Sulfur in Oil Analyzer

The Rigaku NEX QC sulfur in oil analyzer for fuel and lubricating oils
Sulfur in Oil Analyzer

The ASTM D4294 sulfur-in-oil analyzer from Rigaku combines established functionality with unmatched versatility

Sulfur will always be an important element in crude oils and fuel oils. Sulfur content is regulated in many products, and plays an important role in fuel quality and control of polluting emissions. Around the world, regulations are limiting the amount of sulfur allowable in diesel fuels, kerosene, heating oils, etc., thus affecting the price and quality of crude oil based on its sulfur content.

Reliably characterizing the sulfur content of crudes ensures proper quality for the various feedstocks at the refinery, and optimum blending ratios when combining different crudes to meet a desired sulfur concentration. Monitoring sulfur is also very important when characterizing other similar oils, like residual oils and bunker fuels. To meet the needs of the industry, Rigaku offers NEX QC, a simple and versatile benchtop EDXRF analyzer for the analysis of sulfur and other elements in crude oil, petroleum oils and fuels.

Modern design and functionality
As a premium low cost benchtop Energy Dispersive X-ray Fluorescence (EDXRF) analyzer, the Rigaku NEX QC delivers compliance with ASTM D4294 with an easy-to-learn software interface in a robust package specifically designed for the petroleum industry. Optimized for routine determination of sulfur in oil, the new Rigaku NEX QC features an intuitive and modern “icon-driven” touch screen, a smartphone-style interface for easy operation and a built-in thermal printer for convenience.

Up-to-date X-ray source/detector
A 50 kV X-ray tube and Peltier cooled semiconductor detector deliver exceptional repeatability and long-term reproducibility, with excellent element peak resolution. The high voltage, along with multiple automated X-ray tube filters, provides multi-element analysis capability for unmatched performance with low limits-of-detection (LOD).
Computational dexterity
In addition to being remarkably easy to use, the Rigaku NEX QC sulfur analyzer is powered by sophisticated software. Empirical calibration curves may be linear, quadratic or hyperbolic fits. To compensate for the presence of other elements in oil, intensity-based or concentration-based alpha (α) corrections may be enabled. C/H correction is also available to compensate for light element matrix changes in the oil and/or the presence of low atomic number (low-Z) additives and contaminants. All calibration functions are accessible via intuitive icons and with the touch of a finger.

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 their credit, the Rigaku Group of Companies are world leaders in the field of analytical X-ray instrumentation. Rigaku employs over 1,100 people worldwide in operations based in Japan, the U.S., Europe, South America and China.

Instrument status, spectra, and analytical results are icon selectable with the touch of a finger.

Calibration curves and statistics are accessible with familiar smartphone style interface.

Paper copies of analytical results are conveniently available from the front mounted thermal printer.

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**Sample ID:** tank 19  
**Timestamp:** 10:40:07 2012-07-27  
**Instrument:** NEX QC S/N QC1002  
**Product:** S in Diesel  
**Total Acquisition Time:** 100 sec  
**ID Result**  
Sulfur 0.071 %

**Sample ID:** tank 11  
**Timestamp:** 10:10:07 2012-07-27  
**Instrument:** NEX QC S/N QC1002  
**Product:** S in Diesel  
**Total Acquisition Time:** 100 sec  
**ID Result**  
Sulfur 0.071 %

**Sample ID:** tank 19  
**Timestamp:** 10:49:07 2012-07-27  
**Instrument:** NEX QC S/N QC1002  
**Product:** S in Diesel  
**Total Acquisition Time:** 100 sec  
**ID Result**  
Sulfur 0.104 %

**Sample ID:** tank 99  
**Timestamp:** 12:47:07 2012-07-27  
**Instrument:** NEX QC S/N QC1002  
**Product:** S in Diesel  
**Total Acquisition Time:** 100 sec  
**ID Result**  
Sulfur 0.471 %
ASTM D4294 Method for Sulfur

The ASTM D4294 sulfur-in-oil analyzer from Rigaku combines established functionality with unmatched versatility.

**International standardized test methods**

For the determination of traditional levels of sulfur (S) in crude, bunker fuel, diesel and other petroleum raw materials and refined products, the Rigaku NEX QC offers compliance for:

- **ASTM D4294-10**: 16 ppm – 5 wt%
- **ISO 20847**: 30 – 500 mg/kg
- **ISO 8754**: 100 mg/kg – 5 wt%
- **IP 336**: 100 mg/kg – 5 wt%
- **IP 496**: 100 mg/kg – 5 wt%
- **JIS K 2541-4**: 0.01 – 5 wt%

The versatility and performance of the Rigaku NEX QC is also demonstrated by its ability to perform other petroleum related applications like multiple elements (P, S, Ca, Zn, and Ba) in lubricating oils.

- **ASTM D6481**

**Multi-element versatility**

Rigaku NEX QC is capable of measuring more than just sulfur. Multi-element analysis is important for detecting crude oil contamination or adulteration by the presence of salt and other chlorine-bearing compounds. Left undetected, the presence of Cl can bias the S reading high, and potentially leave the presence of Cl unnoticed. NEX QC has the ability to detect Cl and other elements, and correct for the presence of Cl so that it does not bias the S reading.

