysis of diesel, petrol (gasoline) and other fuels, the Rigaku Micro-Z ULS wavelength dispersive X-ray fluorescence (WDXRF) analyzer features a novel design that measures both the sulfur peak and the back ground intensity. The ability to measure and correct for changes in background intensity delivers a better net peak intensity measurement, resulting in superior calibrations and enhanced real world precision. Rigaku Micro-Z ULS complies with ASTM 2622-10, ISO 20884 and JIS K2541-7 methods.

### Euro 5 / US Tier 3 / China National V gasoline production and certification

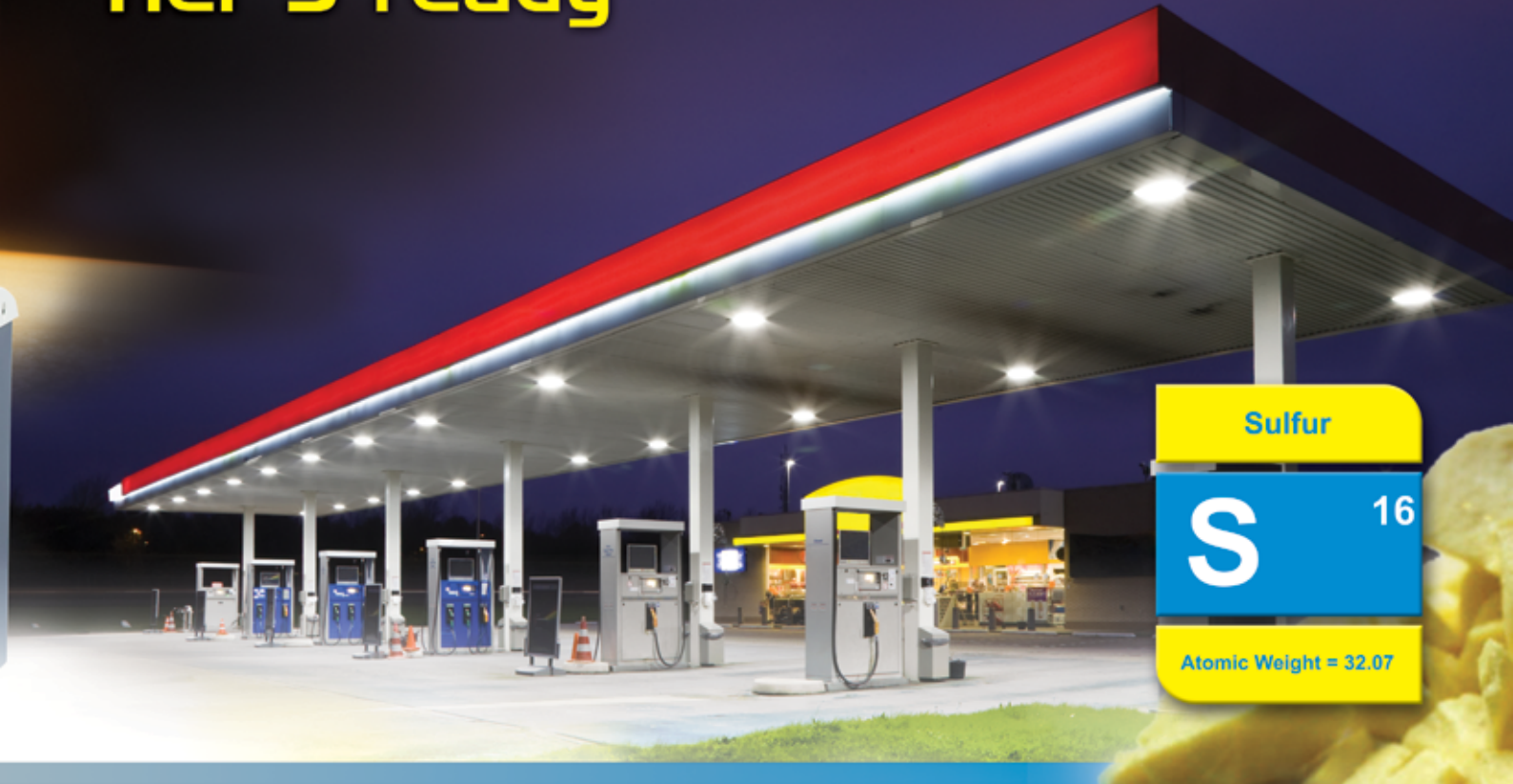
Similar to current European (2003/17/EC, Jan 2009), California (CARB LEV III, 2012), Japanese, Israeli and South Korean regulations, the new US EPA Tier 3 rules specify that gasoline meet an annual average standard of 10 ppm sulfur by January 1, 2017. China will implement their similar National V regulations along this same timeframe. Large US refineries (those producing greater than 75 Mbpd) must comply with Tier 3 standards by 2017. Refiners producing below 75 Mbpd must meet Tier 3 regulation standards by 2020. For Tier 3, and consistent with the US EPA's performance based measurement system (PBMS), no specific analytical method is precluded. However, for laboratories worldwide, the benefit of the Rigaku Micro-Z ULS is that it strictly complies with ASTM D2622-10, eliminating the risk and uncertainty of attempting to implement other methods.

