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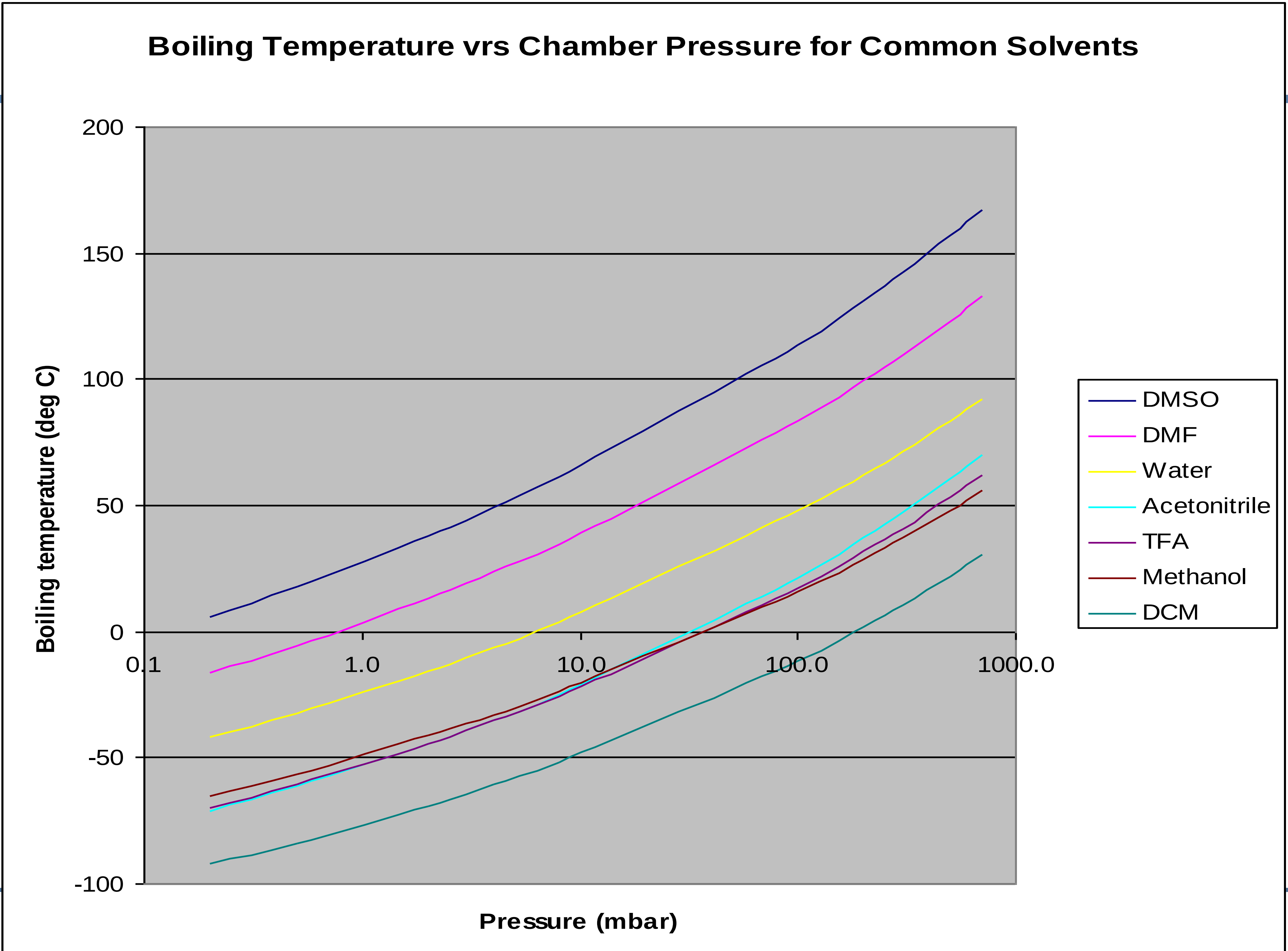
Introduction to Rocket

Do You Wanna Walk Away From Your Evaporating Sample???

Evaporation Principle

- Vacuum evaporators rely on boiling the solvent at low temperature
- Low temperature boiling is achieved by using a vacuum pump to reduce the pressure in the system, and therefore the boiling point of the solvents
- When the boiling point is lower than the sample temperature, the solvent must boil!



