



Thank you for your interest in Milestone's ETHOS X Advanced Microwave Extraction System. This Infopack will help you learn how the ETHOS X can enhance your lab's efficiency in analyzing organic pollutants from environmental matrices.

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SUPERIOR LAB EFFICIENCY IN THE DETERMINATION OF ORGANIC POLLUTANTS



ETHOS X Advanced Microwave Extraction System for Environmental Applications



HANDLING



HIGH THROUGHPUT



SUPERIOR RETURN ON INVESTMENT



CONSISTENCY & REPRODUCIBILITY



SAFETY AND RELIABILITY

ETHOS X DESIGNED BY YOU DEVELOPED FOR YOU

Determination of organic pollutants in environmental matrices is a common task for thousands of laboratories worldwide, as it leads to controlling and protecting our environment from high levels of contaminants. This analysis is often done to evaluate the effectiveness of a remediation process, to assess the contamination in waste, in waste landfills and for general environmental monitoring. Therefore, every day environmental laboratories deal with several challenges to ensure high quality data and fast turnaround time while maintaining their competitiveness. Extraction of pollutants from solid matrices is often performed with techniques that limit the productivity and have high running costs. Milestone listened to the needs of environmental laboratory professionals by developing the ETHOS X with the fastEX-24eT rotor, which allows for simultaneous extraction of 24 samples in 40 minutes with minimal solvent usage. By using large volume disposable glass vials, the fastEX-24eT rotor simplifies handling and allows to achieve lower detection limits.

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| MICROWAVE-ASSISTED EXTRACTION

Solvent extraction is the least evolved and one of the most error-prone steps in pollutant analysis. Many laboratories still use the Soxhlet method that was developed in 1879! Microwave-assisted extraction combined with closed vessels, heats the extraction solvent above its atmospheric boiling point. The elevated temperature of the solvent increases the solubility of the analytes of interest,

leading to a dramatic reduction of extraction time. Microwave extraction is also greener approach, it combines efficient heating with lower consumption of solvents to produce more accurate and precise results. Typical applications of microwave-assisted solvent extraction include chlorinated pesticides, semi-volatile organics, PAHs, PCBs, chlorinated herbicides, phenols, organophosphorus pesticides, dioxins and furans.



2 Video

HIGHER PRODUCTIVITY AT LOWER COST FOR GREATER ROI

When determining organic pollutants, most environmental labs aim to improve productivity and lower detection limits with easy-to-implement solution. The sample preparation technique plays a pivotal role in overcoming this challenge.

EASE OF USE WITH DISPOSABLE GLASS VIALS

The ETHOS X operation is simple: samples are loaded into large volume disposable glass vials (1) with appropriate solvent mixture (2), placed into the vessel (3) and sealed (4). The unique vessel design, in combination with the accurate temperature control, enables extraction of different matrices simultaneously, which enhances the lab's workflow. The accurate contactless temperature sensor ensures full control of the extraction cycle in all positions and displays real-time the temperature of all samples on the dedicated user interface. The 100-mL glass disposable vials accommodate large sample amounts of up to 30 g, enabling lower detection limits with minimal solvent volume. In addition, the memory effects often observed with other technologies are eliminated, expediting even trace analysis of challenging species such as dioxins.



GREATER RETURN ON INVESTMENT

The competitive nature of the environmental analysis market requires today's laboratories to have innovative solutions that provide faster turnaround times. With throughput capabilities of 24 samples in only 40 minutes, the ETHOS X with fastEX-24eT reduces overall analysis costs by increasing productivity. The combination of lower solvent volumes, less maintenance needs, and the use of inexpensive disposable glass vials decreases the cost per sample to enhance your lab's competitiveness and profitability.

