



## SAMPLE PREPARATION OF PETROCHEMICAL SAMPLES FOR TRACE METAL ANALYSIS

### Ensuring high-quality and productivity in elemental analysis of petrochemical samples using the Milestone ETHOS UP

#### | INTRODUCTION

Analysis of heavy metal in QA/QC laboratories in the chemical and petrochemical industries has an important impact, as it directly influences product quality and performance. Inorganic materials in crude oil might have an adverse effect on petroleum refining and product quality, and represent a useful path in correlating production from different wells and sources. Concentrations of inorganic trace elements can be used as a “fingerprint” of crude oil samples, allowing the identification of their origin and their properties. For this reason, it is extremely important to be able to detect a large number of elements to better identify the oil product.

Having stated that, the “dream” of any analytical chemist involved in the petrochemical field is to detect the largest possible amount of elements.

The Milestone ETHOS UP microwave digestion system incorporates all the benefits of closed vessel microwave digestion, providing an easy, fast, effective and highly efficient solution for QA/QC laboratories in the petrochemical industry.



### EXPERIMENTAL

In this technical note, a recovery study on certified reference polymer materials was performed in order to prove the efficacy of the ETHOS UP in sample preparation for metal analysis.

### INSTRUMENT

The ETHOS UP meets the requirements of modern analytical labs. It offers several unique benefits including:

- Increased ease of use and productivity
- Enhanced control in all vessels
- Fast, accurate and traceable
- Superior safety and digestion quality

The ETHOS UP is a flexible and high performing platform used for elemental analysis and routine determination in many applications. Its construction of stainless steel coated with five PTFE layers accommodates both high-pressure and high-throughput rotors.



Figure 1 – Milestone's ETHOS UP

### easyTEMP

Milestone's easyTEMP contactless sensor directly controls the temperature of all samples and solutions, providing accurate temperature feedback to ensure complete digestion in all vessels and high safety. The superior temperature measurement of easyTEMP allows the processing of different samples of similar reactivity, thus reducing labor time and increasing overall throughput.

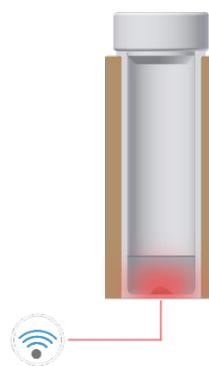


Figure 2 – easyTEMP contactless direct temperature sensor.

This technology combines the fast and accurate reading of an in-situ temperature sensor with the flexibility of an infrared sensor. The ETHOS UP software provides digestion history traceability and temperature measurement for every sample. The temperature diagram and profiles are displayed real-time, and can be subsequently saved on the ETHOS UP terminal.

### SK-15 HIGH PRESSURE ROTOR

The SK-15 rotor perfectly matches the needs of a modern analytical lab to determine trace elements, thanks to its ability to digest large sample amounts at high temperature (up to 300 °C) and pressure (up to 100 bar).



Figure 3 – SK-15 easyTEMP High Pressure Rotor

The 15-position rotor is controlled by a contactless direct temperature sensor that controls the internal temperature of all vessels throughout the digestion cycle. This ensures complete and reproducible digestions of even the most difficult and reactive samples. The SK-15 also features Milestone's patented "vent-and-reseal" technology for controlling the internal pressure of each vessel.

### USER INTERFACE

The ETHOS UP comes with a dedicated touch screen terminal and easyCONTROL software which incorporates our expertise and know-how in microwave sample preparation. The ETHOS UP user-interface provides full control of all digestion parameters, provides complete documentation and expedites the overall digestion procedure. The

terminal is equipped with multiple USB and ethernet ports for interfacing the instrument to external devices and the laboratory network. The ETHOS UP controller is user-friendly, icon-driven, multi-language and 21 CFR Part 11 compliant. To find the method which best suits your application, simply select from the vast library of pre-stored methods. Included with the ETHOS UP is a unique web-based application: Milestone Connect. This app allows you to become a part of the Milestone community and gain exclusive access to a robust library of information: lists of parts, technical notes, user manuals, video tutorials, continuously updated application notes and all relevant scientific articles.



Figure 4 – easyCONTROL built-in library

### ANALYTICAL PROCEDURE

ETHOS UP – SK-15 easyTEMP		
SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Mineral Oil (CONOSTAN S-12)	0.3 g	4 mL of HNO <sub>3</sub> 65%, 1 mL H <sub>2</sub> O <sub>2</sub> 30%

Table 1 - Sample amount and acid mixture used for the microwave digestion run



STEP	TIME	T2	POWER
1	00:20:00	210 °C	1800 W
2	00:15:00	210 °C	1800 W

Table 2 - Microwave program used for digestion of samples

- Final dilution: 50 mL with deionized water

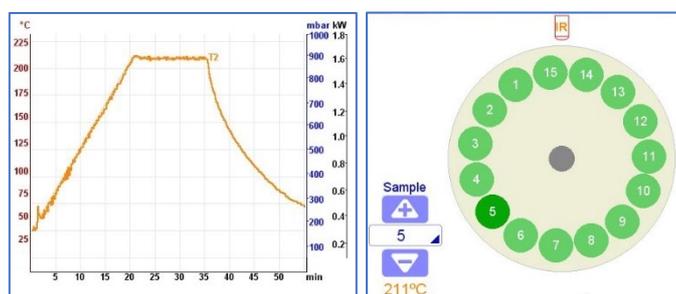


Figure 5 - Microwave Run Report and multiple temperature traceability

## QUANTIFICATION

ICP-OES Instrumental Parameters: RF power (W): 1300; Plasma flow (L/min): 15.0; Auxiliary Flow (L/min): 1.5; Nebulizer Flow (L/min): 0.75; Replicate read time (s): 10; Instrument stabilization delay (s): 15; Sample Uptake Delay (s): 30; Pump Rate (rpm): 15; Rinse Time (s): 10; Replicates: 3.

## RESULTS AND DISCUSSION

The performance of the Milestone ETHOS UP equipped with SK-15 easyTEMP rotor was evaluated through a recovery study on mineral oil (CONOSTAN S-12). The sample was digested with Milestone's ETHOS UP and subsequently analyzed via ICP-OES.

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%)
<b>Al</b>	250	101	1.2
<b>Cr</b>	250	100.4	1.1
<b>Cu</b>	250	99.6	1.3
<b>Fe</b>	250	96.2	1.1
<b>Mg</b>	250	103.4	1.0
<b>Na</b>	250	102.2	1.6
<b>Ni</b>	250	98.7	1.2
<b>Pb</b>	250	103.1	1.3

Table 3- Data of the recovery study on mineral oil (CONOSTAN S-12).

The analytical results are shown in table 3 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the ETHOS UP equipped with SK-15 easyTEMP technology

## CONCLUSION

The data shown in this technical note demonstrates full recovery of the elements reported in the certificates of the reference materials. Highly reactive samples, such as those from the petrochemical industry, can be completely digested, even in large sample amounts. The digestion process was accurately controlled by the easyTEMP sensor, ensuring superior digestion quality and reliable results. In addition to full analyte recovery, microwave digestion using the Milestone ETHOS UP provides the highest level of reproducibility, great ease of use and high productivity.