BUCHI Corporation

Solutions Guide to Sample Prep – Extraction to Clean-up/Water Removal to Concentration to Final Volume

Overview

Concentration of Samples Complying to US EPA 8270

Parallel evaporation technology for high solvent and analyte recoveries

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Introduction

Sample concentration and analytic findings for US EPA method SW-846-8270 to determine the concentration of semivolatile organic compounds (SVOC) in extracts from solid waste matrices, soils, and water samples are highlighted.

During the concentration step traditionally performed by Kuderna-Danish (KD) or nitrogen blowdown devices, organic solvent fumes could escape into the environment. These solvent vapors are harmful to exposed operators and persist in the atmosphere.

Only recently, commercial laboratories were enforced to control their solvent emissions, non-compliance led to high monetary fines.

Here, thanks to the BUCHI Syncore® Analyst more than 95 % of the solvent evaporated during concentration is recovered and high analyte recovery rates reported.

Experimental Set-up

Cooled appendix technology

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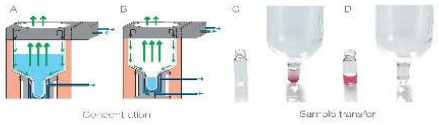

Flushback effect

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Analytical results

Spiked (50/75 ng) and concentrated laboratory control samples were analyzed by GC/MS for all compounds mentioned in US EPA SW 846-8270.

Analytical Method	CAS Number Equivalent	Parameter Name Surrogate Compounds	Spike Level (ng)	Percent Recovery % LCS 1	Percent Recovery % LCS 2
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SW8270	84-66-2	Diethyl phthalate	50	89	93
SW8270	117-84-0	Di-n-octylphthalate	50	104	105
SW8270	118-74-1	Hexachlorobenzene	50	106	102
SW8270	67-72-1	Hexachloroethane	50	87	89
SW8270	91-20-3	Naphthalene	50	91	93
SW8270	621-64-7	N-Nitrosodi-n-propylamine	50	95	91
SW8270	87-86-5	Pentachlorophenol	50	103	111
SW8270	129-00-0	Pyrene	50	101	105

* The elevated % recovery is related to the instrument.
For complete list, please visit http://www.buchi.com/epa_8270

Conclusion

- High solvent recovery > 95 %
- High analyte recovery for SVOC
- Excellent reproducibility
- Compliance with waste minimization and pollution prevention
- Automation and intensification ⟶ cost reduction
- Also applicable for US EPA 8270/625 SVOC, 8081A,B/608 organochlorine pesticides, 8082/608 polychlorinated biphenyls, and 8015 nonhalogenated organics

Physics of good Chemistry

Effects of Temperature, Pressure and Agitation

- 4 Temperature zones
- Positive and negative pressure
- Vortex motion
- Dual Condensation
- Solvent resistant design

