ACHEIVING HIGH HEAT EPOXY FORMULATIONS USING BISDA

Version 1.0

SABIC’s Specialties Business
Thermosets & Additives

GENERAL BUSINESS USE
BISDA PRODUCT INFORMATION

BISDA (4,4-Bisphenol A Dianhydride) (dianhydrides of BPA) can be used as a comonomer in polyimide synthesis or as an anhydride curing agent in epoxy. When used as an epoxy curative, BISDA can be used to enhance thermal performance in comparison to mono anhydrides and aromatic amine curing agents. Application areas include electronics encapsulation, composites, and adhesives. BISDA derived polyimide varnishes, coatings, adhesives, films and wire enamels meet desired solubility/stability in organic solvents even after complete imidization, heat resistance, flexibility/softness, dimensional stability and adhesion properties for ever-increasing design complexity of electronics devices.

<table>
<thead>
<tr>
<th>PROPERTIES</th>
<th>TYPICAL VALUES</th>
<th>UNITS</th>
<th>TEST METHODS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Point</td>
<td>185-190</td>
<td>°C</td>
<td></td>
</tr>
<tr>
<td>Boiling Point</td>
<td>&gt;314</td>
<td>°C</td>
<td></td>
</tr>
<tr>
<td>Solubility, Methyl Ethyl Ketone (75°C)</td>
<td>10 wt%</td>
<td></td>
<td>SABIC method</td>
</tr>
<tr>
<td>Mw</td>
<td>520.49</td>
<td>-</td>
<td>SABIC method</td>
</tr>
</tbody>
</table>

AVAILABLE PRODUCT FORMS

<table>
<thead>
<tr>
<th>GRADE</th>
<th>FORM FACTOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>BISDA-1000</td>
<td>Flake</td>
</tr>
<tr>
<td>ER009614* (Developmental)</td>
<td>Powder</td>
</tr>
</tbody>
</table>
ACHIEVING HIGH HEAT EPOXY FORMULATIONS USING BISDA

**Formulation Data:**

<table>
<thead>
<tr>
<th>BISDA loading (wt %) in overall formulation</th>
<th>Unit</th>
<th>0%</th>
<th>6.7%</th>
<th>13.1%</th>
<th>19.2%</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Thermal Performance</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tg by DMA</td>
<td>°C</td>
<td>145</td>
<td>164</td>
<td>173</td>
<td>182</td>
</tr>
<tr>
<td><strong>Viscosity of BISDA-MTHPA blends</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dynamic Viscosity @ 25°C</td>
<td>cP</td>
<td>400</td>
<td>1100</td>
<td>3000</td>
<td>8000</td>
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<tr>
<td>Dynamic Viscosity @ 90°C</td>
<td>cP</td>
<td>38</td>
<td>42</td>
<td>48</td>
<td>80</td>
</tr>
<tr>
<td><strong>Formulations:</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Epoxy: D.E.R.(TM) 332*</td>
<td>phr</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>MTHPA**</td>
<td>phr</td>
<td>78.1</td>
<td>70.3</td>
<td>62.5</td>
<td>54.7</td>
</tr>
<tr>
<td>BISDA**</td>
<td>phr</td>
<td>0</td>
<td>12.2</td>
<td>24.5</td>
<td>36.7</td>
</tr>
<tr>
<td>2E-4-MI**</td>
<td>phr</td>
<td>1.8</td>
<td>1.8</td>
<td>1.9</td>
<td>1.9</td>
</tr>
</tbody>
</table>

* Anhydride to Epoxy (A/E) ratio: 0.8
** Anhydride \( \text{BISDA}_{1000} / (\text{Anhydride}_{\text{MTHPA}} + \text{Anhydride}_{\text{BISDA}_{1000}}) \): 0, 0.1, 0.2 and 0.3
*** Catalyst content: 1 wt% of total formulation

**Components:**

- Bisphenol-A Diglycidyl Ether (D.E.R.(TM) 332)
- BISDA
- MTHPA: (Methyl-cyclohexene 1,2-dicarboxylic anhydride)
- 2-Ethyl-4-methylimidazole
PROCESSING GUIDELINES

1. Add BISDA into MTHPA
   - Scale: 120-130 gms
   - BISDA content: 6-20 wt %
   - Mixing temperature: 22 °C
   - Time: 3 minutes
   - Observation: Turbid solution

2. Heat mixture at 120 °C
   - BISDA content Vs heating time:
     - 6.7 wt % >>> 15 minutes
     - 13.1 wt % >>> 20 minutes
     - 19.2 wt % >>> 30 minutes
   - Observation: Clear solution

3. Cool down 80 °C and add D.E.R.® 332
   - Scale: 275-285 gms
   - Anhydride to epoxy ratio: 0.8
   - Mixing temperature: 80 °C
   - Time: 3 minutes
   - Observation: Clear solution

4. Add catalyst 2E-4MI at 80 °C
   - Scale: 275-285 gms
   - 2E-4MI content: 1 wt%
   - Mixing temperature: 80 °C
   - Time: 2 minutes
   - Observation: Clear solution

5. Pour into a pre-heated mold
   - Scale: 275-285 gms
   - Mold temperature: 130 °C
   - Pouring time: 2-3 minutes

6. Thermal curing
   - Curing profile:
     - 80 °C >>> 30 minutes
     - 120 °C >>> 30 minutes
     - 150 °C >>> 30 minutes
     - 180 °C >>> 60 minutes
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