BUCHI Corporation

Increase Analyte and Solvent Recoveries for US EPA 8082

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- Overview of PCBs
- Evaporation step after extraction
- Importance of solvent recovery
- US EPA 8082, analytical results
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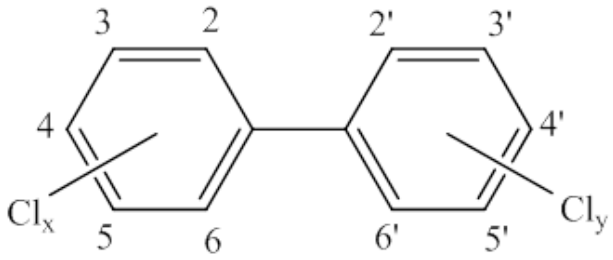
US EPA 8082

Scope of application

- US EPA method 8082 is used to determine the concentration of polychlorinated biphenyls (PCBs) in extracts prepared from many types of solid, tissue, and aqueous matrices.

- A solvent exchange from dichloromethane (DCM) to *n*-hexane is required so the sample can be efficiently analyzed through a gas chromatography / electron capture detector (GC / ECD).

For example:



PCBs

History behind PCBs

What are PCBs

- Polychlorinated biphenyls are a group of chemicals that contain 209 individual compounds with varying harmful effects.
- From 1930 to 1977 Monsanto marketed Aroclors touting chemical stability, low flammability and electrical insulating properties.
- In this time over 600,000 tons of PCBs were produced in the US[1]
- Many capacitors, transformers and other electrical products, as well as their waste from production, have contributed to massive amounts of PCBs released into the environment

1. Breivik, K; Sweetman, A; Pacyna, J; Jones, K (2002). "Towards a global historical emission inventory for selected PCB congeners — a mass balance approach1. Global production and consumption". *The Science of the Total Environment* **290** (1–3): 181–98.

Toxicity associated with PCBs

- Mixtures of PCBs have shown toxic, carcinogenic and mutagenic effects in testing and research.
- Main routes of exposure are by contact, consuming contaminated food and by breathing contaminated air.
- PCBs have not been produced or used in the US since the late 1970s but redistribution of PCBs already present in soil and water continues.
- PCBs released to waste water find their way to soil and sediment which can lead to slow leaching and bioaccumulation in fish.

EPA Technology Transfer Network. (2000). *Polychlroinated biphenyls (PCBs)(Aroclors).* Retrieved from http://www.epa.gov/ttnatw01/hlthef/polychlo.html

US EPA 8082

Workflow

Sample Preparation	• Dependent on sample matrix • Aqueous, solid or tissue
Extraction	• Dependent on sample matrix • Liquid-liquid, Soxhlet, PSE, MAE, etc.
Concentration	• Parallel evaporation
Analysis	• GC/ECD