Capabilities of the Syncore®

One platform multiple functions

- Complete solvent removal or predefined residual volume

Wide range of racks

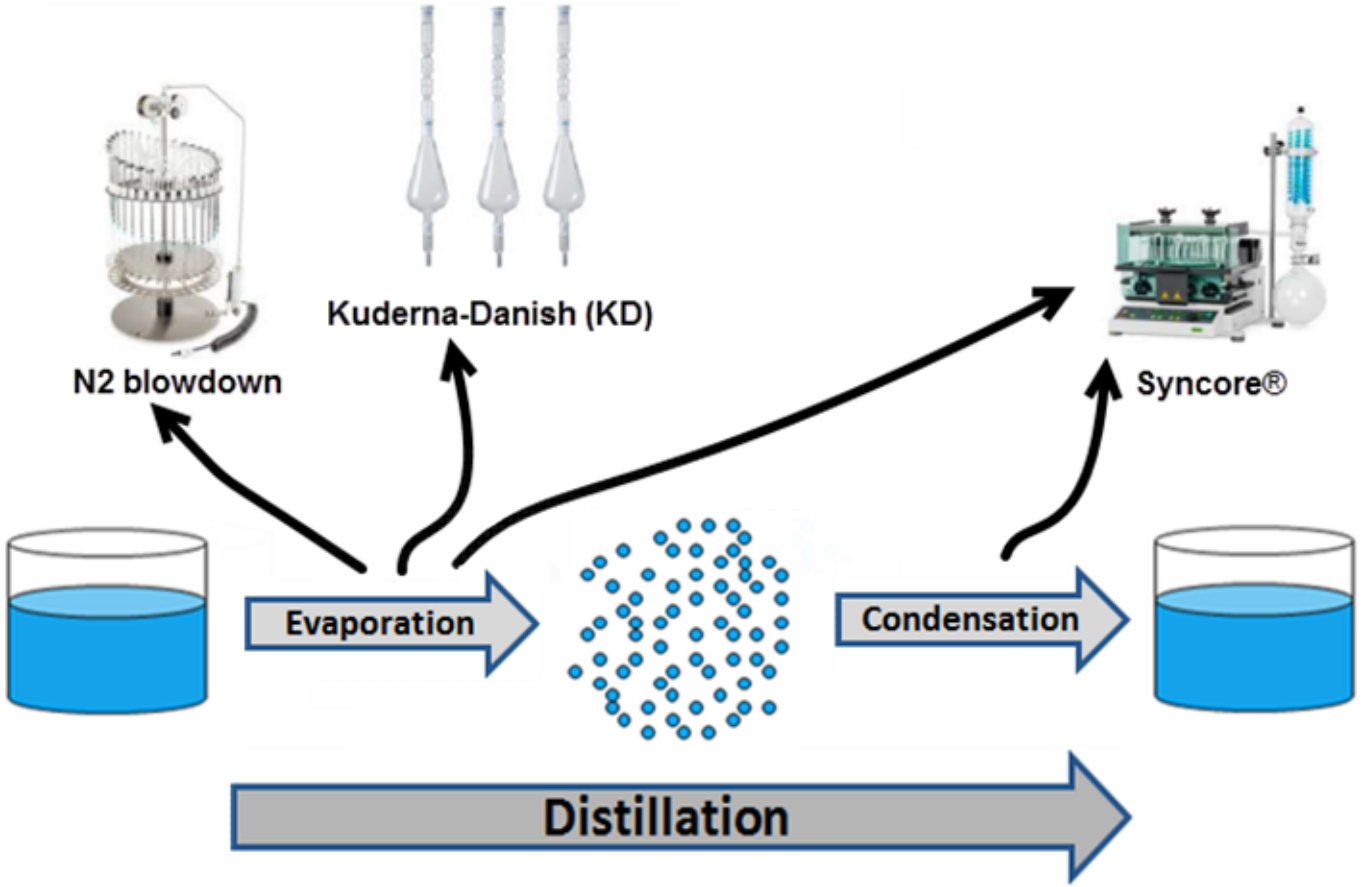
- Working volumes from 0.5–500ml

