

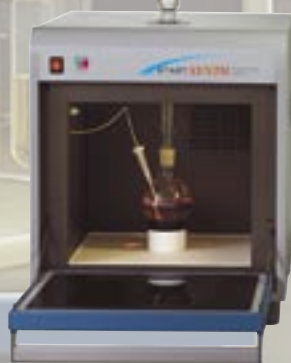
MILESTONE



HELPING  
CHEMISTS

# StartSYNTH

Microwave  
Synthesis  
Labstation





## The benefits of microwave-enhanced synthesis

Microwave enhanced chemistry represents a fundamental step forward in the capabilities of synthetic chemists. Since its introduction, it has allowed to run reactions faster than ever before and with higher yields, and to scale-up experiments reliably from milligrams to much larger quantities, without the need of reaction optimization. The total number of microwave synthesis scientific publications has grown from very few in 1995 to several thousand in 2007! Today, the use of dedicated microwave

instrumentations is becoming popular in many undergraduate laboratories, providing students with an in-depth view on the new advancements of the modern synthesis.



# Introducing the new Milestone StartSYNTH



Domestic or slightly modified commercial microwave units are not suitable for safe and reliable synthesis processes. Based on the experience of thousands of successful installations of the MicroSYNTH, Milestone has developed the StartSYNTH, a new microwave system which combines sophisticated design and high technology with ease of use and safe operation. The StartSYNTH is equipped with an industrial magnetron which delivers up to 1200 Watt. A microwave diffuser located above the microwave chamber evenly distributes the microwaves throughout the cavity, preventing localized hot and cold spots. The chassis of the StartSYNTH is made of corrosion-resistant stainless steel. The cavity and the door are plasma-coated with PTFE to protect the unit. The StartSYNTH door includes a shock resistant double-glass window for an easy

viewing of the reaction vessels. The door opens downward and it can be used by the chemist as working platform to facilitate the loading of the accessories into the microwave cavity.

The StartSYNTH is operated via a compact terminal, with bright, high resolution, touch screen display. The terminal runs the Milestone's unique EasyCONTROL software, to provide an easy, user-friendly control of the microwave process. Simply recall a stored procedure or create a new one, press 'START', and the system will automatically follow the drawn temperature (or pressure) profile, or just apply the set microwave power for the time desired.

The StartSYNTH is equipped with the most advanced sensors for accurate control and reproducibility of all reaction parameters (microwave power, time, temperature, pressure, and stirring speed).

## Direct temperature control

The direct automatic temperature control by fiber optic system enables direct continuous monitoring and control of a reference vessel in a carousel.

## Contact-less infrared temperature control

Focused, high-sensitivity infrared sensor for contact-less temperature monitor and control of all vessels in a carousel.

## Direct pressure control

The automatic pressure control system allows direct monitoring and control up to 55 bar. Furthermore, a contact-less sensor monitors the solvent vapor concentration in the microwave cavity, effectively controlling all vessels.



# *StartSYNTH* Microwave Synthesis Labstation



**START SYNTH** Microwave Labstation

## The *StartSYNTH* Advantages

- ◆ Advanced microwave technology
- ◆ Intuitive user interface
- ◆ Unique application versatility
- ◆ Inherent safety for flexible use by researchers and students
- ◆ Parallel scale-up capability



# Microwave Synthesis Platform for

Although suitable for most synthesis laboratories, thanks to its inherent versatility, the new Milestone StartSYNTH finds its specific working environment in academic laboratories for undergraduate teaching, as well as for research.

Since microwave technology has become so important in every field of chemical science, it is becoming a must for instructors to explore the benefits of this fascinating technology also in undergraduate classes. In this way, students can gain the needed knowledge and technical skill to

be ready for working in the most advanced R&D industrial laboratories, where microwave synthesis is applied at its state of the art. The broad range of reaction vessels available allows groups of up to sixteen students to run their experiments at the same time (with the Milestone Teaching Lab kit), and researchers to investigate their reactions in open vessels (NP kit) as well as in closed vessels (Research Lab kit). Further parallel scale-up is also possible, using a wide selection of specifically designed rotors.

