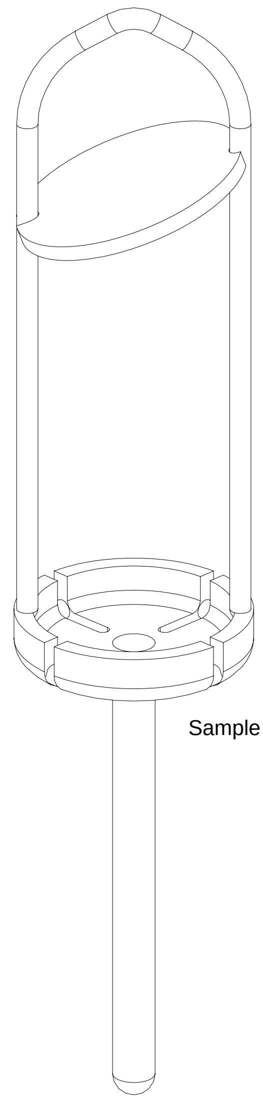
Microwave-induced Oxygen Combustion (MOC)

Anton Paar extends the versatility and flexibility of its Multiwave 3000 Microwave Sample Preparation System by introducing accessories for Microwave-induced Oxygen Combustion (MOC)*.

Introduction

Combustion of organic samples in oxygen-pressurized closed systems (in particular Oxygen Bombs) is widely used for preparation of samples for the subsequent determination of non-metal elements by ion chromatography. During combustion in an oxygen atmosphere, volatile non-metal elements are converted into volatile gaseous anions (e.g. halides) or oxy-anions (e.g. sulphate, phosphate). Those anions can be collected in a suitable absorption solution and are then accessible for analysis by ion chromatography, atomic spectroscopy or voltammetry. Several EPA and ASTM methods for the analysis of combustible solids and liquids based on sample preparation by oxygen combustion are readily available and describe the above-mentioned procedures in more detail.

The analysis of metals or metalloids by AAS or ICP-AES after oxygen combustion is also possible, but limited to suitably volatile elements (Hg, Cd), if the combustion residues cannot be completely dissolved in the absorption solution.



Sample holder quartz

Microwave-induced Oxygen Combustion from Anton Paar

Accessories that enable to perform closed system oxygen combustions of solid samples in a high-performance microwave oven can further extend the wide spectrum of applicability of Anton Paar's Multiwave 3000 Microwave Sample Preparation System in analytical chemistry.

Using the Multiwave 3000 equipped with Anton Paar's 8-position rotor 8XQ80 and slightly adapted high pressure XQ80 vessels loaded with specifically designed high quality quartz sample holders opens the door to well-established oxygen combustion methods for the analysis of volatile ions and thus, extends the capabilities of the Multiwave 3000 to a wide range of new applications.

* Patent pending

How does Microwave-induced Oxygen Combustion work?

High-energy microwave irradiation emitted by the powerful magnetrons of the Multiwave 3000 is used to ignite specifically impregnated filter paper inside the oxygen filled XQ80 vessel and thus initiates the combustion of the solid sample in the high-pressure oxygen atmosphere. Volatile compounds released during this combustion process are subsequently trapped in absorption solution suitable for analysis by e.g. ion chromatography, AAS or ICP-AES. Anions like halides or sulphur are absorbed in weak alkaline absorption solutions (e.g. 1mM $NaHCO_3$ + 8mM Na_2CO_3), while metal ions are absorbed in diluted acids which increases the detection limit by avoiding dilution steps and by reducing blank levels.

For complete dissolution of metal ions it is usually necessary to start an acid digestion program immediately after the combustion step. Samples containing silicate (e.g. plant material, coal) might show lower recovery rates, as metal ions could be partially trapped inside combustion residues, which may not be fully soluble in diluted acids.

Microwave-induced Oxygen Combustion (MOC) Procedure

Absorption solution is dispensed to all XQ80 quartz vessels. Combustible air-dried solid samples can be used in MOC reactions, preferably powdered and pressed to form a sample pellet. Bone-dry, loose samples might burn vigorously and rapidly, eventually resulting in incomplete combustion.

Homogeneous samples must not necessarily be powdered, they can also be used in other forms (e.g. coarse grained or cut into small pieces) if they ignite and burn completely. But care has to be taken, as larger particles might not burn completely, while smaller particles can be swept out of the sample holder by turbulent gases during the combustion. Therefore compression of samples into pellets is recommended.

Typically, 100-500 mg of sample can be used in MOC reactions (depending on the kind of sample used and the oxygen pressure applied, e.g. 300 mg of a PS polymer at 20 bar O_2).

Samples are individually put onto an impregnated filter paper that sits on top of the sample tray of a suitable quartz glass sample holder. Using the quartz lifting hook, the laden sample holders are then carefully placed inside the quartz vessels.

