The Guide To Dry Down Solvent Evaporation

Keywords: sample preparation, ultravap, evaporator, drydown, nitrogen blowdown

In this article:

- Insight to the bottlekneck in chromatography sample preparation workflows
- How blowdown evaporators work
- Enhancing the speed and efficiency of sample dry down
- Guide to choosing the right dry down evaporator for your samples

The Lab Workflow

In any laboratory workflow there is a bottleneck, a step in the process from sample collection, storage, preparation, processing, and analysis (Figure 1.) that takes the longest time and hence the throughput defining step. No matter what efficiency improvements are made elsewhere in the workflow, that bottleneck step controls the overall sample throughput.

In analytical or life science laboratories, samples are typically diluted in a solvent, concentrated or dried down then reconstituted in a known volume of liquid ready for downstream applications. Depending on the volume and type of solvent, dry down is often the bottleneck step. Solvents can be evaporated in ovens, water baths or lyophilisation chambers, but control over cross contamination and the degree of drying can be difficult to achieve with these methods and maintenance can be high.

An alternative solution is the blowdown approach. Heated nitrogen passes through needles positioned above the sample tube or plate and continuously blows (hence 'blowdown') hot air over the surface of the solvent.

Sample Work Flow

Collection - Storage - Preparation - Processing - Analysis

Figure 1. Typical journey of samples in analytical workflows showing stages from collection through to analysis.

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How do blowdown evaporators work?

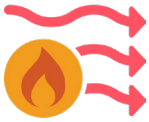
A blowdown sample evaporator consists of a number of set features that define its function. By definition the gas travels from the inlet through a variable flow controller and heater before being passed over the samples held in appropriate adaptors for the sample containers, multiwell plates, vials or tubes.

It is crucial for reproducibility that the temperature and flow rate of the gas across each sample surface is consistent run-to-run and sample-to-sample. By adjusting the gas flow, temperature, needle position and timing, sample concentration can be fast and reproducible. In addition, as the needles are not in contact with the sample, cross contamination is reduced to a minimum.

There are four variables are controlled by a blowdown evaporator; time, temperature, gas flow and needle position. The flexibility, simplicity and range of control over these parameters is what makes blowdown evaporators an attractive and efficient method (See Table 1 & 2 for sample method) for solvent evaporation.

Gas Temperature

The higher the gas flow temperature, the faster the drying effect. Thermal stability of the sample must however be considered to ensure any labile/sensitive samples are not exposed to damaging temperatures. In a blow down evaporator, this risk is minimised since the gas only heats the surface of the sample, leaving the bulk at ambient temperature for most of the drying process. For more sensitive samples, it is recommended that the temperature is reduced at the end of the drying programme to reduce the risk of damage.



Gas Flow

The higher the gas flow, the greater the level of solvent vapour removal and therefore, a faster drying process. However, this should be controlled at two points in the process programming:

At the start: To prevent splashing and the risk of cross contamination
At the end: To prevent dried sample being blown away and lost.

A gradual start and lower flow towards the end are ideal.

Needle Position

The ideal position for the needles is just above the sample, but as the solvent evaporates, the surface level drops in the well or tube. To maintain the tip position, the height of the needle needs to be adjusted to maintain consistent solvent evaporation rates. Too high and the heating effect is reduced, too low and there is the risk of needles touching the sample and causing cross contamination. The rate of needle movement should be matched with the rate of evaporation to ensure a near constant position above the sample surface. In conjunction with gas flow, needle height can be adjusted to slow down the drying process and minimise risk of sample loss.

Time

Each step of the blowdown process should be long enough to complete the objectives of the stage, but as short as possible to maximise throughput and minimise the risk of sample loss and damage.

Developing A Method

The aim of evaporation is to rapidly dry or concentrate a sample while ensuring sample integrity and eliminating cross contamination. To start, some basic information will help with define the evaporation efficiency and some good practices will help avoid the risk of cross contamination and sample damage.

What is your solvent?

This influences the rate of evaporation at a given temperature.

What is the starting volume?

This defines the starting position of the needles and in conjunction with the rate of evaporation of the solvent, the rate of movement of the needle. This also depends on the sample container.

Is your sample thermally sensitive in solution and/or dry form?

This defines the maximum temperature and whether it is necessary to lower temperature exposure at the end of the drydown.

