Silicone coating weight analysis by EDXRF spectroscopy for

- Food packaging
- Medical packaging
- Converting
- Film extruders
- Vacuum forming
- Traditional release coatings
New technology from Rigaku delivers superior performance for ultra-low silicone coating weights and more . . .

Frederic Stanley Kipping and Matt Saunders coined the word silicone in 1901 to describe a polysiloxane. The most common siloxane is linear polydimethylsiloxane (PDMS), a silicone oil. A rubber shortage during World War II propelled interest in the development of silicon-based technologies. The use of silicone release coatings has evolved from tapes and labels to more recent specialized, high-performance applications like vacuum forming, converting, plastic films, food packaging and medical devices. Better chemistries and cost-effective performance, driven by ultra-low coating weights, provide an increasing number of coaters with performance solutions to demanding application requirements.

New technology unlocks 21st century performance

The Rigaku NEX QC Series next generation of advanced Energy Dispersive X-ray Fluorescence (EDXRF) technology enables the measurement of very low silicone coating weights and metal catalysts in silicone coatings – as well as other types of metallic coatings and barriers – all with one instrument. Difficult applications, that were either marginal or not possible with earlier technologies, are now a reality. As premium, cost-effective EDXRF analyzers, the Rigaku NEX QC series deliver superior performance with an easy-to-learn software interface in a robust package specifically designed for today’s coating industry.
High-performance X-ray source and detector

Unlike previous generations of benchtop EDXRF, with their limited X-ray source performance, poor resolution proportional counter detectors and high maintenance requirements, the NEX QC series feature 21st century technology, including a mono-block 50 kV X-ray tube and solid state Peltier cooled semiconductor detector. This technology delivers exceptional short-term repeatability and long-term reproducibility with excellent element peak resolution and high sensitivity (see comparison at top of previous page).

Flexibility to meet your changing business needs

The advanced technology employed in the NEX QC means that no customization of the instrument is required to accommodate different applications. This is not true of older proportional counter based EDXRF analyzers, as they required custom filter configurations for almost all applications. All NEX QC instruments (of the same model) are interchangeable and can analyze all elements from sodium (Na) through uranium (U), thus allowing the analysis of metals in metal catalyzed release coatings.

Two models: NEX QC and NEX QC+

Rigaku offers two models to accommodate the needs of the silicone coating marketplace. The low cost NEX QC greatly expands the performance envelope, as compared to traditional EDXRF analyzers. A step up in performance is available with the NEX QC+ instrument. Featuring a silicon drift detector (SDD), the NEX QC+ offers higher resolution, better sensitivity and shorter analysis times. Detection limits for silicone release coatings, either on paper or plastic, are shown in the adjacent tables as a function of analysis time.

Detection limits with NEX QC (Environment: Air)

<table>
<thead>
<tr>
<th>Product</th>
<th>Silicone LLD @ 100 sec</th>
<th>Silicone LLD @ 50 sec</th>
<th>Silicone LLD @ 25 sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper</td>
<td>0.0023 g/m²</td>
<td>0.0032 g/m²</td>
<td>0.0045 g/m²</td>
</tr>
<tr>
<td>Plastic</td>
<td>0.0019 g/m²</td>
<td>0.0027 g/m²</td>
<td>0.0038 g/m²</td>
</tr>
</tbody>
</table>

Detection limits with NEX QC+ (Environment: Air)

<table>
<thead>
<tr>
<th>Product</th>
<th>Silicone LLD @ 100 sec</th>
<th>Silicone LLD @ 50 sec</th>
<th>Silicone LLD @ 25 sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper</td>
<td>0.0011 g/m²</td>
<td>0.0015 g/m²</td>
<td>0.0022 g/m²</td>
</tr>
<tr>
<td>Plastic</td>
<td>0.0009 g/m²</td>
<td>0.0013 g/m²</td>
<td>0.0018 g/m²</td>
</tr>
</tbody>
</table>

• Ultra-low silicone coating weights
• Sn, Bi, Ti, Pt, etc. in metal-catalyzed silicone release coatings
Rigaku NEX QC series for demanding applications

The use of silicone release coatings has evolved from tapes and labels to more recent specialized, high-performance applications like vacuum forming, converting, plastic films, food and medical packaging. Since the late 1980s, energy dispersive X-ray fluorescence (EDXRF) analyzers have been employed to measure the silicone coating weight for these applications.

Clay coated paper method
Solid state detector and proprietary method for enhanced performance on clay coated paper.

Metals in metal catalyzed coatings
Broad elemental range, from sodium (Na) through uranium (U), means that the analyzer can quantify other metals in addition to silicon (Si).

Modern touchscreen interface
Familiar easy-to-understand operation is like using a smartphone or tablet computer.

No helium
No helium is required for standard release coatings.

Up to 38 calibrations
Large number of calibrations is available, at the touch of a finger, supporting a vast array of applications and sample types.

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

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

Single position or autosampler
Standard single-position configuration can be supplemented with an optional autosampler. Interchangeable optional autosampler trays may be pre-loaded, and swapped in and out.
Silicone on clay coated papers – solving a difficult application

The Si X-ray signal comes from both the clay coat and the silicone coating. The clay coat weight can vary by about ±10% along a roll and from batch to batch of paper and still be within specification. The variations in the Si signal from the clay coat can bias the measurement of the silicone layer coat weight. Previous generation EDXRFs used either a tare or background subtraction to correct for the bias, but those methods assumed the clay coating to be constant ... leading to errors.

Because the NEX QC series can resolve Al from Si, the aluminum measurement can be employed for an absolute correction. The Rigaku software includes a correction algorithm based on the Al measurement in the clay to adjust the silicone layer measurement for variations in the clay coat weight. Simply calibrate the analyzer using standards that have different clay coat weights, the actual clay coat weight does not need to be known, it simply needs to be known to vary. Enable the correction algorithm and the software automatically corrects the calibration and adjusts the silicone layer measurement.
Touchscreen interface and built in printer

Intuitive software with modern interface

Availability of hardened high-resolution touchscreen displays has allowed Rigaku to redefine the user interface experience for the 21st century. Membrane keyboards and primitive displays are now a thing of the past. Operating the NEX QC series of elemental analyzers is a familiar experience, with finger selectable icons guiding users through routine analysis operations. Touchscreen interface technology lowers the cost of ownership because it simplifies operator training and reduces the potential for operator error.

- Touchscreen top level menu allows the operator to select the desired analysis with the touch of an icon.
- With an autosampler, enter the sample identification for each sample tray position and touch the “start” icon.
- Set desired upper and lower bounds and use the validation feature to indicate when coating trends too thick or too thin.
- Results screen is easy-to-read; obtaining a hard copy via the built-in thermal printer is just the touch of an icon.
- Calibration curves and statistics are accessible with a familiar touchscreen interface.
- Tare function for determining and subtracting the baseline concentration of a new substrate.
### Specifications

#### Excitation
- 50 kV X-ray tube
- 4 W max power
- 6 tube filter positions

#### 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 (optional)
- 5-position 40 mm automatic sample changer (optional)
- Single-position 32 mm sample spinner (optional)
- Analysis in air or helium (optional)

#### Software
- Qualitative and quantitative analysis
- Normalization and validation feature
- 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 touchscreen interface
- Embedded computer
- Internal thermal printer
- USB and ethernet connections

#### 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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**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.