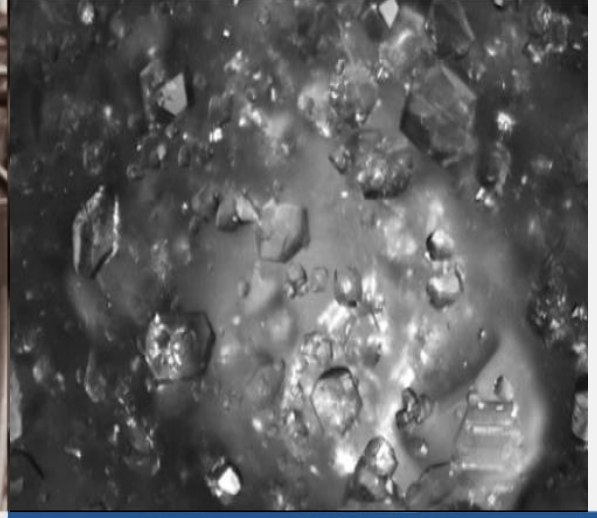
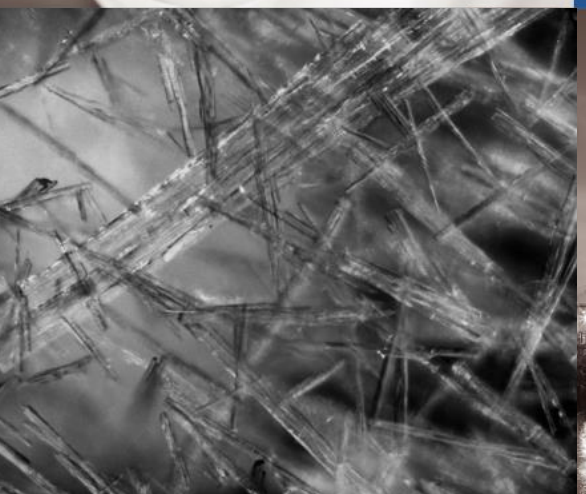
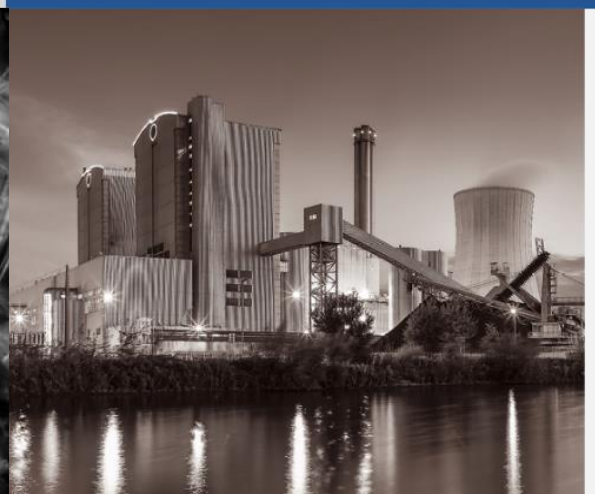



GWB

Workflow Concept in Laboratory

Tomi Kemppinen

MILESTONE
HELPING CHEMISTS

Introduction

- **GWB** in nut shell
- **Milestone Agilent collaboration**
- **Reagent dosing -** easyFILL
- **Sample filtration**: - SFS-24
- **Acid steam cleaning**: - traceCLEAN
- **Sub-Boiling Distillation**: - duoPUR & subCLEAN

OY G.W. BERG & CO AB - GWB

Sales:

- Laboratory instruments
- Research instruments (i.e. analysis instruments for laboratory)
- Microscopy & Imaging
- Industrial Sales

Service:

- Balance service team
- Analysis instruments service team
- PI service team

Research Instruments Team

- **Sample preparation**: Microwave digestion and extraction (Milestone)
- **Elemental analysis**: Mercury (Milestone); XRF (Rigaku) and CS & ONH (Eltra)
- **Material Characterization**: XRD (Rigaku); BET, particle size (Micromeritics) and Thermal Analysis (Mettler-Toledo)
- **Reaction Kinetics Analysis**: probes for particle size and spectroscopy analysis (Mettler-Toledo)
- **Reactors**: batch reactors (PARR); flow through reactors (Micromeritics) and automated reactors (Mettler-Toledo)

