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# Choosing a bath fluid

## Application Note

*by Steve Iman*

Fluke's Hart Scientific Division manufactures a large number of constant temperature baths that cover a temperature range from –100 °C to 550 °C. However, the usable range of each bath highly depends on the fluid chosen. Hart's definition of "usable range", is the temperature range over which the fluid will provide the best performance. The ideal bath fluid would have a low viscosity, high heat capacity, very low vapor pressure, and a high flash point. It would also need to cover a very wide temperature range. Unfortunately, no single fluid has all of these attributes, so care must be taken when choosing a bath fluid. From both an operational and safety standpoint, some considerations are: safety precautions, flash points, viscosity, heat capacity, thermal conductivity, fluid expansion, specific gravity, vapor pressure, gel time, usable life and storage.

## 1.0 Fluid Safety

Always obtain data sheets and/or material safety data sheets for the fluids that will be used. It is important to read and understand all safety requirements. When it comes to safety, Hart strongly recommends the following:

1. Have the appropriate fire extinguishing equipment nearby in case of a fire.
2. Never mix fluids or put any chemicals into the fluid. Doing so may cause contamination or an adverse chemical reaction.
3. Always exercise caution when working around extremely cold or hot bath fluids. Wear protective clothing to prevent accidental injury.
4. Use adequate ventilation for fluids at elevated temperatures. (See Figure 1).
5. Never operate a bath on or around combustible materials.
6. Provide safety training for all personnel who will either use the baths or be around them.
7. Abide by federal and state laws regarding storage and disposal of any hazardous or flammable liquid.

## 2.0 Terms and Definitions

Now that you have the data sheets, what do all those terms mean?

### 2.1 Flash point

This is the temperature at which an adequate mixture of fluid vapor and air will ignite if in the presence of an open flame or spark. It is important to note that if the fluid is nonflammable it's only the vapor that will burn and not the fluid. There are two units of measure for flash point.

1. Open Cup (oc). As the term implies, the air and fluid vapor are not enclosed. In an open cup, there is a higher ratio of air to fluid vapor.
2. Closed Cup (cc). The mixture of air and fluid vapor are contained in an enclosure. In this instance, the ratio of fluid vapor to air is higher.

When specifying the flash point, fluid manufacturers only have a list of options to pick from. They do not have open text fields that they can fill in. So when it says >101.1 °C, this is just stating that the flash point is greater than 101.1 °C. This is just used to classify the material (ie. flammable or combustible, etc. for the hazard profile, not as an actual value). It's like saying how old are you? Then giving you choices of >15, >25, >35, etc. So when it comes to the flash point definitely go with the product data sheet.


***Figure 1*** *Ventilation system for removing oil vapors.*

### 2.2 Viscosity

Viscosity is the unit of measure for the thickness of a fluid at 25 °C. Generally it is a constant consistency under fixed pressure and temperature. Ideal fluids offer no resistance to shear and have zero consistency. Viscosity dimensions are force per area x time. The unit of viscosity is the poise (P) = 1g/(cm) (sec) and is a measure of mass flow of a liquid. One poise is equal to 0.1 Pa.s in SI units. Where Pa.s is the Pascal-second, the SI unit for viscosity, equaling 1kg(m.s) or 10 poise.

A common unit of measure with bath fluids is kinematic viscosity. This differs from viscosity in that it is the measure of volume flow of a liquid, defined as a stoke (st). A stoke equals 1 $cm^2$/sec or $10^{-4} m^2$/sec. A centistoke, cst = 0.01 St = 1 $mm^2$/sec. Kinematic viscosity can be converted to viscosity (poise) by multiplying by the density of the fluid.

Since bath fluids generally are used at more than one temperature, the viscosity will change when it is heated or cooled. Because the viscosity changes, each fluid will have a "viscosity temperature coefficient." Example: Over a range of 0 °C to 100 °C, VTC = 1–(viscosity@100 °C/viscosity@0 °C). Thus the lower the VTC, the less change there will be in viscosity over the range.