Boiling Temperature vrs Chamber Pressure for Common Solvents
Boiling temperature (deg C)
200
150
100
50
0
-50
-100
0.1
1.0
10.0
100.0
1000.0
Pressure (mbar)
DMSO
DMF
Water
Acetonitrile
TFA
Methanol
DCM

Solvent Properties

- Boiling
  - Boiling Point = f(pressure)
    - No good tea shop (café) on top of Mt. Everest
      - Tea needs 95°C to brew
      - Water boils at < 90°C at this altitude
  - Boiling Point = f(analyte, concentration of analyte)
    - This is why some analytes are tougher to dry than others, even with the same solvents
    - This why evaporation may slow down towards the end when the concentration of analyte increases

What we do currently

- Energy is needed to speed up boiling – this is normally supplied as heat energy
- The energy needs to reach the samples somehow

1. Hot chamber Radiation heats the samples Limited by heat transfer because there is no atmosphere to conduct heat	2. IR Heat Lamp IR heats the sample holders Heat transfer better because light can cross a vacuum. Limited by the heat transfer from holder, to tube to sample

Note - this energy does not increase the sample <u>temperature</u> – this is controlled by the <u>boiling</u> point of the solvents, set by the pressure

Efficiency Limit

- Most evaporators are limited by the rate at which heat energy can get to the samples
- Consider 3 heat sources at or near to 100°C

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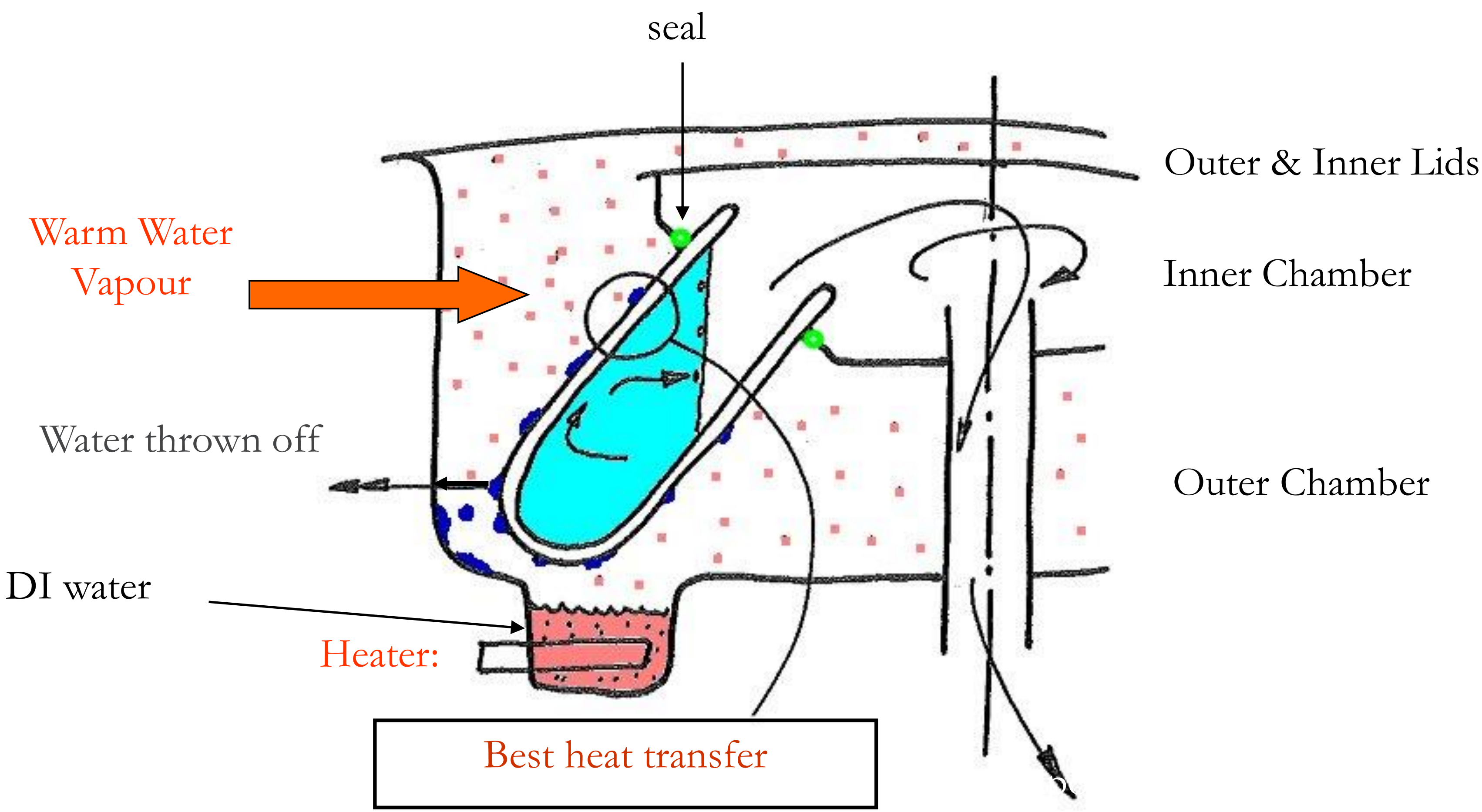
Best Heat Source