	Soxhlet	Sonication	Pressurized Liquid Extraction	ETHOS X
Sample Size (g)	10-30	30	10-20	2-30
Solvent Volume per sample (mL)	300-500	300-400	15-30	15-30
Extraction time (min)	Days	Hours	270 min - 24 samples	40 min - 24 samples
Productivity (8 hours)	Low	Low	Moderate	Very high
Initial investment	Low	Low	High	Moderate
Cost per sample [*]	Moderate	Moderate	High	Low

*Including: solvent - handling - time - productivity - maintenance

| PRODUCTIVITY

Although new extraction technologies have been developed in recent years, they have fallen short on addressing the needs of environmental labs. The ETHOS X with fastEX-24eT overcomes productivity limitations of other conventional approaches by enabling the extraction of a large number of samples in a single workday. Other technologies process one sample at a time sequentially, requiring long extraction cycles. The ETHOS X simultaneously processes 24 samples in only 40 minutes and eliminates cleaning steps by using disposable glass vials. The graphic below compares the typical daily productivity using the Milestone ETHOS X, Sequential Pressurized Liquid Extraction and Soxhlet.





ACHIEVING LOWER DETECTION LIMITS WITH HIGHER SAMPLE AMOUNT



30 gram soil sample in disposable glass vials (actual size)

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RELIABLE SOLUTION FOR UP-TO-DATE ANALYSIS OF ORGANIC POLLUTANTS

COMPLIANCE

Several official methods describe the use of microwave closed-vessel technology to enhance the extraction efficiency of organic pollutants, such as US EPA 3546, ASTM and other national methods. The *Table 1* reports the typical extraction conditions.

The ETHOS X with fastEX-24eT further enhances the performance of microwave technology for the extraction of water-insoluble or slightly water-soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. In fact, the ETHOS X with fastEX-24eT can be used for solvent extraction of chlorinated pesticides, semi-volatile organics, PAHs, PCBs, chlorinated herbicides, phenols, organophosphorus pesticides, dioxins and furans. Table 1: Typical extraction conditions

Sample amount	2-20 gram
Solvents type	Hexane and Acetone (1:1)
Solvents volume	25 mL
Temperature	100-115°C
Time at temperature	10-20 minutes

Compound	Typical samples
PCBs	
PAHs	
Semivolatile organics	
Phenols	Solls, clays, sediments, sludges, and solid wastes
Chlorinated pesticides	
Organophosphorus pesticides	
Chlorinated herbicides	
Dioxins	

CONSISTENCY

Data quality and reliability are key features for environmental labs. The ETHOS X with the fastEX-24eT rotor provides fast, accurate and precise analysis. The vessels, in combination with the temperature sensor, allow to simultaneously process various matrices ensuring optimal extraction efficiency. In addition, the unique design of the fastEX-24eT rotor with its large volume disposable glass vials (100 mL) eliminates cross contamination and memory effect, providing reproducible results.



Soxhlet ETHOS X

Milestone ETHOS X recovery study and comparison vs Soxhlet for various organic compounds.



The ETHOS X offers a seamless integration of microwave hardware, user interface, contactless temperature control and rotor technology. This combination results in a powerful sample preparation tool for the determination of pollutants in environmental samples, overcoming the limitations of conventional extraction techniques.

SAFETY AND CONTROL

The ETHOS X cavity delivers high microwave power of up to 1900 watts, resulting in faster heating and shorter extraction times. Robustness and reliability are essential characteristics in an environmental lab to avoid any lapses in the daily operation. The ETHOS X has a rugged stainless-steel construction to ensure a longer lifetime and to eliminate lab's downtime. The system is equipped with a contactless sensor, the easyTEMP, that directly controls the temperature during the whole extraction process in all vessels, providing higher safety and reproducibility. The use of a non-invasive sensor simplify user operation while delivering accurate temperature control.

USER INTERFACE

The user interface runs an icon-driven, multi-language software with pre-loaded extraction methods. Using the touchscreen, the operator simply selects a method and presses "START" to begin a new extraction process. The extraction parameters are displayed real time on the terminal and can be stored or transferred via LIMS.





| FAST AND SIMULTANEOUS FILTRATION

The Milestone Simultaneous Filtration System (SFS) is a compact and complete workstation to filter up to 24 samples simultaneously in a few minutes. Samples are filtered into standard evaporation tubes, using the Milestone disposable funnels, providing easier handling and reducing fume hood space.