Hart baths generally perform the best with a viscosity that does not exceed 10 cst. But in the real world, the viscosity will change with temperature. So viscosities that do not exceed 50 cst will work satisfactorily. Any higher than this, and the stability and uniformity may be very poor. In addition, if the viscosity is too high, it will put too much of a load on the stirring motor. This may cause it to overheat or to stop altogether.

## 2.3 Heat Capacity

The specific heat capacity of a solid or liquid is defined as the heat required to raise a unit of mass or substance by one degree of temperature.

$DQ = mcDT$

Where:

$\Delta Q$ = *heat applied to fluid.*

$m$ = *fluid mass.*

$c$ = *specific heat capacity.*

$\Delta T$ = *rise in temperature.*

## 2.4 Thermal Conductivity

Thermal conductivity is a fluid's ability to transfer heat from one molecule to another. This can be determined by:

$\lambda(T) = a(T) \times c_p(T) \times p(T)$

Where:

$\lambda$ = *thermal conductivity*

$T$ = *temperature*

$a$ = *diffusivity of material*

$cp$ = *specific heat*

$p$ = *density*

The better the heat transfer, the quicker the fluid will heat or cool. Better thermal conduction will help with bath uniformity.

## 2.5 Coefficient of Volume Expansion

All fluids have a thermal expansion coefficient. This unit of measure tells how much the fluid will either expand or contract with changes in temperature. Unless the bath is equipped with an overflow device, it must be considered, otherwise the bath may overflow.

## 2.6 Specific Gravity

The specific gravity is a specification of the density or weight of a fluid as compared to that of water. The specific gravity of water is 1. A cubic foot of water weighs 62.4 pounds.

The higher the specific gravity, the more the fluid will weigh. If the fluid is too heavy, it may not work well in a bath equipped with a pump mechanism or circulator.

## 2.7 Vapor Pressure/Volatility

The temperature at which a liquid is on the verge of vaporization is called vapor pressure. At this temperature, the vapor pressure of the liquid is equal to that of ambient pressure. Another way of saying this is that the vapor and ambient pressures are at equilibrium. If the temperature is below this point, the vapor will condense into liquid. Conversely, if the temperature is above this point, the liquid will vaporize. A fluid that has high vapor pressure such as alcohol will evaporate quickly and require frequent replenishment. Furthermore, rapid evaporation at the fluid surface will have a cooling effect, making temperature control more difficult. These fluids generally are only suitable for low temperature use.

With some liquids, the processes of condensation and evaporation can be delayed, which is referred to as supersaturation and superheating, respectively. A good example is adding ethylene glycol to water. This raises the boiling point of the water as well as the vapor pressure.

The specific gravity of water is $= \frac{62.4}{62.4} = 1$

To calculate an unknown specific gravity:

$$Sp\ gr = \frac{\text{Weight of the fluid}}{\text{Weight of an equal volume of water}}$$

$$Sp\ gr = \frac{\text{Density of a fluid}}{\text{Density of water}}$$

***Figure 2*** *Calculation of an unknown specific gravity.*

## 2.8 Gel Time

Gel time is usually associated with silicone oils when used at elevated temperatures. This is the time that it takes silicone oil to gel or polymerize. Oxidation of the oil is the root cause. When this occurs, it's a molecular chain reaction that happens instantly and can cause the fluid to nearly double in volume. Polymerization is a metrologist's worst nightmare; the oil will either turn to a jelly-like substance or even worse, a "molasses in winter" goop! It can be very difficult to remove from the bath and its parts. Fortunately, Dow Corning makes a solvent that can be used to remove polymerized oil. The solvent is called OS-2 and can be purchased from an authorized distributor of Dow Corning fluids. It will require approximately 2 gallons of OS-2 for every 7 gallons of polymerized oil.

Polymerization of silicone oil in an open system may not be avoidable. However, there are steps that can be taken to prolong the oil's life.

1. Keep the time that the bath is at high temperatures to a minimum.
2. If the bath isn't being used, either turn it off or set the

idling temperature below its oxidation temperature.

3. Avoid cross-contamination of oils.
4. Keep oxidizers such as bath salts out of the oil.
5. Change the oil if it becomes too dark in color, to viscous, or there is a notable difference in bath stability.
6. Keep a cover on the bath. Drill holes in the cover to put the probes through. Keep the holes not in use plugged.

