An Easier QuEChERS Solution for Diverse Matrices

Learn more at www.phenomenex.com/roQ
What is roQ™ QuEChERS?

QuEChERS is a descriptive acronym that stands for Quick, Easy, Cheap, Effective, Rugged, and Safe. Improved with you in mind, the unique design of the roQ QuEChERS kits eliminates common problems seen with current QuEChERS kits. By incorporating features such as trays to hold centrifuge tubes, easy pour salt packets, and stand alone centrifuge tubes, roQ QuEChERS Kits make the QuEChERS procedure even easier!

Stay Organized with Built-In Test Tube Racks

roQ boxes are designed with removable racks that can be used as test tube racks

Easily Weigh Out Samples with Stand Alone Centrifuge Tubes

Simultaneously add sample while weighing, no transfer steps required
No Salt Spills with Easy Pour Salt Packets

Our easy open, easy pour salt packets have a narrow spout which makes salt addition easy.

Cleaner Extracts with Low Extractable Tubes

Reduce background noise in chromatograms

Avoid Leaky Tubes with No Leak Caps

We guarantee our tubes won’t leak!

15 mL roQ centrifuge tube extracted with 1% Acetic acid in Acetonitrile and then with Toluene.
How does it work?

The QuEChERS technique addresses shortcomings of traditional sample preparation methods, such as long extraction procedures and the use of hazardous solvents, and radically simplifies multi-residue pesticide analysis in food or environmental samples.

The QuEChERS technique consists of two steps; Extraction and Dispersive SPE (dSPE)

Step 1: Extraction

Pesticides and analytes of interest must first be extracted from the sample. This process relies on the combination of organic solvent and various salts to partition the analytes from samples into an organic layer (typically acetonitrile).

Blend fruits or vegetables to be analyzed.

Weigh blended sample.

Add salts and acetonitrile.

Shake tube for 1 minute.

Centrifuge tube for 5 minutes.

Easily Weigh Out Samples
Stand alone centrifuge tubes make sample analysis easy

No Mess Salt Packets
Easy pour spouts eliminate spills during salt addition
Step 2: Dispersive SPE (dSPE)

An aliquot of the organic layer from the extraction step is subjected to further clean up by dispersive SPE. This step selectively removes unwanted interferences such as lipids and pigments.

Add
supernatant from extraction procedure into a roQ dSPE tube.

Avoid Leaky Tubes
Our tubes are designed to seal completely

Shake
dSPE tube for 30 seconds.

Centrifuge
dSPE tube for 5 minutes.

Cleaner Extracts
roQ kits contain low extractable centrifuge tubes reducing background noise in chromatograms

Analyze
supernatant by GC or HPLC.
Step 1

**roQ™ Extraction Kits**

QuEChERS can be performed by following 3 different methods: the AOAC 2007.01 Method, the EN 15662 Method, or the Original Non-Buffered Method.

Use the chart below to select the most appropriate kits for your work and sample size/type.

### AOAC 2007.01 Method

Salts used:
- **Magnesium Sulfate (MgSO₄)**
  - Induces phase separation between water content in sample and acetonitrile layer
- **Sodium Acetate (NaOAc)**
  - Buffers the sample to stabilize pH

### Original Non-Buffered Method

Salts used:
- **Magnesium Sulfate (MgSO₄)**
  - Induces phase separation between water content in sample and acetonitrile layer
- **Sodium Chloride (NaCl)**
  - Induces phase separation between water content in sample and acetonitrile layer

### EN 15662 Method

Salts used:
- **Magnesium Sulfate (MgSO₄)**
  - Induces phase separation between water content in sample and acetonitrile layer
- **Sodium Chloride (NaCl)**
  - Induces phase separation between water content in sample and acetonitrile layer
- **Sodium Citrate Tribasic Dihydrate (SCTD)**
  - Buffers the sample to stabilize pH
- **Sodium Citrate Dibasic Sesquihydrate (SCDS)**
  - Buffers the sample to stabilize pH

<table>
<thead>
<tr>
<th>Sample Size</th>
<th>Part No.</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 g</td>
<td>KS0-8911</td>
<td></td>
</tr>
<tr>
<td>15 g</td>
<td>KS0-8912</td>
<td>to be used with AOAC 2007.01 dSPE procedure</td>
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<tr>
<td>10 g</td>
<td>KS0-8910</td>
<td>to be used with EN 15662 dSPE procedure</td>
</tr>
<tr>
<td>10 g</td>
<td>KS0-8909</td>
<td></td>
</tr>
</tbody>
</table>

