



Microwave-assisted UV Digestion (MUV)

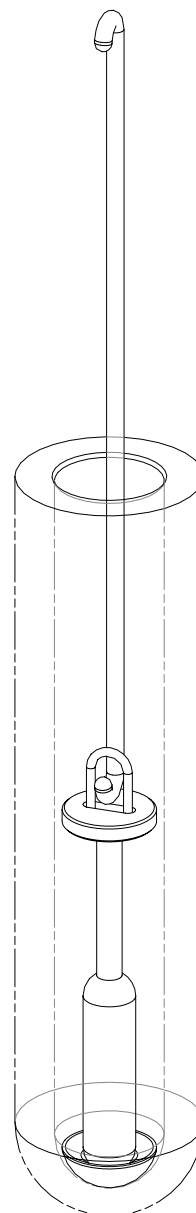
Anton Paar extends the versatility and flexibility of its Multiwave 3000 Microwave Sample Preparation System by introducing accessories for Microwave-assisted UV Digestion* (MUV).

Introduction

Many laboratories determine heavy metals, nitrogen, phosphorus or carbon in liquid samples containing organic compounds, e.g. drinking water, seawater, wastewater, sewage, body fluids or beverages. Trace element analysis is often carried out using atomic absorption spectrometry (AAS) or electrochemical methods (e.g. voltammetry). Those methods require homogeneous samples, which are free of organic matter. Organic compounds present in the sample can either directly (e.g. by interacting with electrode material) or indirectly (e.g. by forming complexes with metal ions) adversely affect the analytical procedure and thus, significantly decrease the detection power of trace analytical measurement techniques. Therefore it is very important to completely remove dissolved organic matter prior to analysis. In standard wet digestion procedures this is achieved by using highly corrosive reagents like concentrated mineral acids. But the photochemical process of UV oxidation represents a promising enhancement for the decomposition of organic and to some extent also inorganic matter (e.g. polyphosphates) prior to analysis.

UV irradiation of dissolved compounds leads to the formation of several highly reactive oxidative species (H_2O_2 , singlet oxygen, superoxide ions, organoperoxy radicals, hydroxyl radicals, halogen radicals) which accelerate the decomposition of interfering substances or enable the determination of total dissolved nitrogen, phosphorus and carbon by converting various N, P, C containing compounds to nitrate/nitrite, orthophosphate and CO_2 .

The destruction of organic matter by UV photo-oxidation is also well established for industrial decomposition of toxic substances, in treatment of urban or industrial waste waters, the production of ultrapure water for trace analysis, or the sterilisation of water and air.



*Florian, D.; Knapp, G.; Anal. Chem. 2001, 73 (7), 1515-1520

Anton Paar introduces Microwave-assisted UV Digestion (MUV)

Conventional open UV digestion systems consist of a UV lamp symmetrically surrounded by quartz test tubes and are operated at atmospheric pressure. Therefore they suffer from several drawbacks, such as limited reaction temperature, danger of loss of volatile analytes and solvent evaporation, extended degradation times up to several hours, as well as decreased decomposition rates.

The wide spectrum of applicability of Anton Paar's Multiwave 3000 Microwave Sample Preparation System in analytical chemistry can be further extended by accessories, which enable to perform high temperature/high pressure UV digestions of natural samples using a high-performance microwave oven.

The Multiwave 3000 with Anton Paar's 8-position rotor 8XQ80 and respective reaction vessels individually equipped with microwave-operated UV lamps opens the door to high temperature UV digestion methods extending the capabilities of the Multiwave 3000 to a wide range of new applications.

How does MUV (Microwave-assisted UV Digestion) work?

Digestion of organic samples is routinely carried out in closed, either microwave or conventionally heated pressurized devices, utilising concentrated mineral acids. By introducing photochemical UV oxidation to conventional acid digestion procedures only reduced concentrations of mineral acids or oxidants are necessary for the decomposition of organic matter prior to analysis of natural samples.