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Established in 1988, Milestone is headquartered in Italy with its R&D and manufacturing centre in Germany and Switzerland and offices in the United States, China, Japan and Korea. We

operate worldwide through a network of over 100 exclusive distributors, all providing our customers with premium application and service support. Milestone's mission is to help chemists by offering them the most advanced instrumentation for sample preparation and direct mercury analysis in the world. Our industry-leading technology, in combination with fast, responsive service and applications support, allows Milestone to support our goal of giving you the highest return on investment possible.

ADDITIONAL MILESTONE SOLUTIONS FOR ENVIRONMENTAL ANALYSIS



ultraWAVE The Game Changer in Microwave Digestion



ETHOS UP High Performance Microwave Digestion System



DMA-80 🕬 Direct Mercury Analyzer

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ETHOS X VIDEO



The ETHOS X offers higher sample throughput and easier handling in microwave environmental extraction, while ensuring analysis quality, cost-effectiveness and faster turnaround time in organic pollutant determination.

ightarrow see the ETHOS X Video



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Green, fast and easy microwave extraction of organic pollutants from soils, sediments and sludges

This industry report will discuss the use of ETHOS X Microwave Extraction System utilizing fastEX24 eT rotor with disposable glass vials to extract organic pollutants from certified soils during a recovery study following US EPA Method 3546. Samples were analyzed using GC-MS.

INTRODUCTION

The United States Environmental Protection Agency's (USEPA's) Test Methods for Evaluating Solid Waste (SW846) provides a comprehensive source of information on sampling, sample preparation, analysis, and reporting. US EPA 3546 is a Microwave-Assisted Solvent Extraction (MASE) procedure for extracting water insoluble or slightly water soluble organic compounds such as organochlorine pesticides, semivolatile organics, PAHs, PCBs, phenoxyacid herbicides, phenols, dioxins, and furans from soils, clays, sediments, sludges, and solid waste. This method was formally included in SW846 in 2008 (1) and most of these compounds have been identified by the US EPA as priority pollutants.

MASE results in a rapid sample preparation technique that enables extractions with reduced amounts of solvents while working at higher temperatures and pressures. The process consists in a partitioning of the

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compounds of interest from the sample matrix into the solvent within a closed vessel. This accelerates the extraction process, yielding results equivalent to the standard Soxhlet method, but in a fraction of the time and using significantly less solvent.

Milestone's new ETHOS X benchtop microwave extraction system offers the ability to extract up to 24 samples simultaneously. With the new fastEX 24 eT rotor, ETHOS X is fully compliant with US EPA 3546 (100-115 °C and 50-150 psi). In addition, disposable glass vials can accommodate sample up to 30 grams of sample if needed, thereby improving the limit of quantitation (LOQ) for analysis. This exceeds by far both the throughput and sample size capabilities of all the other automated techniques, such as pressurized fluid extraction. MASE also uses far less conventional than Soxhlet solvent extractions. This combination of performance and reduced solvent usage provides for the lowest cost per test of any technique available. Today, is possible to process up to 30g of sample thanks to the introduction by Milestone of the largest ever 145 mL weflon vessel in combination with the largest ever 100 mL disposable glass vials that fits inside the new fastEX 24 eT vessel. This vial improves productivity by providing inexpensive, disposable glass tubes that eliminate the need to clean vessels between batches.

The synergy between the weflon construction and the contact less temperature control in all positions ensure a perfect temperature uniformity and make fastEX24 eT a unique and innovative solution for the extraction of contaminants from soils, providing unmatched ease of use and low running costs. MASE has definitely become the preferred technique used by the most analytical laboratories for priority pollutants.

EXPERIMENTAL

INSTRUMENT

- Milestone ETHOS X microwave system equipped with fastEX-24 eT extraction rotor
- 100-mL disposable glass vials (PN GB00122)
- Gas chromatograph with Mass Spectrometer detector (GC-MS)
- Analytical balance
- Vials for collection of extracts
- Glass funnels for filtration
- Glass fiber filters



STANDARD AND REAGENTS

Pesticide grade or grade solvents and chemicals must be used in all tests. Samples should be extracted using a solvent system that gives optimum,

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reproducible recovery of the analytes of interest from the sample matrix, at the concentration of interest. The choice of extraction solvent will depend on the analytes of interest. Generally the most applied solvent mixtures are acetonehexane and acetone methylene chloride as recommended in the EPA analytical methods.

