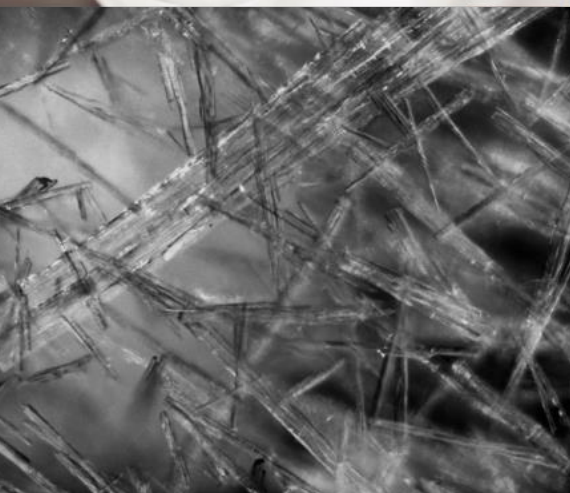
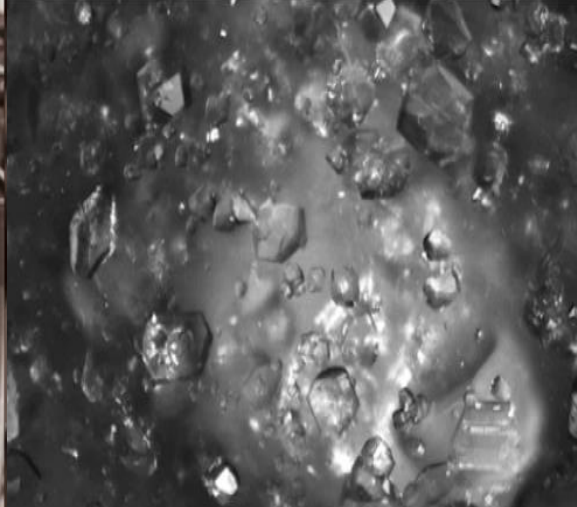




Workflow Concept in Laboratory

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
H E L P I N G
C H E M I S T S

World wide collaboration



- 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



Direct determination of Cu, Fe, Mn, P, Pb and Ti in HF acid-digested soils using the Agilent 4200 Microwave Plasma-Atomic Emission Spectrometer

Application note

Agriculture

Authors

Dave McDonald and Alejandro Amorin, Agilent Technologies Melbourne, Australia



Introduction

Accurate elemental analysis of soils is extremely important for environmental effects. Lead and cadmium are known for their environmental effects. Phosphorus, copper, iron, and zinc are important macro and micro nutrients for plants. Titanium minerals in soil are also of interest for agricultural science, crop studies.

Laboratories that analyze soil and rock samples (HF) during sample preparation to ensure where silicate based minerals may be present. Introduction systems are not suitable for them unless the samples are neutralized prior to and degrades the glass and quartz components.

Unfortunately, the HF neutralization step introduces a potential source of contamination routinely analyze samples prepared using



Determination of major elements in milk using the Agilent 4200 MP-AES

Application note

Food testing & agriculture

Authors

Courtney Tanabe, University of California, Davis, California, USA

Fabio Silva, Agilent Technologies Brasil Ltda, Brazil

Greg Gilleland, Agilent Technologies, Inc., USA

Jenny Nelson, Agilent Technologies, Inc., USA



Introduction

Milk is one of the most important food sources. As a substantial source of several essential elements, milk consumption has grown, particularly in developed countries.

As a substantial source of several essential elements, milk consumption has grown, particularly in developed countries. As a substantial source of several essential elements, milk consumption has grown, particularly in developed countries.

Essential elements such as Ca, P, and K are important for human health. Deficiencies in these elements can lead to various health issues.