### Ultra-low sulfur diesel (ULSD) production and certification

ULSD fuel enables the use of cleaner technology diesel engines equipped with advanced emissions control devices, resulting in significantly improved air quality. Annual emission reductions are estimated to be the equivalent of removing the pollution from >90 % of today's trucks and buses, when the current vehicle fleet is replaced (est. 2030). A 15 ppm specification, known as Ultra Low Sulfur Diesel (ULSD), was phased in for highway diesel fuel in the US beginning in 2006. Similar, albeit lower, 10 ppm limits are currently in effect in Europe, Japan, Israel, New Zealand and Australia. Other countries, like Chile and Canada, have the 15 ppm limit like the US. In addition, Russia and China are both rapidly phasing in ULSD. Likewise for ultra-low sulfur gasoline, for laboratories worldwide, the benefit of the Rigaku Micro-Z ULS is that it strictly complies with ASTM D2622-10, delivering easy-to-use and cost-effective compliance validation.

# PETROL · DIESEL · ULSD · GASOLINE

## Tier 3 ready



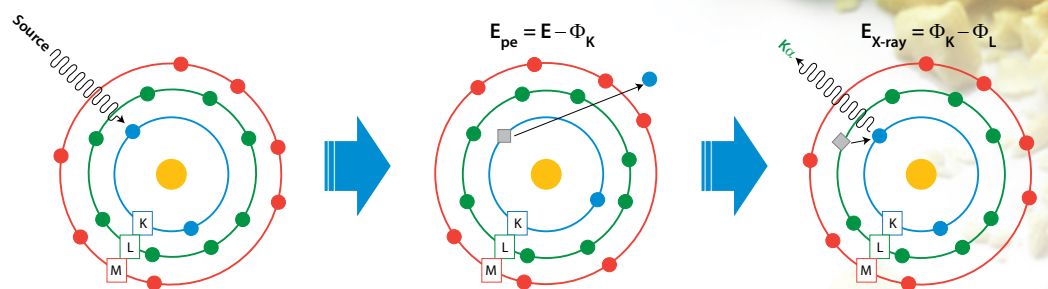
**Sulfur**

**S** 16

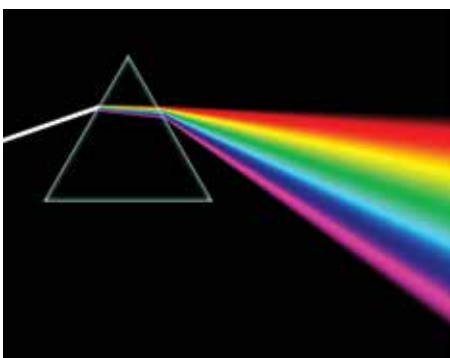
Atomic Weight = 32.07

### What is XRF?

In X-ray fluorescence (XRF), an electron can be ejected from its atomic orbital by the absorption of light (photon) from an X-ray tube. When an inner orbital electron is ejected (middle image), a higher energy electron transfers to fill the vacancy. During this transition, a characteristic photon may be emitted (right image) that is of a unique energy for each type of atom. The number of characteristic photons per unit time (counts per second or cps) is proportional to the amount of that element in a sample. Thus, qualitative and quantitative elemental analysis is achieved by determining the energy of X-ray peaks in a sample spectrum and measuring their associate count rates.