All roQ Extraction Kits include 50 easy-pour salt packets and fifty 50 mL stand-alone centrifuge tubes.
### Step 2

**Select Your roQ™ dSPE Kit**

#### Salts and sorbents used in roQ dSPE kits

<table>
<thead>
<tr>
<th>Sorbent/Salt</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnesium Sulfate ($\text{MgSO}_4$)</td>
<td>Removes excess water from sample</td>
</tr>
<tr>
<td>Primary/Secondary Amine (PSA)</td>
<td>Removes organic acids, fatty acids, sugars, and anthocyanin pigments from sample</td>
</tr>
<tr>
<td>Endcapped C18 Sorbent (C18E)</td>
<td>Removes fats, sterols, and other non-polar interferences from sample</td>
</tr>
</tbody>
</table>
| Graphitized Carbon Black (GCB) | Removes pigments from sample *NOT FOR USE WITH PLANAR PESTICIDES*

All roQ dSPE Kits include pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes.

#### Fruit and Vegetable Type

<table>
<thead>
<tr>
<th>Method</th>
<th>Sample Size</th>
<th>General</th>
<th>Fats and Waxes</th>
<th>Pigmented</th>
<th>Highly Pigmented</th>
<th>Pigments and Fats</th>
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<tr>
<td><strong>AOAC 2007.01</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>1 mL</td>
<td>150 mg $\text{MgSO}_4$ 50 mg PSA</td>
<td>150 mg $\text{MgSO}_4$ 50 mg PSA 50 mg C18E</td>
<td>150 mg $\text{MgSO}_4$ 50 mg PSA 50 mg GCB</td>
<td>---</td>
<td>---</td>
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<tr>
<td></td>
<td>8 mL</td>
<td>1200 mg $\text{MgSO}_4$ 400 mg PSA</td>
<td>1200 mg $\text{MgSO}_4$ 400 mg PSA 400 mg C18E</td>
<td>1200 mg $\text{MgSO}_4$ 400 mg PSA 400 mg GCB</td>
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<td>---</td>
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<td><strong>EN 15662</strong></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>1 mL</td>
<td>150 mg $\text{MgSO}_4$ 25 mg PSA</td>
<td>150 mg $\text{MgSO}_4$ 25 mg PSA 25 mg C18E</td>
<td>150 mg $\text{MgSO}_4$ 25 mg PSA 2.5 mg GCB</td>
<td>150 mg $\text{MgSO}_4$ 25 mg PSA 7.5 mg GCB</td>
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<tr>
<td></td>
<td>6 mL</td>
<td>900 mg $\text{MgSO}_4$ 150 mg PSA</td>
<td>900 mg $\text{MgSO}_4$ 150 mg PSA 150 mg C18E</td>
<td>900 mg $\text{MgSO}_4$ 150 mg PSA 15 mg GCB</td>
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</tr>
</tbody>
</table>

Phenomenex | WEB: www.phenomenex.com
Extraction of Pesticide Residues from Kale and Grapes Using roQ™ EN 15662 QuEChERS Kit

**LC-MS Analysis**

Over 200 pesticides were screened at concentration ranges between 0.01 ppm to 1 ppm with the majority of analytes having a recovery range of 70-130%. The method produced excellent selectivity and reproducibility for the earlier eluting polar pesticides owing to the use of QuEChERS for removing sample matrix interferences and the biphenyl bonded-phase chemistry of the Kinetex® Biphenyl LC column for the chromatographic separation across the entire range of pesticides.

**QuEChERS Procedure (EN 15662 Method)**

Kale and grapes samples were prepared at four concentrations: no spike (0.0 ppm), low spike (0.1 ppm), medium spike (0.5 ppm), and high spike (1 ppm)

**Pretreatment:** Kale and grapes were frozen at ~-80 °C overnight

**Step 1: Extraction**

1. Homogenize sample using a blender or similar apparatus.
2. Weigh 10 g of homogenized sample into a clean 50 mL tube (provided in roQ Extraction Kits).
3. Add 10 mL of Acetonitrile containing internal standard.
4. Dispense contents of the included QuEChERS salt packet into the 50 mL tube containing homogenized sample.
5. Shake vigorously by hand for 1 minute.
6. Centrifuge for 5 minutes @ 4000 rpm, making sure that the solid material is at the bottom of the tube and a liquid layer forms on top of the solid material.