- The best heat source is steam
- Problem:
  - steam at 100°C could damage samples and may be dangerous to user!
- Solution:
  - Use vacuum control to create steam at 40°C
  - Is at low pressure – approx 70mbar
  - Is at a safe temperature – 40°C

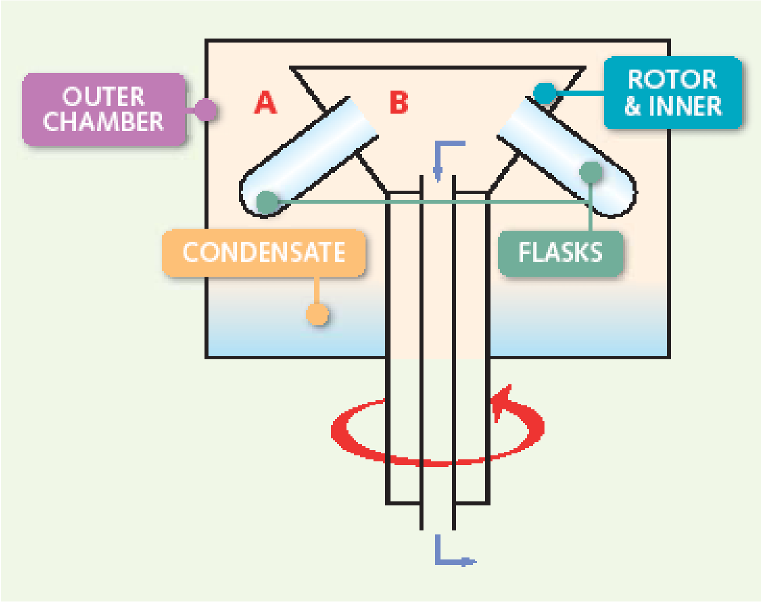
How it works



In this system many of the limiting factors have been eliminated to provide excellent heat transfer

Pressure is the Key

- Two separate pressure systems
- A – pressure to create low pressure steam, e.g. 70mbar = 40°C. Varies with control temperature
- B – pressure to boil solvents, e.g. 10mbar to boil methanol
- Vacuum pump shared between the two chambers