MILESTONE
HELPING
CHEMISTS

World wide collaboration

Agilent

Sweden
Finland
Estonia
Bosnia, Croatia
Bulgaria
Macedonia
Moldavia
Azerbaijan
Indonesia
Saudi & UAE
US
Ungheria
Slovacchia
India
Mexico
Czech
KSA, Qatar, UAE,
Kuwait
Uruguay & Paraguay
Trinidad & Tobago
Bolivia
Costa Rica, Honduras e
Nicaragua
Panama

Application Reports collaboration

Webinar collaborations

MILESTONE

Agilent
Trusted Answers

SUPERIOR SAMPLE PREPARATION FOR ELEMENTAL ANALYSIS OF LI-ION BATTERY MATERIALS

Oct. 28th, 2022 - 1pm EDT | 10am PDT

Join us for a special **Chat with an Expert**, where you will learn how critical the reliability and the quality assessing of batteries is and how microwave digestion can overcome the bottleneck associated with sample preparation for trace metals analysis.

What you'll learn:

- Understand why microwave closed-vessel digestion is an essential tool for elemental analysis.
- Learn how the latest microwave digestion systems address your acid digestion needs.
- See practical examples of elemental analysis of samples with varying levels of reactivity.
- Learn tips and tricks to improve the quality of your battery-related elemental analysis using ICP-OES.
- Find out how smart ICP-OES software and hardware can help you improve accuracy and verify purity in Li-ion battery materials.

REGISTER TO LIVE EVENT

When

October 28th, 2022
3pm CEST GMT+2

Who should attend

- Testing labs
- Li-ion battery R&D
- Manufacturers and Recyclers
- QC labs

Speakers

Gianpaolo Rota
Application Specialist
Milestone Srl

Ana Garcia Gonzalez
Application Scientist
Agilent Technologies

REAGENT ADDITION TODAY

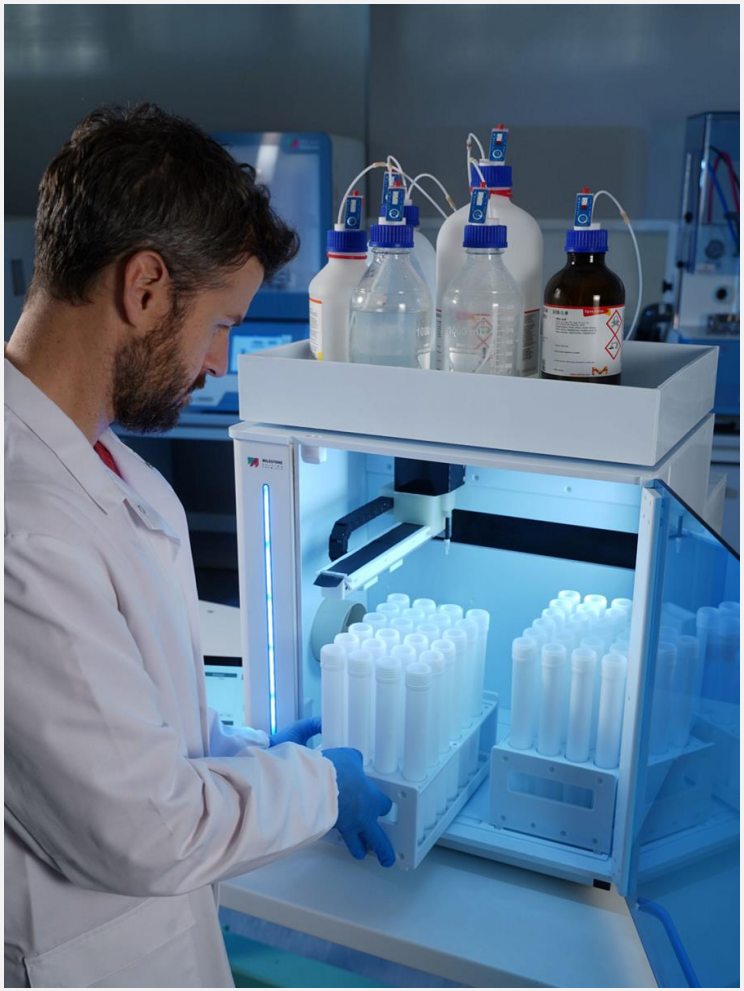
- Reagent addition is a tedious and unpleasant step
- Still done manually, with several drawbacks:
  - Safety Concerns, operator's exposure to acid vapours
  - Risk of contamination and human error
  - Time-consuming operation

