



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

Think Blank

The analyst must 'think blank'. He must be aware as to the effect on the blank of every step of the analytical procedure.

He must ask himself "If I do this, what is the effect on the blank?"

(T. Murphy, The role of analytical blank in accurate trace analysis)

Clean Chemistry Line

“The analytical blank may be considered the “Achilles’ heel” of trace analysis. The analytical blank is simply the contamination by the element or compound being determined from all sources external to the sample. Modern methods of analysis have lowered the threshold of determining trace elements to the low parts per billion for many elements but the inability to control the analytical blank has seriously affected the accuracy of these methods.”

T. Murphy, The role of analytical blank in accurate trace analysis, in: P.D. LaFleur (Ed.), National Bureau of Standards Special Publication 422: Accuracy in Trace Analysis: Sampling, Sample Handling, and Analysis, National Bureau of Standards, Gaithersburg, Maryland, 1976, pp. 509-539.

The Milestone Clean Chemistry Line is an innovative and complete portfolio of systems and accessories for reducing and controlling the analytical blank in ultra-trace elemental analysis. There is a growing awareness that sample preparation should evolve to the same standards of the most modern analytical techniques, such as ICP-MS, and there are a number of factors that can critically impact the quality of the data:

- The purity of the reagents
- The cleanliness of the material in contact with the sample
- The sample preparation method

Each of these factors is related to the reduction and the control of the analytical blank. To address this issue, Milestone has developed a comprehensive line of products and accessories aimed to reduce and control the analytical blank, which perfectly complement its ETHOS UP and ETHOS EASY microwave digestion systems.

DUOPUR

Quartz sub-boiling distillation system

SUBCLEAN

PTFE sub-boiling distillation system

TRACECLEAN

Acid steam cleaning system for PTFE, quartz and glassware

ULTRATRACE INSERTS

TFM and quartz accessories for microwave sample preparation



traceCLEAN

duoPUR

subCLEAN



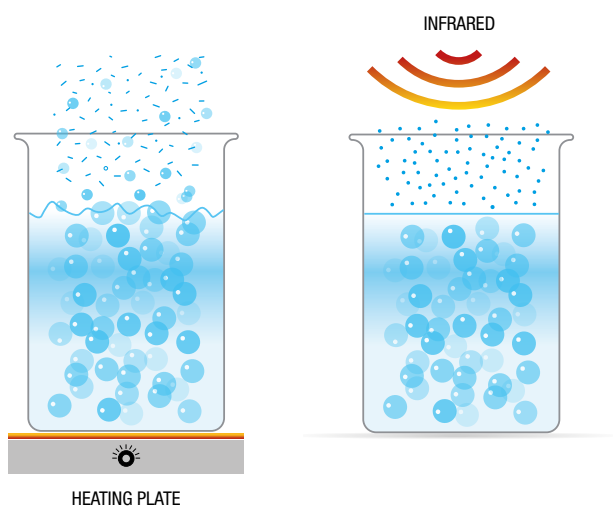
duoPUR

“In the preparation of high purity reagents, there is only one significant and practical choice for the method of purification: sub-boiling distillation.”

US EPA SW-846, Chapter 3, Update IVB.

SUB-BOILING TECHNOLOGY

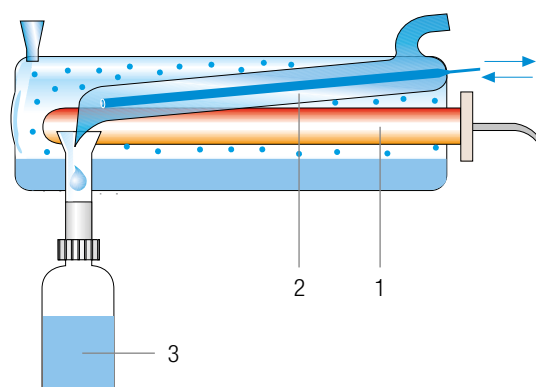
The chemical reagents used during the analysis are an important source of the analytical blank. Sub-boiling distillation has been demonstrated to be the best method of acid purification. It uses contactless infrared lamps to vaporize the surface liquid at a temperature typically 20 C below the boiling point. In contrast to conventional distillation, where strong boiling action generating aerosolized particles results in contamination of the original liquid with the distillate, a gentle surface evaporation during sub-boiling distillation prevents the formation of spray or droplets and yields to a very high pure acid.