Direct determination of Al, B, Ti, V and Zr in HF acid-digested alloys using the Agilent 4210 Microwave Plasma-Atomic Emission Spectrometer

Application note

Author

Alejandro Amorin, Agilent Technologies Melbourne, Australia



Introduction

Nickel alloys are used when good high- and low-temperature strength and corrosion resistance are needed. Typical industrial applications are for fabrication of chemical and petrochemical process vessels and gas turbine parts. The aerospace and military industries are also important users of nickel based alloys, which are frequently used in the manufacture of jet engine parts including turbine blades. The additive elements in the nickel alloy and their concentrations are carefully selected in order to obtain the desired material properties. For example, titanium [Ti] is added to improve corrosion resistance and increase the strength-to-density ratio of the alloy.



High throughput, low cost environmental samples analyzed by EPA 6010C using the Agilent 5100 ICP-OES

Application note

Environmental

Author

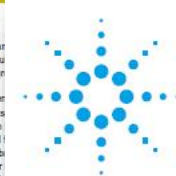
Neel Dvodelic, Agilent Technologies Melbourne, Australia



Introduction

Many laboratories concerned with the analysis of elements in environmental samples, such as ground waters, industrial wastes, soils, sludge and sediment by ICP-OES, work to United States Environmental Protection Agency (US EPA) method 6010C guidelines. Fast sample throughput and low cost analysis is desirable for these labs but can be challenging to achieve using spectrochemical techniques due to the wide range of elements and their varying concentrations in typical samples.

Traditionally, radial ICP-OES with a vertical torch or a dual view (DV) ICP-OES have been used for the determination of major, minor and trace elements in complex environmental samples. However, the unique Synchronous Vertical Dual View (SVDV) configuration of the Agilent 5100 ICP-OES ensures that the instrument can be operated in the best mode for the application (axial, radial, vertical dual view or synchronous vertical dual view) providing full flexibility with established methods and application requirements [1].



Analysis of domestic sludge using the Agilent 4200 MP-AES

Application note

Environmental

Authors

Neel Dvodelic, Agilent Technologies, Melbourne, Australia



Introduction

Managing the treatment and disposal of domestic sludge is an important activity and one that is highly regulated in many countries. After treatment, the effluent may be discharged leaving a mixture of water, organic solids and chemicals, nutrients, heavy metals, and inorganic ions. This sludge can be further treated and the resulting biosolids can then be applied to the land as a fertilizer, sent to landfill or incinerated. It is vital that the sludge or biosolids are tested and conform to regulatory levels in order to protect public health and the environment.

The United States federal biosolids rule is contained in 40 CFR Part 503. The European Union Sludge Directive 86/278/EEC is expected to be revised as several Member States have enacted and implemented stricter limit values for heavy metals and set requirements for other contaminants.

In this work, an Agilent 4200 MP-AES was used to determine major and minor elements in domestic sludge, following microwave digestion.