The vessels are subsequently closed and put into the 8XQ80 rotor. After closing the rotor lid, the quartz vessels are finally filled with 10-20 bar of oxygen with the help of a special oxygen loading device. This oxygen loading device can be directly connected to the laboratory oxygen supply and to the PEK seal holder of the quartz vessel via a specifically designed gas loading adaptor that replaces the standard syringe connector of the XQ80 quartz vessel.

After placing the rotor inside the oven cavity of the Multiwave 3000, the impregnated filter paper inside each pressure vessel can be ignited using microwave irradiation, and, in turn, the burning filter paper ignites the sample. If necessary, the combustion step can be followed by a wet digestion and a subsequent cooling step. Incomplete combustion might result from either using too much sample or from lack of O_2 (caused by leakage of the vessel or insufficient O_2 pressure).

To enable thorough absorption of volatile species after complete combustion of samples, the rotor including all pressurized vessels should be left inside the oven cavity for at least 1h, only interrupted every 10 minutes by a short mixing procedure outside the instrument. Sample solutions can then directly be used for analysis e.g. by ion chromatography without any additional dilution steps.

Please refer to the Microwave-induced Oxygen Combustion (MOC) – Instruction Sheet for a more detailed description of the MOC procedure.

Advantages of MOC in comparison to Oxygen Steel Bombs

Assembling / disassembling of XQ80 quartz vessels for oxygen combustion is much easier than handling of Oxygen Steel Bombs

No need to fumble with ignition wires

No handling of electrical connections necessary

Simultaneous combustion of up to 8 samples possible

Increased chemical resistance of XQ80 quartz vessels versus steel-made oxygen bombs

No metal parts inside the MOC combustion chamber, therefore reduced risk of contamination

Lower detection limit for analysis of certain metal ions (e.g. Hg) due to use of diluted acids for sample digestion (no additional dilution steps necessary)

XQ80 quartz vessels can be used for all combustion applications, no need for costly oxygen bombs made of special alloys or additional equipment (e.g. fragile quartz liners) when handling highly corrosive substances

Typical Applications of Microwave-induced Oxygen Combustion

Sample preparation for analysis of non-metal and metal elements in combustible solids

e.g.

Biological samples

Food, animal feed

Wood, chipboard

Wall board, paper board, paper

Resins, polymers (e.g. PS, PP, PE)

Organic compounds

Solid waste

Coal, coke

Product Information

MOC Accessories and spare parts

Description	Cat. No.
Accessory Sets	
Oxygen Combustion Set 4XQ80 Accessories necessary to adapt 4 pieces of XQ80 quartz vessels for use in MOC (4x Vessel Cap Gas*, 4x Protective Cap Gas, 4x Sample Holder (Quartz), Lifting Hook (Quartz), Accessory Rack, Punching Tool)	**17091**
Gas Loading Accessories Set Accessories necessary to enable loading of modified XQ80 vessels with gas (4-way Valve Station, Loading Nozzle Gas, Rotor Lid 8 Gas)	**17265**
Accessories	
Protective Cap Gas (Protective cap with additional slit for gas loading adaptor)	**17075**
Sample Holder Quartz	**16427**
Lifting Hook Quartz	**16428**
Accessory Rack (4 positions) Rack that holds up to 4 sample holders, 4 UV lamps (for microwave-assisted UV digestion) and a lifting hook	**16447**
Punching Tool (diameter 16 mm) Tool to punch out circular filter papers with 16 mm diameter, incl. protective pad	**17263**
4-way Valve Station Gas loading device with 4-way valve which enables flow control of gas between oxygen supply and reaction vessel (4-way Valve, Valve Holder, Screw Plug, Metal Tripod, Hose 0.5m + Hose 2.0m incl. Connectors, NPT-Adaptor 1/8” male)	**17224**
Loading Nozzle Gas Bayonet connector male that extents the 0.5m hose of the 4-way valve station and fits to the gas loading adaptor of the vessel cap - gas	**17074**
Rotor Lid 8 Gas Rotor lid 8 with additional openings that enable coupling of gas loading nozzle to gas loading adaptor and thus, loading of XQ80 vessels with gas, while rotor lid is securely closed	**17076**
Spare parts	
Seal XQ Gas (Seal XQ with additional O-ring)	**17413**
Loading Adaptor Gas **(**Replaces syringe connector to provide bayonet connector female for gas loading nozzle)	**17073**
4-way Valve	**17202**
Valve Holder	**17226**
NPT Adaptor 1/8" (male)	**17216**
Screw Plug	**17215**
Metal Tripod	**17201**
Hose 0.5m incl. Connectors	**17082**
Hose 2.0m incl. Connectors	**17085**

* **Vessel Cap Gas** consists of Seal Holder X (10335), Safety Disk X (10359), Safety Disk Holder X (10358), Venting Screw (5692), Seal XQ Gas (17413) and Loading Adaptor Gas (17073)

Anton Paar does neither supply oxygen nor appliances for control of oxygen loading pressure (10 – 20 bar).