Analyte	EPA analytical method	Solvent mixture
Semivolatile Organics	8270	1:1 acetone - hexane or methylene chloride
PCBs	8082	1:1 acetone - hexane or methylene chloride
PAHs	8270, 8100	1:1 acetone - hexane or methylene chloride
Phenols	8151	1:1 acetone – hexane and phosphate buffer
Chlorinated Pesticides	8081	1:1 acetone - hexane or methylene chloride
Organophosphorus pesticides	8141	1:1 acetone -hexane and phosphate buffer
Chlorinated herbicides	8141	1:1 acetone - hexane or methylene chloride
Dioxins and Furans	-	1:1 acetone - hexane or methylene chloride

Tabella 1 Recommended solvents and EPA analytical methods by analyte of interest.

Sodium sulfate anhydrous, silica gel and glass wool or paper filter were used in the work up procedure. According to the analytical method surrogate and internal standard could be used.

SAMPLE INFORMATION

The sandy loam soil standard reference material LGC6115 was used for the determination of PAHs and PCBs (2).

The certified standard reference mineral oil contaminated sediment sample BAM-U015b was used for the determination of TPH (3).

ANALYTICAL PROCEDURE

Samples, wet or dried and ground, were weighed directly into the 100-mL extraction disposable glass vials. An aliquot of the surrogate solution were added to the samples just prior to solvent addition then, the glass vials were closed. According to the moisture content, the best suitable built-in method were choose. The extraction procedure so described follows the detailed method provided by U.S. EPA SW-846 Method 3546.

Sample amount (g)	Solvent mixture (mL)
Up to 10	25
10-20	35
20-30	50

Table 2 - Suggest solvent volumes according to the used sample amounts

Temperature

(°C)

110

110

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QUANTIFICATION

PCBs and PAHs analyses of the soil extract were performed according to the following method. Injection was through a splitless injector in a GC-MS equipped with VF-17-MS 30 m \times 0.25 mm i.d. capillary columns with 5 m guard column. The injector was maintained at 280 °C. The injection was 2 µL at 2mL/min flow rate. The oven was held at 80 °C for 2 min, from 80-300 °C at 20 °C/min then hold for 29 min at 300 °C. The detector worked with electron impact chemical ionization mass spectrometer. TPHs analyses of the soil extract were performed according to the UNI EN 16703 method. Injection was through on column injector in a GC-FID equipped with Select Mineral Oil 15 m \times 320 μ m i.d. (film 0.1 μ m) columns. The injector was maintained at 320 °C. The injection was 1 µL with 2 mL/min flow rate. The oven was held at 70 °C for 2 min, from, 70-320°C at °C/min. The FID detector were 30 programmed at flow rates of 400 mL/min air and 30 mL/min H₂, make up 30 mL/min He.

Table 3 - Microwave Program

Time

(min)

15

10

Step

1

2

*The power applied depends on the moisture content. Dedicated methods are pre-loaded in the ETHOS X software according to the moisture content.

Power (W)

up to 1600*

up to 1600*



After the extraction, samples were filtered on glass fiber filters and sodium sulfate anhydrous and the vials were rinsed with additional solvent aliquots. Extracts and rinsates were collected together.

RESULTS AND DISCUSSION

Results from extractions of sandy loam soil and sediment sample are shown in Table 4 through 9. The tables show the recovery and the RDSs (%) for PCBs, PAHs and TPH content of these matrices. Recovery for all compounds are in the range 70-120% of the certified standard reference material.

The results demonstrate the efficiency of the ETHOS X as sample preparation method for the determination of contaminants. ETHOS X provides extracts with the lowest solvent usage and significant time compared to all the other extraction technique.

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PCB Cogener	Certified value (mg/kg)	Ethos X (mg/kg)	Recovery (%)	RSD (%)
PCB 101	93	74	80	1.75
PCB 118	116	86	74	4.94
PCB 138	16	14	88	0.2
PCB 153	19	17	89	3.2
PCB 180	9.6	10	104	2.6

Table 4 - PCBs recovery from 1g sandy loam soil standard reference material (LGC6115)(n=4).

