

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 industry has an important impact since directly impacts 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 of all their properties. For this reason, it is extremely important to be able to detect a large number of elements in

order to have a better identification of 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. Milestone’s Ethos UP, microwave digestion system, incorporates all of the benefits of closed vessel microwave digestion providing an easy and highly efficient sample preparation for the QA/QC laboratories in the petrochemical industries. Milestone’s Ethos UP the sample preparation is fast, easy, effective, and the highest quality.



| EXPERIMENTAL

In this industry report, a recovery study on certified reference polymer materials has been performed in order to prove the efficacy of ETHOS UP in the sample preparation for metal analysis.

INSTRUMENT

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

- Ultimate ease of use and control
- Unrivaled performance and productivity
- Expertise and know-how
- Superior safety and reliability

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



Figure 1 – Milestone's ETHOS UP

easyTEMP

The 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.

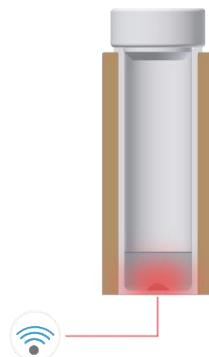


Figure 2 – EasyTEMP, contactless direct temperature sensor.

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 then can be 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 capability for digesting large sample amounts at high temperature (up to 300 °C) and pressure (up to 100 bar).



Figure 3 – SK15 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 all 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 the easyCONTROL software which incorporates our expertise and know-how in microwave sample preparation. The ETHOS UP user-interface fully control all the digestion parameters, provides complete documentation and expedite the overall

digestion procedure. The terminal is equipped with multiple USB and Ethernet ports for interfacing the instrument to external devices, and to the laboratory network. The ETHOS UP controller is user-friendly, icon-driven, Multilanguage and CFR-21 Part 11 compliant. To find the method which best suits your application simply select 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 ₃ 65%, 1 mL H ₂ O ₂ 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

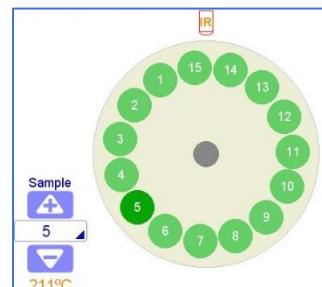
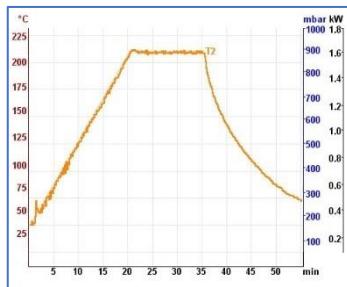


Figure 5 - Microwave Run Report and Multiple temperature traceability

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

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%)
Al	250	101	1.2
Cr	250	100.4	1.1
Cu	250	99.6	1.3
Fe	250	96.2	1.1
Mg	250	103.4	1.0
Na	250	102.2	1.6
Ni	250	98.7	1.2
Pb	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 element reported in the certificates of the reference material. Highly reactive samples such as petrochemical have been completely digested even in large sample amounts. The digestion process has been accurately controlled by the easyTEMP sensor, ensuring same digestion quality and reliable results. In addition to full analyte recovery, microwave digestion using Milestone ETHOS UP provides the highest level of reproducibility, great ease of use and high productivity.

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.