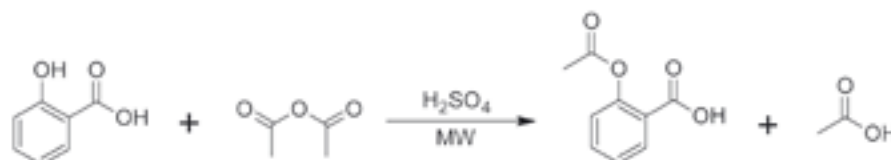
## Teaching Lab kit

The Teaching Lab kit with its 16-positions rotor allows several students to run up to 16 reactions simultaneously. All reactions are carried out at limited pressure (up to 1,5 bar-20 psig) to ensure the

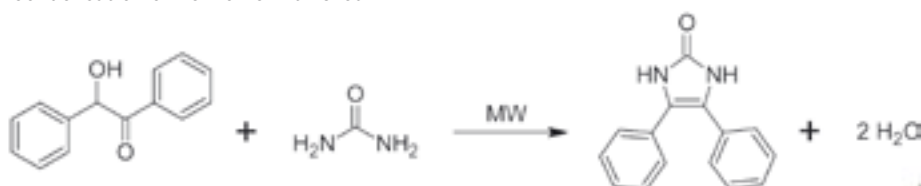
highest safety of operation. Anyway, temperatures higher than the normal boiling point of the used solvents can be attained to explore the advantages of microwave superheating effect.

Examples of microwave-assisted synthesis for undergraduate classes

### Synthesis of Aspirin



### Condensation of Benzoin with Urea



Reaction	Method	Reaction Time (min)	Yield (%)
Aspirin	Conv. Heating	120	85
	Microwave	1	92
Condensation of Benzoin	Conv. Heating	60	70
	Microwave	8	73



Teaching Lab Kit

## Research Lab kit

The Research Lab kit provides all the vessels for full optimization of a reaction for research or teaching purposes. The QV50, single quartz vessel, allows to perform reactions in closed vessel at temperature up to 250°C and pressure up to 40 bar. It is made of a high-purity quartz tube, hosted in a glass-reinforced thermosetting shield. The shield features a built-in pressure control through a preloaded spring with vent-and-reseal mechanism. The special TFM cover is equipped with two ports for direct temperature and pressure control. An optional cover permits to flush the reactor with inert gas, or to preload a gaseous reactant for gas-liquid reactions. Rapid cooling is ensured by a stream of compressed air. The SK-10 segment vessel, TFM reaction tube with PEEK shield, is recommended for all the

applications where improved chemical resistance is required, or higher pressures (up to 100 bar) are needed or expected. It is also useful for extraction purposes, allowing to work with low boiling points solvents at high temperature (up to 250°C), gaining the advantage of highly efficient extraction with easy product recovery.



Research Lab Kit



## NP Setup

The normal pressure NP reactors allow chemists to perform synthetic reactions under reflux. Actually, ordinary laboratory glassware can also be used in the StartSYNTH. In this way, any chemical reaction currently performed with a hot plate, heating mantle or oil bath, can be immediately transferred to the microwave. This will permit the chemist to investigate the effects of microwaves on reaction rates and mechanism.

- 50, 100 and 500 ml reflux reactors for optimization purpose and easy scale-up
- Stirring of thick reaction mixture with overhead mechanical stirrer

StartSYNTH with  
NP Setup

# Parallel Format Scale-up



Only Milestone's StartSYNTH platform, with its highly homogeneous field, gives chemists the ability to perform a reliable scale-up using a

parallel set-up. With Milestone's parallel rotors, up to 20 reactions can be processed at the same time.

