DEVELOPMENT OF A NEW, HIGH POWER CONDENSATION HEATING TECHNOLOGY TO ENABLE VERY FAST PARALLEL EVAPORATION, IN A SAFE AND CONTROLLED MANNER

INTRODUCTION

The optimum method for removing solvent from large numbers of small (up to 20ml) samples at the same time is by centrifugal evaporation. Larger samples (e.g. synthesis products in round bottom flasks) can be fairly rapidly processed with a rotary evaporator, but this method accommodates one sample at a time. In addition, optimisation of a rotary evaporator for maximum throughput requires continuous monitoring by the user. Larger vessels can, with appropriate adapters, be placed in a centrifugal evaporator, however, the rate of evaporation is typically a lot less than that achieved by a rotary evaporator.

This paper details a new development which enables a centrifugal evaporation system to achieve evaporation speeds comparable to a rotary evaporator.

EVAPORATION RATE IN A CENTRIFUGAL EVAPORATOR

A high performance centrifugal evaporator requires:

- the use of a centrifugal to force the samples to boil only at the surface and thereby prevent spitting (or bumping)
- the use of vacuum to lower the boiling point (BP) of the solvent so that it can be rapidly boiled off without raising the solution temperature above what is deemed safe for the sample
- the use of a refrigerated solvent condenser, situated before the vacuum pump to trap the large volumes of solvent vapour produced. This is necessary for several reasons.
  - it is not feasible for any commercial vacuum pump to pump away the total volume of vapour and maintain the required vacuum level
  - the vapour is ideally not to be released into the atmosphere
  - some pump technologies are not compatible with organic solvent vapours in great quantity
- The application of heat in some way.

There are three factors controlling the rate of evaporation:

1. Vacuum level
2. rate at which heat is added
3. rate at which solvent is condensed

Given that the vacuum level is stable, then the rate at which solvent from the sample solutions is converted to vapour and removed is governed by the rate at which heat is supplied into the solution. This holds until heat is added so fast that the condenser is insufficiently powerful, at which point it will not be possible to maintain the target vacuum level. Trying to add more heat to samples in this situation will have little effect.

Conversely, just making the condenser 10 times more powerful will produce little increase in evaporation speed because the limiting factor becomes the level of heat being supplied to the samples. A typical high performance evaporation system is specified so that the heat flow into the samples is slightly below the level of the other system components so as to provide a small amount of excess performance. In such a situation heat flow will govern the rate of evaporation.

HEAT FLOW INTO THE SAMPLES

Controlled application of heat to samples in a centrifugal evaporator is far more technically challenging than in a rotary evaporator.

In the latter, the flask is partly submerged in a water bath held at a specified temperature. This means that

- there is a large and highly conductive surface area of water in contact with the vessel
- the sample can never be heated above the maximum bath temperature, and hence sample temperature control is easily and inexpensively achieved.

In a centrifugal evaporator, the vessel is carried in a rotor revolving at high speed and so getting heat in to the samples in a controlled and efficient fashion must be achieved via remote means. One simple (but not so effective!) method is to heat the vacuum chamber in which the samples are spinning. This in turn heats the gases in the chamber which carry heat by forced convection to the sample vessels. There are a number of limitations of this method which render the approach unsuitable for high performance systems:

- the method works best when the pressure in the chamber is high (lots of gas molecules) whereas high performance evaporation requires a very low pressure.
- most of the gas in the chamber is recently evaporated solvent which is generally cold
- heating the gas in the chamber forces the condenser (which needs to cool the solvent vapour and condense it) to do more work.

A more suitable approach is to apply heat with infra red lamps. In order that the samples do not become overheated, the samples must not be heated directly. Instead, they are placed in conductive blocks and the infra red lamps heat the blocks, and heat is conducted to the sample vessels. Using a remote means of temperature measurement, the system can maintain the blocks at temperature which is safe for the samples.

The comparison between a rotary evaporator and a centrifugal evaporator in terms of heat flow can now be seen clearly. In the rotary evaporator, a sample which must not exceed 40°C is evaporated in a flask which is partly immersed in liquid at 40°C. In the centrifugal evaporator, a similar sample is evaporated in a flask that is supported in an conductive holder at 40°C. However well designed the holder, it can never hope to conduct heat into the flask as well as the rotary evaporator's liquid can because the contact area for heat transfer is too small. To enable centrifugal evaporation to proceed much faster, it is necessary to

Laboratory Products Focus

Author Details:
Roger Lee-Smith, Senior Project Engineer
David Griffin, Applications Engineer
Genevac Ltd, Farthing Road,
Ipswich, IP1 5AP, UK
Tel: +44 1473 240000
Email: applications@genevac.com

conduct heat into the flask in a more effective way. The technology described below achieves this.