IMPROVING YOUR WORKFLOW

- easyFILL – Automatic Dosing Station offers
  - Enhanced safety
  - Consistency
  - Improved workflow
  - High flexibility

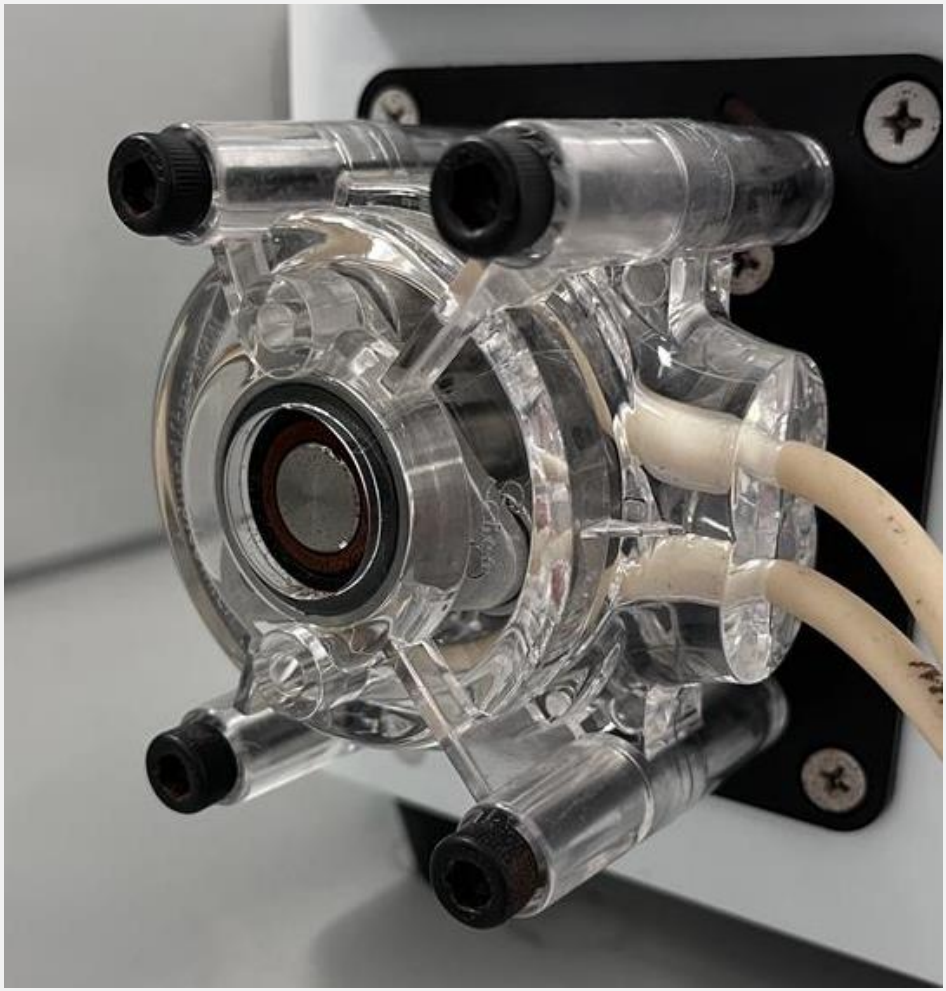
RUGGED CABINET

- Dosing compartment made of anticorrosive polypropylene
- Built-in exhaust
  - With dedicated inlet filter
- All acid-contact parts made of high-purity PTFE
  - Compatible with any acid