**Step 2: Dispersive SPE (dSPE)**

1. Transfer 6 mL of supernatant from Step 6 of the extraction process into the 15 mL tube containing the QuEChERS dSPE sorbents.
2. Shake vigorously by hand for 30 seconds.
3. Centrifuge for 5 minutes @ 4000 rpm to separate solid material from the liquid layer.
4. Transfer desired supernatant to an autosampler vial.
5. Dilute samples 1:10 in mobile phase A prior to injection or LC-MS analysis.
**Standard Chromatogram (0.5 ppm standard)**

**Kale Sample (1 ppm spike)**

**Grapes Sample (0.5 ppm spike)**

**LC-MS/MS Conditions**

- **Column:** Kinetex® 5 µm Biphenyl
- **Dimensions:** 50 x 4.6 mm
- **Part No.:** 00B-4627-E0
- **SecurityGuard™ ULTRA:** AJ0-9207
- **Mobile Phase:**
  - A: 10mM Ammonium Formate in Water
  - B: Methanol
- **Gradient:**
  - 0-2 min: 2%
  - 2-10 min: 2-100%
  - 10-13.1 min: 100%
  - 13.1-16 min: 2%
- **Flow Rate:** 0.5 mL/min
- **Inj. Volume:** 20 µL
- **Temperature:** 35°C
- **Detection:** MS/MS (ESI+), scheduled MRM
- **Instrument:** SCIEX 4000 QTRAP®

Sample: For full list of 210 pesticides, go to www.phenomenex.com and Search: TN-0115
Mycotoxins from Corn Meal Products Using roQ™ EN 15662 QuEChERS Kit

The use of roQ QuEChERS and Kinetex® XB-C18 Core-Shell Technology LC columns deliver a rapid and simple approach for mycotoxin screening from corn products.

QuEChERS Procedure (EN 15662 Method)

Step 1: Extraction from Ground Corn
1. Homogenize sample using a blender or similar apparatus
2. Weigh and transfer 5 g of ground corn meal to a 50 mL roQ QuEChERS extraction tube
3. Add 10 mL of water and 10 mL of acetonitrile with 1.0 % formic acid
4. Dispense contents of the included roQ QuEChERS extraction packet (KS0-8909) into the 50 mL tube containing homogenized sample
5. Shake vigorously by hand for 1 minute
6. Centrifuge for 5 minutes @ 4000 rpm, making sure that the solid material is at the bottom of the tube and a liquid layer forms on top of the solid material

Step 2: Clean up using dispersive Solid Phase Extraction (dSPE)
1. Transfer the supernatant from Step 6 of the extraction process into a roQ QuEChERS 15 mL centrifuge tube containing 900 mg MgSO4 and 150 mg PSA (KS0-9507)
2. Shake vigorously by hand for 30 seconds
3. Centrifuge for 5 minutes at 4000 rpm to separate solid material from the liquid layer
4. Transfer the supernatant to a vessel for evaporation

LC-MS/MS Conditions
- **Column:** Kinetex® 2.6 µm XB-C18
- **Dimensions:** 50 x 2.1 mm
- **Part No.:** 00B-4496-AN
- **SecurityGuard Cartridge:** AJ0-8782
- **Mobile Phase:**
  - A: 5 mM Ammonium acetate with 0.5 % Acetic acid
  - B: 5 mM Ammonium acetate in Methanol with 0.5 % Acetic acid
- **Gradient:**
  - Time (min) % B
  - 0 5
  - 2 5
  - 5 80
  - 5.2 98
  - 8 98
- **Flow Rate:** 0.45 mL/min
- **Temperature:** Ambient
- **Injection Volume:** 25µL
- **Detection:** Tandem Mass Spec (MS/MS)
- **Instrument:** SCIEX API 5000™