## 3.0 Fluid Life and Storage

The life of a fluid depends on how it is used, at what temperature it is used, and the length of time at that temperature. Generally, most bath fluids will have a long life as long as their limitations are not exceeded. Unused liquids should be left in their original unopened container. If storage life is a concern, please check with the fluid manufacturer for specifics about shelf life and storage requirements.

## 4.0 Choosing a Fluid

Having a bath for each temperature or fluid type is the "ideal." However, this may not be within your budget, so more than one fluid may be needed. There are many bath fluids on the market that may work well for the temperature range of interest. As stated earlier, a single fluid may not be available to cover the desired temperature range. So try to choose fluids with the widest range, lowest viscosity, and the highest flash point. There may be some overlap in the temperature ranges of each fluid, but this is fine. It is probably better to have some overlap rather than being right on the "fringe" of a fluid's minimum or maximum temperature.

To avoid cross-contamination of fluids, thoroughly clean all wetted parts before putting in the next fluid. Another source for cross-contamination is moving thermometers from a salt bath to an oil bath without cleaning off the salt.

### 4.1 Water

Water is one of the most commonly used bath fluids. It is an ideal fluid over its usable range. It's inexpensive, has a low viscosity, and good thermal characteristics. The drawbacks are limited temperature range, hard water deposits, and the formation of algae. Water generally has a usable range from about 5 to 60 °C but its upper limit depends on atmospheric pressure. The elevation at Hart Scientific is about 4500 ft above sea level, so here water has a higher vapor pressure. This limits the usable upper temperature to about 40 to 45 °C. Higher temperature settings will cause instability due to the rapid cooling effect from evaporation. The vapor pressure can be reduced using a 50/50 mix of Ethylene Glycol and water. In Utah, this will raise the usable high temperature to about 75 °C.

To eliminate hard water deposits, we recommend using either distilled or deionized water. To reduce the growth of algae, use a good algaecide.

### 4.2 Silicone Oils

Silicone oils have unique properties because they are not petroleum or organic based. They were the first and only polymer products made from inorganic chemistry. Silicone oils vary in viscosity and cover a broad temperature range. (See Table 1.) They have good thermal characteristics and low flammability.

Even with their good qualities, there are a few disadvantages. At high temperatures, fuming occurs and cleanup will require a solvent. Because baths are "open" systems, prolonged use at high temperatures cause the thinner properties to boil off. As the hot oil comes in contact with air, the oil will oxidize. The oxidation accumulates over time and will eventually cause the oil to gel or polymerize. When the color changes from light honey to a darker color, this is a sign that oxidation has taken place. The oxidation rate also depends on contaminates. Bath salts are a heavy oxidizer; so avoid getting them into the oil

To minimize oxidation, turn the bath off when it's not in use or, keep it at low idling temperature. To avoid polymerization, it's recommended that the oil be changed any time it becomes too dark or the temperature becomes less stable.

Some silicone oils are designed for low-temperature use. However, at low temperatures condensation will form. In locations where the humidity is particularly high, more moisture will condense. As with many oils, water and oil remain separated. At cold temperatures, the water and oil will pass over the cooling coils or cooled tank walls. Generally, the water will freeze to these parts of the bath. As the ice thickens, it will reduce the heat transfer between the cooling coil or plate and the liquid. This will prevent the bath from reaching cold temperatures.

### 4.3 Cooking Oils

Hydrogenated vegetable oils and coconut oil can be used, but they have a limited temperature range and tend to fume more at high temperatures than do silicone oils. Please note that vegetable and coconut oils are subject to gelling, just as silicone oils are.

### 4.4 White Mineral Oil

White mineral oil is a good choice for resistor baths. It is inexpensive, and has good thermal and electrical resistive properties. Over time however, the electrical resistivity may decline. Generally this is due to water contamination.