Microwave irradiation emitted by the powerful magnetrons of the Multiwave 3000 is used to operate specifically designed electrodeless Cadmium low-pressure discharge microwave lamps (MWL), which are immersed in sample solution inside each reaction vessel and emit short-wavelength UV irradiation (emission domain at 228 nm and 326 nm). Operation of a MWL does not require a separate power supply; its operation is fully initiated and maintained by the microwave field in the oven cavity. The microwave energy absorbed by the lamp antenna determines the UV irradiation intensity.

Reaction temperatures far above the boiling point of the sample solution can be easily achieved in a closed pressurized microwave digestion system without risking solvent evaporation or loss of volatile elements. As reaction temperature turned out to be the key factor for UV mineralization, enormous acceleration of UV induced degradation can be achieved at much higher initial concentration of organic matter.

High temperature reactions also enable dissolution of solid organic matrixes by diluted mineral acids and thus, render this UV digestion process applicable to slurries of solid organic samples as well. Reaction temperature and pressure are controlled by the microwave system's electronics and, in addition, are continuously documented which enables control of quality and reproducibility of digestion reactions.

Microwave-assisted UV Digestion (MUV) Procedure

High-pressure quartz reaction vessels (XQ80) offered by Anton Paar for the 8-position rotor 8XQ80 are suitable for Microwave-assisted UV digestion reactions.

Sample and reagents are transferred to a digestion vessel equipped with an UV lamp. For XQ80 quartz vessels, a lamp spacer and a lamp base ring should be used to avoid contact of the microwave UV lamp to and thus, possible damage of the quartz glass. After sealing, all vessels are positioned in rotor 8XQ80 and heated in the Multiwave 3000 according to an adequate microwave program.

Please refer to the Microwave-assisted UV Digestion (MUV) – Instruction Sheet for a more detailed description of the MUV procedure.



Advantages of MUV in a Closed System

Combination of pressurized microwave digestion and UV digestion offers convincing advantages over both independent methods:

- Maximum reaction temperature up to 280°C (compared to <95°C in conventional open UV digestion systems)
- No loss of volatile analytes or solvent evaporation in a closed system
- No need for additional cooling to eliminate heat generated by UV lamp
- Tremendous increase of mineralization efficiency
- Reduced decomposition time (30 min vs. up to several hours)
- Samples of much higher carbon content can be digested (7500mgL⁻¹ DOC vs. 300 mgL⁻¹ DOC)
- MUV applicable to heavily polluted samples of high turbidity and also to particular solid organic samples
- Direct contact of UV lamp with sample solution increases efficiency of UV irradiation
- No need for concentrated mineral acids
- Only reduced amount of oxidizing reagents necessary
- Reduced amount of reagents per sample reduces cost
- Reduced risk of contamination by reagents
- Reduced background levels
- Reaction pressure and temperature controlled by electronics of Multiwave 3000
- Continuous documentation of pressure and temperature possible
- Enables control of characteristics and reproducibility of UV digestion reactions

Typical Applications of Microwave-assisted UV Digestion (MUV)

Preparation of samples with high DOC for ultratrace analysis:

- Body fluids (blood, serum, liquor, urine)
- Sea water / brackwater analysis
- Waste water, sewage
- Beverages (wine)
- Animal / plant tissue



MUV Accessories and Spare Parts

Description	Cat. No.
<p>Microwave-assisted UV Digestion – Set (4x) Accessories needed to adapt 4 pieces of high pressure vessels for use in MUV (4x Microwave UV Lamp, 4x Lamp Base Ring, 4x Lamp Spacer, 1x Lifting Hook (Quartz), 1x Accessory Rack)</p>	<p>16846</p>
<p>Microwave UV Lamp Cadmium low-pressure discharge microwave lamp filled with Argon</p>	<p>16847</p>
<p>Lamp Base Ring Protective Teflon ring to prevent damage of XQ80 vessel by bulb of UV lamp</p>	<p>16446</p>
<p>Lamp Spacer Teflon disk to keep UV lamp centered and to prevent damage of XQ80 vessel by strap of UV lamp</p>	<p>16445</p>
<p>Lifting Hook (Quartz)</p>	<p>16428</p>
<p>Accessory Rack (4x) Rack that holds up to 4 UV lamps, 4 sample holders (for microwave-induced oxygen combustion) and a lifting hook</p>	<p>16447</p>