Calibration curves and statistics are accessible with familiar smartphone style interface.
**Touchscreen interface**
High-resolution, modern, user-friendly touch screen navigation and instrument control.

**5 ppm sulfur (S) detection limit**
ASTM D4294 method is met for diesel oil as analyzed in air. Repeatability is ±3.4 ppm at 100 ppm with a 120 s measurement in air.

**Built-in printer**
Thermal printer provides fast hard copy results when and where you need them.

**X-ray tube conservation**
By operating only during data collection, X-ray tube wear and tear is minimized.

**C/H correction**
Correction is available to compensate for calibration error caused by different types of oil.

**Detector protection mechanism**
An easily changeable plastic film is positioned in between the detector and the sample holder to protect the instrument in the event of leaks or spills.

**Up to 38 calibrations**
A large number of calibrations is available, at the touch of a finger, to support a vast array of applications and sample types.

**Accepts standard XRF cups**
No special cups needed. Use industry standard Chemplex™, Spex™, VHGM™ or Premier™ cups.

**Digital data output**
Ethernet RJ-45 jack and USB port for output to LIMS or memory stick. Data is available in either CSV or PDF formats.

**Single position or autosampler**
Standard single position configuration can be supplemented with an optional autosampler.

**Removable sample trays**
Interchangeable optional autosampler trays may be pre-loaded, or swapped in and out, to increase efficiency or where throughput is important. Supports 32 mm and 40 mm cups.
Calibration

Sulfur in Bunker Fuel - High Range 0,50 - 5,00% S

**Calibration**
An empirical calibration was built using a set of commercially available certified bunker oil standards, which is similar to crude or residual oil. Accuracy is optimized by developing separate calibration methods for different sulfur ranges, if needed. A summary of the calibration for 0,50 – 5,00% S is shown here, using a measurement time of 100 sec.

<table>
<thead>
<tr>
<th>Sample ID.</th>
<th>Standard Value</th>
<th>Calculated Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>STD 6</td>
<td>0,50</td>
<td>0,505</td>
</tr>
<tr>
<td>STD 7</td>
<td>1,00</td>
<td>0,994</td>
</tr>
<tr>
<td>STD 8</td>
<td>2,50</td>
<td>2,488</td>
</tr>
<tr>
<td>STD 9</td>
<td>3,00</td>
<td>3,011</td>
</tr>
<tr>
<td>STD 10</td>
<td>4,00</td>
<td>4,015</td>
</tr>
<tr>
<td>STD 11</td>
<td>5,00</td>
<td>4,988</td>
</tr>
</tbody>
</table>

**Repeatability**
To demonstrate repeatability (precision), the select samples were chosen from the set of calibration standards. Each sample was measured in static position for ten repeat analyses using a total analysis time of 100 sec per measurement, with typical results shown below.

<table>
<thead>
<tr>
<th>Sample ID.</th>
<th>Standard Value</th>
<th>Calculated Value</th>
<th>Standard Dev</th>
<th>% Relative</th>
</tr>
</thead>
<tbody>
<tr>
<td>STD 7</td>
<td>1,00</td>
<td>0,993</td>
<td>0,004</td>
<td>0,4</td>
</tr>
<tr>
<td>STD 11</td>
<td>5,00</td>
<td>5,006</td>
<td>0,015</td>
<td>0,3</td>
</tr>
</tbody>
</table>
Specifications

**Excitation**
- 50 kV X-ray tube
- 4 W max power
- 6 tube filter positions with shutter

**Detection**
- High performance semiconductor detector
- Peltier thermo-electric cooling
- Optimum balance of spectral resolution and count rate

**Sample chamber**
- Large 190 x 165 x 60 mm sample chamber
- Single position 32 mm sample aperture
- Single position 40 mm sample aperture
- Bulk sample aperture
- 6-position 32 mm automatic sample changer
- 5-position 40 mm automatic sample changer
- Single position 32 mm sample spinner
- Analysis in air or helium

**Software and application packages**
- Qualitative and quantitative analysis
- Normalization and validation feature
- Fundamental parameters
- Data export function with LIMS compatibility
- User selectable shaping times
- Simple flow bar wizard to create new applications
- Icon driven graphical user interface
- Password protection

**Environmental conditions**
- Ambient temperatures 10 – 35 °C (50 – 95 °F)
- Relative humidity <85% non condensing
- Vibration undetectable by human
- Free from corrosive gas, dust, and particles

**User interface**
- 8” WVGA touch screen interface
- Embedded computer
- Internal thermal printer
- USB and ethernet connections

**Options**
- 6-position 32 mm automatic sample changer
- 5-position 40 mm automatic sample changer
- Single position 32 mm sample spinner
- Helium purge
- Fundamental parameters
- 2.0 megapixel CMOS color camera with manual collimators

**Spectrometer data**
- Single phase AC 100/240 V, 1.4 A (50/60 Hz)
- Dimensions: 331 (W) x 432 (D) x 376 (H) mm (13 x 17 x 14.8 in)
- Weight: 16 kg (35 lbs.)
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