Webinar collaborations



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

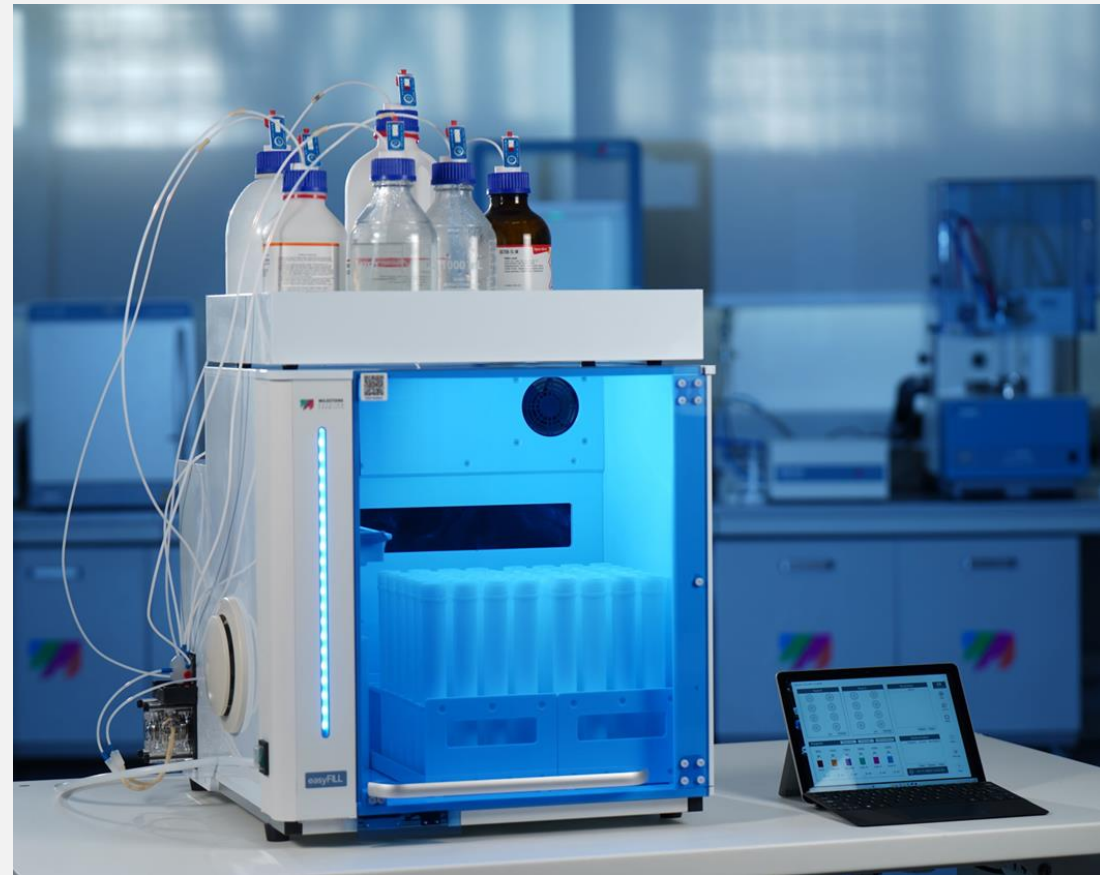
REAGENT ADDITION TODAY

- 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



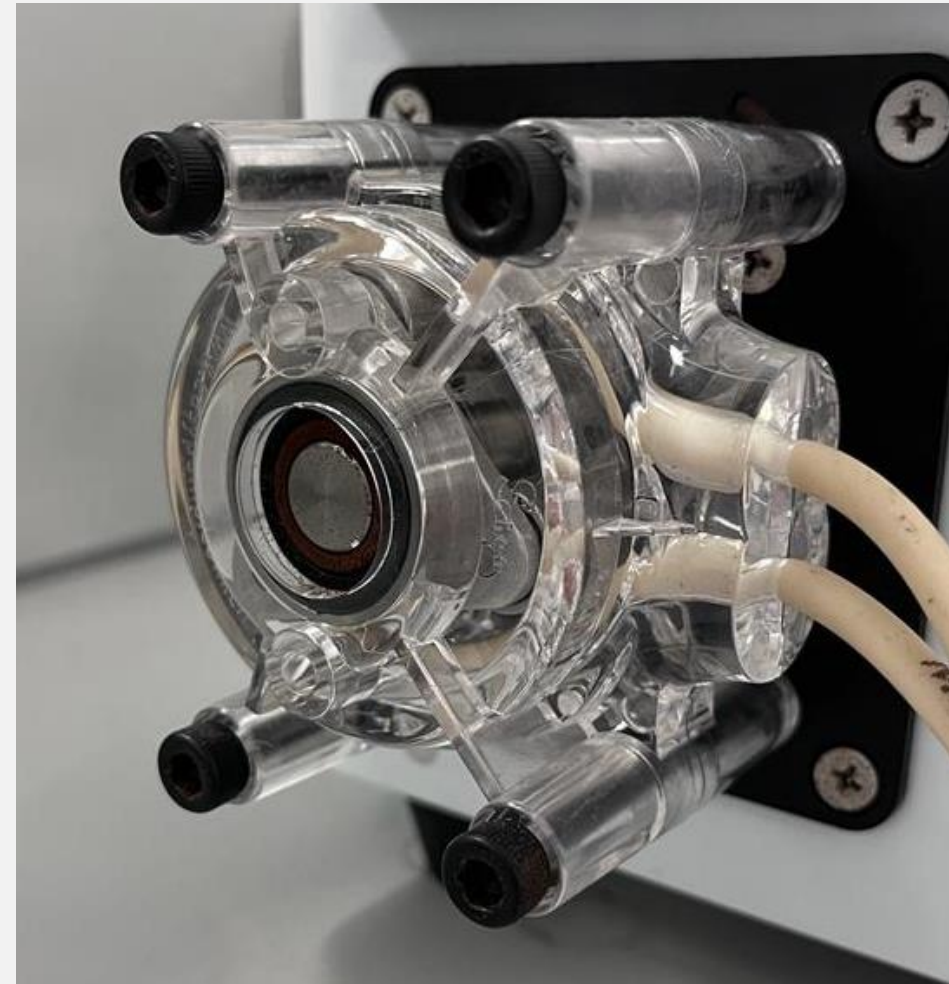
RUGGED CABINET

- 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