Sub-boiling vs conventional technology

QUARTZ SUB-BOILING SYSTEM

The duoPUR consists of two quartz distillation units. Each unit contains two infrared heating elements (1), a water cooled condenser (2), a high-purity PFA collection bottle (3), and a fully automatic acid loading/discharge system. The vaporized liquid is collected on the inclined water-cooled condenser and drips into the collection bottle. The distillation process is microprocessor controlled, allowing the user to set the distillation time and power level by using a compact control terminal with easy-to-read, bright, full-color, touch-screen display. The distillation rate ranges from 50 to 400 mL per hour, depending on the power setting and the temperature of the cooling water.



duoPUR schematics

- 90% cost saving of ultra-pure acids by purifying low-cost reagent grade acids
- High productivity, up to 400 mL per hour (duoPUR) and up to 60 mL per hour (subCLEAN)
- On-demand acid purification
- Re-purification of contaminated acids

subCLEAN

TYPICAL SUBBOILING QUALITY

| Element | Single distillation | Double distillation |
|---------|---------------------|---------------------|
| Ag | <46 | <1,5 |
| Al | <557 | <147 |
| As | <3 | <0,9 |
| Ba | <25 | <3,5 |
| Ca | <900 | <157 |
| Cd | <8,1 | <1,8 |
| Co | <6 | <1 |
| Cr | <118 | <4,6 |
| Cu | <58 | <21 |
| Fe | <1.000 | <210 |
| Mg | <195 | <42 |
| Mn | <9,7 | <2,1 |
| Mo | <7,1 | <0,4 |
| Ni | <155 | <23 |
| Pb | <10 | <2,5 |
| Sb | <6,1 | <0,5 |
| Se | <3,9 | <1,2 |
| Sn | <22 | <9,1 |
| Ti | <59 | <8,1 |
| V | <51 | <11 |
| Zn | <261 | <49 |

PTFE SUB-BOILING SYSTEM

The Milestone subCLEAN is a compact and easy-to-use sub-boiling system, where all parts in contact with acids are made of high-purity fluoropolymers. The subCLEAN is therefore suitable for the purification of HF, as well as for HNO₃ and HCl. The acid is automatically loaded into the distillation container, where it is gently heated below its boiling temperature. All process is microprocessor controlled by using a compact control terminal with easy-to-read, bright, full-color, touch-screen display.

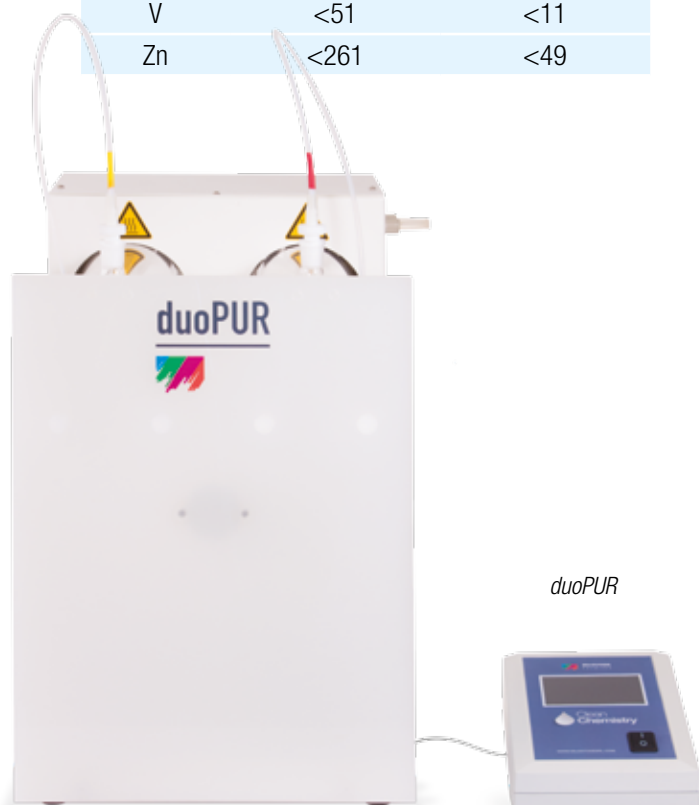
The subCLEAN does not require cooling water or a chiller, as acid vapors rapidly condense into a collection bottle by forced air cooling.

Nitric Acid, all concentrations in pg/g (ppt).

Concentrations expressed as the upper limit of the 99% confidence limit of the measured result (n=4).

On-Demand Production of High-Purity Acids in the Analytical Laboratory. Robert Richter, Dirk Link and H.M. (Skip) Kingston.

