### Determining the Gel Content of Ethylene Vinyl Acetate

**Purpose**
The gel content (insoluble fraction) produced in ethylene vinyl acetate (EVA) plastics by cross-linking can be determined by extracting with toluene. This procedure describes the method used to determine the gel content of ethylene vinyl acetate.

**Safety Precautions**
- Toluene is a flammable substance. Do not use near open flames.
- Use this chemical in a fume hood.
- Wear safety glasses.
- Wear chemical resistant gloves.
- Read the Material Safety Data Sheet for toluene before using.

**Principle**
The purpose of the method is to determine the gel content of the EVA. Gel content provides a means of both controlling the process and rating finished product quality.

**Apparatus**
- Glass jars equipped with foil-lined caps
- "Low Form" weighing bottles with covers
- Filter Paper (Whatman No. 54 size 15.0 cm)
- Glass funnels (large enough to hold the filter paper)
- Glass beakers (for toluene waste filtrate)
- Vented calibrated oven
- Analytical balance
- Desiccator
- Tongs

**Reagents**
- Reagent grade toluene
- BHT antioxidant ("Ionol," 2,6-di-Tert-butyl-4-methylphenol)

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### Hazardous Material

Toluene is a flammable chemical and should be handled and disposed of accordingly. Be sure to wear safety glasses, gloves, and a respirator when working with this chemical.

### Prepare EVA Sample

<table>
<thead>
<tr>
<th>Step</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cut approximately two inches from all four sides of the cured 12 inch x 12 inch sample.</td>
</tr>
<tr>
<td>2</td>
<td>Discard this two inch border.</td>
</tr>
<tr>
<td>3</td>
<td>Cut a 1.0 gram specimen from the middle section of the cured sheet.</td>
</tr>
</tbody>
</table>
| 4    | Weigh the specimen to the nearest 0.0001 g.  
   **Note:** This is your W1 weight. |
| 5    | Place the weighed specimen in a glass sample jar. |

### Combine Sample with Reagents

<table>
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| 1    | Fill a glass container with 100 ml reagent grade toluene per sample.  
   **Note:** For duplicate specimens you will need 200 ml total. |
| 2    | Add enough antioxidant to obtain a concentration of 0.1 percent.  
   **Example:** 100 ml of toluene will require 0.0865 g of BHT.  
   (density of toluene is 0.865 g/ml). |
| 3    | Add toluene to sample jars. |
| 4    | Swirl the contents two times to distribute the BHT.  
   **Note:** Two swirls performed at once. |
| 5    | Cap the jars |
| 6    | Place the sample jar immediately in the oven preheated to 60°C.  
   **Note:** Maintain the sample jar at 60°C for a minimum 20 to 24 hours. |

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<td>1</td>
<td>Preheat an oven at 105°C.</td>
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| 2 | Place the filter paper, weighing bottle, and cover in the oven at 105°C for two hours.  
   **Note1**: This can be done prior to removing the specimen from the oven at the end of the minimum 20-24 hour period.  
   **Note2**: Do not handle these materials with your hands, use tongs. |
| 3 | At the end of the time period **immediately cap the weighing bottle** and place the bottle and filter paper in a desiccator until cooled to room temperature. |
| 4 | Once at room temperature weigh the weighing bottle, cover, and filter paper to the nearest 0.0001 g.  
   **Note**: This is your W2 weight. |
| 5 | Return to the desiccator until ready to use. |

### Dry Filter, Bottles, Covers

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<tr>
<td>1</td>
<td>After the 20-24 hour period immediately remove the sample jar from the oven and place in a fume hood.</td>
</tr>
</tbody>
</table>
| 2 | Place a glass funnel in the toluene waste beaker.  
   **Note**: A ring stand may be used to hold funnel. |
| 3 | Place the pre-weighed filter paper in each funnel.  
   **Note1**: Use a small amount of clean reagent grade toluene to secure the filter placement.  
   **Note2**: This is done by pouring the solvent down the sides of the funnel/filter paper. |
| 4 | Pour contents of the sample jar into the funnel/filter paper setup. |
| 5 | Rinse the sample jar and filter paper again with clean solvent.  
   **Note**: A reagent grade toluene filled squeeze bottle is convenient. |
| 6 | Place the filter paper with residue into a weighing bottle. |
| 7 | Place the weighing bottle and corresponding cover side by side in an oven at 105°C. |

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### Dry Insoluble Portion

<table>
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<tr>
<td>1</td>
<td>Allow the sample to dry at least four hours, until a constant weight is achieved.</td>
</tr>
<tr>
<td>2</td>
<td>At the end of this time period <em>cap the weighing bottle</em> and place in a desiccator until cooled to room temperature.</td>
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</tbody>
</table>
| 3    | Weigh the weighing bottle and its contents to the nearest 0.0001 g.  
      | **Note:** This is your **W3** weight. |

### Calculation

\[
\frac{W_3 - W_2}{W_1} \times 100 = \% \text{ Crosslinked Material}
\]

Where:

- \( W_1 \) = Weight of the original specimen in grams.
- \( W_2 \) = Weight of the weighing bottle, cover, and dried filter paper in grams.
- \( W_3 \) = Weight of the weighing bottle, cover, dried filter paper, and dried residue.
Apparatus Used for Determining Gel Content

- Analytical Balance
- Desiccator
- Sample Jar
- Weighing Bottle
- Filter Paper
- Filter Paper
- Glass Funnel
- Weighing Bottle w/sample
- Filter / Funnel