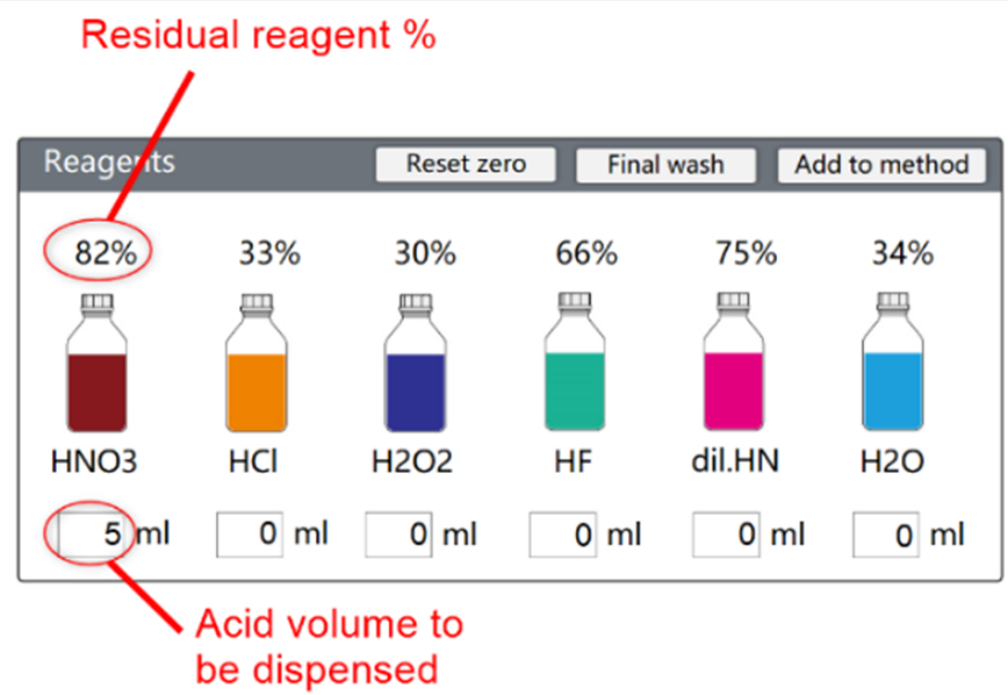
PERISTALTIC PUMP

- Peristaltic pump with dispensing speed of 1.5 mL/sec
- Dispensing Accuracy: 1%
- Working range from 0,5 mL to 50 mL (higher volume can be dosed too)



EASYFILL CAPABILITIES

- Allows for the use of up to 6 reagents
- Full reagent compatibility, including HF
- Automatic line flushing when changing acids
- Waste drains into a carboy for easy disposal
- Software tracks reagents and waste volume:
  - Specific acids require refilling
  - The waste carboy needs to be emptied



SMART SOFTWARE

- Touch screen terminal with built-in software
- Pre-stored rack configurations
- Customized methods saved for routine processes
  - Operator simply recalls a method and press “Run”
- Unlimited flexibility
  - Any # of vessels
  - Any type and volume of reagents
  - Same dosing in all batch or individual dosing in each vessel

BLANKS EVALUATION

- Comparison between Manual (bottle top dispenser) and easyFILL addition on:
  - 5 mL of TraceMetal HNO_3
  - 45 mL of UltraPure water

Result	Bias	Color code
Lower blank achieved with easyFILL	<0.00%	green
Same results	0.01-10.00%	black
Lower blank achieved with Bottle-top dispenser	>10.01	orange

MAJOR ELEMENTS (µg/L)			
Element	**Manual Addition (µg/L)**	**easyFILL addition (µg/L)**	**Bias (%)**
11B	0,23	0,22	-3
23Na	1,52	1,08	-29
24Mg	0,10	0,06	-36
27Al	0,15	0,04	-76
29Si	9,60	10,01	4
31P	0,47	0,37	-21
32S	0,21	0,12	-44
39K	6,75	6,37	-6
44Ca	0,35	0,15	-57

*Analysis done by ICP-MS TQ. Data on trace elements available in the easyFILL technology report.

WHY EASYFILL?

- Improved workflow
  - Automated process
  - Less operator time and more time for other tasks

- High reliability
  - Limited risk of contamination
  - No human error
  - Precise peristaltic pump

- Enhanced safety
  - No direct contact with acids
  - No exposure to acid vapors