**X-ray fluorescence schematic**



**Like a prism spreads the spectrum of visible light, so the analyzing crystals disperse light in the X-rays spectral region.**

### How WDXRF works

Wavelength dispersive X-ray spectroscopy (WDXRF) is a method used to separate and measure the characteristic fluorescent X-rays emitted from a sample. The technique employs an analyzing crystal to spatially spread the X-ray light, much like a prism spreads visible light into its component colors. The wavelength of the impinging X-ray and the crystal's lattice spacings are related by Bragg's law and produce constructive interference when they satisfy the Bragg equation. The X-rays emitted by the sample are collimated by parallel metal blades (collimator) and irradiate an automatically selected analyzing crystal at a precise angle. X-ray light diffracted by the analyzing crystal is spatially spread out, so that characteristic photons may be collected by a detector positioned at a precise angle to record an X-ray intensity of a specific element.





Sulfur

**S** 16

Atomic Weight = 32.07

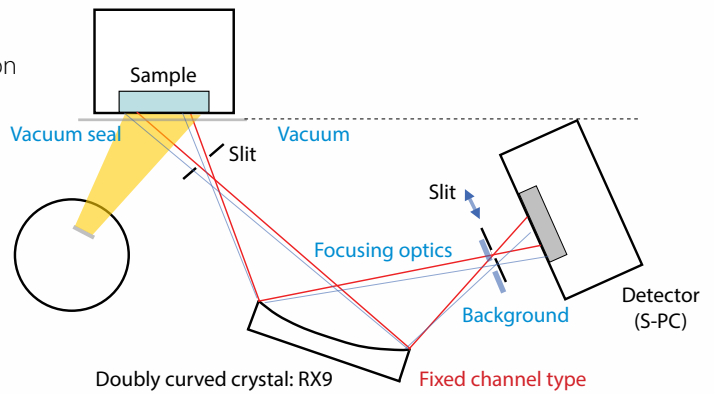
# ASTM D2622-10, ISO 20884 and JIS K12541-7 compliant

## Superior optics for reliable performance

The Rigaku Micro-Z ULS is the ideal solution for sulfur analysis of petroleum based fuels, with a lower limit of detection (LLD) of 0.3 ppm sulfur. Employing robust fixed optics in a vacuum environment, and featuring a specially designed doubly curved RX-9 analyzing crystal, the Micro-Z ULS delivers consistent high-intensity measurements.

## Movable slit provides background correction

The unique Rigaku automatic background correction enhances the goodness of the calibration fit, providing unmatched precision for short-term repeatability and long-term reproducibility.



Schematic of the close-coupled optical design. Note that the moveable receiving slit allows for measurement of both peak and background intensity.





# Micro-Z ULS

## Designed for routine non-technical operation

Specifically designed for non-technical users, all operations—from calibration through routine analysis—can be performed via the easy-to-use interface. The analyzer can be powered by any standard wall AC outlet.

## High resolution WDXRF spectrometer

A unique doubly curved analyzing crystal delivers superior resolution for sulfur that ensures the required performance for production and validation of ULSD and Tier 3 gasolines. With the ability to perform automatic background corrections, Micro-Z ULS is strictly compliant with ASTM D2622-10. As a vacuum type design, no helium (He) gas is ever required. And the spectrometer features easy maintenance for the vacuum seal system.



Instrument status, through to analytical results, are selectable with the touch of a finger.

### Calibration results using Micro-Z ULS (units: ppm)

Material	Diesel fuel	Isooctane (for gasoline)
Calibration range	0 – 100	0 – 100
Accuracy	0.49	0.37
LLD	0.3	0.3

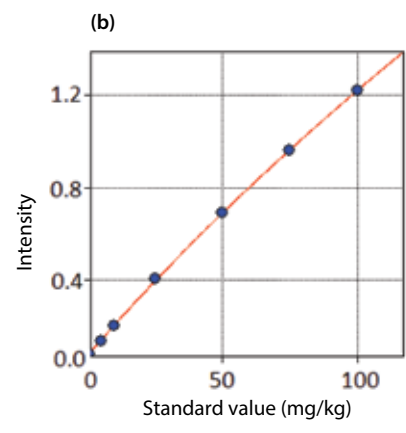
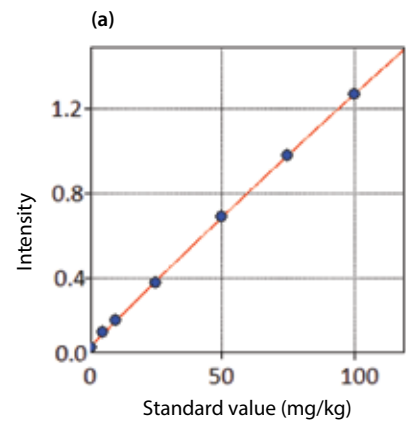
### (a) Diesel fuel

