Ice Blocks

Usually an ice block forms in an orange cryostat when air is sucked into the sample well or needle valve. Here we outline the two most common types of ice blocks, how to recognize if you have an ice block, and how to fix one if you do.

Needle Valve Ice Block

How to Avoid:
Ice blocks in the needle valve are the most common type of block, and there is no surefire way to avoid them. They can happen if a helium dewar is contaminated or if some air sneaks in during a helium transfer. However following these steps can greatly reduce the frequency of needle valve ice blocks.

1. ALWAYS close the warm valve when pumping on the annulus. Otherwise you will pump in air through the annulus and needle valve.
2. Don't pump on the annulus when you have less than 10% helium in the helium reservoir. The helium exhaust valve is supposed to be one way, but they don't always work like they're supposed to!
3. After doing a helium fill and replacing the cap to the helium reservoir, heat it with the heat gun and re-tighten it. Otherwise the cap will loosen as it warms and you could suck in air (cryopump).

Symptoms:

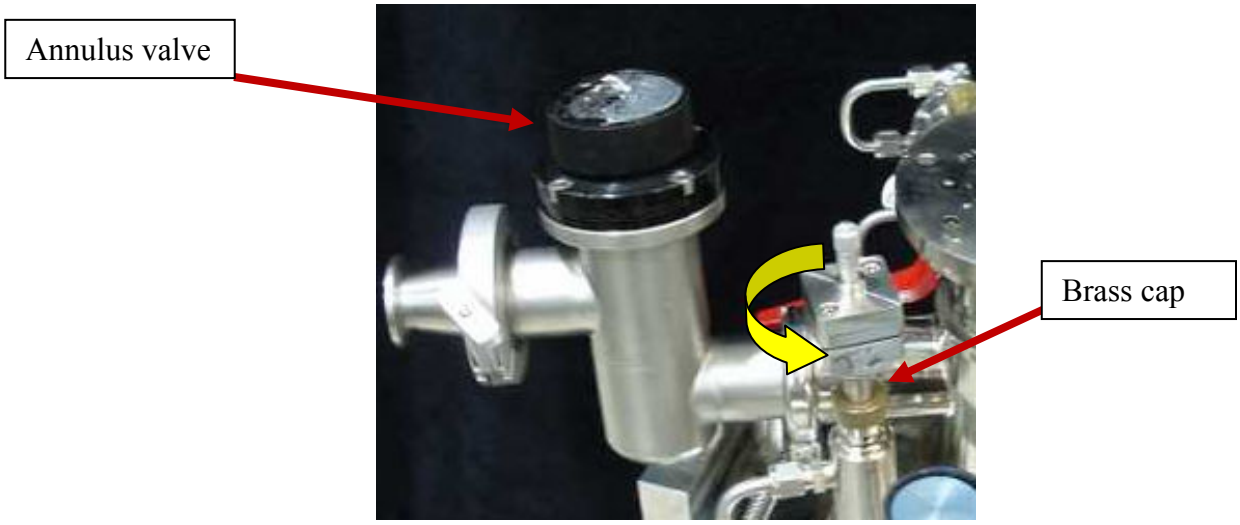
1. The cryostat temperature cools much slower than usual or stops cooling all together.

Treatment:

1. Test to see if the needle valve is indeed blocked by opening the needle valve while pumping on the annulus. If the temperature stays the same or increases, you have a block. If it decreases very slowly the needle valve is only partially blocked.
2. Now that you know you have ice in the needle valve, make sure you have at least 30% helium, and continue pumping on the annulus with the needle valve open. Everything else on the cryostat should be closed tightly (the helium exhaust, black exhaust knob, helium reservoir cap).
3. There should be a valve heater in the cryostat cart. Plug it into the attachment hanging off of the 'cryostat' sensor cable. Press down on the button. The red light next to the button will light up if the heater is working. If it does not light up, contact a member of the sample environment team and request new batteries or a replacement valve heater.
4. Hold the button down for two minutes and release. Repeat this a few times. If the ice in the needle valve clears you will hear the pump suddenly start to work harder and the pressure inside the pump line will increase. The cryostat will immediately start to cool. If this does not work, heat the cryostat up to 80K and apply the valve heater again. If this does not work, proceed to step 5.
5. If the valve heater method did not clear the block, contact a member of the sample environment team. If a sample environment staff member is not available (at night or on a weekend), continue with the following instructions. Note that this process requires rapid

movements and results in massive helium boil off, so be prepared and do not attempt it if you are uneasy about any of the following steps! Now, try to apply heat directly to the ice via the needle valve. Use safety glasses and gloves for this step!