Analyte	Certified value	Ethos X	Recovery	RSD
	(mg/kg)	(mg/kg)	(%)	(%)
TPH	920 ± 100	841.8	91.5	2.4

Table 5 - Semivolatile organics, TPH recovery from 1g certified standard reference mineral oil contaminated sediment sample (BAM-U015b) (n=4).

Analyte	Certified value (mg/kg)	Ethos X (mg/kg)	Recovery (%)	RSD (%)
Phenanthrene	178 ± 6	200.72	113	4.52
Fluoranthene	312 ± 7	297.29	95	5.41
Benz[a]anthracene	36 ± 1	33.40	93	2.09
Benzo[a]pyrene	0.13 ± 0.02	0.16	123	11.5
Benzo[ghi]perylene	0.33 ± 0.06	0.25	76	0.3

Table 6 - PAHs recovery from 1g sandy loam soil standard reference material (LGC6115) (n=4).

Analyte	Soxhlet (mg/kg)	Ethos X (Recovery % of Soxhlet)	RSD (%)
TPH	11354 ± 122	111	5.2

Table 7 - Recovery of TPH from solid waste sample (1g) - Ethos X compared to Soxhlet extraction (n=4).

▶



Analyte	Soxhlet (mg/kg)	Ethos X (Recovery % of Soxhlet)	RSD (%)
PCB 28	4.09	88	5.2
PCB 52	3.70	88	4.8
PCB 95	2.46	79	6.2
PCB 99	1.40	73	3.1
PCB 101	3.18	72	2.6
PCB 105	1.22	90	6.4
PCB 114	0.07	85	7.3
PCB 118	2.68	79	2.0
PCB 123	0.07	114	5.6
PCB 126	0.16	118	4.2
PCB 128	0.55	82	3.4
PCB 138	1.79	80	8.3
PCB 146	0.25	116	6.2
PCB 151	0.17	105	7.4
PCB 153	1.46	90	6.1
PCB 156	0.29	110	7.9
PCB 157	0.10	100	6.5
PCB 169	0.45	104	3.1
PCB 170	0.41	78	2.2
PCB 180	0.36	81	7.7
PCB 183	0.20	80	2.3
PCB 187	0.35	100	5.3
PCB 189	0.21	114	4.7
PCB 77+149	2.37	71	4.9
PCB 81+110	7.03	76	6.1

Table 8 - Recovery of PCBs from solid waste sample (1g) – Ethos X compared to Soxhlet (n=4).

Analyte	Certified value (µg/kg)	Ethos X (µg/kg)	Recovery (%)	RSD (%)
2,3,7,8-TCDD	4500±0.6	4236	94	3.4
1,2,3,7,8-PeCDD	440±0.05	515	117	2.8
1,2,3,4,7,8-HxCDD	1220±0.21	1298	106	3.1
1,2,3,6,7,8-HxCDD	5400±0.9	4610	85	2.1

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1,2,3,7,8,9-HxCDD	3000±0.4	2522	84	1.9
2,3,7,8-TCDF	78±0.013	75	96	2.7
1,2,3,7,8-PeCDF	145±0.028	116	80	3.5
2,3,4,7,8-PeCDF	360±0.07	329	91	2.6
1,2,3,4,7,8-HxCDF	3400±0.5	3402	100	1.9
1,2,3,6,7,8-HxCDF	1090±0.15	1082	99	3.8
1,2,3,7,8,9-HxCDF	22±0.010	18	82	3.6
2,3,4,6,7,8-HxCDF	370±0.05	445	120	2.2

Table 9 - Recovery (n=4) of PCDD and PCDF from sandy soil standard reference material BCR-529 (2g)

CONCLUSION

The ETHOS X enables simultaneous solvent extraction of up to 24 samples (from weighing to filtration steps) in only 40 minutes. This in turns means that is able to extract over 200 samples in 8-hour workday. Contamination, memory effects, and cleaning are completely eliminated due to the use of disposable glass vials. The use of contactless temperature control ensures high reproducibility and full recovery of the target analytes for full compliance with EPA 3546. Moreover thanks to the unique design, ETHOS X ensures reliable extraction also on difficult samples such as solid waste. The ETHOS X with all its unique features fully addresses the need of environmental laboratories in terms of productivity, ease of use, running costs, and extraction quality.