US EPA 8082

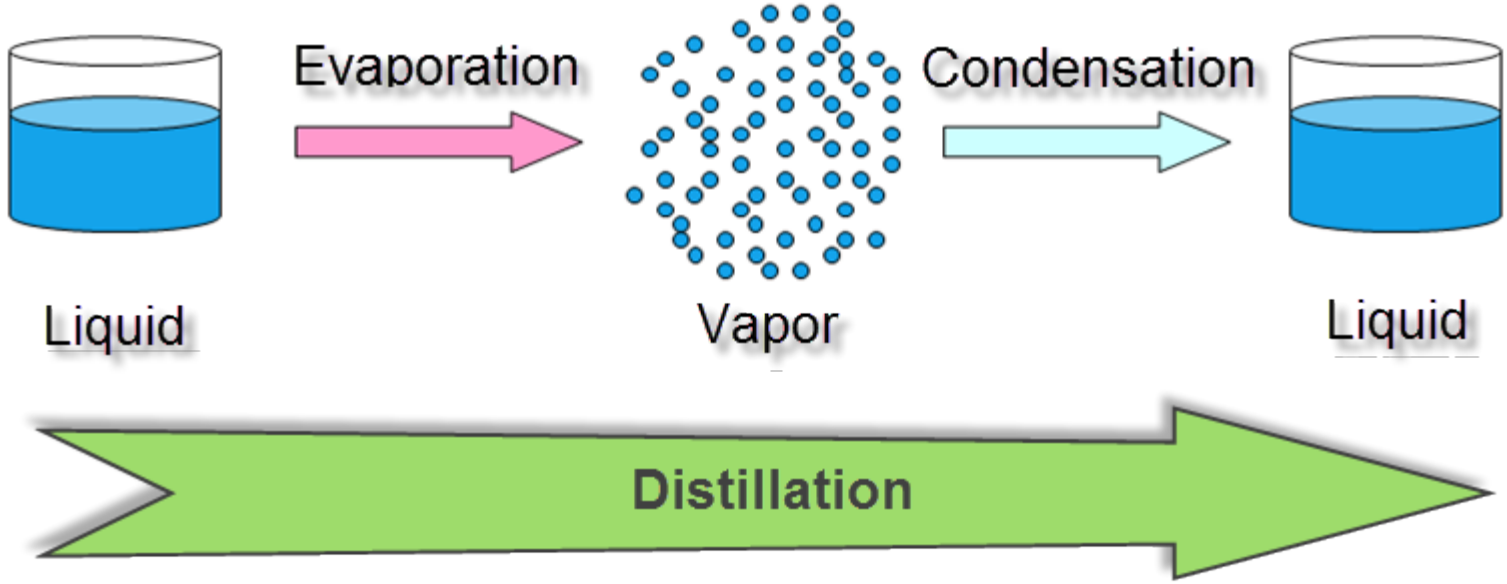
Concentration Step

- The extraction step for US EPA 8082 uses dichloromethane (DCM) as a solvent as well as variants with acetone.

- Use of electron capture detection (ECD) requires a solvent exchange because even a small amount of DCM will overwhelm the ECD.

- DCM from the extraction step is concentrated to a residual volume followed by the addition of n-hexane and further concentration to a residual volume to remove any traces of DCM.

Overview of parallel evaporation



Utilization of a vacuum source

Why evaporate under vacuum?

- Lowering the pressure lowers the boiling point
- Not as much heat or cooling is needed
- Nearly a closed system

Utilization of a vacuum source

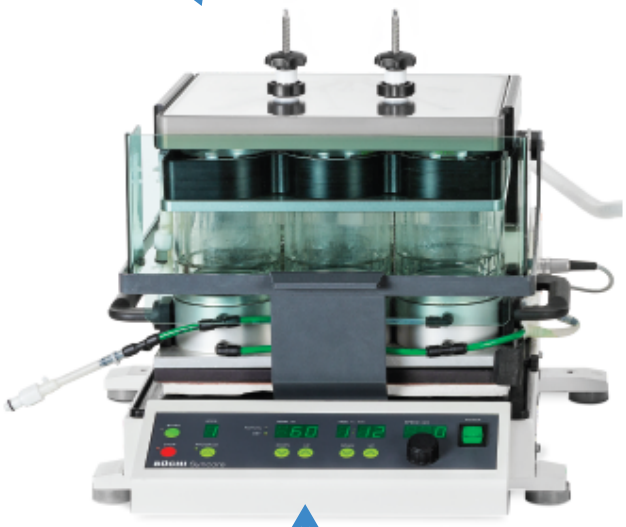
Advantages:

- Protect temperature-sensitive samples
- Speed up evaporation
- Eliminate N_2 usage
- Minimized exposure