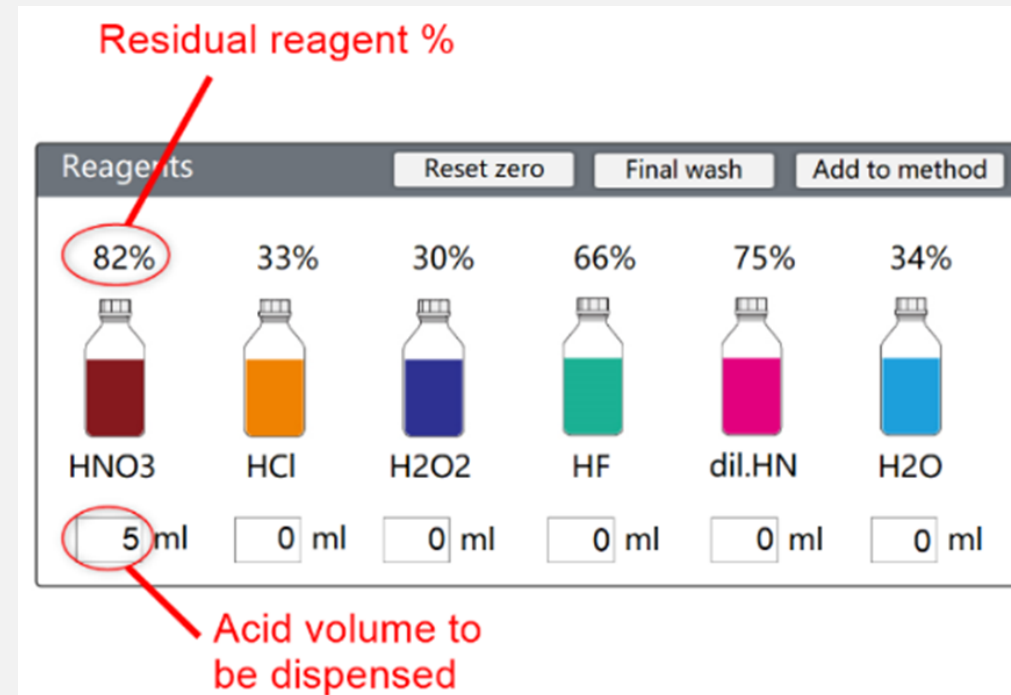
PERISTALTIC PUMP

- 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 HNO₃
 - 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

Element	MAJOR ELEMENTS (µg/L)		Bias (%)
	Manual Addition (µg/L)	easyFILL addition (µg/L)	
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