Wide range of applications

- Environmental
- Food & Beverage
- Feed
- Pharmaceutical & Nutraceutical

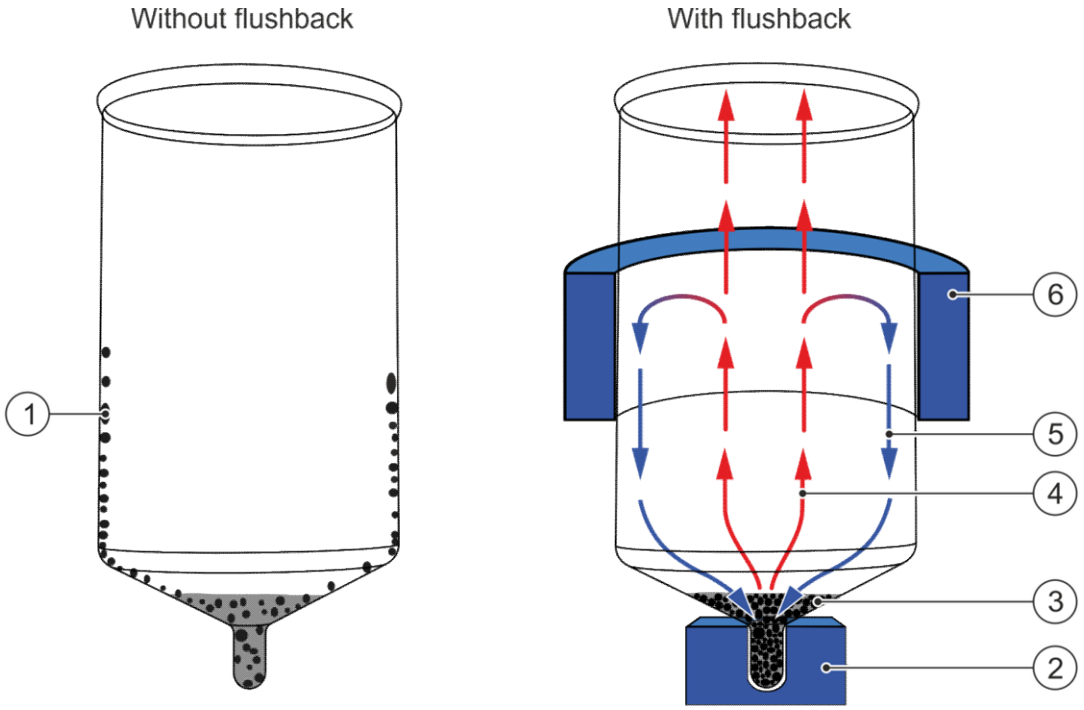
Evaporation vs. distillation

Parallel evaporation