Run #	Average (mg/kg)	Difference (mg/kg)
1	8.0	0.3
2	8.2	0.1
3	8.1	0.3
4	8.0	0.3
5	8.2	0.1
6	8.1	0.3
7	8.1	0.3
8	8.2	0.1
9	8.1	0.0
10	8.1	0.1
11	8.2	0.5
12	8.5	0.0
13	8.6	0.2
14	8.7	0.0
15	8.4	0.7
16	8.3	0.6
17	8.3	0.5
18	8.0	0.2
19	8.2	0.7
20	8.3	0.5
Avg.	8.2	
Maximum		0.7
ASTM limit		0.8

### (b) Gasoline

Run #	Average (mg/kg)	Difference (mg/kg)
1	11.8	0.8
2	11.7	0.7
3	12.1	0.1
4	11.8	0.8
5	11.5	0.1
6	11.5	0.1
7	11.8	0.4
8	12.1	0.4
9	12.1	0.4
10	11.7	0.4
11	11.7	0.4
12	11.9	0.2
13	11.7	0.1
14	12.0	0.6
15	12.1	0.4
16	11.6	0.6
17	11.5	0.3
18	11.6	0.0
19	11.6	0.1
20	11.8	0.4
Avg.	11.8	
Maximum		0.8
ASTM limit		1.1

Repeatability results for Micro-Z ULS (ASTM D2622-10)



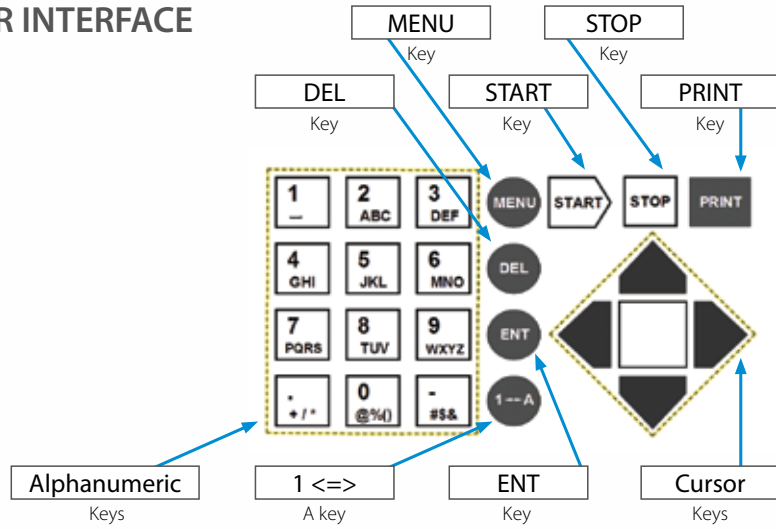
Calibration curve for each material of Micro-Z ULS

- (a) Diesel fuel
- (b) Isooctane (for gasoline)