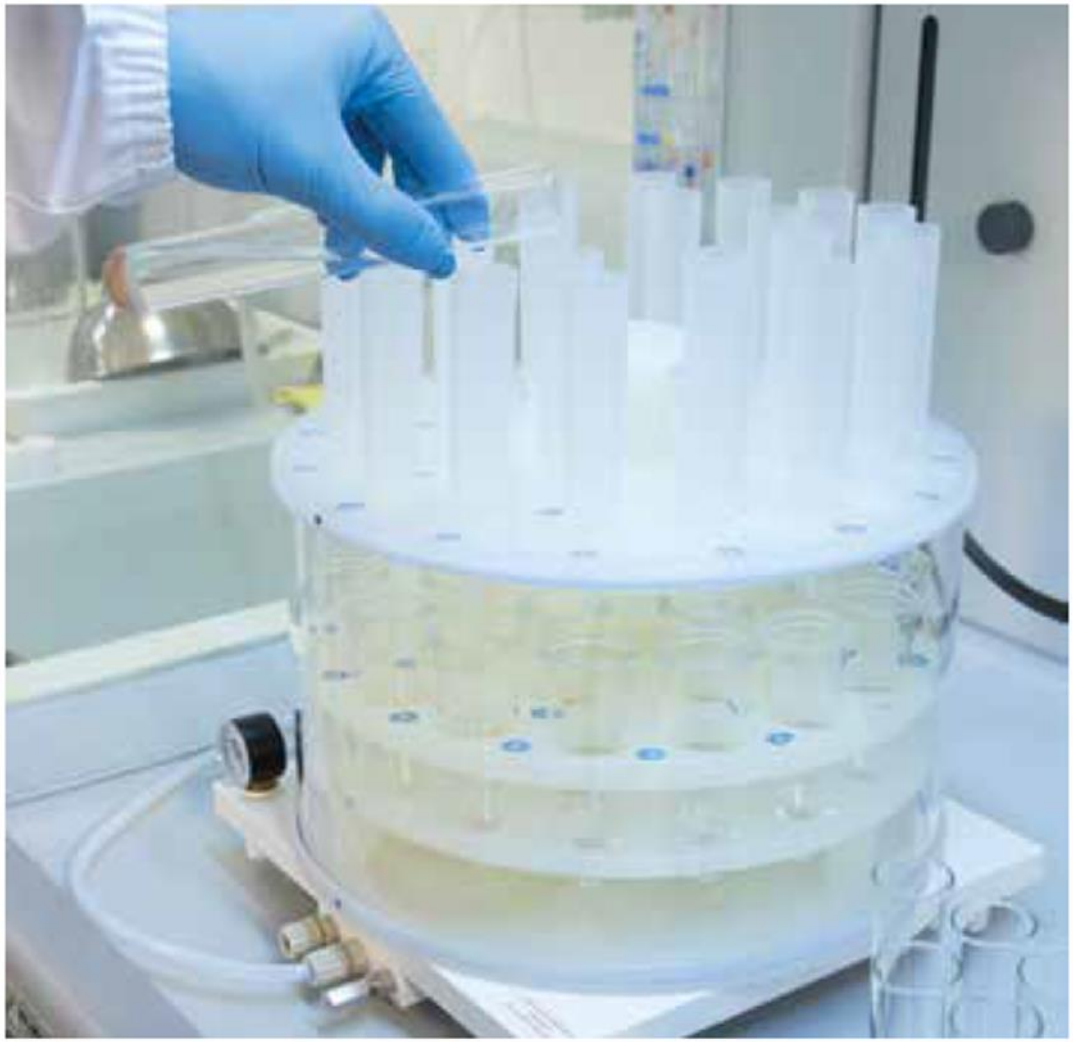
- High flexibility
  - Suitable for reagent addition and pre-dilution
  - Compatibility with all current Milestone and Non-Milestone rotors and racks

SAMPLE FILTRATION TODAY

- Sample filtration is a repetitive process
- Requires fume hood space
- Stressful for operator
- Time-consuming

IMPROVED SAMPLE FILTRATION

- SFS-24 – vacuum filtration
  - Max. 24 sample positions
  - Volume 50 ml/position
  - High sample through-put
- Existing laboratory vacuum system can be used