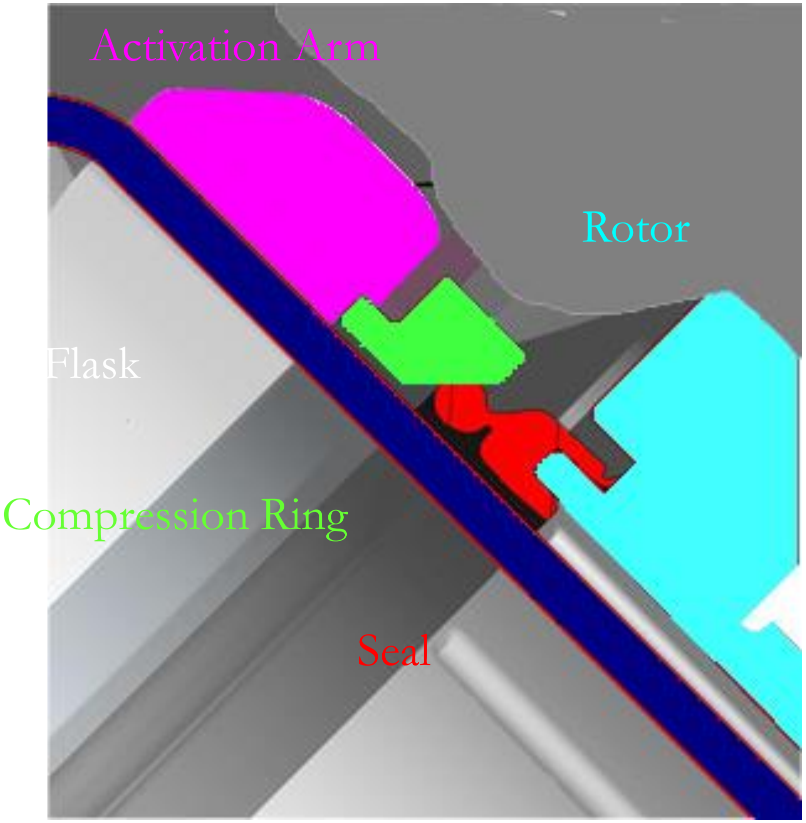
Technology Challenges

- To make this system work effectively in the laboratory, sealing the two chambers is critical issue
- The outer chamber (steam) must remain separate from the inner chamber (solvent).
- The lid for the inner chamber, is simple and easy to use.

Sealing the Flasks

- Seals present no resistance when loading flasks
  - The flask slides into position

- The flask is automatically sealed into position by the action of spinning the rotor
  - A compression ring and a weighted seal activation arm close the seal due to g-force generated by the spinning rotor.



Sample Safety

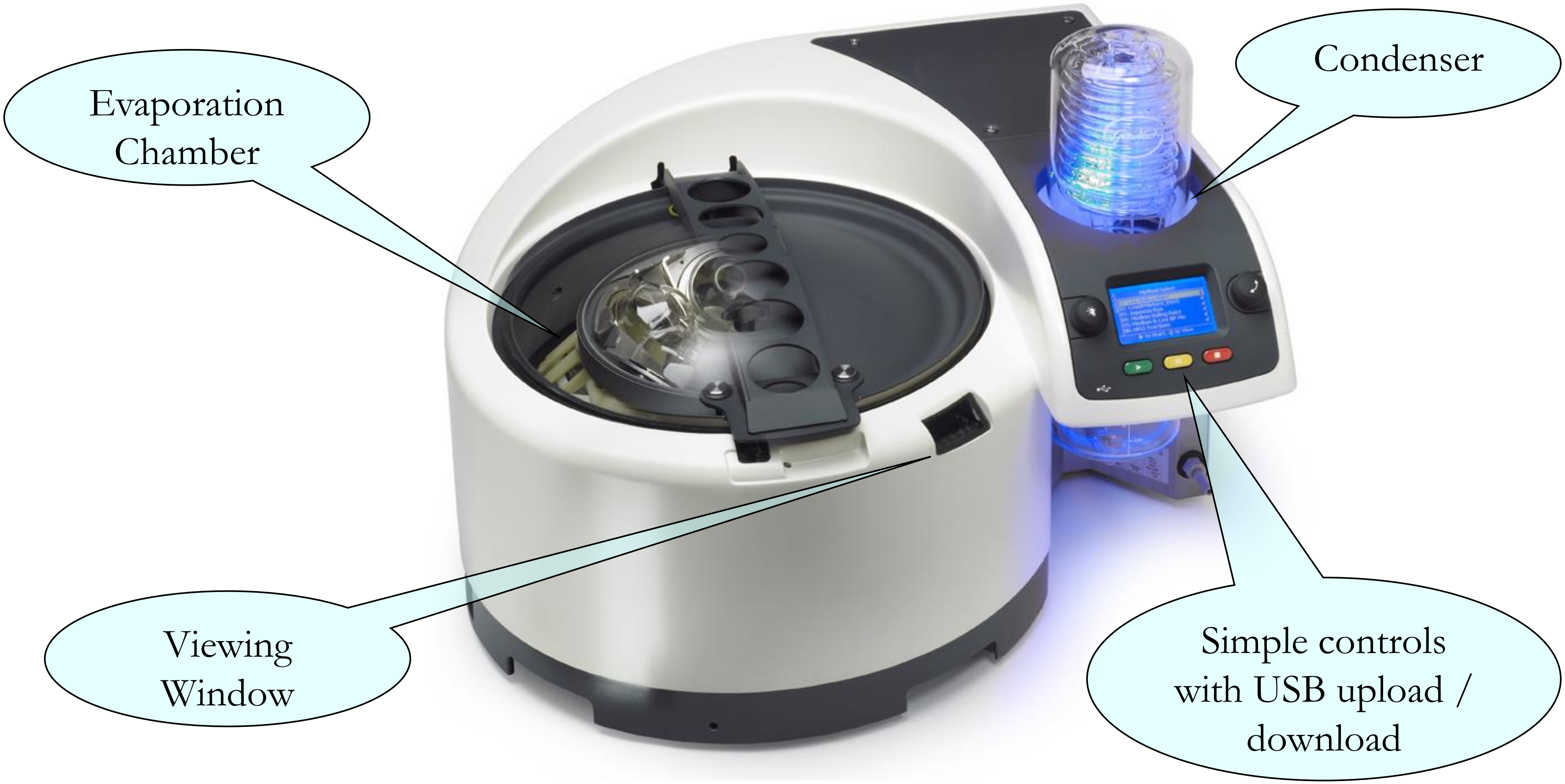
- The samples are safe from thermal damage, because:
  - Pressure controls the boiling point, and the sample temperature, while sample is wet
  - Steam is accurately temperature controlled