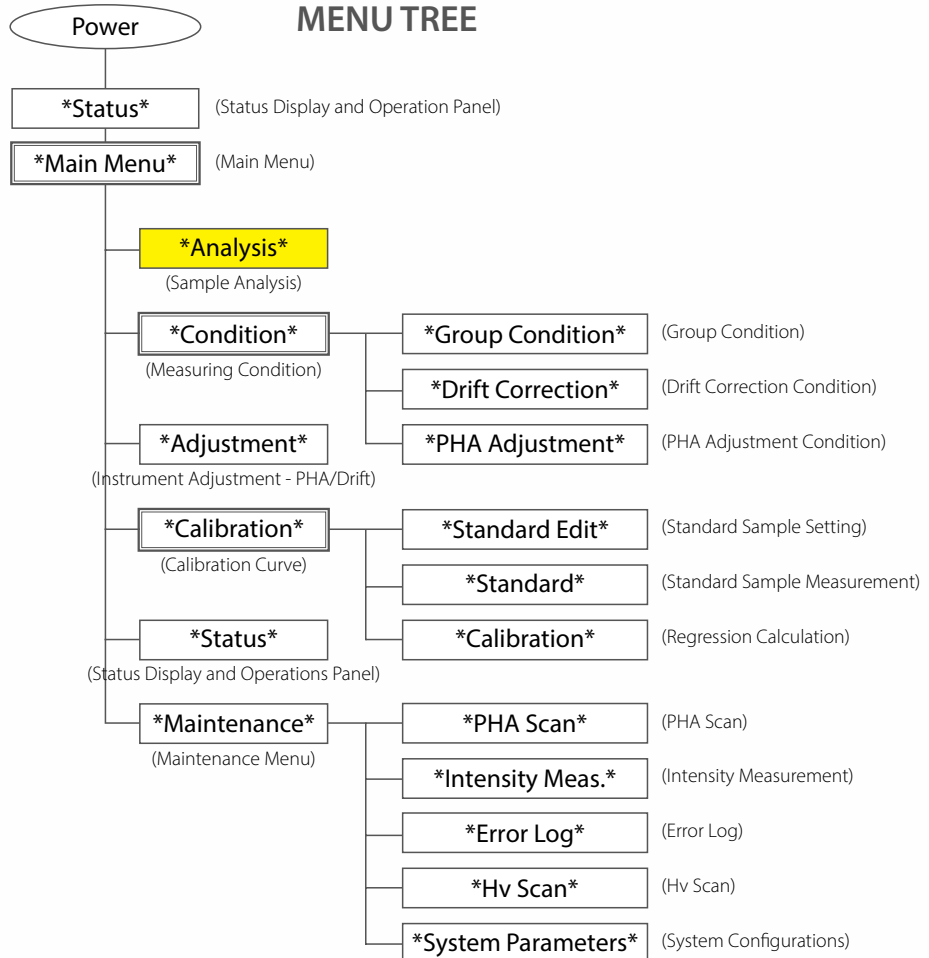


# Operational Guide

## USER INTERFACE



## MENU TREE





# Micro-Z ULS

## Specifications

### Instrument:

- Wavelength dispersive X-ray fluorescence analyzer
  - Includes vacuum pump
- Element range: sulfur (S), LLD = 0.3 ppm
- Application: analysis of fuels for sulfur content
- Single measurement position (std. 35 mm cup)

### Supported methods:

- ASTM D2622-10
- ISO 20884 and JIS K12541-7

### Excitation:

- 40 kV Cr-anode X-ray tube
- 40 W max power

### Detection:

- Doubly curved RX-9 analyzing crystal
- Sealed proportional counter
- Fixed optics with switchable receiving slit
  - To select peak or background
- Pulse height analyzer (PHA)

### Atmosphere:

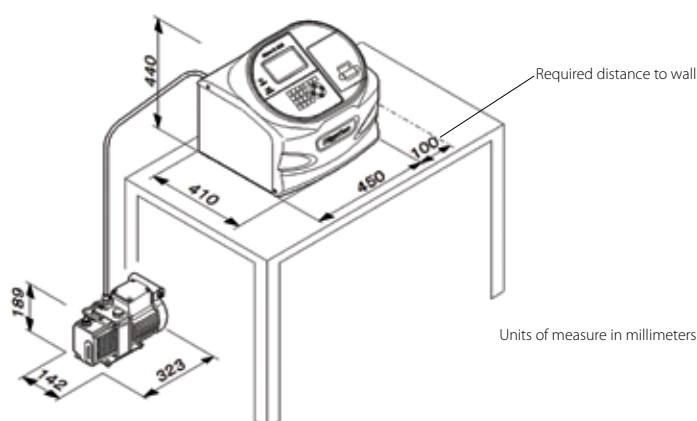
- Sample analysis in air
- Optics are under vacuum

### Environmental conditions:

- Ambient temperatures 15 – 28°C
- Relative humidity <75% non condensing
- Vibration undetectable by human
- Free from corrosive gas, dust, and particles

### Dimensions / power:

- Analyzer: 450 x 410 x 440 mm, 36 kg
- Pump: 323 x 142 x 180 mm, 10 kg
- 100-120 VAC, 15 A or 200-240 VAC, 10 A



## Backed by Rigaku

Since its inception in 1951, Rigaku has been at the forefront of analytical and industrial instrumentation technology. Today, with hundreds of major innovations to our credit, the Rigaku Group of Companies are world leaders in the field of analytical X-ray instrumentation. Rigaku employs over 1,400 people worldwide in operations based in Japan, the U.S., Europe, South America and China.

### Computer:

- Embedded dedicated processor
- Proprietary operating system

### Software:

- Up to 10 calibrations
- Up to 30 standards per calibration
- Selectable analysis time (up to 900 s)
- Drift correction
- Pulse height analyzer (PHA) adjustment

### User interface:

- LCD display
- Embedded computer
- Membrane keypad and navigation

### Options:

- Thermal dot printer
- RS-232C data output



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[www.rigaku.com/products/wdxf](http://www.rigaku.com/products/wdxf)



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