Overview of parallel evaporation

Syncore® vortex evaporator overview

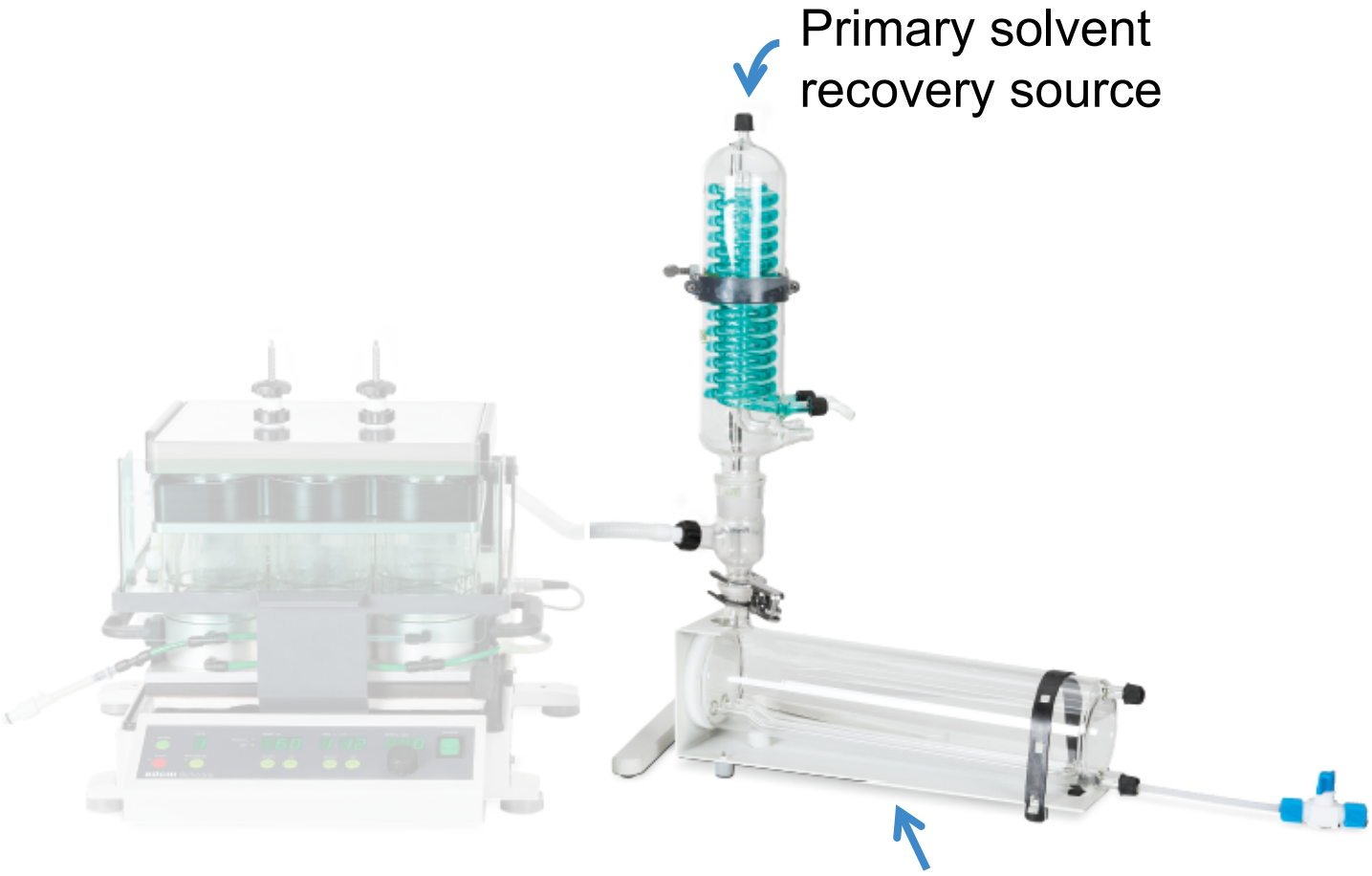
Rack and
vacuum cover



Platform

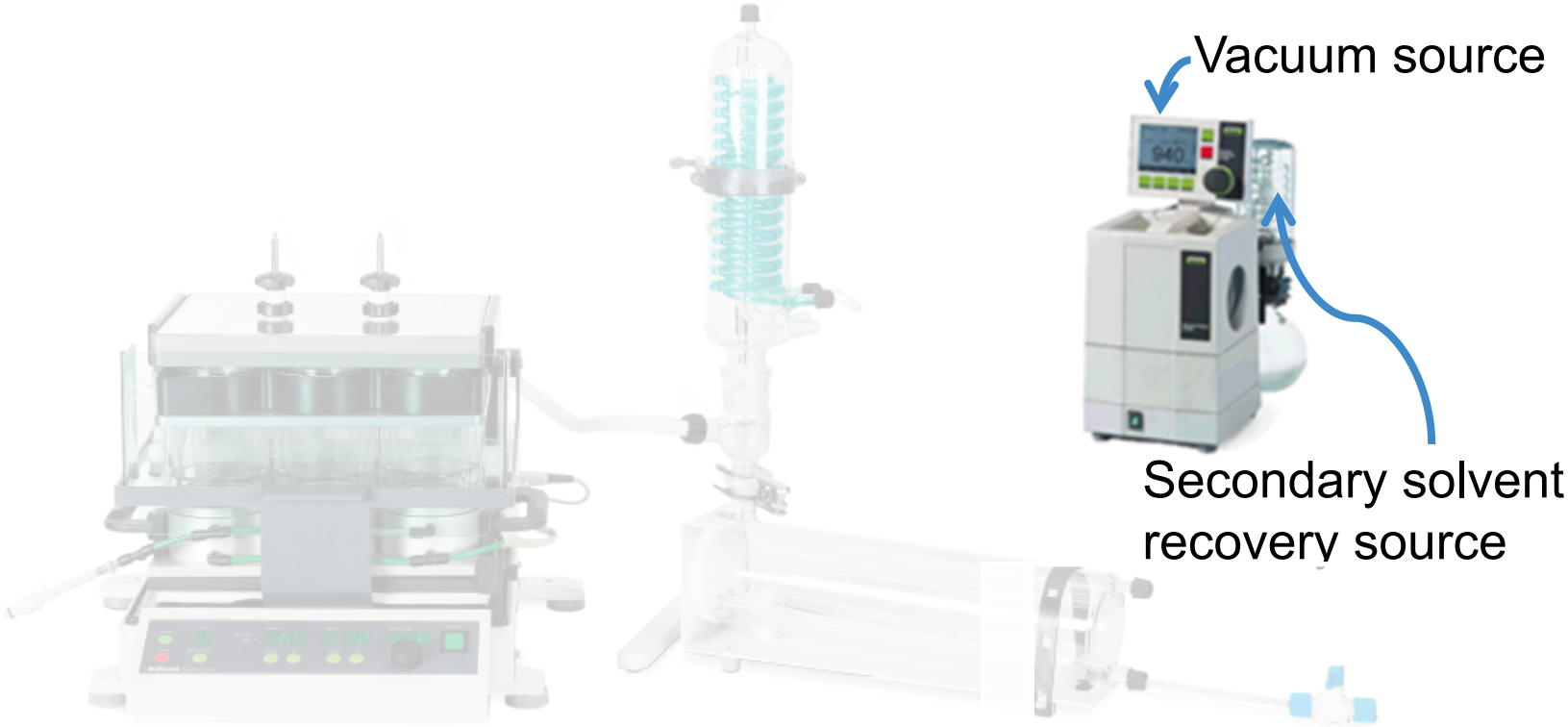
Overview of parallel evaporation

Syncore® vortex evaporator overview



Overview of parallel evaporation

Syncore® vortex evaporator overview



Overview of parallel evaporation

Syncore® vortex evaporator overview

> 95 % solvent recovery

Importance of solvent recovery

"Emissions Reduction Program"

US EPA Method 3500C and other concentration methods

- Summary of Methods; section 2.2 → "Solvent recovery apparatus is recommended for use in evaporative concentrators. EPA recommends that incorporation type of reclamation system as a method to implement an emissions reduction program."

Source:

- http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3500c.pdf

Importance of solvent recovery

Environmental aspects

No air pollution by organic or chlorinated solvents

- Chlorinated solvents are (potential) carcinogens
- Safe laboratory
- Clean atmosphere

Safe

Sustainable

US EPA 8082 results

Analytical results: Accutest® Laboratories

Spiked (0.05/0.5 ng) and concentrated laboratory control samples were analyzed for all compounds mentioned in US EPA 8082

Analytical Method	CAS Number Equivalent	Parameter Name	Spike Level (ng)	% Recovery LCS #1	% Recovery LCS #2	% Recovery LCS #3/ 2:1	% Recovery LCS #4/ 2:1
Surrogate Compounds							
SW8082	877-09-8	% Tetrachloro-m-xylene	0.05	86	88	80	82
SW8082	2051-24-3	% Decachlorobiphenyl	0.05	134	120	114	116
Target Analytes							
SW8082	12674-11-2	Aroclor 1016	0.5	94	96	86	86
SW8082	11096-82-5	Aroclor 1260	0.5	124	113	107	109

