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Choosing Fluids for Calibration Baths

To perform a high quality, safe and trouble-free calibration by using liquid baths, the appropriate fluid must be selected as part of the configuration. As many factors have to be taken into account, Ellab has performed several tests in order to make selecting the ideal liquid for the required application easier.

The relevant specifications and important facts regarding each fluid, have been set up in the table below:

<table>
<thead>
<tr>
<th>Fluid</th>
<th>Lower Temperature Limit*</th>
<th>Upper Temperature Limit*</th>
<th>Flash Point</th>
<th>Viscosity (centistokes)</th>
<th>Specific Gravity</th>
<th>Specific Heat (cal/g/°C)</th>
<th>Thermal Conductivity (cal/cm°C)</th>
<th>Thermal Expansion (cm/cm°C)</th>
<th>Resistivity (10^12 Ω-cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>-97°C (fr)</td>
<td>16°C (fl,cc)</td>
<td>16°C</td>
<td>1@20°C</td>
<td>0.809@20°C</td>
<td>0.61</td>
<td>0.00047</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ellab Low Temp Liquid</td>
<td>-89°C (fr)</td>
<td>10°C (fl,cc)</td>
<td>12°C</td>
<td>20@20°C</td>
<td>0.79@20°C/4°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>0°C (fr)</td>
<td>95°C (b)</td>
<td>none</td>
<td>1@25°C</td>
<td>1.00</td>
<td>1.00</td>
<td>0.0014</td>
<td>0.0002@25°C</td>
<td></td>
</tr>
<tr>
<td>Ethylene Glycol - 50%</td>
<td>-30°C (fr)</td>
<td>90°C (b)</td>
<td>none</td>
<td>7@0°C</td>
<td>1.05</td>
<td>0.8@0°C</td>
<td>0.001</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ellab Standard Oil 200.10</td>
<td>-30°C (v)**</td>
<td>209°C (fl,cc)</td>
<td>211°C</td>
<td>10@25°C</td>
<td>0.934@25°C</td>
<td>0.43@40°C</td>
<td>0.00032@25°C</td>
<td>0.00108</td>
<td>1000@25°C/50@150°C</td>
</tr>
<tr>
<td>Ellab High Temp Oil 710</td>
<td>80°C (v)</td>
<td>300°C (fl,oc)</td>
<td>302°C</td>
<td>50@80°C</td>
<td>1.11@25°C</td>
<td>0.363@40°C</td>
<td>0.00035@25°C</td>
<td>0.00077</td>
<td>100@25°C/1@150°C</td>
</tr>
</tbody>
</table>

*Limiting Factors - B-boiling point, e-high evaporation, fl-flash point, fr-freeze point, v-viscosity / Flash point test - oc-open cup, cc-closed cup
**Very low water solubility, ice will form as a slush from condensation below freezing

Usable Range – Lower/Upper Temperature Limits

The usable range is the temperature range in which a fluid can safely provide a good environment for the sensors and probes being compared. The range and specifications may differ depending on the manufacturer and could be limited by factors such as viscosity, flash points, freeze and boiling points, evaporation rates, etc.

While a single fluid may not cover the full range of the instrument calibrations, a combination of 2 or 3 may be sufficient. Ellab recommends dedicating one fluid to one liquid-bath in order to shorten calibration procedures by eliminating the need to change out fluids, which can be cumbersome and inefficient.
Flash Point

The flash point is the temperature at which a mixture of fluid vapor and air would ignite if exposed to a spark or flame.

There are two ways to measure the flash point, either by using the “open cup” or “closed cup” method. With the “open cup” method, neither fluid or the surrounding air is closed off, resulting in there being a higher ratio of air to fluid vapor. With the “closed cup” method, the fluid, fluid vapor and air are sealed away within the bath.

“Closed cup” flash points are typically lower than “open cup” flash points. In the material safety data sheets (MSDS), the flash point is often presented in a general and purposely lowered version in order to fit into a classification scheme used for hazard control, e.g. “>101.1°C”.

Product specification sheets usually give more specific information, such as “211°C cc”. Ellab fluids with flash points are listed using the “closed cup” method and the upper range limit is set slightly below the flash point.

Viscosity

Viscosity, often referred to as “thickness”, is the measuring of a fluid’s ability to resist flow. Kinematic viscosity is considered the ratio between density and absolute viscosity and is measured in “centistokes”. The more centistokes, the thicker or more viscous a fluid is. Viscosity will always be stated at a specific temperature and increase as the temperature of the fluid decreases (and vice versa). Bath fluids that are too viscous create a strain on the stirring and pumping mechanisms and do not adequately transfer heat uniformly from the temperature sources to the thermometers.

Using fluids with less than 25 centistokes are recommended, which is shown within the usable ranges stated for each of the fluids. Picking fluids with less than 10 centistokes of viscosity is, however, deemed optimal. As low-uncertainty calibrations require a homogeneous temperature within the “calibration zone” of a bath, high-viscosity fluids often provide temperature gradients.
Specific Gravity

The specific gravity is the ratio of a fluid’s density to that of water. The higher the specific gravity, the denser and heavier a fluid is. If the fluid is too heavy, it may not work well in a bath equipped with a pump mechanism or circulator.