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.

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CUSTOMER ACEA

acea (acronym for

Municipal Energy and Environment Company) is an Italian multiservice company active in the management and development of networks in the water, energy and environmental sector. Its activities are mainly concentrated in Italy but they are also present in some Latin American countries including Colombia, Honduras, Peru and the Dominican Republic.

CHALLENGE The main reason why they decided to switch to microwave assisted extraction technique was the need to reduce time per analysis and to process some of the most difficult and polluted matrices avoiding high maintenance costs and tedious cleaning steps.

SOLUTION

With the ETHOS X implementation, ACEA could process a full suite of matrices, even those with high plastic content that could not be processed with PLE. This allowed ACEA to expand its department's application range and increase its productivity.

BACKGROUND

The ACEA Group deals with waste management, handling over 1 million tons of waste every year. Since 2006, it has been managing waste-to-energy plants, composting plants and in 2019 the company also entered the plastic treatment sector. ACEA operates two incinerators that burn waste and pulp.

ACEA is also the top national operator in the water sector, serving 9 million customers in the Lazio, Tuscany, Umbria, Molise and Campania regions of Italy.

The laboratory gets involved in several aspects: water analysis; characterization of wastes from purification plants (exhausted sewage sludge, soils) and from incinerators (ashes, soils, slags). Moreover, it is active in the industrial emissions and environmental monitoring.

| CHALLENGE

The ACEA lab performs SVOCs analysis on solid waste and it is equipped for organic pollutant testing with the latest instrumentation.

The previous adopted technique for the extraction process was Pressurized Liquid Extraction technique (PLE).

They implemented the Milestone ETHOS X microwave extraction system with the fastEX-24 rotor for the determination of the following classes of compounds:

- Hydrocarbons
- PCBs
- PAHs
- Dioxins

LAB PROFILE MICROWAVE EXTRACTION | ENVIRO



ETHOS X IMPLEMENTATION

2 Video

At ACEA, they were impressed by the robustness of the ETHOS X with the fastEX-24 rotor that is able to process every kind of matrix, even the most difficult and polluted ones. Matrices with high plastic content could not be processed with the sequential technique, whereas the ETHOS X is capable of extracting them, allowing ACEA to expand its department capabilities and increase its productivity.

Moreover, the ETHOS X ease of use and high throughput resulted in significantly lower cost per analysis. It can process 24 samples in 40 minutes, including cooling time.

The main benefits achieved by ACEA are the following:

- Reduced extraction time. PLE is able to process 24 samples in 6 hours, while ETHOS X is able to process 24 samples in less than 1 hour.
- Wide application range. Thanks to the robustness of the ETHOS X hardware, ACEA could process all kind of solid matrices, even those with high plastic content or highly polluted ones, eliminating any clogging problem that typically occurs with the previous sequential technique.
- No cleaning of vials. A common and very critical issue for all sample preparation techniques is the subsequent memory effect/carry-over in the extraction cells. For instance, the stability of dioxins requires long and tedious cleaning procedures of the extraction vessels. The implementation of the fastEX-24 rotor eliminates the cleaning steps by using **disposable and inexpensive glass vials** as reaction vessels. This approach leads to a simplified handling and workflow.



"The extraction time per sample was reduced by 90% with the ETHOS X, thanks to the simplified handling and the elimination of the cleaning steps" - dr. Pier Francesco Gigliucci

- The initial investment is very competitive and also consumable costs are negligible, because disposable glass vials are very economical.
- ETHOS X can operate **without any gas** tank or flow, thus improving the overall lab workflow.

CONCLUSION

When adopting Milestone's microwave technology instead of conventional extraction techniques, ACEA gained advantages in terms of time-saving, application capabilities, maintenance costs and productivity on any kind of matrix.



ABOUT MILESTONE

With over 50 patents and more than 20,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 30 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.