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Technical information bulletin

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Moisture removal from Therminol heat transfer fluid cooling systems

For single organic heat transfer media used in cooling systems, it is important to prevent the chiller's heat exchange surfaces from being coated with ice. Icing will reduce the efficiency of the chiller and can occasionally cause blockage in the system piping. Moisture will also cause volatility problems during heating cycles, such as pump cavitation and two-phase flow. Various methods of moisture removal are presented in the following sections.

Water removal from the heat transfer system prior to Eastman Therminol® heat transfer fluid introduction

1. All the low points in the piping system and the expansion tank should be checked for water and drained.

2. The system should be purged with dry air or nitrogen for a period of 12 to 72 hours. The amount of moisture removal can be increased by increasing the flow rate of the purge gas stream. A convenient hookup point for the purge gas supply is the system surge expansion tank with the purge stream discharging through the system low-point drains and high-point vents. **(Caution: all system circulation pumps should be secured to prevent rotation during the purging operation and avoid dry running.)** Monitoring the purge gas stream humidity will assure the effectiveness of the drying operation. After purging, leaving a slight nitrogen pressure on the system will prevent moist air intrusion.

3. As an alternative method for moisture removal, if the system is heat traced, it can be heated under vacuum while monitoring the dew point of the system atmosphere. A slight nitrogen gas purge into the system under vacuum will aid the moisture removal. **(Caution: be sure the vessels in the system are rated for the vacuum level.)**

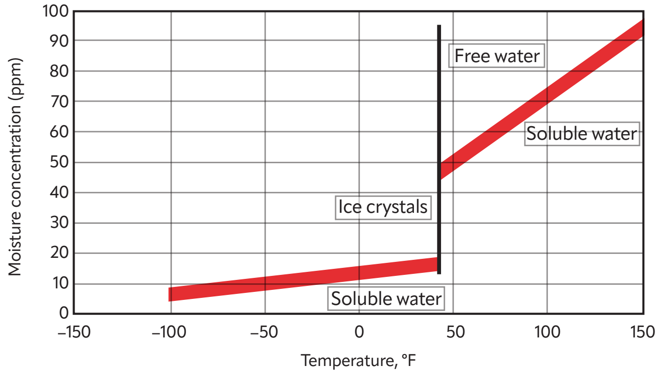
4. All low points in the system should be checked again. If the system is not completely dry, repeat the purging. When dry, a low-pressure nitrogen blanket should be left on the system until it is ready to be filled with Therminol fluid.

5. To prevent moisture contamination of Therminol fluid in drums, the drums should be kept under cover. The change in the drums' vapor space pressure as a result of ambient temperature changes can cause moisture intrusion of freestanding water on the drum tops.

Free-water removal from Therminol D-12 in the system

If moisture is present in the system after Therminol D-12 is charged to the system, the moisture is often above the saturation level. This visible moisture, known as free water, is seen as water droplets on the bottom of clean glass containers. The free water can be removed by opening the system low-point drains periodically after brief system circulations. The water should be allowed to settle for at least one hour before the low-point drains are checked for moisture. Once the system is in operation, low levels of free water can be removed in the chiller system by filtration of ice particles in 100-mesh strainers. Dual strainer systems are recommended for continuous ice particle removal until the leakage source can be plugged. The presence of ice particles in the strainers may indicate a significant amount is present on the chiller's heat exchanger tubes. After the free water is removed, the moisture level in the Therminol D-12 is at the saturation level (Figure 1). Although the Therminol D-12 moisture saturation level is low compared to other organic coolants, the soluble moisture may still cause icing problems in the cooling system.

Figure 1. Therminol D-12 water solubility



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Saturated water removal from Therminol D-12

The saturated moisture drying of Therminol D-12 in the system can be accomplished by three different methods: molecular sieves, azeotropic separation (Dean-Stark trap) or nitrogen purge.

Water removal by molecular sieves

Water can be removed from Therminol D-12 by circulating the fluid over molecular sieves. Molecular sieve towers are placed in a side stream for bypass flow control. Contact your Eastman Therminol representative for information on molecular sieve manufacturers.