- Bumping is a potential risk … however,
- Genevac patented bumping control system, Dri-Pure® is used
  - Controls pressure gradient at start
  - Uses very high G-force to keep sample in flask

System Overview

ROCKET
MAKING TIME FOR SCIENCE

System Overview



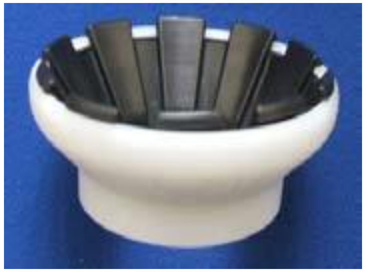
System Overview

External Chiller

- Cools and pumps coolant into Rocket condenser automatically
- Solvent capture
- Interfaces directly to the Rocket system
- Automatically adjusts coolant temp to method loaded onto Rocket
- Included in the Rocket purchase

Sample flasks with GC vial

250 Vial for evaporation to dryness



Sample Genie to evaporate 250 mL directly to GC vial

Sample Genie with Flasks

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Specification

- 3mm borosilicate glass flask, 250ml working volume (excludes vial)
- Vials from:
  - 12mm to 28mm diameter
  - 30mm to 70mm height
- Seal is low leachable white viton
- Optional Perfluoro

Rocket Flip-Flop™ Vial System

- Allows concentration from ASE vial directly into 2-mL autosampler vial
- Eliminates manual transfer step
- Simple, Fast, and Repeatable

Pucks

- Needed if working with 60 mL collections vials and the Flip Flops

Puck Performance Data

Solvent	# tubes	Volume	Time
Methanol	6	55ml (330ml)	48 min
Hexane	6	55ml (330ml)	30 min
Hexane	18	55ml (990ml)	33 min
Water	6	55ml (330ml)	74 min
Water	18	55ml (990ml)	192 min

- All tests done at 40°C

Handling HPLC Fractions

- We know that some HPLC fractions stick to the glass when the sample starts to crash out.
- Work done on SG for HT series shows that use of glass treatment can help prevent this
- A better method is to stop it sticking in the first place!
- Add a co-solvent to keep the sample in solution until it is all in the vial

