



**Sulfur**

**S** 16

Atomic Weight = 32.07  
\*

## NEXQC+



- **Model**  
NEX QC+
- **X-ray tube**  
4 W Ag-anode
- **Detector**  
SDD
- **Sample Type**  
Diesel and isoctane to simulate gasoline
- **Film**  
Polypropylene 4  $\mu$ m (diesel)  
Ultralene<sup>®</sup> 4  $\mu$ m (gasoline)
- **Analysis Time**  
300 sec
- **Environment**  
Helium
- **Sample Ring**  
Single Position 32 mm

## SCOPE

The analysis of sulfur in ULSD (ultra-low sulfur diesel) is demonstrated, as well as the measurement of ultra-low sulfur in gasoline.

## BACKGROUND

Regulations around the world have limited the amount of sulfur in various fuels oils with particular attention to diesel fuel. For many years, road diesel has been limited to a maximum sulfur concentration between 10 – 15 ppm, depending on the global region. Now, these limits are expanded to all diesel fuel, including use in large engines and off-road diesel engines.

## SAMPLE PREPARATION

To measure a sample, gently shake the sample bottle, allow bubbles to settle, and fill a 32 mm XRF sample cup with 4.0 g of sample. For ultra-low sulfur measurements, make a fresh cup, prepare a fresh sample and measure immediately after the sample is prepared. Remove sample from analysis chamber immediately after measurement completes. All samples must be homogeneous and stable.

**ULTRA-LOW SULFUR  
HIGHWAY DIESEL FUEL  
(15 ppm Sulfur Maximum)**

**Required** for use in all model year 2007 and later highway diesel vehicles and engines.

Recommended for use in all diesel vehicles and engines.

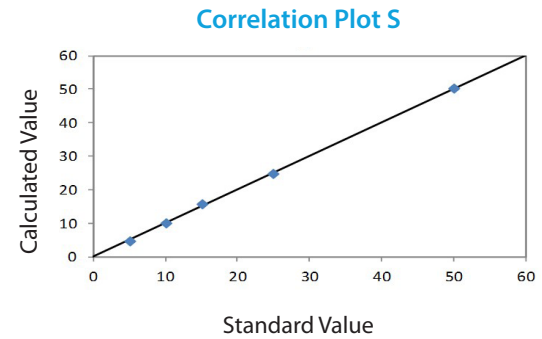
Ultralene is a registered trademark of Metuchen Scientific, Inc. DBA Spex CertiPrep Corporation.

## DIESEL

### CALIBRATION

Empirical calibration was built using a suite of 6 certified diesel calibration standards.

Element: S Units: ppm		RMS Deviation: 0.31 Correlation: 0.99969	
Sample I.D.	Standard Value	Calculated Value	
STD 1	5.0	4.7	
STD 2	10.0	10.1	
STD 3	15.0	15.5	
STD 5	25.0	24.7	
STD 6	50.0	50.0	



### PRECISION

Instrument repeatability (precision) is determined by ten repeat analyses of a sample in static position using a 300 sec analysis time.

Element: S Units: ppm				
Sample	Standard Value	Average Value	Std. Dev	% Relative
STD 2	10.0	10.2	0.48	4.8
STD 6	50.0	50.9	0.50	1.0

### DETECTION LIMITS

Ten repeat analyses of a blank diesel sample containing no sulfur are taken with the sample in static position and the standard deviation is determined. The lower limit of detection (LLD) is then defined as three times the standard deviation.

Atmosphere	LLD	Analysis Time
Helium	1.3 ppm	300 sec
Helium	0.9 ppm	600 sec
Air	3.0 ppm	300 sec
Air	2.0 ppm	600 sec

LLDs shown are typical and may differ depending on measurement time used and the overall elemental composition of the sample being tested.

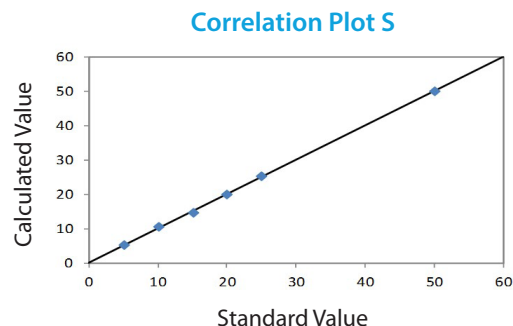
Note: 1 ppm = 1 mg/kg

## GASOLINE

### CALIBRATION

Empirical calibration was built using a suite of 7 certified isooctane calibration standards to simulate gasoline.

Element: S Units: ppm		RMS Deviation: 0.32 Correlation: 0.99958	
Sample I.D.	Standard Value	Calculated Value	
STD 1	5.0	5.1	
STD 2	10.0	10.4	
STD 3	15.0	14.5	
STD 4	20.0	19.8	
STD 5	25.0	25.1	
STD 6	50.0	50.1	



### PRECISION

Instrument repeatability (precision) is determined by ten repeat analyses of a sample in static position using a 300 sec analysis time.

Element: S Units: ppm				
Sample	Standard Value	Average Value	Std. Dev	% Relative
STD 2	10.0	9.71	0.20	2.0%
STD 5	50.0	50.2	0.47	0.9%

### DETECTION LIMITS

Ten repeat analyses of a blank isooctane sample containing no sulfur are taken with the sample in static position and the standard deviation is determined. The lower limit of detection (LLD) is then defined as three times the standard deviation.

Atmosphere	LLD	Analysis Time
Helium	0.7 ppm	300 sec
Helium	0.5 ppm	600 sec
Air	1.2 ppm	300 sec
Air	0.9 ppm	600 sec

LLDs shown are typical and may differ depending on measurement time used and the overall elemental composition of the sample being tested.

Note: 1 ppm = 1 mg/kg

## INTERNATIONAL STANDARD TEST METHODS

The Rigaku NEX QC+ complies with ISO 13032 for the measurement of ultra-low sulfur between 8 – 50 mg/kg in diesel fuels and gasoline. NEX QC+ also complies with the following ASTM and international standard test methods for the measurement of sulfur up to 5% in various petroleum oils and fuels.

ASTM D4294	ISO 20847	ISO 8754	IP 496	IP 336	JIS K 2541-4
16 ppm - 5%	30 - 500 mg/kg	100 mg/kg - 5%	100 mg/kg - 5%	100 mg/kg - 5%	0.01 - 5%

## CONCLUSION

The results shown here indicate the Rigaku NEX QC+ EDXRF analyzer gives excellent performance for the measurement of ULSD and ultra-low sulfur gasoline in accordance with ISO 13032. The versatility of the NEX QC+ also makes it an ideal tool for the measurement of many other elements and oil matrices throughout the petroleum industry.