General operating parameters for molecular sieve systems

1. Molecular sieves are not efficient at removing free water. Free water will saturate the molecular sieves, which will require frequent molecular sieve replacement or regeneration. To bring the water level down to the saturation level where molecular sieves are effective, a coalescer (or the other free-water removal techniques mentioned previously) needs to be employed first.

2. Molecular sieve sizes 3A and 4A (8–12 mesh) are recommended for moisture removal from Therminol D-12. Typical sieves have the capacity of approximately 2 lb (1 kg) of moisture per 100 lb (50 kg) of sieves. Moisture can be removed down to the 1 ppm concentration level by this method.

3. Ambient temperatures are recommended for moisture removal. At temperatures above ambient, the absorbent effectiveness decreases. Temperatures below ambient can be used until the viscosity reaches 5 cP, but lower temperatures require special equipment considerations. The sieves will lose their effectiveness if the viscosity increases above 2 cP.

4. Optimum flow rates can vary, depending on system size and water concentration. In general, higher flow rates require larger molecular sieve beds due to reduced contact time. Slow flows less than 10 GPM (2.3 m^3/h) are usually acceptable in typical beds, provided they do not channel. The molecular sieve manufacturers need to be consulted on the operating conditions for the specific heat transfer system using Therminol D-12 heat transfer fluid.

5. The molecular sieves can be reactivated with heated nitrogen flow through the molecular sieve bed, followed by cooling with ambient-temperature nitrogen. Reactivation with heated nitrogen can be accomplished with an electric heater or through a combination of heating with steam and electric heat for larger molecular sieve towers.

6. Generally, a small pressure drop will occur through a molecular sieve bed. For example, a bed with a diameter of 1.5 ft (0.46 m) and a height of 4.5 ft (1.4 m) could have a pressure drop of 2 psi (13.8 kPa) for a flow of 50 GPM (11.5 m^3/h) and 0.13 psi (0.9 kPa) for a flow of 5 GPM (1.15 m^3/h).

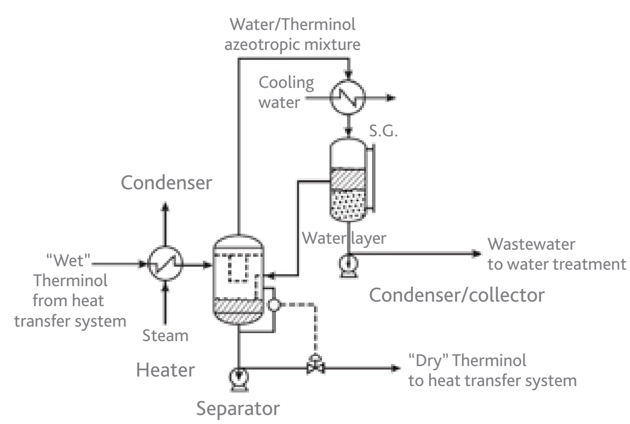
A design example

A single tower unit with 215 lb (98 kg) of 4A molecular sieves will dry 11,300 gal (42.8 m^3) of Therminol D-12 containing 200 ppm water. The drying time will be 15–16 hours with the recommended flow rate of 12 GPM (2.8 m^3/h). This tower system is based on an open-circuit model using reactivation with an electric heater only.

Azeotropic separation (Dean-Stark trap) method

A collector/separator and condenser can be added to the system (Figure 2). To start, a low-pressure nitrogen blanket is placed on the system. The Therminol D-12 and water mixture is heated initially to the saturation temperature of water, and the temperature is gradually increased to the boiling point of Therminol D-12 as the water is removed from the system. The water and Therminol D-12 fluid vapors are taken overhead from the separator, condensed and collected in the collector until the visible water/Therminol D-12 interface is seen high in the sight gauge. The water is below the interface. The water is then drained from the collector for disposal, and the Therminol D-12 is passed

Figure 2. Azeotropic separation



back to the separator. This procedure should be repeated until only clear Therminol D-12 collects in the collector. The "dry" Therminol D-12 is returned to the system. Drying by this method is practiced commercially and is effective for large systems and those that experience frequent moisture leakage to the cooling system. There are no packaged systems available, but small heater/separator/condenser systems have been designed and provided as packages by engineering firms.