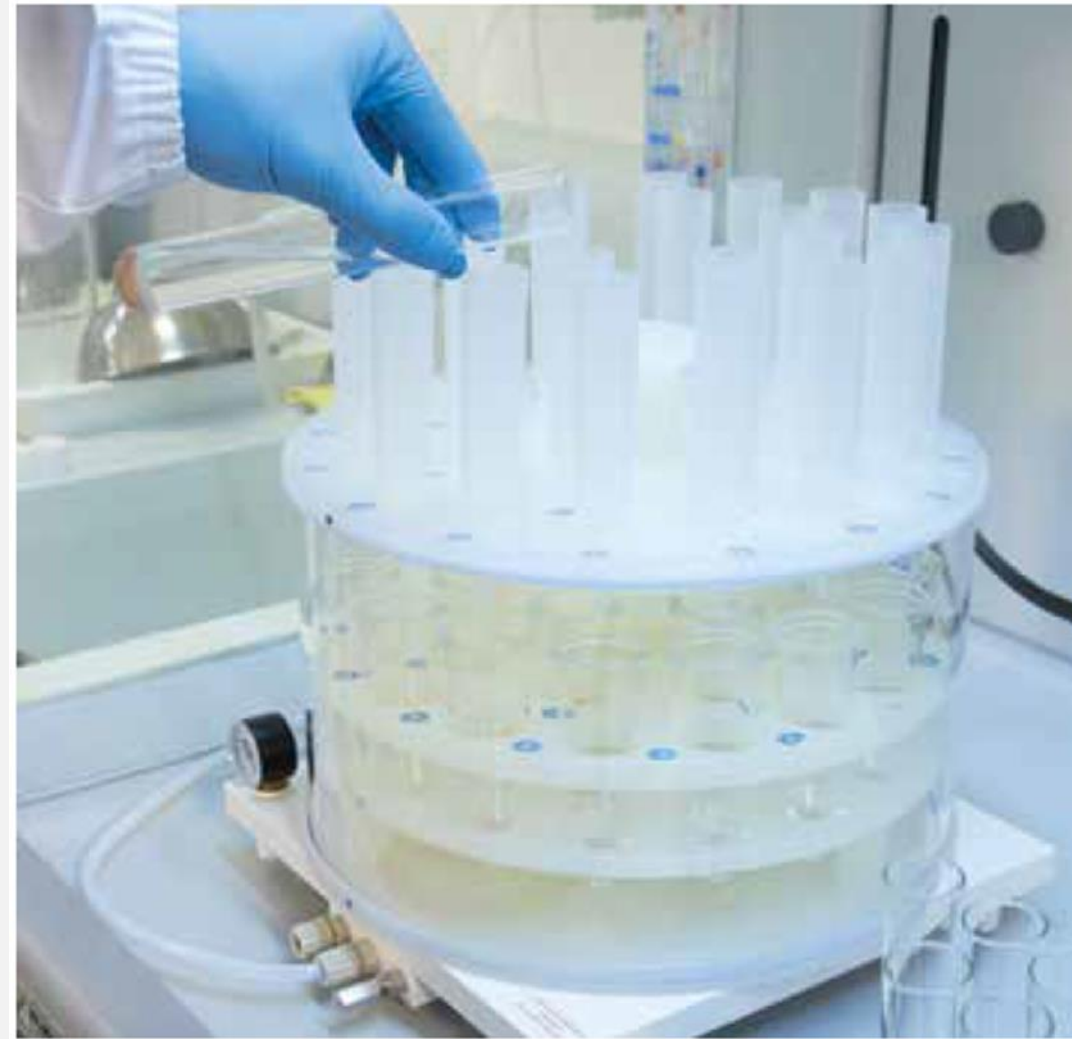
SAMPLE FILTRATION TODAY

- 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



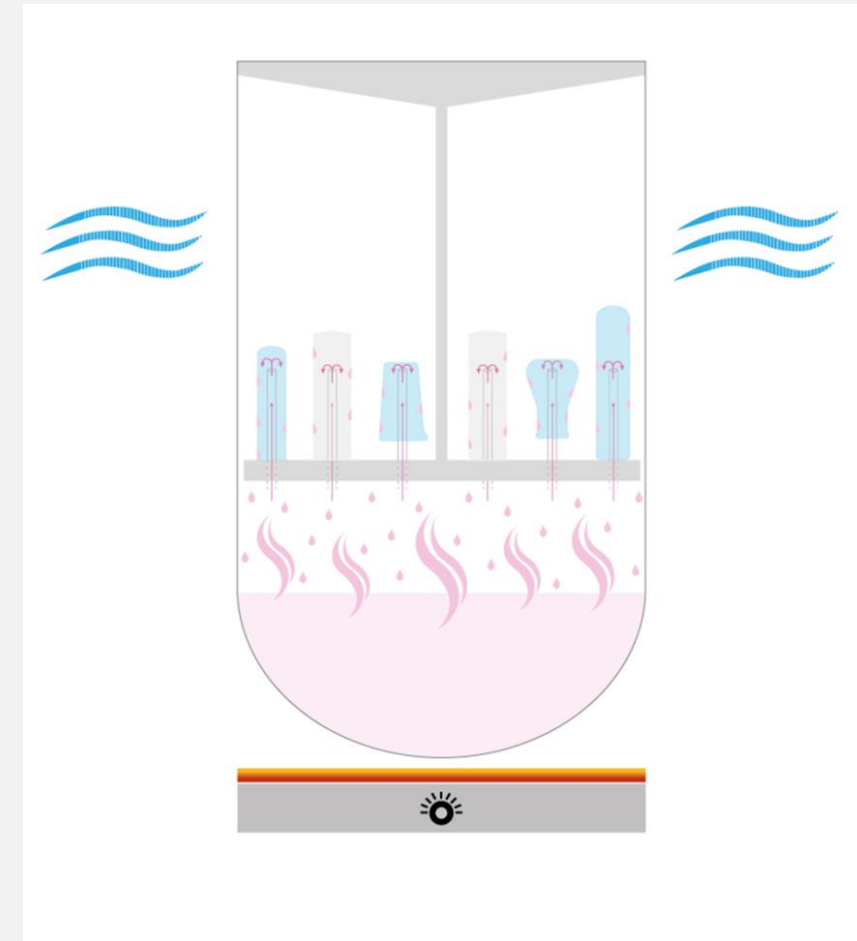
ACID CLEANING FOR TRACE METAL ANALYSIS

- 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% HNO₃ blanks prepared after cleaning are listed below. The acid leaching was performed at 180°C with mixture of HCl and HNO₃. The steam cleaning performed with HNO₃ 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