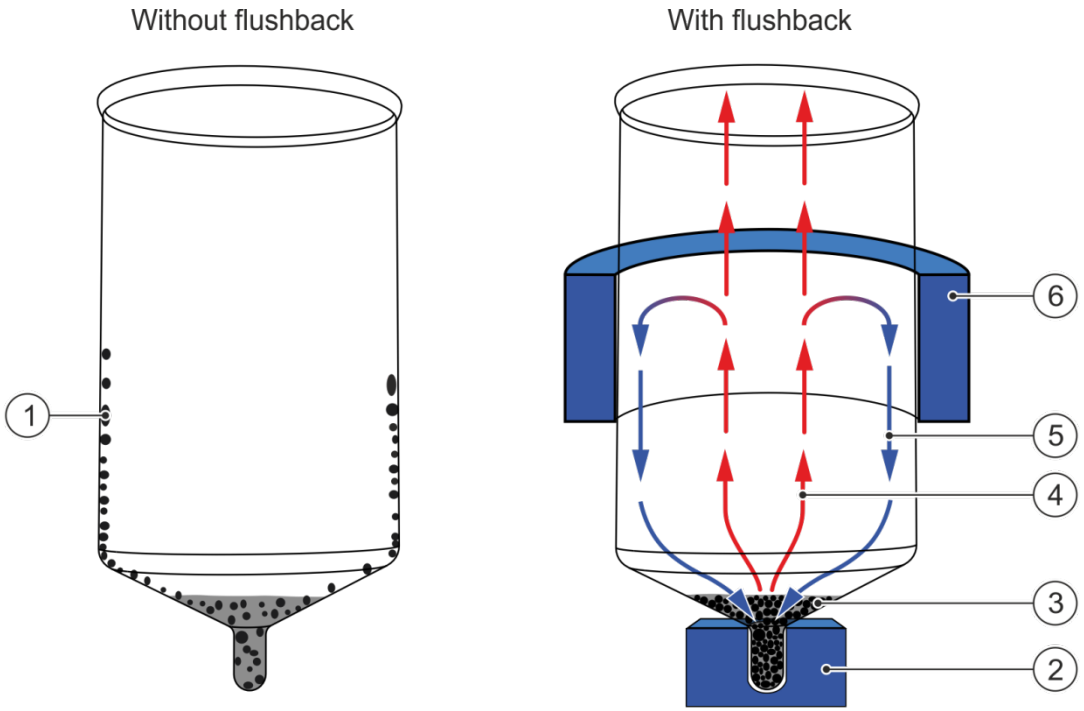
* Recoveries over 100% are due to analytical uncertainty

Analyte recovery

Which one would you choose to sample for analysis?

Analyte recovery

Flushback module



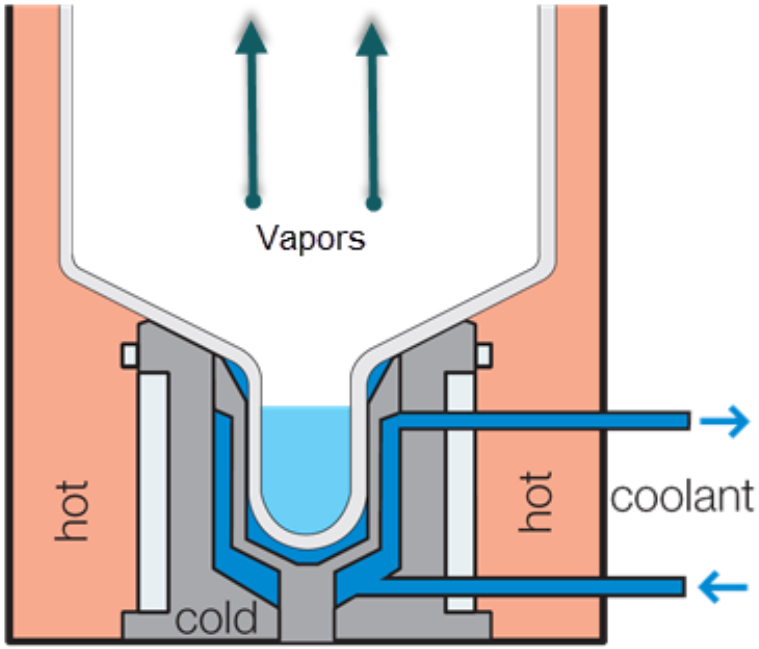
1 Analyte sticks to the wall
2 Cooling
3 Concentrated analyte