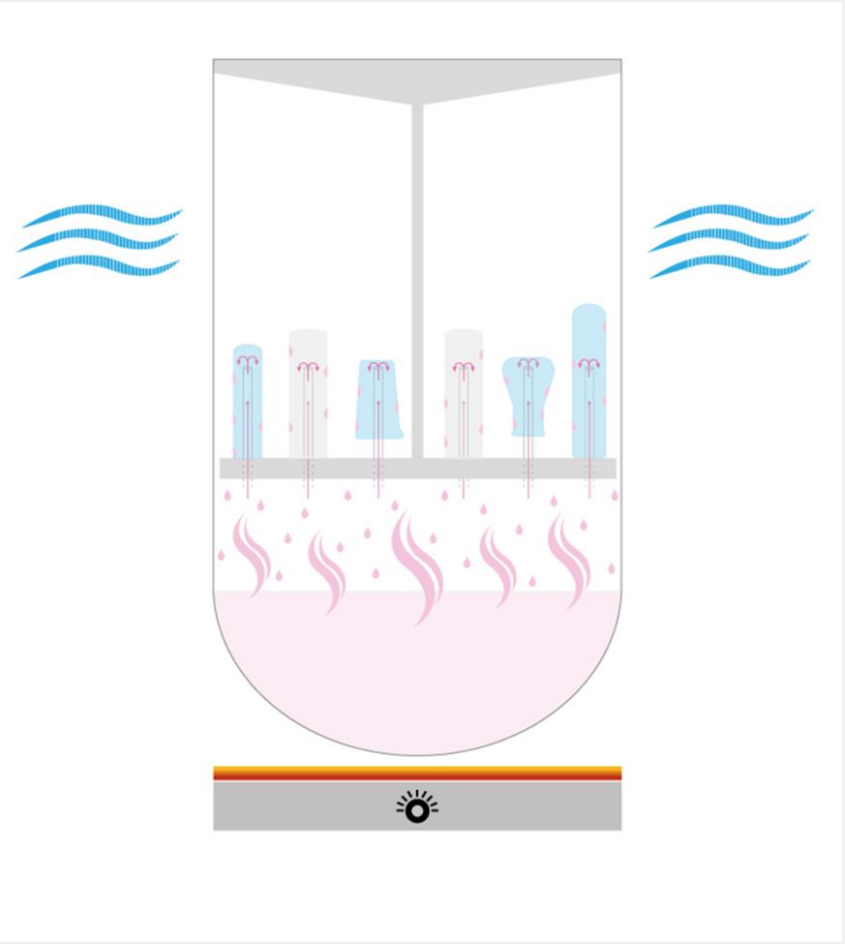


ACID CLEANING FOR TRACE METAL ANALYSIS

- Preconditioning methods seems to be the most effective method to control contamination
- The procedure suggested by US EPA (3052) is:
  - Soak in hot 1:1 HCl for minimum 2 h
  - Soak in hot 1:1 HNO3 for minimum 2 h
- Temperature must be > 80°C and < boiling point
- Steam cleaning with HNO3 vapors is very effective for routine cleaning

ACID STEAM CLEANING

- Steam cleaning with HNO3 vapors is very effective for routine cleaning
- Containers/ flasks are placed on rods
- Acid reservoir will be heated
- Condensed acid will remove the contamination



AUTOMATED ACID STEAM CLEANING

- traceCLEAN
- Suitable for Teflon, quartz and glass items
- Fast, typically < 1 hour
- Preset programs or own method

traceCLEAN PERFORMANCE

Table 9. *Comparison of high-temperature acid leaching cleaning vs acid steam cleaning. Trace metal contamination (ng/L) in 5% HNO_3 blanks prepared after cleaning are listed below. The acid leaching was performed at 180°C with mixture of HCl and HNO_3. The steam cleaning performed with HNO_3 only.*[29]

Element	TFM Teflon Vessel		Quartz Vessel	
	Acid Leaching	Steam Cleaning	Acid Leaching	Steam Cleaning
Al	287 ± 46	258 ± 24	398 ± 28	327 ± 18
Mg	289 ± 22	232 ± 15	441 ± 56	347 ± 26
Na	< 121	< 121	1190 ± 350	608 ± 67
Fe	< 474	< 474	< 474	< 474
Ni	< 55	< 55	< 55	< 55
Co	< 56	< 56	< 56	< 56
Cu	144 ± 39	117 ± 12	170 ± 15	109 ± 9
Cr	< 85	< 85	176 ± 57	< 85
Cd	< 72	< 72	< 72	< 72
Tl	< 261	< 261	< 261	< 261
Pb	< 57	< 57	< 57	< 57
Zn	995 ± 80	< 876	1640 ± 1000	1005 ± 124

Error expressed as one standard deviation (n =3)