Syncore® Analyst

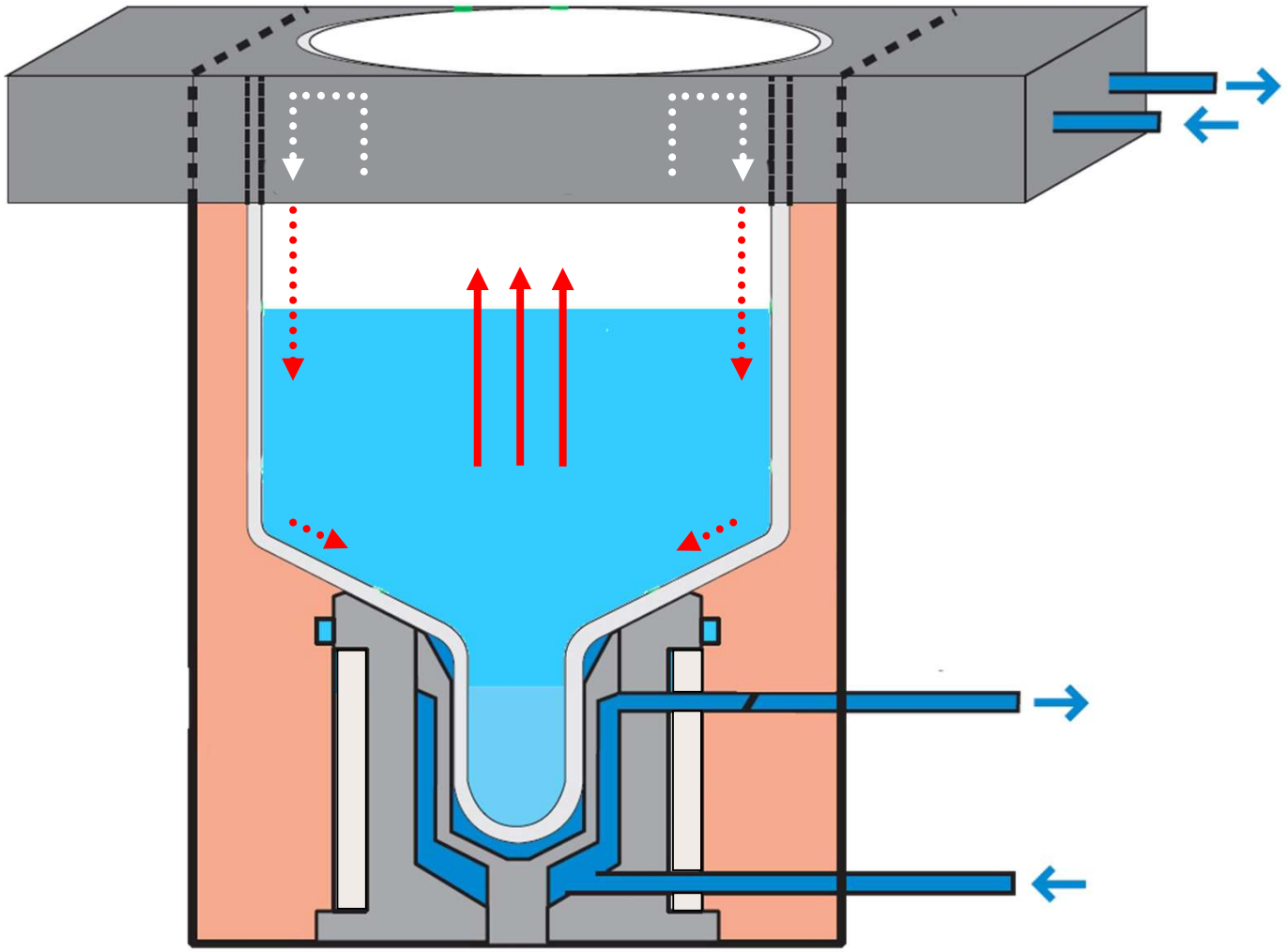
Enhance analyte recoveries



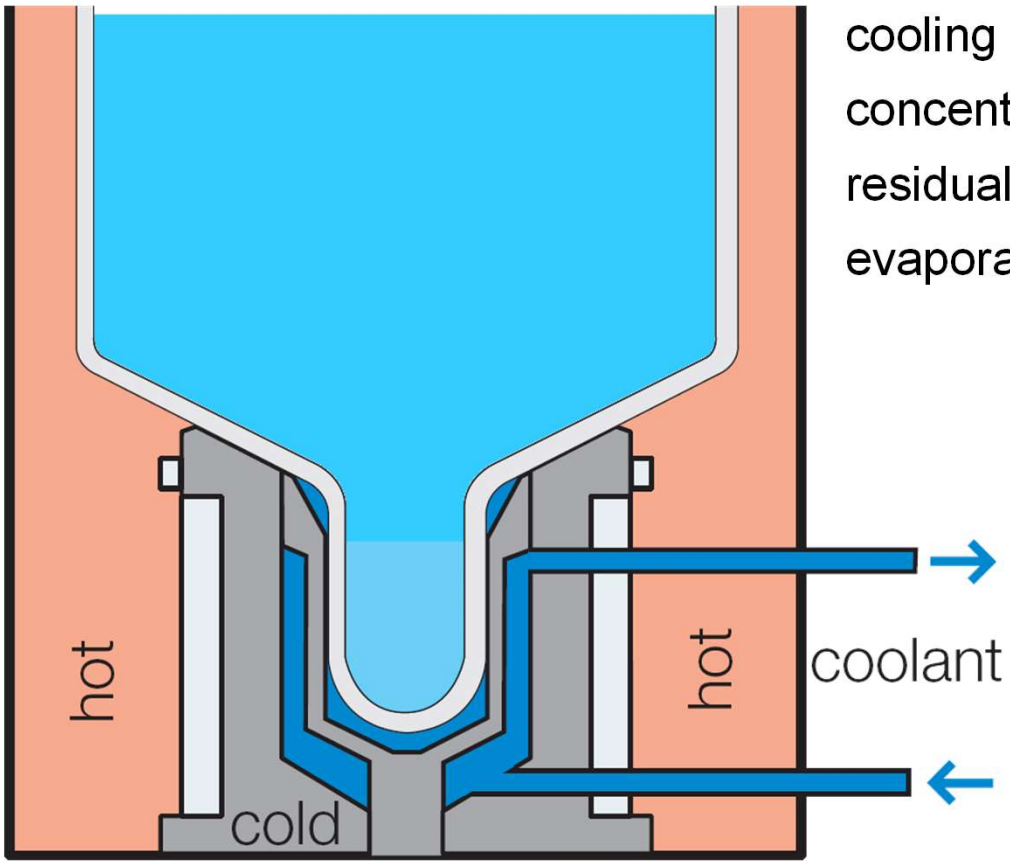
1 Analyte sticks to the wall
2 Cooling
3 Concentrated analyte