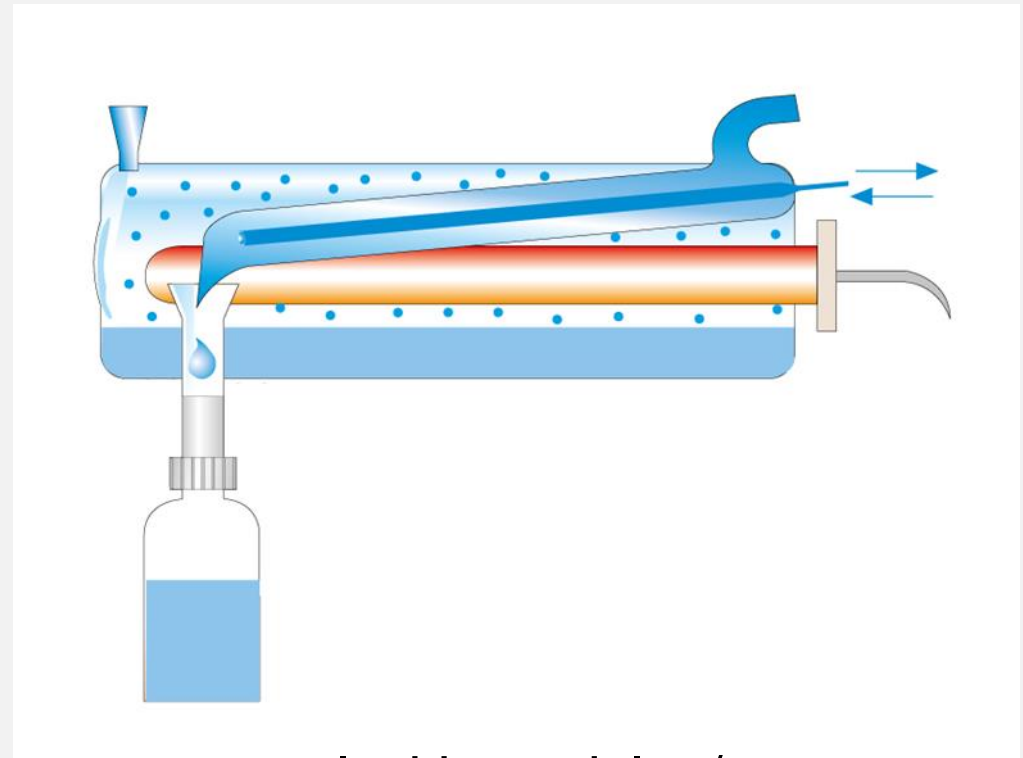
SUB-BOILING VS TRADITIONAL DISTILLATION

Comparison of metal contamination ($\mu\text{g/L}$) 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 $\mu\text{g/L}$	220 $\mu\text{g/L}$

SUB-BOILING DISTILLATION SYSTEM

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

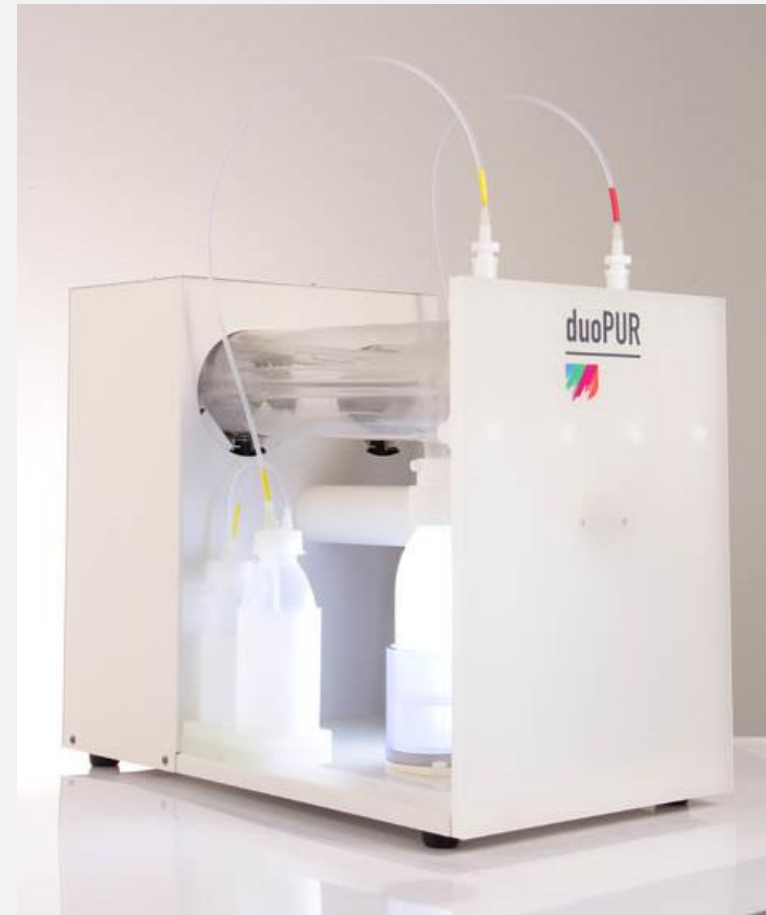


Vaporization without boiling prevents aerosol with particles/
contaminates

MILESTONE DUOPUR

QUARTZ SUB-BOILING DISTILLATION SYSTEM

- Double Quartz sub-boiling system
- Cost savings of ultra-pure acid
- High productivity
- Water cooling
- Suitable for HNO₃, HCl, H₂O



SUBCLEAN DISTILLATION SYSTEM

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



Questions?