WAX APPEARANCE TEMPERATURE BY CROSS POLARIZATION MICROSCOPY

This test method consists of determining the wax appearance temperature (WAT, or cloud point) of a crude oil by measuring the temperature at which wax crystals can be seen via microscopy under cross polarized light. The basis for the determination is that light is prevented from passing through two polarizing filters that are set 90° apart. The oil sample is placed between the cross-polarized filters, and any crystalline material present will rotate light allowing the light to pass through the second polarization filter. Therefore, during the experiment what is observed is a dark sample in which spots (or other shapes depending on magnification) are seen as the temperature is lowered.

The experiment entails initially looking at an oil sample under the microscope at high temperature (set temperature of 60°C) to ensure that no significant amounts of water and/or crystalline debris are present to interfere with observation of waxes. If water and debris are present, then the sample is centrifuged. The clean supernatant is transferred via syringe, to a second bottle and reheated to 65°C before standard sample preparation.

After initial conditioning, the clean oil supernatant is reheated to 65°C to dissolve all the wax. Micro-capillary samples are prepared by opening the sample container while still hot and dropping in all the capillary cells to be used in testing. The bottle is then quickly recapped. When the sample has slowly cooled to room temperature, an individual cell is withdrawn and attached to a standard microscope slide with epoxy. A micro-thermocouple placed close to the capillary cell is also attached to the slide with epoxy. The sample is then heated and cooled with a Peltier thermoelectric device.

The minimum requirements for a valid wax appearance temperature are:

- Acquisition of a representative sample in a sealed micro-capillary cell.
- The sample is heated to a sufficiently high temperature so that no crystalline material can be seen.
- A minimum of 10 minutes elapses between temperature setting and observation.
- The sample is cooled to a temperature near, but above, the wax appearance temperature so that crystalline material is still not seen.
- The sample is cooled in increments of 1°C, until crystalline material can be seen.
- The sample is cooled to at least 1°C beyond the point where crystalline material is first seen and it is observed that additional crystalline material is precipitating from solution.

There are a number of methods by which WAT can be determined, including: Cross Polarization Microscopy (CPM), Differential Scanning Calorimetry (DSC), filter plugging, and Fourier Transform Infrared Spectroscopy (FT-IR). Of these methods, Cross Polarization Microscopy is well accepted by the industry as the preferred method to determine WAT.
## Determination of Wax Appearance Temperature

*by Cross Polar Microscopy Method*

<table>
<thead>
<tr>
<th>Observation Time</th>
<th>Sample Temperature (°F)</th>
<th>(°C)</th>
<th>Observation</th>
</tr>
</thead>
<tbody>
<tr>
<td>14:40</td>
<td>180.0</td>
<td>82.2</td>
<td>Clear field</td>
</tr>
<tr>
<td>14:50</td>
<td>152.6</td>
<td>67.0</td>
<td>Clear field</td>
</tr>
<tr>
<td>15:00</td>
<td>145.0</td>
<td>62.8</td>
<td>Clear field</td>
</tr>
<tr>
<td>15:10</td>
<td>138.2</td>
<td>59.0</td>
<td>Clear field</td>
</tr>
<tr>
<td>15:20</td>
<td>131.7</td>
<td>55.4</td>
<td>Clear field</td>
</tr>
<tr>
<td>15:30</td>
<td>126.0</td>
<td>52.2</td>
<td>20+ crystals 3.0 to 5.0 microns</td>
</tr>
<tr>
<td>15:40</td>
<td>179.2</td>
<td>81.8</td>
<td>Clear field</td>
</tr>
<tr>
<td>16:30</td>
<td>132.3</td>
<td>55.7</td>
<td>Clear field</td>
</tr>
<tr>
<td>17:30</td>
<td>131.2</td>
<td>55.1</td>
<td>Clear field</td>
</tr>
<tr>
<td><strong>17:40</strong></td>
<td><strong>130.6</strong></td>
<td><strong>54.8</strong></td>
<td>5 crystals 1.0 to 3.0 microns</td>
</tr>
<tr>
<td>17:50</td>
<td>128.7</td>
<td>53.7</td>
<td>20+ crystals 1.0 to 3.0 microns</td>
</tr>
<tr>
<td>18:00</td>
<td>126.9</td>
<td>52.7</td>
<td>20+ crystals 1.0 to 10 microns</td>
</tr>
<tr>
<td>18:10</td>
<td>126.1</td>
<td>52.3</td>
<td>20+ crystals 1.0 to 10 microns</td>
</tr>
</tbody>
</table>

*Power setting for Objective Lens is 20X.*

Wax Appearance Temperature = 130.6°F

*Picture Taken at 126.1°F*

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Contact Us:

Intertek Westport Technology Center
Houston, Texas USA
Telephone: 713.844.3263
Email: cbwnalabs@intertek.com
Website: www.intertek-cb.com