

What's it Worth?

Did you ever calculate the value of the samples you put through your evaporator? In many cases, the value of a month's throughput is more than the value of the instrument itself. We're not talking about the value added by the evaporation step, but all the value added up to now in getting the samples this far.

Why is this figure important? Because if your evaporation step were to damage your samples, and you had to remake them, this is how much money you would be throwing away.

Now, there's no reason why you should do anything to damage samples in your evaporator, provided you use it correctly. The point we're making, though, is that your primary focus must be to get your samples through the process intact, and uncompromised. Getting them dry *fast* should be the second, not the first, priority.

Here we'll be dealing with the various aspects of sample protection, and the things to watch out for.

What could go wrong?

There are four main ways in which a sample could be damaged in a centrifugal evaporator if the correct precautions are not taken:

- Overheating
- Cross contamination
- UV damage caused by heat lamps
- Unintended evaporation of the actual compound

You might add "over drying" but in fact this is rarely a source of actual damage, it simply makes re-dissolving the solid more difficult afterwards.

We'll deal with each of these problems in turn.

Preventing exposure to damaging Ultra Violet Light

This is the simplest of all to deal with, so we'll cover it first.

There are three ways to prevent UV exposure in a Genevac system.

The first and least helpful is not to use the Coolheat™ lamps at all. This extends runtimes hugely, however, and so is definitely not recommended.

The next is to use “shrouded” sample swings. Thus, your samples are enclosed in a light tight box. The fact that the samples are sheltered from the air currents inside the chamber means that drying is up to 10% slower. Though these swings are available, this approach is also not really recommended as there is a far better way (see below).

“Cool Heat Shades”

If your compounds are (or might be) sensitive to UV, Genevac have a couple of window options for preventing the UV portion of the light from the Coolheat™ lamps from ever reaching your sample.

The standard window glass placed between the Coolheat™ lamps and the chamber will block out all frequencies up to 330nm (*)

Then if this is not good enough there is an optional special glass, which blocks out frequencies up to 550nm (*).

This special glass may be retrofitted to existing systems or ordered with a new system.

(*) Independent test data, not manufacturer's figures.

Tests carried out by the National Physical Laboratories, in the UK.

Prevent Overheating of Samples

In many cases, users prefer not to heat their compounds above 40-45 °C. Yet for rapid drying the system must feed a lot of heat into the samples while the solvent is being removed. Most modern evaporators (including Genevac) do this using infrared lamps. When the sample dries, there is no longer a flow of heat out of the sample, but if the lamps are still on, heat is still flowing in. This can cause the sample temperature to rise far above safe temperatures.

Happily, Genevac's SampleGuard™ system protects against that. Heat is applied only as much as is safe, by controlling the sample holder at the chosen maximum temperature.

On systems without SampleGuard™, the only safe way to use the heat lamps is to turn them off well before the samples are dry, so that heat is never applied in an uncontrolled fashion to samples which have no solvent present. This has two significant drawbacks.

- You need to already know the likely "with lamps" runtime before you can adopt this approach safely, which will mean "practising" on a dummy solution containing no valuable compounds
- Once the lamps are turned off, the final part of the run happens slowly. Extremely slowly if the solvent is a high boiling point solvent such as DMSO.

Additionally the temperature control system allows newer Genevac systems to automatically determine the end of the drying run and hence to stop when samples are dry.

All Genevac systems have temperature control.

If you would like to know more about Genevac's sample guard system, or if you would like some additional training/advice on using it, please contact your Genevac sales rep or email applications@genevac.com.

Eliminating Cross Contamination

If suitable steps are not taken to prevent bumping, samples in plates and tubes can contaminate each other.

This is easily prevented on a Genevac HT Series II system. Simple select Dri-Pure™ (called VacRamp II on early systems) as the pressure control regime when programming the system.

You can read a far more in depth article on this topic at www.genevac-news.com (look for the March 2002 issue).

Evaporation of Compounds

This is not as common a problem but for those users that it affects, it is very real. What happens is that the compound itself is evaporated in the drying run, usually by subliming after the solvent has all been removed.

One drawback of this is that solid compound will reform elsewhere in the system eventually, potentially causing problems.

A second possible problem is that compound subliming from a dry sample may re-solidify in a colder, not yet dry sample in an adjacent tube, resulting in a tiny amount of cross contamination.

But the most important drawback is that precious compound has been lost.

Thankfully, there are a number of ways to prevent compound loss due to this mechanism.

The first is to adjust the run time settings. Typically this problem is encountered when medium to high boiling point solvents are being used (from DMF up to DMSO or NMP). With these, you will often have the system programmed to run at full vacuum with the SampleGuard™ control temperature set to the highest value your compound can take without damage. And at these conditions, volatile compounds can also be lost.

Note that by **either**

- Controlling the vacuum level at something other than just “full vacuum”
- Lowering the SampleGuard™ control temperature

it is possible to reduce the extent to which compound is removed. Note that with a high BP solvent this is almost certain to slow the run down, but this is a trade-off you have to make.

If you go too far with this strategy, of course, you'll never get the samples dry.

For example

- 2mbar and 40°C will barely remove DMSO (at 2 mbar DMSO boils at almost 40°C)
- Full vacuum and 20°C will barely remove DMSO. (Since “full vacuum” at the pump often equates realistically to as much as 0.6mbar in the chamber, and at 0.6mbar DMSO boils at nearly 20°C).

But preventing the problem can be even simpler than that.

Stop at the End!

This may sound obvious, but you should stop a run when the samples are dry.

The time in a run when most of a volatile compound will be lost is the period after the solvent has gone and the dry sample rises to the same temperature as the sample holder. This is because,

- In the absence of any solvent vapour, the machine achieves a better vacuum
- The compound is now warmer than it has been all through the run so far.

The longer this situation persists; the more compound might be lost.

So it is clearly a benefit to be able to stop the run as soon as the samples are dry. It is certainly bad to leave the run needlessly going overnight when the samples may have been dry for hours.

The options for stopping at the right time are:

- Learn by experiment what the optimal runtime is and then run the machine for no longer than this.
- Use a second temperature probe (HT series II systems only) in channel 2 and watch the temperature of a typical sample. When this rises to meet the temperature of channel 1, the sample is pretty much dry. Experience will show that a bit more time after this is required.
- When the new software becomes available for HT series II (see April 2002 story on www.genevac-news.com), use the "Automatic End of Run detection" feature to stop the system when it is dry.

How can I find out which compounds are volatile?

Genevac have far more information on this subject, beyond the scope of this article. If this is a topic that interests you, email applications@genevac.com and tell us about your application.