CONDENSATION AS A HEAT TRANSFER METHOD

In this alternative system, heat is transported from the chamber walls to the vessels by water. The approach (like a heat pipe) takes advantage of the very high specific latent heat of water. A small quantity of water circulates continuously, carrying heat from chamber wall to sample vessels, and cycling between vapour and liquid states.

The simplified schematic *(Figure 1)* shows how heat is transferred.

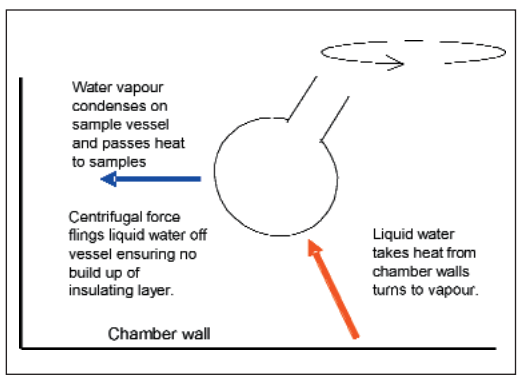

Figure 1. Schematic showing heat flow

In order for this to work, the conditions in the chamber must be such that the water boils and re-condenses at the chosen maximum sample temperature. For example, for a 40°C sample temperature 68mbar is required.

What this simplified diagram does not show is that the "chamber" must be separated into two entirely different volumes, see *Figure 2*.

The system shown in *Figure 1* is controlled at the right pressure for water to boil at the chosen sample temperature. This is the "condensation chamber" in *Figure 2*. The other volume, the "evaporation chamber" must serve all the usual functions of an evaporator, namely:

- the pressure should be controlled so as to achieve the solvent boiling point required
- it must be connected to the condenser for removal of solvent vapour.

Perhaps the greatest challenge is that the sample vessel itself must form part of the seal between the two volumes.

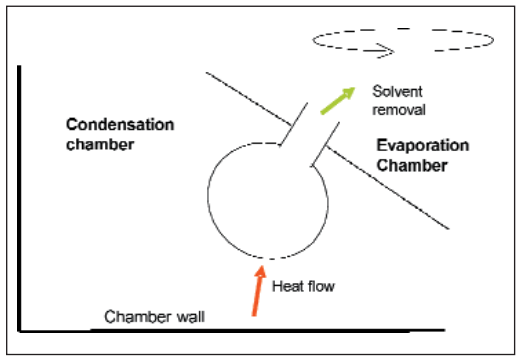

Figure 2. Schematic showing two separate chamber volumes

In the implementation that has been developed, the condensation chamber is stationary and the evaporation chamber rotates within the condensation chamber. The two chambers kept separate from each other until the sample flasks are removed.

BENEFITS OF A CONDENSATION EVAPORATOR

This method has a number of benefits:

- The condensation chamber and the rotating evaporation chamber are equally and accurately heated to the control temperature which prevents solvent vapour condensing in the evaporation chamber before leaving. This eliminates problems which might be caused by higher boiling point solvents or aggressive solvents condensing.
- The heating medium reaches all areas, therefore the time to reach the control temperature is very rapid and without any risk of overshoot.
- It is possible to ensure no part of the chamber is heated above the control temperature. This has safety and reliability implications.
- The temperature sensor is mounted externally using existing rugged reliable and accurate technology. No sensing of temperature on the rotating part of the system is necessary.
- Heat is provided in a controlled way ensuring that minimum heat is wasted thus increasing the efficiency of the whole system.
- The evaporated vapour is ducted directly out of the evaporation chamber and is not superheated prior to entering the condenser, thus improving efficiency and reducing condenser load.
- The sample tubes are not enclosed in a sample holder, therefore the progress of the drying can be visualised using a strobe.

TECHNICAL CHALLENGES OF A CONDENSATION EVAPORATOR

There are very few limitations to this technology, and those that exist are associated with

- Making the seal between the condensation chamber and the evaporation chamber (i.e. sealing the sample vessels into the rotor)
- Communicating the evaporation chamber to the condenser which is outside of both chambers.

This requires robust, simple sealing technology which has been developed especially for this application. The seal that forms on the sample vessel to separate the condensation chamber from the evaporation chamber has been designed to actuate as a result of the centrifugal force acting as the rotor spins up. It therefore offers no resistance to the user when the sample vessel is being loaded into (or removed from) the stationary rotor.

Provision also needed to be made for increased mid-evaporation imbalance tolerance which can be caused by samples evaporating at different rates, but this is well within the remit of existing technologies.

SUMMARY

Condensation evaporation provides a quantum increase in available evaporation rate whilst offering a host of additional benefits to the evaporator: principally

- Simple design.
- Rugged component choice.
- Inherent temperature control.
- Efficient energy use.
- Visible evaporation.

This method has proven so advantageous that it has been developed into a novel centrifugal evaporator, providing rotary evaporator speed and utility with centrifugal benefits of bump and foam free evaporation as well as "load and forget" operation, freeing laboratory staff for higher priority tasks.