

Monowave™ 300 – A valued Microwave Synthesis Reactor

Abstract



The Anton Paar Monowave™ 300 monomode microwave reactor opens up pathways to completely new reaction routes in small scale applications.

In addition to unique temperature and pressure limits it offers simultaneous IR and fiber optic temperature control for accurate reaction monitoring and safe process control. An extremely high field density ensures reproducible and outstanding heating rates in any scale and for any solvent, overriding the need for any kind of heating elements. Two different vials allow for a small-range scale up, and easy-to-handle, durable caps and silicone seals assure reduced costs for consumables.

The combination of highest safety standards, utmost accuracy, impressive performance and handling convenience make Monowave 300 a powerful tool for microwave-assisted organic synthesis.

Introduction

Microwave-assisted organic synthesis (MAOS) has become a tremendously growing field in chemistry during the last 20 years. Whereas at the beginning temperature and pressure measurement was not possible at all, nowadays there is a call for highly accurate monitoring in order to ensure comparability and reproducibility in chemical synthesis. Not only for the investigation of *specific* microwave effects, which is an extremely controversial topic in the scientific community, but also for method development and optimization in drug discovery utmost safety and accuracy are required in order to fulfil general GMP standards.

Besides the principle application of microwaves for chemical reactions, another growing interest currently challenges reactor manufacturers. Since the application of microwave irradiation has proven to be highly advantageous in chemical research, there is an urgent demand for scale-up production of pharmaceutical targets.

Anton Paar now covers impressive solutions to both matters in microwave synthesis: *Monowave 300* represents a valuable solution in terms of trouble-free processing at enhanced conditions for **small scale** applications, thus being a useful and important tool in drug discovery, method development and optimization, while *Synthos 3000* offers direct **scale-up** possibilities of well-established small scale synthesis protocols up to 1 mol of product within one run.

General Features of Monowave 300

Monowave 300 is equipped with a **powerful magnetron** delivering up to **850 W**. Due to its special design the cavity provides the highest available field density resulting in outstanding heating rates in any scale and for any solvent. Even low absorbing solvents can be heated efficiently far above their boiling point without the use of invasive or non-invasive heating elements.

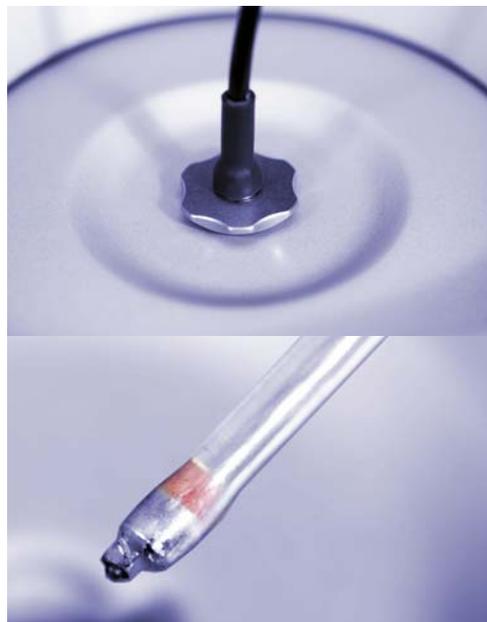
The temperature limit of Monowave 300 is **300 °C**. This can be either controlled via the built-in IR sensor, or additionally, the instrument offers the possibility of simultaneous temperature monitoring with a fiber optic probe, which is completely unique in the field of microwave assisted small scale applications. The **fiber optic probe** consists of a **ruby crystal** which is periodically excited, while a sensor measures the decay time of the ruby crystal's luminescence (being temperature dependent). This setup is completely **calibration-free** and applicable for any vial by applying a seal with a corresponding immersion tube. As neither the ruby crystal nor any other sensor material interferes with electromagnetic irradiation ultimate monitoring accuracy across the whole range as well as excellent reproducibility can be ensured.

Besides the broad temperature range Monowave 300 offers a pressure limit which is unique for single-mode microwave reactors. A non-invasive hydraulic system integrated in the swiveling cover provides immediate and accurate **pressure control up to 30 bar**. In case of overpressure during the reaction the pneumatic pressure release ensures safe and controlled pressure decrease while exhaust vapors are safely removed through the exhaust channel.

Due to continuous microwave power output over the whole power range, 850 W installed magnetron power guarantee homogeneous **heating with a rate of up to 9 °C/sec**. Nevertheless the sophisticated software controller prevents temperature overshoots which results in cleaner conversions and less formation of by-products.