Positive Mode Sample:
1. 15-Acetyldeoxynivalenol (15-AcDON)
2. Aflatoxin G2 (AFG2)
3. Aflatoxin G1 (AFG1)
4. Unknown Interference
5. Aflatoxin B2 (AFB2)
6. Aflatoxin B1 (AFB1)
7. Diacetoxyscirpenol (DAS)
8. HT2 Toxin (HT2)
9. Fumonisin B1 (FB1)
10. T-2 Toxin (T2)
11. Ochratoxin A (OTA)
12. Fumonisin B2 (FB2)

Negative Mode Sample:
1. Patulin
2. Nivalenol (NIV)
3. Deoxynivalenol (DON)
4. Fusarenone-X (FUS X)
5. 3-Acetyldeoxynivalenol (3-AcDON)
6. Zearalenone (ZEA)
7. Verruculogen
Free Method Development Support and Guidance

Did you know?

You have a Phenomenex technical support team dedicated to supporting your lab’s needs! We collaborate with chemists every day to improve productivity and results while working within the bounds of your established methods.

Email: info@phenomenex.com
Determination of PFASs in Sediments Using roQ™ AOAC 2007.01 QuEChERS Kits

**LC-MS Analysis**

In order to extract Perfluoroalkyl substances (PFASs) from marine and freshwater sediment, QuEChERS was introduced to save time and offer reliable results.

**QuEChERS Procedure (AOAC 2007.01 Method)**

**Step 1: Extraction from Sediments**

1. Weigh 2.0 g of suitably dried sediment into a polypropylene container and spike with isotopically-labeled internal standards. PPCPs, Steroids, and Pyrethroids can be extracted concurrently with this method by adding the appropriate internal standard and spiking solutions to the samples and QCs.

2. Add 10 mL deionized water and vortex. Add 10 mL acidified acetonitrile (1 % Acetic acid) to the slurry and vortex.

3. Add the extraction salts (1.5 g Sodium acetate and 2 g MgSO₄) to the sample and vortex for 1 minute.

4. Centrifuge the samples for 5 minutes at 4000 rpm.

5. Place the samples in a rack and freeze at -20 °C for 30-60 minutes. This freezing step allows for easier extraction of the supernatant.

**Step 2: Clean up Using dispersive Solid Phase Extraction (dSPE)**

1. Transfer 8-9 mL of the acetonitrile supernatant into a roQ QuEChERS PSA/C18 dSPE clean-up tube (Part no. KS0-9516).

2. Vortex for one minute.

3. Centrifuge the dSPE tubes for 10 minutes at 3000 rpm.

4. Place an aliquot of the extract in a polypropylene HPLC vial and dilute 1:1 with deionized water. The sample is now ready for analysis.

**LC-MS/MS Conditions**

- **Column:** Gemini® 3 μm C18
- **Dimensions:** 100 x 3.0 mm
- **Part No.:** 000-4439-Y0
- **Inline Filter:** Phenomenex KrudKatcher™ Ultra (AFO-8497)
- **Delay Column:** Luna® 5 µm C18(2) 30 x 2.0 mm
- **Part No.:** 00A-4252-B0
- **Mobile Phase:**
  - A: 20 mM Ammonium acetate in Water
  - B: Methanol
- **Gradient:**
  - | Time (min) | % B |
  - | 0      | 10  |
  - | 1.5    | 65  |
  - | 8      | 95  |
  - | 8.1    | 99  |
  - | 12     | 99  |
- **Flow Rate:** 0.6 mL/min
- **Injection:** 90 μL
- **Temperature:** 40 °C
- **Detector:** SCIEX 5500 QTRAP®
- **Detection:** MS/MS ESI Negative (sMRM)
- **Samples:**
  - 1. PFBA
  - 2. PFPeA
  - 3. PFBS
  - 4. PFHxA
  - 5. PFBS
  - 6. PFHxS
  - 7. PFHpA
  - 8. PFHpS
  - 9. FFOA
  - 10. PFOS

Images of sediment samples are included throughout the text.
Learn more about roQ QuEChERS
Visit: www.phenomenex.com/roQ