Heat Capacity

Specific heat is the amount of heat required to raise the temperature of an object (mass 1 kg) by 1°C. The higher the heat capacity is, the more energy is required to change the temperature, which results in a slower and more stable process.

For example, water has a high heat capacity, which means that it absorbs a lot of heat before it starts warming up. Due to its high heat capacity and viscosity, water would be one of the best medias for calibration if not for its highly limited liquid form temperature range.

Thermal Conductivity

A fluid’s ability to transfer heat between molecules is known as thermal conductivity. Fluids with a high heat transfer will heat or cool faster, whereas a low heat transfer will be slower at reaching the same goals. Having good thermal conductivity can significantly improve the uniformity of calibration baths.

Thermal Expansion

Every calibration fluid has a coefficient of thermal expansion. This coefficient indicates exactly how much a fluid will contract or expand (change in volume) as temperatures change. The expansion of fluids can have critical consequences for cleanliness, state of equipment and most importantly, safety. If baths are filled with too much liquid while at low temperatures, heating it without regard for the potential increase in volume can cause liquids to spill. As opposed to this, if a bath is not filled with enough liquid, it can lead to the bath heaters being exposed and result in damages. It is not unusual that silicone oils can expand by 10-15% when heated over a 100 °C interval.
Polymerization

Given enough time, temperature and catalysts, silicone oils will eventually polymerize. This means that they suddenly turn into molasses-like “syrup”, doubling in volume and creating a mess. This phenomenon is caused by oxidation.

While silicone oils can safely be used near their flash points, the risk of polymerization increases when above their oxidation points.

Avoid polymerization by:
1. Limiting the time baths are kept above or around its vapor point.
2. Ensure that contaminants are kept out of the liquid (like foreign oils, salts and oxidizers).
3. If an oil becomes too dark, viscous or unstable, it should be changed to avoid further polymerization.

Water

There are a few things to know about water in non-water baths. First and foremost, water should never be introduced into a salt or hot oil bath, as performing such an action is highly hazardous for operators. Water can also freeze to cooling surfaces and result in poor stirring conditions if used at low temperatures. Water therefore occasionally needs to be boiled off these surfaces.

Alcohol absorbs water, allowing for combinations like 5% water in methanol, which then allows the methanol to be used at -100 °C. If too much water is absorbed however, alcohol can become saturated and take slurry, icy forms, which has a negative effect on uniformity and stability. If this ever becomes the case, fluids will have to be replaced.
Fluids with high amounts of vapor pressure (like water and alcohols), evaporate faster than other liquids and require frequent refills. Additionally, accelerated evaporation at the fluid surface, cools down the fluid, resulting in temperatures being harder to control. These kinds of fluids are generally only suitable for low temperatures. Health hazards are an additional factor that needs to be taken into consideration, as these vapors can be hazardous and should therefore be vented accordingly.

Always ensure that there is proper ventilation to the baths in order to prevent operators from breathing in potentially hazardous fumes. The best solution for this issue is having suction devices that suck the fumes from a bath's opening and exhaust them outdoors. Baths should be kept sealed whenever possible to ensure that fumes do not escape into the work environment. Doing so will increase the lifetime of the oil and increase work space safety, as oil vapor might otherwise settled on surfaces or eyes, causing discomfort.

**Using Multiple Fluids**

Although using a single fluid would be optimal, this is rarely possible for metrology departments due to the wide range of equipment that they are responsible for maintaining and calibrating. Every calibration fluid has a specific temperature range for a reason. Not only would there occur issues in regard to freezing and boiling, but evaporation, viscosity changes and flash points determine the limit of a fluid’s temperature range.

This means that a single fluid might not cover the entire range required for a single bath, resulting in users having to choose between the inconvenient process of changing fluids or having several baths dedicated for specific temperature areas. As a rule of thumb, Ellab usually recommends choosing a bath for each of the required liquids in order to cover the temperature ranges, thereby avoiding time consuming liquid changes.
Safety Tips

When using different kinds of oil and alcohol at high temperatures, it is extremely important to follow safety regulations and good practices.

Here are some recommendations:

• Always wear appropriate protective equipment. This includes gloves, aprons and face shields consisting of adequate material for the temperatures and liquids being worked with.
• Be familiar with the fluids being used. A helpful tool for getting familiar with these oils is the MSDS sheets or product specification sheets from the manufacturers.
• As previously mentioned, ventilating properly is of utmost importance.
• Never mix fluids or apply any chemicals into the fluid.
• Never add anything to the bath fluid that could cause a chemical or physical reaction.
• Never allow water to come into contact with hot salts or oils.
• Only insert clean sensors or probes into the active baths.
• Always ensure that baths are not operated around (or on) combustible materials. Keep the surrounding area of all baths clean.
• Always ensure that appropriate fire extinguishing equipment is stationed nearby.
• All personnel who deal with or operate near baths must understand the relevant precautions and know how to appropriately deal with any potential emergencies.
• Ensure that all laws and regulations are followed regarding the storage and disposal of hazardous or flammable fluids used in calibration baths.
• Always avoid using a fluid above the designated flash point. Using alcohols require special safety considerations, as their flash points can be below ambient temperature.