a. Close the valve to the annulus and stop pumping on it. Release the pressure in the helium reservoir by opening then closing the helium exhaust valve.
b. Now unscrew the base of the needle valve from the cryostat by holding the main body of the needle valve (not just the knob on top!) and turning counter-clockwise. You will probably need to unscrew the brass cap and o-ring holding the needle valve in place on the top of the cryostat. When the needle valve is un-screwed, pull it completely out of the cryostat. Helium should be boiling out of the cryostat where you just removed the needle valve.



c. Heat the needle valve using a heat gun. Make sure that there is no condensation left on the shaft and that the bottom is warm to the touch. You are ready to insert the needle valve back into the cryostat.
d. OPEN THE RED HELIUM EXHAUST VALVE.
e. Wearing gloves, stick the needle valve quickly into the cryostat. If it gets stuck part way, raise it a little bit and then try to stick it back down. You will boil off a lot of helium in this process. When the needle valve is as far down as it will go, turn the body clockwise until it is tight and re-tighten the brass fitting on top of the cryostat.
f. Close the pressure relief valve and start pumping on the annulus again. If the cryostat cools, you fixed the block! If not, repeat the steps above. If after two tries the block is not cleared, continue to step 6.

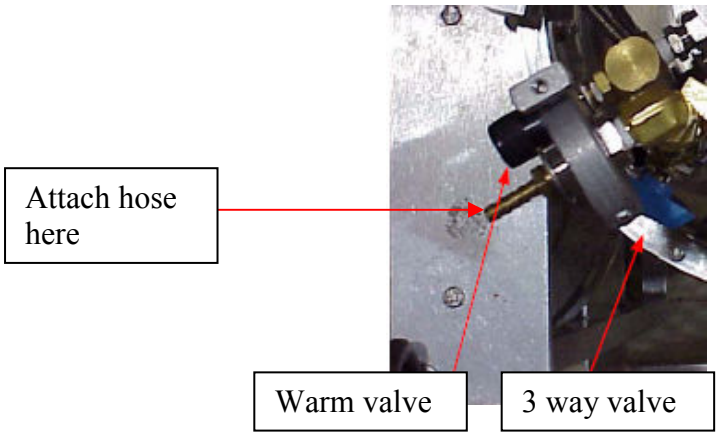
6. Your cryostat needs to be warmed to room temperature in order to fix the ice block. It will take at least 2 hours to clear the block and reach base temperature again. Contact a member of the sample environment team and request a replacement cryostat if one is available.

Sample Well Ice Block

How to Avoid:

Sample well ice blocks most commonly occur while changing samples in a cold cryostat. Follow these steps to avoid sucking air into the sample space while you are changing samples:

1. Make sure helium gas is flowing *before* you attach the hose to the nipple leading to the sample well. This is to make sure you don't also blow air into the sample space.
2. Turn the blue three way valve to the 'up' position so that the gas flows into the sample space.



3. Start unscrewing the Allen bolts on top of the sample well. You should hear a 'pop' when you loosen the last bolt. Do not start to unload your sample until you are sure there is a positive helium pressure inside the sample well. Otherwise you will let air in when you lift up on the stick.
4. Take your sample out and put the new one in. Continue to flow helium gas into the sample space until you have re-tightened the bolts on top of the cryostat.

Symptoms:

1. The cryostat temperature (sensor B) reaches base, but the sample temperature (sensor A) does not.

OR

2. The sample temperature cools much slower than the cryostat temperature or stops cooling all together.

Treatment:

1. The first thing you should always do if you suspect a sample space ice block is to determine whether your sample stick is stuck inside the cryostat. To do this pump helium gas into the sample space until it is over-pressurized and unscrew the top Allen bolts part way. Then see if the sample stick can move up and down. If it can, re-tighten the bolts and continue with the next step. If it cannot, contact a member of the sample environment staff immediately. Do not heat the sample or cryostat if your sample stick is stuck inside the sample well.

2. The cryostat must be warmed up to at least 80K to pump out the nitrogen ice. Change the set temperature to 80K and attach a rough pump to the sample space. When the sample temperature reaches 80K the pressure on the rough pump should increase, signifying the nitrogen ice has started to melt. You can expedite the pumping process by pump-purging the sample space with warm helium gas. This process can take up to 2 hours, so it might be more time efficient to request a new cryostat.