***Table 1*** *A few silicone oils and their characteristics.*

| Hart Model # | Description | Usable Range | Flash Point |
|---|---|---|---|
| 5010 | Silicone oil Type 200.05 | –40 to 130°C | 133°C |
| 5012 | Silicone oil Type 200.10 | –30 to 209°C | 211°C |
| 5013 | Silicone oil Type 200.20 | 10 to 230°C | 232°C |
| 5014 | Silicone oil Type 200.50 | 30 to 278°C | 280°C |
| 5017 | Silicone oil Type 710 | 80 to 300°C | 302°C |

## 4.5 Perfluorocarbons
Perfluorocarbon fluids are excellent for low temperature baths. They are thermally and chemically stable, nonflammable, and have low levels of toxicity. They have a high dielectric strength and non-solvent characteristics, which make them ideal for electronic control testing. Cleanup is easy, as they leave practically no residue. The disadvantages are evaporation and cost.

## 4.6 Alcohols
Ethanol and methanol are excellent cold bath fluids especially where the ambient humidity is high. All alcohols absorb moisture, which can be an advantage. Unlike oils where the water and oil remain separated, ice will not form on cooling coils or tank walls like it will with oils. However, alcohols can become overly saturated with water. When saturation is reached, the mixture forms a slurry of ice and alcohol. At this stage, the stirring will be impeded resulting in poor stability and uniformity. When the alcohol becomes too saturated with water, it must be changed.

Normally methanol freezes at –98 °C, but by adding water it will lower the freezing point. This is called the "freezing point depression." If water is not added for –100 °C operation, the methanol will freeze to the bath cooling coils as shown in Figure 3. When ice forms on the cooling coil, the bath will not reach temperature. By adding 5% water by volume, –100 °C may be achieved.

Methanol is very volatile, has a low flash point, and has a high degree of toxicity which can be absorbed through the skin. Ethanol also is very volatile, but unless ingested it is less toxic. Isopropyl is not as toxic or volatile as methanol, but it can become highly viscous at low temperatures because it tends to be more hydroscopic. Because of alcohol's high volatility and low flash points (see Table 2), all alcohols should only be used for low temperature work. When not in use, alcohols should be kept in an appropriate container and stored in a cabinet designed for flammable liquids.

If alcohol is left in the bath, the temperature should be maintained at 0 °C or lower to minimize evaporation.

***Figure 3*** *Cooling coil coated with ice. The fluid is pure methanol (no water added).*

***Table 2*** *Usable temperature range for alcohols*

| Type | Temperature Range (°C) | Flash Point |
|---|---|---|
| Isopropyl | –10 to 20 | 11.7°C |
| Ethanol | –80 to 10 | 11.1°C |
| Methanol | –100 to 10 (add 5% water by volume for < –90°C) | 12.8°C |

## 4.7 Bath Salts
Tempering A heat transfer salt is very stable and experience has shown that in the absence of contaminants, it can give many years of service if used at temperatures of 454 °C and below. Salt is usable from 200 °C to 550 °C, but at temperatures above 454 °C, the salt undergoes a slow thermal decomposition. This is accompanied by a gradual rise in the freezing point. Tempering salt is somewhat hygroscopic, so it is recommended that it be stored in a dry place to prevent caking. Organic chemicals and combustible materials should not be stored in the same area.

Bath salt can be easily removed from thermometers by rinsing it off with warm water. ***NEVER*** *allow water to come in contact with molten salt!*

## 5.0 Conclusion
Bath fluids are an integral part of constant temperature baths. It is important to know their advantages as well as their limitations, so that you can get the maximum benefit from their use. It is our hope that by sharing our knowledge and experience, that we can help you make the right fluid choices. Should you have any questions, please call or email us. We are more than happy to be of service.

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**Fluke Corporation**
**Hart Scientific Division**
799 E Utah Valley Drive
American Fork, UT 84003

Tel: 801.763.1600
Fax: 801.763.1010
E-Mail: info@hartscientific.com
www.hartscientific.com

**Fluke Eurpoe B. V., Hart Scientific Division**
PO Box 1186, 5602 BD Eindhoven
The Netherlands
Tel: +31 (0)40 2675 403
Fax: +31 (0)40 2675 404
E-mail: Hart.Logistics@Fluke.NL

All other countries:
Tel: +1 801.763.1600
Fax: +1 801.763.1010


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