The nitrogen purge method

By running the system expansion tank hot, the moisture can be removed by purging nitrogen through the expansion tank vapor space. The fluid can be circulated through the expansion tank from 160°–340°F (71°–172°C). Nitrogen is purged through the vapor space in the expansion tank for 4 to 24 hours. Lower tank operating temperatures and slower nitrogen flow rates will increase the time required for drying. Equal portions of Therminol D-12 and water may be removed during this operation. This method is convenient for removing large quantities of free moisture from small- to medium-size systems that can be heated to higher temperatures. Since Therminol D-12 is combustible, the purge stream must be vented to a safe area and collected for disposal. The molecular sieve procedure described previously can be used in conjunction with this method to remove the moisture that is soluble down to 32°F (0°C).

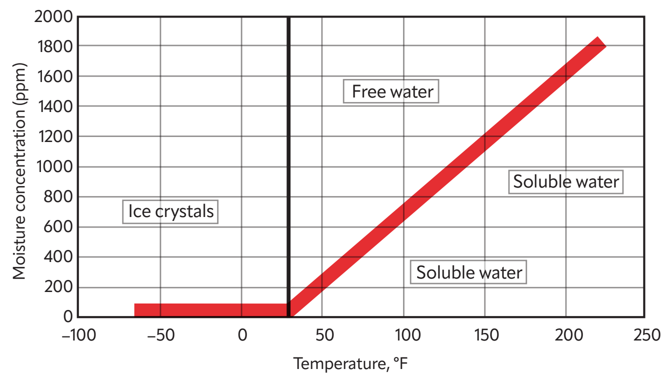
Water saturation in Therminol D-12 at cold temperatures

The concentration of water saturated in Therminol D-12 was measured at various temperatures. The moisture saturation chart (Figure 1) provides the approximate moisture solubility limits. These data are a guide in determining the level of drying necessary for any given application. Moisture analysis of fluid samples taken from the system should be conducted regularly to avoid the associated operational problems. The moisture analysis technique should be able to detect moisture levels in the 10 to 100 ppm concentrations. A coulometric Karl Fischer titration system provides accurate and rapid moisture analysis at these low concentrations. Contact your Eastman Therminol representative for analytical equipment resources.

Water removal from Therminol LT systems

The removal of moisture from Therminol LT systems is similar to the procedure for Therminol D-12 with the following additional considerations. The concentrations of moisture saturation are much higher than for Therminol D-12 and will generally require longer time periods for the moisture removal processes (Figure 3). Larger quantities of molecular sieves will be needed to effectively remove moisture from Therminol LT. With the somewhat strong aromatic odor of Therminol LT, the nitrogen purging of the system expansion tank vapor space to remove moisture is not recommended.

Figure 3. Therminol LT water solubility



System design and operating considerations

Moisture in the system can often cause operational problems such as loss of chiller efficiency, increasing pressure drops across strainers, and cavitation in pumps operating near the boiling point of water. Ice crystal filtration and the soluble moisture removal methods described previously should be employed immediately before the chilling utility is shut down by ice fouling. Good design practices should be followed to avoid moisture contamination of the cooling system. Cooling water heat exchangers should utilize double tube sheets and strength welds. The nitrogen inert gas supply on the system expansion tanks should be maintained at a low dew point, and the possible use of dryers to assure a dry nitrogen supply should be considered. Effective moisture traps can be designed into the system. For example, many systems use low-temperature thermal surge tanks to store large quantities of cold Therminol D-12. The discharge from these tanks can employ a standoff pipe in the bottom of the tank where free moisture can accumulate on start-up. A drain can be placed in the bottom of the tank near the stand pipe to effectively remove free moisture. Nitrogen solubility is also a consideration in systems where nitrogen comes out of solution at higher operating temperatures and can form gas pockets or degas in pump sections. High-point vents in process equipment are necessary for insoluble gas removal, and vent valves may need to be used for continuous removal.

EASTMAN

Eastman Corporate Headquarters
P.O. Box 431
Kingsport, TN 37662-5280 U.S.A.

U.S.A. and Canada, 800-EASTMAN (800-327-8626)
Other locations, +(1) 423-229-2000

eastman.com/locations