



Acid digestion of samples using ultrapure quartz inserts with low acid volumes ICP and AAS metal analysis

I INTRODUCTION

Clinical laboratory testing plays a crucial role in the detection, diagnosis, and treatment of disease. Clinical laboratory personnel examine and analyze the chemical content of fluids. They test for drug levels in the blood that show how a patient is responding to treatment. They also prepare specimens for examination. With increasing automation and the use of computer technology, the work of technologists and technicians has become less hands-on and more analytical. The complexity of tests performed implicates the need of technological advanced instrumentation. Many laboratories often need to perform analysis of low-level trace metal samples. This is due to the small sample quantity they have available, since it often comes from a biopsy. Many laboratories specialized in analyzing biological materials are equipping

their lab with instrumentation suitable for trace analysis, such as ICP-OES or ICP-MS. In these conditions, sample preparation becomes a crucial operation before analysis. This is why Milestone has developed new micro-inserts for SK-15 rotor, capable to prepare samples employing small volume of acids.

Micro-inserts, based on so-called *Vessel-Inside-Vessel Technology*, are smaller secondary vials that can be placed inside the primary high-pressure microwave vessel of SK-15 rotor.

This configuration reduces the amount of acid required for digestion to near stoichiometric quantities, which reduces the dilution factor and decreases the final blank level.



I EXPERIMENTAL

The objective is that of completing a digestion of biological sample (with an internal standard), with a maximum of 2mL of nitric acid and to verify the recoveries. Moreover, we will test (with a different sample) that no cross contamination occurs

INSTRUMENT

The ETHOS UP matches the main requirements of many laboratories, thanks to its unique benefits, such as:

- High productivity
- Ease of use
- High safety
- High flexibility

The Milestone ETHOS UP is a very flexible and high-performing platform used to prepare samples for trace elements and routine analysis. The ETHOS UP is available with multiple configurations. The most suitable one for employing micro-inserts is the SK-15 high-pressure rotor.

The SK-15 rotor works with the Milestone “vent-and-reseal” technology for controlling and limiting the internal pressure of each vessel.



Figure 1 – Milestone's ETHOS UP

SK-15 HIGH PRESSURE ROTOR

The SK-15 perfectly matches the laboratories needs to determine trace elements, thanks to its capability to digest large sample amount and its high temperature/pressure capabilities.

The 15 positions high-pressure rotor is safely controlled via direct temperature sensor that constantly controls the digestion temperature during the run, ensuring perfect digestion of even the most difficult and reactive samples.



Figure 2 – SK-15 easyTEMP High Pressure Rotor

MICRO-INSERTS TECHNOLOGY (VESSEL-INSIDE- VESSEL TECHNOLOGY)

Below the inserts configuration used for the test.

Description	Picture	Working volume
TFM rack for 3 micro-inserts (PH00054) + Quartz vials (QB00039) or Teflon vials (MKE0006A) or Glass vial (70017)		4 mL ≤ Volume ≥ 1 mL



SAMPLE PREPARATION AND ICP-OES PARAMETERS

Test 1

The micro-sampling inserts have been used to digest Hair samples, with 50 ppb of an internal standard containing the following elements: As, Cd, Cr, Pb, Se, Ni, Mn.

Test 2

We performed a second test with a sample of Animal Blood (non-certified), to check that any cross contaminations occur between each micro-insert vials.

Here is the list of samples that we have performed in micro-inserts configurations:

	Sample Name	Sample weight	Reagents into the vials	Reagents into the SK15 vessel	Micro-sampling config.
Test 1	Human hair IAEA - 085	100 mg	HNO ₃ - 2 ml	H ₂ O dist. - 10 ml	TFM rack for 3 micro-inserts. Quartz vials
Test 2	Animal Blood sample	100 mg	HNO ₃ - 2 ml	H ₂ O dist. - 10 ml	TFM rack for 3 micro-inserts. Quartz vials.

	Sample	Method	Temperature profile
Test 1	Human hair IAEA-085		
Test 2	Animal blood		

INDUSTRY REPORT

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ICP-OES PARAMETERS, AGILENT ICP-OES (710 SERIES)

Power	1.30 kW
Plasma Flow	15.0 L/min
Auxiliary Flow	1.50 L/min
Nebulizer Flow	0.75 L/min
Replicate read time	10 s
Instrument stabilization delay	15 s
Sample Uptake Delay	30 s
Pump Rate	15 rpm
Rinse Time	10 s
Replicates	3

I ANALYTICAL RESULTS

RECOVERY TESTS WITH HUMAN HAIR (spike recovery results of 50 ppb As, Cd, Cr, Pb, Se, Ni and Mn)
(Results expressed in ppb)

Replicate	As	Cd	Cr	Pb	Se	Ni	Mn
1	49.2	48.1	54.9	47.9	51.9	50.7	51.0
2	39.0	47.5	53.0	49.4	49.4	49.9	51.4
3	49.2	48.3	53.2	48.3	51.9	50.4	52.3
Average	45.8 ± 5.9	48.0 ± 0.4	53.7 ± 1.0	48.5 ± 0.8	51.1 ± 1.4	50.3 ± 0.4	51.6 ± 0.7
% Recovery	91.6	95.9	107.4	97.1	102.1	100.7	103.1

CROSS CONTAMINATION TESTS WITH ANIMAL BLOOD

(Results expressed in ppb)

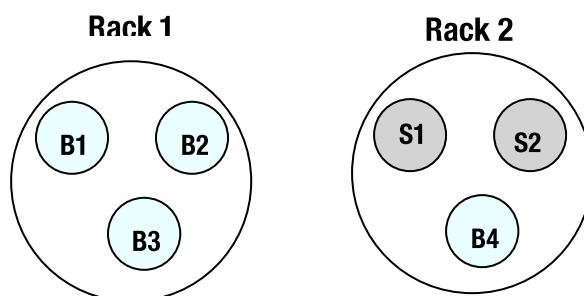


Figure 1. Scheme of sample positions in SK-15 vessels. B = Blank; S = Sample

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Sample	Cr ($\mu\text{g/L}$)	Cu ($\mu\text{g/L}$)	Mn ($\mu\text{g/L}$)	Ni ($\mu\text{g/L}$)	Pb ($\mu\text{g/L}$)	Zn ($\mu\text{g/L}$)
B1	< 2	< 2	< 2	< 2	< 2	< 10
B2	< 2	< 2	< 2	< 2	< 2	< 10
B3	< 2	< 2	< 2	< 2	< 2	< 10

S1	54,79	39,56	14,38	31,61	11,00	148,5
S2	56,08	39,52	14,30	32,59	10,80	148,2
B4	< 2	< 2	< 2	< 2	< 2	< 10
Average	55,4	39,5	14,3	32,1	10,9	148,4
Std. Dev.	0,9	0,0	0,1	0,7	0,1	0,2

CONCLUSION

Human Hair and Animal Blood samples were prepared for elemental analysis using quartz micro-inserts configuration (vessel-inside-vessel technology).

Micro-inserts demonstrate to provide a robust and reproducible way to prepare biological samples for trace metal analysis with low acid volumes and small quantity of sample. The data reported in this industry report shows that the “Vessel-Inside-Vessel Technology” is a great solution for clinical laboratories that need to examine and analyze biological material employing small volume of acids and small quantity of sample.

ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.

Interested in learning more?
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