REAGENTS FOR TRACE ELEMENT ANALYSIS

- For liquid reagents distillation has been used to improve their quality
  - This approach create significant contamination due to the bubble generation
- An alternative technique is the sub-boiling distillation
  - Sub-boiling eliminates the problems associates with the traditional distillation

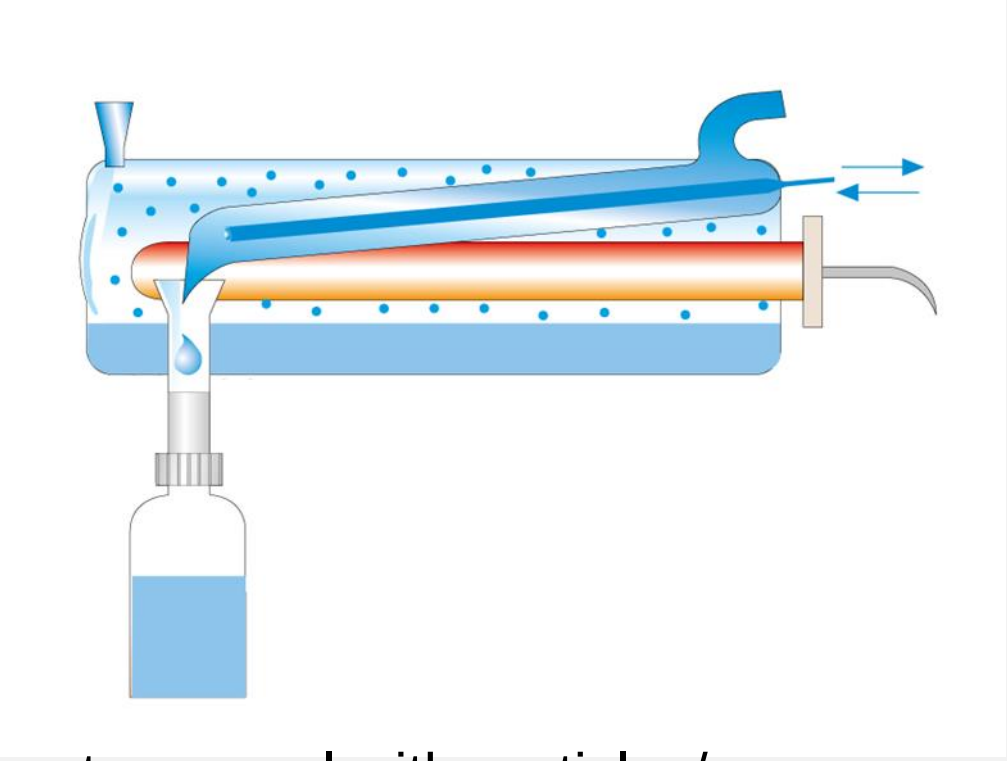
SUB-BOILING VS TRADITIONAL DISTILLATION

Comparison of metal contamination (µg/L) in nitric acid purified by traditional and sub-boiling distillation

Element	Sub-Boiled Distilled	Traditional Distillation
Pb	0.02	0.2
Tl	-	0.2
Ba	0.01	8
Te	0.01	0.1
Sn	0.01	0.1
In	0.01	-
Cd	0.01	0.1
Ag	0.1	0.03
Sr	0.01	2
Se	0.09	0.2
Zn	0.04	4
Cu	0.04	20
Ni	0.05	20
Fe	0.3	24
Cr	0.05	6
Ca	0.2	30
K	0.2	10
Mg	0.1	13
Na	1	80
Total	2.3 µg/L	220 µg/L

SUB-BOILING DISTILLATION SYSTEM

- Sub-boiling distillation uses infrared heaters
- Infrared heating vaporize the surface liquid
- Vaporized liquid is condensate by a cooling system



Vaporization without boiling prevents aerosol with particles/ contaminates

MILESTONE DUOPUR
QUARTZ SUB-BOILING DISTILLATION SYSTEM

- Double Quartz sub-boiling system
- Cost savings of ultra-pure acid
- High productivity
- Water cooling
- Suitable for HNO_3, HCl, H_2O

SUBCLEAN DISTILLATION SYSTEM

- PTFE sub-boiling system
- Cost savings of ultra-pure acid
- Air cooling
- Suitable for HF, HNO3, HCl, H2O
- Affordable

Questions?