Add Dioxane

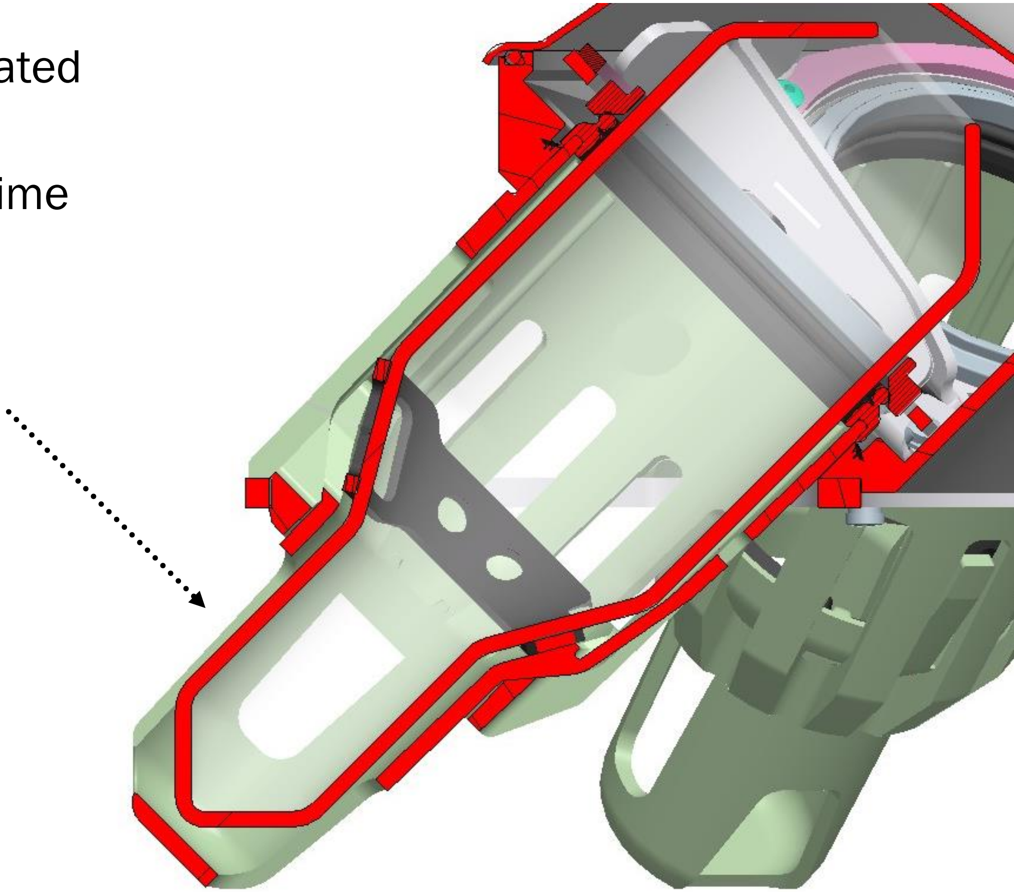
Fraction % Organic		Fraction Volume	
		100ml	200ml
	90	-	-
	80	0-5	30-50
	70	20-30	50
	60	20-30	50
	50	20	50
	40	0-20	0-20
	30	0-20	0-20
	20	0-20	0-20
	10	-	-

No Sticking!

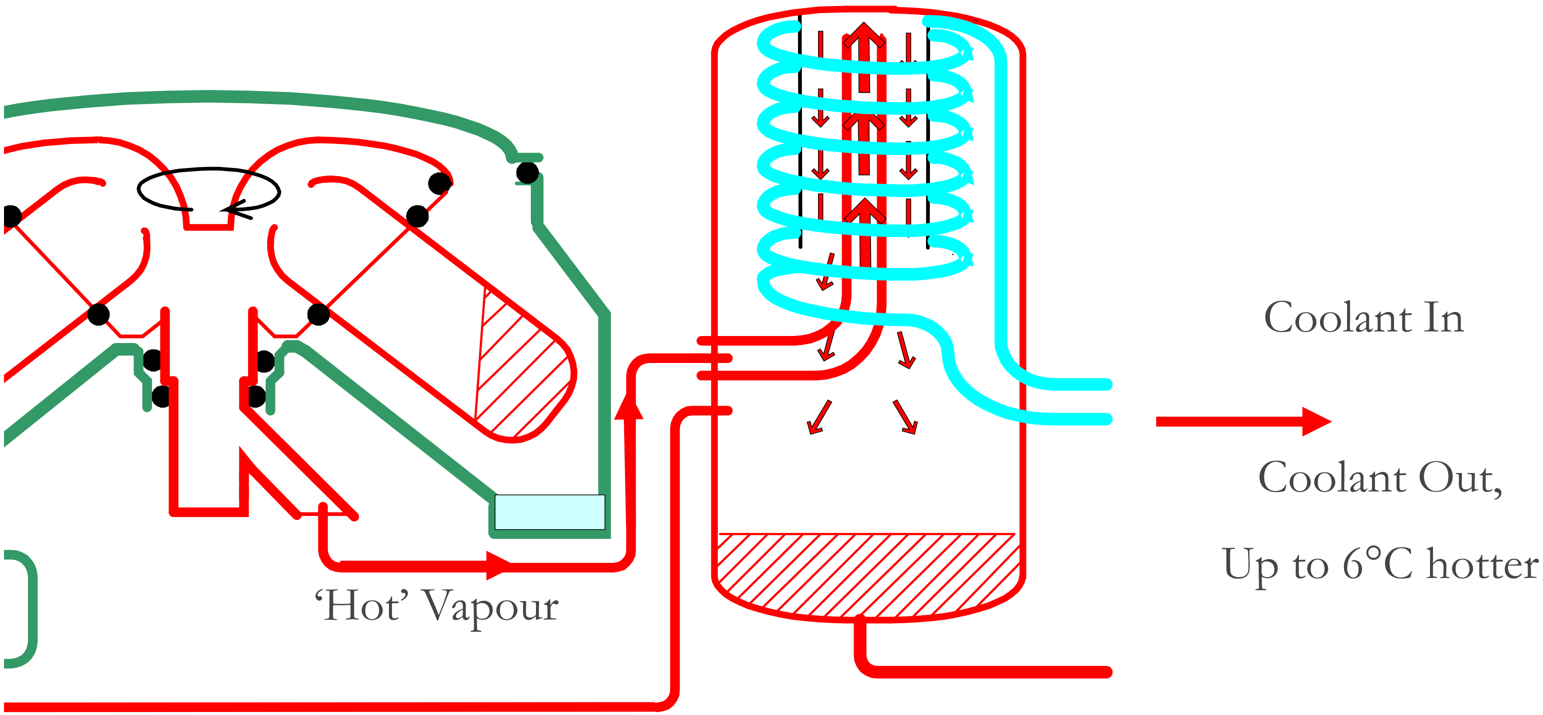
- There is a trace in the flask, but the vast majority is in the vial