Spectroscopy, Volume 15, Number 1, Pages 38-40, January 2000.



traceCLEAN

ACID STEAM CLEANING SYSTEM

Cleaning various items used in ultra-trace analysis work is a critically important laboratory routine. To minimize contamination, traditional cleaning methods require soaking items in hot acids, often for several hours. To be effective large volumes of acid are consumed and need to be changed regularly. There is also a substantial risk of exposure to hot acids and acid vapors using traditional soaking techniques. To address these issues, Milestone has developed the traceCLEAN, a fully automated, self-contained, acid steam cleaning system for trace metal analysis accessories. Place the items to be cleaned in the traceCLEAN system, program the time and temperature required, then press "Start". Freshly distilled acid vapors

will continuously reflux within the sealed unit, thoroughly leaching any metal contaminants from the items. Various holders are available for vials, microwave digestion vessels, flasks, glassware, and ICP-MS accessories.

- Fast, convenient and fully automated
- Best cleaning quality of your TFM, glass and quartz parts
- Perfect accessory for your microwave digestion system
- No operator's exposure to acid vapors



| Element | Microwave cleaning | traceCLEAN cleaning |
|---------|--------------------|---------------------|
| Al | 287 ± 46 | 258 ± 24 |
| Cd | <72 | <72 |
| Co | <56 | <56 |
| Cr | <85 | <85 |
| Cu | 144 ± 39 | 117 ± 12 |
| Fe | <474 | <474 |
| Mg | 289 ± 22 | 232 ± 15 |
| Na | <121 | <121 |
| Ni | <55 | <55 |
| Pb | <57 | <57 |
| Tl | <261 | <261 |
| Zn | 995 ± 80 | <876 |

Microwave cleaning vs TraceCLEAN cleaning. Trace metal contamination (pg/g, ppt) in 5% HNO₃ blanks prepared after cleaning. The microwave cleaning was performed at 180°C with a mixture of HCl and HNO₃. The traceCLEAN cleaning performed with HNO₃. *New Developments in Automated Cleaning of PTFE, Glass, and Quartz Components used in Ultra-Trace Analysis. Robert Richter. Spectroscopy, June 2001.*

Ultratrace Inserts

“To keep blank levels as low as possible the analyst must exercise care in keeping the volume of reagents to a minimum.”

T. Murphy, The role of analytical blank in accurate trace analysis.

VESSEL-INSIDE-VESSEL

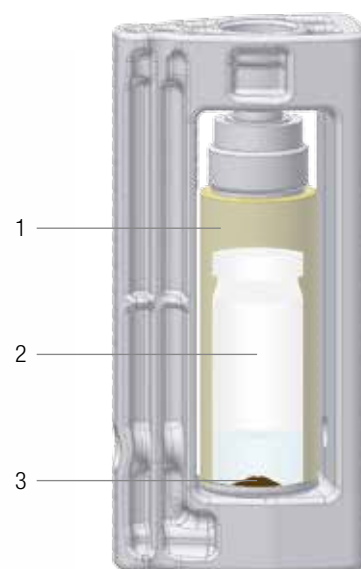
Microwave digestion techniques typically require a minimum volume of reagents of about 5 mL to achieve accurate temperature monitoring of the reaction conditions. However sometimes it is important to maintain a very low dilution factor, particularly when working at concentrations near the method detection limit. This has led Milestone to develop a “vessel-inside-vessel” technology. Vessel-inside-vessel technology uses smaller secondary vessel (1) inside the primary microwave vessel (2). The secondary vessel contains the sample and digestion reagents (3), while the primary vessels contains the solution in which temperature control takes place. This setup reduces the amount of

acid required for digestion, lowering the dilution factor and increasing the detection limit. A variety of inserts are available from Milestone, in different materials (Quartz or TFM) and with different sizes and shapes, to accomplish all application requirements.

- Less acid volume
- Higher sample amount
- Lower dilution factor
- Increased method detection limit
- Less surface contamination



ETHOS UP



Vessel-inside-vessel schematics

HELPING CHEMISTS

Milestone has been active since 1988 in the field of microwave sample preparation. With over 20000 instruments installed worldwide, we are the acknowledged industry leader in microwave technology.

Milestone vision is to help chemists by providing the most technologically advanced instrumentation for research and quality control.

Our products offer a wide range of applications, such as microwave acid digestion, solvent extraction, synthesis and ashing. Furthermore we create products for acid purification and direct mercury determination in solid, liquid and gas samples.

We offer our customers the highest level of application support, building up over the years a relationship based on trust and commitment.



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HELPING
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