4 Evaporation
5 Flushback effect
6 Cooling

Flushback effect – increased recoveries



Concentration to pre-defined residual volumes



cooling of the appendix → concentration to pre-defined residual volume and gentle evaporation

Analyte recovery

Flushback effect – increased recoveries

Which one would you choose to sample for analysis?

Syncore® Analyst

Utilization of a vacuum source

$$PV = nRT$$

Why evaporate under vacuum?

- **Boiling point depends strongly on pressure**
- **Lower pressure → lower boiling point**

Advantages:

- **Protect thermo-sensitive samples**
- **Speed up evaporation**
- **Eliminate N_2 usage**

Syncore® Analyst

Solvent Recovery

Dual Condensation

- **Primary solvent recovery (65-70%)**
- **Post-pump recovery (25-30%)**

Advantage:

- **Maximizes solvent recovery**
- **Safer working environment**
- **Clean air & clean water**

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

Global initiative

- **The gases in question are known as “very short-lived substances” (VSLS) such as dichloromethane, which is used in a variety of industrial processes.”**

Source:
http://www.usatoday.com/story/weather/2015/02/16/ozone-layer-depletion/23494063/

How to profit from recovered solvent

Public perception

ESC Laboratories

- Promoting “environmental stewardship”
- Increase customer base due to environmental friendly approach

Importance of solvent recovery

Environmental/ health aspect

No air pollution by organic or chlorinated solvents

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

Safe

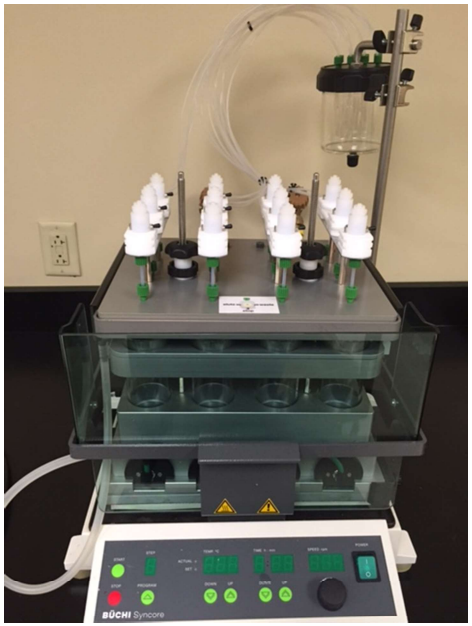
Sustainable

Syncore® Analyst

Latest innovation

Luer connection through lid

- **Solvent exchange**
  - **Pest/PCB/Herb**
- **Sample/Solvent addition**
  - **Large volumes of extracts**
- **Water removal**
  - **Hydrophobic membrane**



Summary

- Data quality
  - No gas blowdown (Nitrogen) necessary, minimizes stripping of low boiling point analytes
  - Vortex action brings analytes to the appendix
  - Vacuum rapidly removes solvent
  - Solvent addition and solvent exchange simplified
- Solvent recovery
  - Safety of lab personnel
  - Environmental responsibility

Analytical Summary - 8270

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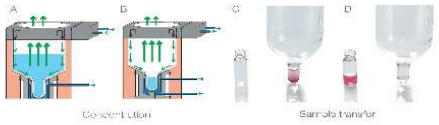

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