When is the Sample ready?

- System features:
  - AutoStop when Dry or Concentrated technology
  - Strobe to view samples in real time



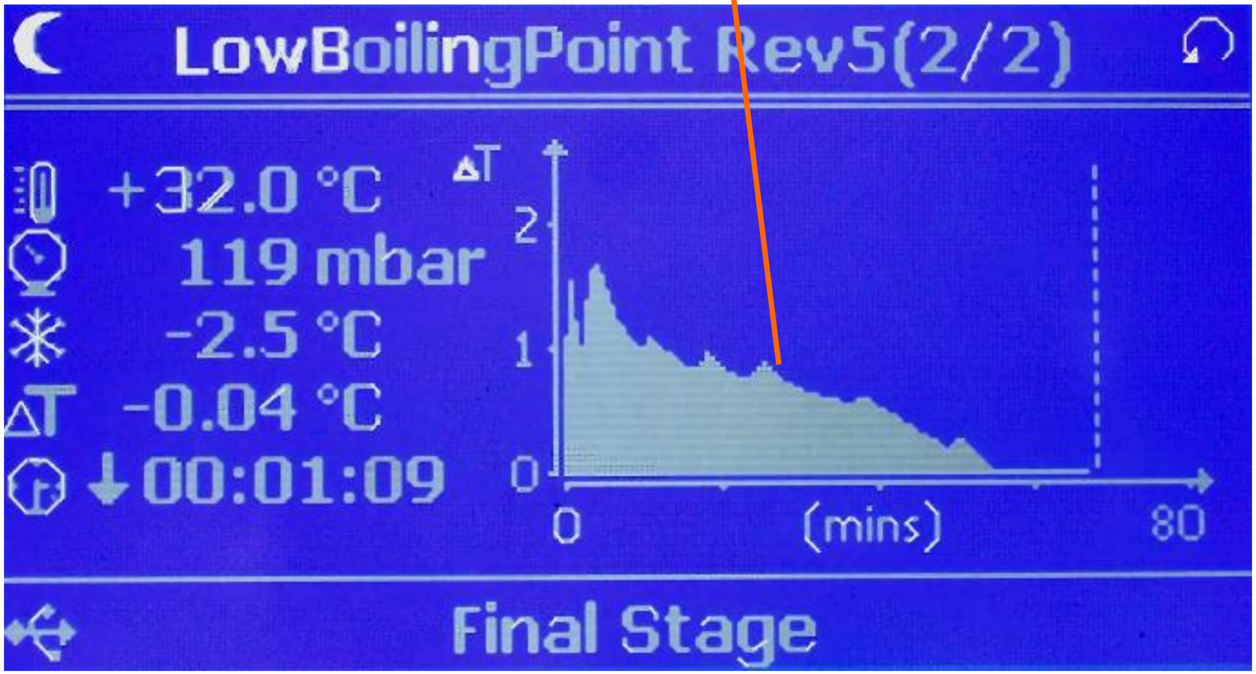
AutoStop – how it works



Delta T= Coolant Out - Coolant In

Which Looks Like:

Chiller Delta T' = Tin-Tout

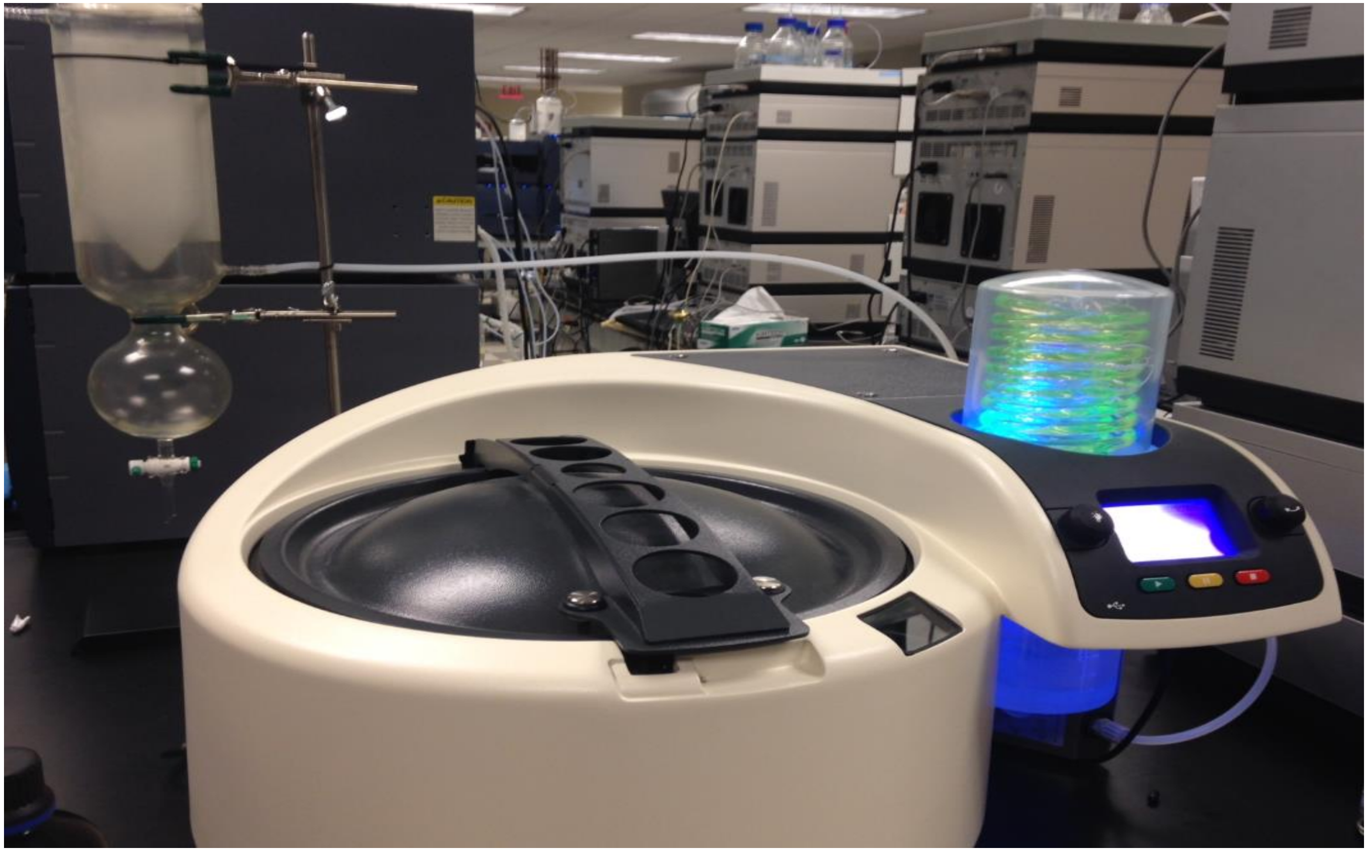


AutoStop for Concentrating

- When concentrating, the SampleGenie system with an insulating adaptor must be used
  - Vial is in an steam free environment
  - No heat input, means no evaporation
  - When sample is all in the vial, the system detects that there is no evaporation, and stops **stage 1** of evaporation
  - First time out – user must adjust **stage 2** to get desired level in vial
  - Lock in for next use

Cold Finger for MeCL2 Recovery

Acetone & Dry Ice in Cold Finger
~ 94% Recovery of MeCL2



Thank you!

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