4 Evaporation
5 Flushback effect
6 Cooling

Reproducibility

Chilled appendage technology

- Less supervision
- High reproducibility
- Low risk of losing sample
- Optical sensor not required



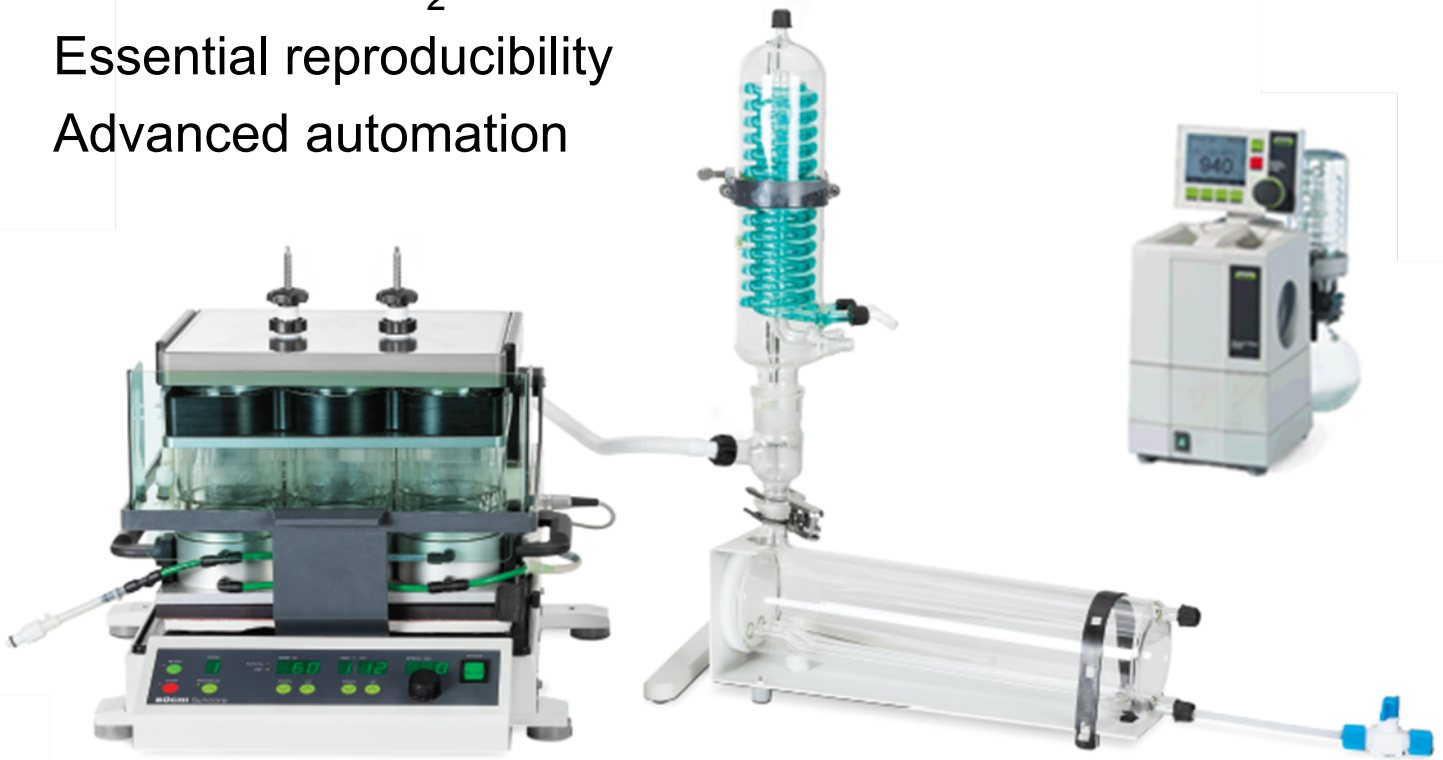
Reproducibility

- Vacuum pump
- Vacuum controller
- Chiller

- Gradient function
- Reproducible results
- Environmentally friendly

Summary

- > 95 % solvent recovery
- High analyte recovery
- Elimination of N_2 cost
- Essential reproducibility
- Advanced automation



Thank you

Questions and answers

Find out more:
http://www.buchi.com/us-en/syncore-epa

Additional “Thank You” to:
Lionel Thomas
Laboratory Manager
Accutest® Laboratories