Method Performance Data for Sediments Spiked at 1 ng/g of the Target Analytes (n=4)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Average % Recovery</th>
<th>% RSD</th>
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<tbody>
<tr>
<td>PFBA</td>
<td>91.7</td>
<td>0.76</td>
</tr>
<tr>
<td>PFPeA</td>
<td>86.3</td>
<td>6</td>
</tr>
<tr>
<td>PFHxA</td>
<td>89.4</td>
<td>1.2</td>
</tr>
<tr>
<td>PFHpA</td>
<td>93.1</td>
<td>2.9</td>
</tr>
<tr>
<td>PFOA</td>
<td>98.3</td>
<td>1.5</td>
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<tr>
<td>PFNA</td>
<td>93</td>
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<tr>
<td>PFDA</td>
<td>87.7</td>
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<td>PFUdA</td>
<td>92.3</td>
<td>2.1</td>
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<tr>
<td>PFDoA</td>
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<td>4.1</td>
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<td>PFTiDA</td>
<td>88.2</td>
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<td>PFTeDA</td>
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<tr>
<td>PFDS</td>
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<td>6.7</td>
</tr>
</tbody>
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Acknowledgement
Special thanks to Syljohn Estil and the Sanitation Districts of Los Angeles County - San Jose Water Quality Laboratory for contributing this method.
Analysis Tools You Can Depend On

Achieve resolution of multi-residues with Phenomenex HPLC, UHPLC, and GC columns.

**UHPLC/HPLC**

**Luna Omega High Performance Silica-based HPLC Columns**

Enhanced with 20 years of technology, innovation, and experience, Luna Omega columns build upon the Luna legacy to now provide incredible UHPLC/HPLC performance and selectivity. With Luna Omega columns you gain:

- Industry benchmark for LC columns
- Wide pH stability for long column lifetime
- Extensive method validation document for proven reproducibility

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**Kinetex Core-Shell LC Columns**

Leverage the power of Kinetex to achieve ultra-high performance on any LC system.

- Increase resolution, throughput, and sensitivity
- Decrease solvent consumption
- Save time and money
- Transfer methods anywhere

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**GC**

**Zebron High Performance ZB-MultiResidue™ Columns**

ZB-MultiResidue-1 & -2 columns are optimized for pesticides, herbicides, and insecticide analysis. These columns have an orthogonal selectivity that allows them to be used together in dual column confirmation analysis.

- Low activity, decreased breakdown of sensitive pesticides such as DDT
- MS Certified phases provide low bleed performance for pesticide confirmation by MS
- Long column lifetimes

Visit [www.phenomenex.com/zebron](http://www.phenomenex.com/zebron) for more information.
Ordering Info

roQ Extraction Kits
Extraction kits contain fifty easy-pour salt packets and fifty 50 mL stand-alone centrifuge tubes

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Part No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>AOAC 2007.01 Method Extraction Kits</td>
<td>50/pk</td>
<td>KS0-8911</td>
</tr>
<tr>
<td>EN 15662 Method Extraction Kits</td>
<td>50/pk</td>
<td>KS0-8909</td>
</tr>
<tr>
<td>Original Non-buffered Method Extraction Kits</td>
<td>50/pk</td>
<td>KS0-8910</td>
</tr>
<tr>
<td></td>
<td>50/pk</td>
<td>KS0-8912</td>
</tr>
</tbody>
</table>

roQ dSPE Kits
dSPE kits contain pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
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</thead>
<tbody>
<tr>
<td>2 mL dSPE Kits</td>
<td></td>
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</tr>
<tr>
<td>150 mg MgSO₄, 25 mg PSA, 25 mg C18E</td>
<td>100/pk</td>
<td>KS0-9504</td>
</tr>
<tr>
<td>150 mg MgSO₄, 25 mg PSA, 2.5 mg GCB</td>
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<td>KS0-9505</td>
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<td>KS0-9503</td>
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<td>150 mg MgSO₄, 50 mg PSA, 50 mg C18E, 50 mg GCB</td>
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<td>150 mg MgSO₄, 50 mg PSA</td>
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</tr>
<tr>
<td>15 mL dSPE Kits</td>
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<td>KS0-9516</td>
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<td>1200 mg MgSO₄, 400 mg PSA, 400 mg GCB</td>
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<td>KS0-9517</td>
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<tr>
<td>1200 mg MgSO₄, 400 mg PSA</td>
<td>100/pk</td>
<td>KS0-9515</td>
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Bulk roQ QuEChERS Sorbents

<table>
<thead>
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<th>Phase</th>
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<td>04G-4348</td>
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<tr>
<td>GCB (Graphitized Carbon Black)</td>
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<td>PSA</td>
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