Best practice for blowdown methods:

An exact guidance is difficult due to the wide range of samples, solvents, volumes, plates, etc. but here are some starting points:

- To prevent cross contamination through sample splashing, consider a gentle build-up of gas flow at the start of the procedure.
- Once a method has started it is recommended to observe the first few runs using "pure" solvents (i.e. without sample to eliminate the requirement for needle head cleaning). The needle heads should remain just above the solvent surface, without touching and without being too close to cause droplets to form. If the needles touch, slow the platform movement, if they become too far away (i.e. losing efficiency), speed it up slightly.
- Use the highest temperature while staying inside the safe range for the stability of the sample. If the sample is sensitive to heat in its dry form, a lower temperature at the towards the end of the drying stage will prevent damage of the now dry sample.
- If the method is designed to completely dry the sample, consider a lower gas flow at the end to prevent sample being blown out of its container.

! Tip for Needle Contamination !

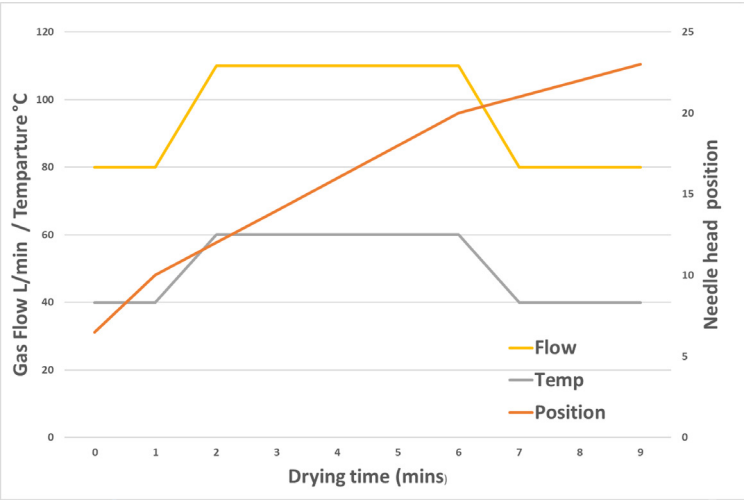
If the needles become contaminated, the head should be removed from the evaporator and cleaned by either autoclaving, or soaking the needles overnight in a strong solvent. Ensure that the cleaning solvent is only in contact with the needle portion of the head. Nitric acid is often used but strong alkali solution should be avoided. After soaking, rinse in ionised water and dry on the evaporator.

Sample Method in Action

A typical three stage method for solvent evaporation on a blowdown evaporator may be set up as the table below.

Stage	Needle start (mm)	Needle end (mm)	Length (mins)	Gas Temp (°C)	Gas Flow (L/min)	Stage description
1	6.5	10	1	40	40	Gentle build up to reduce sample disturbance.
2	10	20	5	60	50	Main drying phase, fastest step considering sample parameters.
3	20	25	3	40	40	Complete drying with a gentle finish.

Table 1. Example of a dry drown method using a blowdown evaporator.



Reference Information

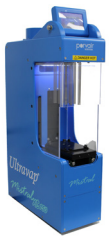
Methanol	12 mins
Acetonitrile	15 mins
Methanol:Acetonitrile (50:50)	12 mins
Water	60 mins
DMSO	90 mins

Table 2. Drying times for 1ml at 60°C and flow rate of 80L/min

The Ultravap® Range of Blowdown Evaporators

Porvair Sciences offer 5 different instruments that range from an entry level manual system that is ideal for the occasional user, to systems that are more automated and can be built into lab automation systems for higher throughput of samples.



	Ultravap® MiniVap	Ultravap® Gemini	Ultravap® Levante	Ultravap® Mistral	Ultravap® Mistral XT150
Gas Flow	Manual	Dual, manual	Programmable	Programmable	Programmable
Temperature Control (Max°C)	Digital (60°C)	Dual, Digital (60°C)	Programmable (80°C)	Programmable (80°C)	Programmable (80°C)
Plate Height	Manual	Single manual	Programmable	Programmable	Programmable
Timing (Max drying length, hrs)	Operator, No limit	Operator, No limit	Software 5 x 3 steps 3 hours	Software 15 x 5 steps 5 hours	Software 15 x 5 steps 20 hours
Features	Compact	Twin plates	Robotics ready	Master/slave Robotics ready	Master/slave Robotics ready
Extraction	None	None	Optional	Standard	Standard
Max Sample Height	50 mm	50 mm	50 mm	50 mm	150 mm

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