MILESTONE TECHNOLOGIES

analis

NEOS

Rapid, Solvent-Free Microwave Extraction (SFME) of Essential Oil

THE MICROWAVE CLEVENGER™

Analis sa/nv Tel 09 243 77 10 chemical@analis.be

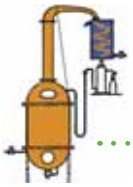
A brief history of essential oil extraction

Around 500 BC
Greeks developed the "Enfleurage"

Around 2500 BC
Egyptians invented the fumigation

8th Century
The alembic by Avicenna

16th Century
Florentine vase by Della Porta

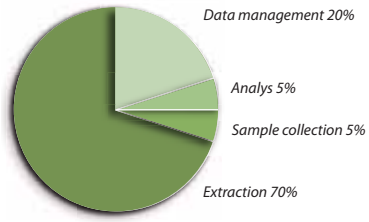


19th-20th Century
Modern alembic, Steam extraction. Cold pressing

21th Century
NEOS Solvent-Free Microwave Extraction (SFME)Technology

Analysis of essential oils

The analytical procedure for essential oils or aromas from plants or spices comprises two steps: extraction by steam distillation or hydro-distillation, and analysis by GC or GC-MS.
While the analysis step is complete after **15 to 30 minutes**, extraction takes at least **several hours**. Even today the standard method is the **Clevenger** Method* wich was the first published in **1928.** The extraction step of essential oils represents **70 percent** of the total processing time.



Time spent (%) for each step in essential oil analysis

The NEOS System

The NEOS system is based on the Solvent-Free Microwave Extraction (SFME) technology for rapid extraction of essential oils from aromatic herbs, spices and dry seeds. The SFME technology was developed at the Laboratory of Chemistry of Natural Substances and Food Sciences at the Université de la Réunion, France. The NEOS system is the result of the partnership between Milestone and this laboratory, which lead to the grant of the European patents 1439218, 1618798 and 1629725.
Microwave extraction makes use of physical and chemical phenomena that are fundamentally different compared with those applied in conventional isolation techniques.
These novel processes can produce essential oil in concentrate form, free from any residual solvents, contaminants, or artefacts.
The NEOS system confirms that microwave isolation offers net advantages in terms of yield and selectivity, with better isolation time, essential oil composition, and is environmental friendly.**
SFME is not a conventional microwave extraction process which utilizes polar and non-polar solvents nor a modified hydro-distillation method.

** Apparatus for Volatile Oil determination. J.F. Clevenger. American Perfumer & Essential Oil Review October 1928.*
*** F. Chemat, M.E. Lucchesi, Chapter 22: Microwave assisted extraction of essential oils in "Microwaves in Organic Synthesis" second edition. Edited by A. Loupy Wiley, Weinheim, 2006. ISBN: 978-3-527-31452-2.*

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NEOS Solvent-free microwave extraction of essential oils

The Milestone NEOS performs solvent-free microwave extraction at atmospheric pressure of essential oil in plant material. The method involves placing the sample in the microwave reactor, without any added solvent or water. The internal heating of the water within the sample distends its cells and leads to rupture of the glands and oleiferous receptacles. This process thus frees essential oil, which is evaporated by the in-situ water of the plant material. A cooling system outside the microwave oven continuously condenses the vapours which are collected on specific glassware. The excess of water is refluxed back to the extraction vessel in order to restore the in-situ water to the sample. Once the essential oils have been extracted they can be analyzed directly by GC-MS without any preliminary clean-up or solvent exchange steps.

Collection of the essential oil. The oil is separated from water simply by decantation.

High definition video system for visual control of process.

Icon-driven programs provide full control of the extraction method parameters.

Samples are placed in dedicated easy-to-handle glass modules. Loading/unloading of samples are immediate and easy.

Microwave labstation, microprocessor controlled with infrared automatic temperature system.

Special lab-grade water chiller for optimal extraction performance (option)

Mobile module for flexibility of use (option)

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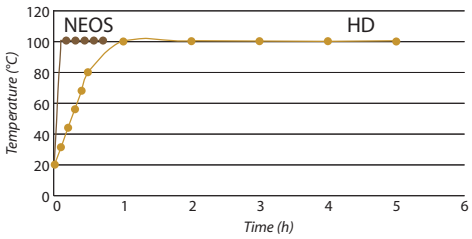
Typical applications

Extraction of essential oils from aromatic herbs*

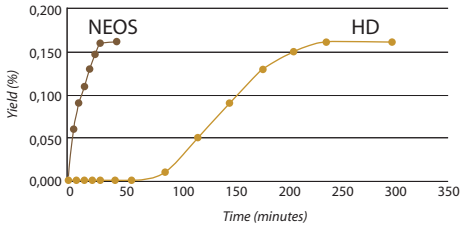
	NEOS (SFME)		Hydro Distillation	
	Time (minutes)	*Yield (%)*	*Time (minutes)*	*Yield (%)*
Basil *(Ocimum Basilicum L.)*	**30**	0,029	**270**	0,028
Garden mint *(Mentha Crispa L.)*	**30**	0,095	**250**	0,095
Thyme *(Thymus Vulgaris L.)*	**30**	0,160	**250**	0,161