The built-in **high performance stirrer** ensures homogeneous distribution even of biphasic reaction mixtures which further enhances reaction rates by providing intense contact between the reaction partners. This makes the equipment also highly applicable for solid phase organic synthesis (SPOS).

Fast and effective cooling of the reaction mixture can be provided by leading compressed air along the vial through the cavity. The air stream leads from the top of the vial into the exhaust pipe on the bottom of the cavity. This guarantees rapid cooling and ensures safe handling even though the instrument is not installed in a fume hood.



Ruby thermometer



Reaction vials and caps

Monowave 300 can operate **10 mL and 30 mL vials** allowing a 10-fold increase of the reaction volume (2 -> 20 mL). The special design tolerates the extended operation range of up to 300 °C and 30 bar.

Easy and leak-tight sealing works by the press of a thumb with **reusable snap caps**. The PEEK caps as well as the PTFE-coated silicone septa and the vials are reusable providing reduced running costs and increased economic efficiency. In case of remaining overpressure after the reaction, the caps allow depressurization during the opening process of the cavity lid.

User's Convenience

Monowave 300 has been designed and programmed in order to ensure **lowest maintenance efforts** minimizing the need of service technicians at **maximum convenience** to the user. The calibration-free optional fiber optic probe only needs to be connected to the sensor port of the instrument without installation (plug-and-play).

The integrated, colored LCD touch screen enables easy and **intuitive software navigation** for on-the-fly control of reaction parameters. Documentation is possible at the push of a button as well as *in situ* printing of pdf reports and exporting data files.

Several built-in connection ports facilitate the implementation of Monowave 300 in a laboratory's network system. Besides the Windows-based embedded PC with touch screen interface the instrument features two USB ports, an Ethernet LAN connection, and serial connectors for the ruby thermometer and a scheduled auto sampler upgrade.

Safety Aspects

The treatment of chemicals under extreme temperature/pressure conditions can lead to unexpected side reactions and may cause severe damage to the instrument and environment if no appropriate precautions are taken. Since user safety is one of Anton Paar's major concerns Monowave 300 guarantees **highest safety standards**. The instrument is designed with respect to safe handling for the operator. Thus various safety measures protect the users against unexpected spontaneous chemical reactions.

Before an experiment is processed the **swiveling cover** places a **non-invasive hydraulic system** on the top of the vial seal ensuring immediate and accurate pressure control during the reaction. An additional bayonet lock avoids unintended starting of the experiment as the magnetron will not supply microwave power until an electrical contact has been established.

The software actively controls pressure/temperature limits which are far below the operation limits of the utilized sensors and vials. Besides avoiding pressure peaks by sophisticated software-mediated power adjustment, **safe venting** in case of overpressure is provided via pneumatic pressure release. Nevertheless, installation in a fume hood is NOT required since a **spill bin** at the rear of the instrument collects solid and liquid material while an exhaust channel for solvent vapors leads exhaust fumes directly in a safe area.

Various tests have proven best performance and reliability, and corresponding certifications guarantee safe handling of Monowave 300 at any stage of the reaction process.



Swiveling cover

Advantages of Microwave-Assisted Organic Synthesis with Monowave 300

- Highest temperature and pressure limits opening up new reaction pathways for single-mode applications
- Utmost field density provide outstanding heating rates in any scale and for any solvent
- High-performance magnetic stirrer
- No need for invasive or non-invasive heating elements
- Rapid and efficient heating leading to drastically reduced reaction times and increase of yields
- Simultaneous internal and external temperature measurement for precise reaction control
- High purity of products due to prevention of temperature overshoots
- Simplified, software-aided reaction parameter control
- Scalability of protocols – from 10 mL to 30 mL vial as well as from Monowave 300 to Synthos 3000

Typical Applications of Microwave-Assisted Organic Synthesis

- of standard chemical reactions, *e.g.* solid phase synthesis, metal catalyzed coupling reactions, multicomponent reactions, high temperature protocols, etc.
- Method development and optimization for further applications in large scale production
- Basic research in academic and industrial laboratories (pharmaceutical, biomedical, agrochemical R&D) as extreme temperatures become easily accessible and open up new reaction routes in chemical synthesis.
- Scientific research regarding investigations of possibly existing specific microwave effects to contribute to this highly controversial topic
- Generation of small compound libraries with the aid of automated sample handling tools which allows for a high-throughput approach in drug design.
- Student education as MAOS nowadays plays a key role in research and development applications