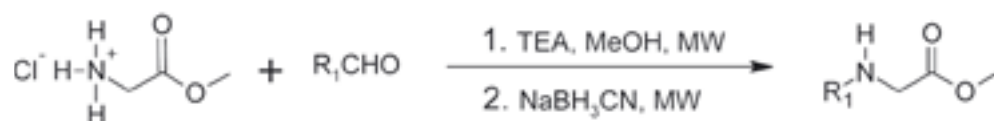
Accessory	Q20	HPR	LV
Vessel material	Quartz	High-purity TFM	High-purity TFM
Number of vessels	20	10	6
Vessel volume	45 ml	100 ml	270 ml
Working volume	3 to 30 ml	10 to 60 ml	50 to 160 ml
Maximum temperature	250°C	250°C	250°C
Maximum pressure	40 bar	100 bar	10 bar

## Q20 Rotor

The Q20 rotor uses 20 quartz vessels QV-50 and offers elevated throughput, very fast heating and

cooling, and easy handling. A fully automated capping system is also available from Milestone.

Synthesis of *N*-Alkyl-glycine methyl esters by microwave irradiation, (10 examples reported).



V. Santagada *et al.*, *Journal of Combinatorial Chemistry*, 7, **2005**, 618.

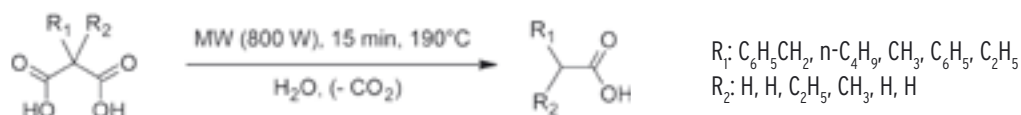


## HPR Rotor

When scaling up a reaction under conditions of extremely high temperature and pressure, there is no better choice than the HPR rotor. This rotor

accommodates 10 SK-10 reactors, each of which can be individually inserted in the MICROSYNTH microwave cavity.

Decarboxylation of malonic acid derivatives in water.



C. L. Zara, T. Jin, R. J. Giguere, *Synthetic Communications*, 30 (12), **2000**, 2099.



## LV Rotor

As an intermediate step in the process of scaling up a reaction, Milestone offers the large volume LV rotor. In its 270 ml reactors, chemists can process as many as 6 large scale reactions simultaneously, at temperatures up to 250°C and pressures up to

10 bar. The wide spacing between vessels facilitate adequate cooling, which, in conjunction with Milestone's superior pressure relief valves and full reaction control, ensures safe operation.





# StartSYNTH *Technical Specifications*

## StartSYNTH Labstation

### Microwave hardware

- Single magnetron system with rotating diffuser for homogeneous microwave distribution in the cavity.
- Magnetron protected from reflected microwave power.
- 1200 Watt installed power.
- Up to 1200 Watt output power, controlled via microprocessor.
- Large microwave cavity 37 x 34,5 x 33,5 (h) cm.
- Cavity illumination.
- Microwave cavity entirely made of 18/8 stainless steel housing with innovative multi-layer PTFE plasma coating applied at over 350°C.
- All hardware protected against acids/organic solvents with polymer coating both on inside and outside surfaces.
- Total of safety interlocks 4 micro-switches to prevent microwave emission with door open.
- Exhaust located in the side of the cavity, separate from electronics to prevent corrosion.

### Reaction sensors

- Direct temperature monitoring and control in a reference vessel.
- Contact-less temperature monitoring and control in all vessels.
- Direct pressure monitoring and control in a reference vessel.
- Contact-less pressure monitor and control up to the vessels highest working pressure.

### Control terminal

- 5" industrial grade monochrome touch screen controller.
- Resolution 240 x 128 dots for sharp process graphic.
- 1 PS2 port for mouse, 1 RS 484 port for microwave unit, and 1 RS 232 port for external devices.
- Methods and process reporting data saved on internal memory.
- Weight ~ 75 kg.
- External dimensions 57 x 51 x 61 (h) cm.
- Power 220V/50-60Hz, 2,4 kW.

*Specifications are subject to change without notice.*



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