Thyme
NEOS vs. Hydro Distillation (HD)

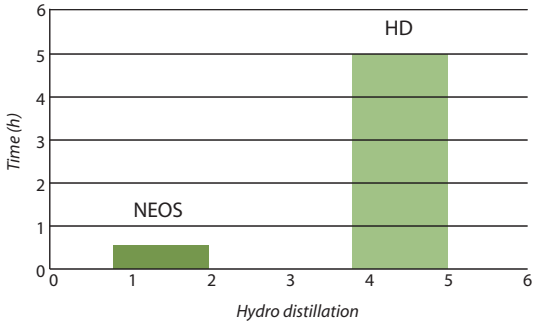
Temperature profile



Yield



Extraction time



** Solvent-free microwave extraction of essential oils from aromatic herbs: comparison with conventional hydro-distillation. Journal of Chromatography A, 1043 (2004) 323-327.*

Extraction of essential oils from orange peel**

	NEOS*	Hydro-distillation	Cold pressing
Extraction time (min.)	**10**	180	60
Yield (%)	**0,4**	0,4	0,15
Total oxygenated compounds (%)	**1,6**	0,9	1,1
Total non oxygenated compounds (%)	**97,4**	98,5	98,2

*** Solvent free microwave extraction of essential oils. Chemistry Today, Vol. 26 n. 2 / March-April 2008.*

Other applications of the NEOS system

Microwave-Accelerated Dean Stark (MADS)

A recently published article "*New and rapid analytical procedure for water determination: microwave accelerated Dean Stark*"* describes for the first time how heating by microwave has been used as an energy source for rapid water determination with a Dean Stark apparatus.
The validation of this new procedure has been checked by applying it to water determination in olive fruits.
When using this new procedure fully reproducible results could be found in a substantially shorter time (less than **10 minutes** compared to about **120 minutes** with conventional method).
The efficiency of this new MADS technique is considerably higher than the conventional Dean Stark procedure if we take into account the short distillation times required, cost and energy used and cleanliness of the process.
This MADS method could be appropriate for routine analysis of quality in the food, cosmetic and pharmaceutical industry.

**"New and rapid analytical procedure for water determination: microwave accelerated Dean Stark".*
Sébastien Veillet, Valérie Tomao, Franco Visinoni ,Farid Chemat. Analytica Chimica Acta 632(2009)203-207.

Microwave Steam Diffusion (MASD)

In a recent published article "*Microwave accelerated steam distillation of essential oil from lavender: rapid, clean and environmentally friendly approach*"*, the authors describe a new process design and operation for microwave accelerated steam distillation (MASD) of essential oils.
A packed bed of lavender flowers (Lavandula angustifolia Mill., Lamiaceae) sits above the steam source generated by microwave heating. Only steam passes through it without the boiling water mixing with vegetable raw material, as is the case in hydro-distillation. MASD has been compared with a conventional technique (steam distillation - SD), for the extraction of essential oil from lavender flower. Extraction of essential oils from lavender with MASD was better than SD in terms of energy saving, rapidity (**10 min vs 90 min**), product yield, cleanliness and product quality.

F. Chemat, M.E. Lucchesi, J. Smadja, L. Favretto, G. Colnaghi, F. Visinoni
Microwave accelerated steam distillation of essential oil from lavender: rapid, clean and environmentally friendly approach.
Analytica Chimica Acta, 2006, 555, 157-160.

Microwave-Integrated Soxhlet (MIS)

A recently appeared article "*Microwave-integrated Soxhlet extraction of total fat in oils*"* describes an improved process of Soxhlet extraction assisted by microwaves called Microwave-Integrated Soxhlet (MIS) for the extraction of oils and fats from different food matrices such as oleaginous seeds, meat and bakery products.
After **32 minutes** of MIS extraction it was possible to extract and concentrate the total amount of fat from foodstuff against the **several hours** required by conventional Soxhlet method which requires an additional step to concentrate fats and oils by evaporation of the solvent using a vacuum rotary evaporator.

**"Microwave-Integrated extraction of total fats and oils".*
Matthieu Virot, Valérie Tomao, Christian Ginies, Franco Visinoni, Farid Chemat.
Journal of Chromatography 1196-1197 (2008) 57-74.

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The simplicity of the NEOS


T/T Programs


Power/Time Programs


Favorites


User Programs


Notes

Through a touch screen, the icon-driven NEOS, can run any protocols both for time-at-temperature or for microwave power and time.

Documenting NEOS protocols

Through USB port programs can be easily uploaded and downloaded for documentation and QC purposes.

Scaling-up

The NEOS system includes a 2 liter dedicated glass module. When larger sample sizes are to be processed a 5 liter module is also available.


2 liter


5 liter

The advantages of NEOS

- No Solvents – Green Chemistry
- Shorter extraction time (minutes)
- Recovery of volatile compounds
- Less work-up. Absence of solvents.
- Higher purity of extracts

More about NEOS ?

Just log-in at **www.milestonesrl.com/essential** for scientific papers published in major journals using the technologies of the NEOS and NEOS-GR System.

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