

Procedures and Methods sheet: 009

Local Rules for the Use of the Ozonolysis Equipment

Prior to carrying out any ozonolysis experiments the researcher must receive suitable training from the operator of the equipment from the group who own it, Mr. Huanming Huang (GE.006).

Each individual experiment must have its own specific CRA assessment, which must be displayed by the reaction.

Hazards:

1. Oxygen can be harmful at high concentrations. Breathing high concentrations of O₂ (greater than 75%) causes symptoms of hyperoxia which include cramps, nausea, dizziness, hyperthermia, amblyopia, respiratory difficulties, bradycardia, fainting spells and convulsions capable of leading to death. Anyone affected should be taken to an area with fresh air.
2. High concentrations of oxygen increase the risk of explosion and fire. Compounds normally not flammable at low (room) temperatures will react much more readily with O₂ under pressure – oxygen is a high pressure oxidising gas which vigorously supports combustion (contact with other material may cause fire).
3. Ozone is harmful and toxic.
4. Ozonolysis produces highly reactive ozonides and peroxides which may be shock-sensitive explosives.

Reference to recorded hazards can be found in Bretherick's Handbook of Reactive Chemical Hazards available from the School Safety Advisor's office.

Experimental Procedure

OZONOLYSIS MUST BE CARRIED OUT IN A TESTED AND CERTIFIED FUME HOOD AND THE SASH MUST REMAIN DOWN WHILST OZONE IS BEING GENERATED. OZONE IS VERY TOXIC!

If the reaction must be stopped due to emergency (or at the end) switch OFF power to the ozone generator BUT DO NOT STOP THE OXYGEN FLOW! This is required to purge ozone from the system.

Set up. (See Figure 1)

1. Check ozone generator and cylinder are correctly connected, check for damage to tubing (especially blockages or kinks and cracking or leaks).
2. Make sure that the reaction vessel is a 3-necked one:
 - i) 1st neck for the O₂/O₃ inlet; this should be made of glass. Do not use a stainless steel needle as this will cause a blockage - it will not be wide enough.
 - ii) 2nd neck should be stoppered (DO NOT USE A SUBA SEAL).
 - iii) 3rd neck should be open to the atmosphere to allow gas to escape.
3. Bubble oxygen through the reaction solvent, make sure that you have a suitable pressure and flow rate (often lower than 1 litre per minute (LPM)) and that the gas can freely escape.
4. Plug in the ozone generator. Depress the main ON-OFF switch on the generator front panel, which will illuminate indicating that ozone gas is being produced.
5. After a couple of seconds, ozone will be produced. Ozonolysis is often fast, reaction times of fewer than 5 minutes are not uncommon. To sample your reaction (for TLC, LCMS, NMR) switch OFF the ozone generator wait 20 seconds (DO NOT TURN OFF OXYGEN FLOW, this is purging the O₃ from the system) then sample your reaction. Carry out step 4 again if the transformation is incomplete.

After Reaction is Complete

1. Switch OFF power to the ozone generator.
2. Allow oxygen to flow through your reaction, this removes any ozone from the ozone generator and your reaction flask. ENSURE OXYGEN HAS BEEN PASSED THROUGH THE OZONE GENERATOR FOR AT LEAST 5mins.

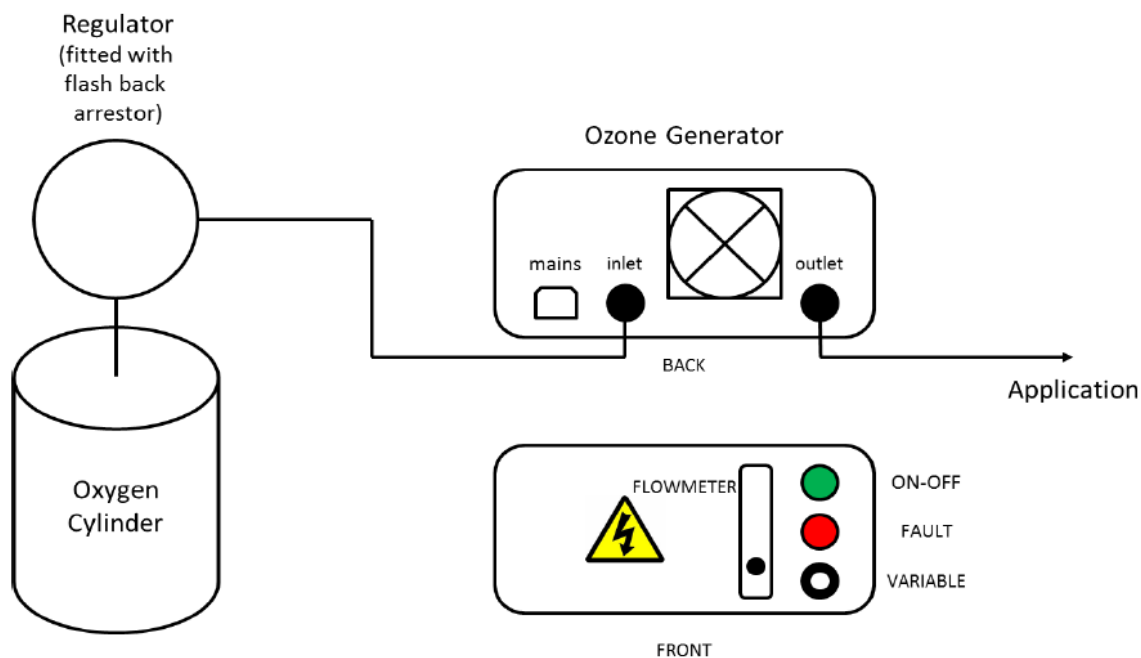
Then allow a flow of N₂ or Ar₂ through the reaction vessel to purge O₂ from the vessel.

3. Unplug the ozone generator and turn off the oxygen.

Notes:

1. The red fault indicator will illuminate briefly after pressing the ON-OFF switch; this is no cause for concern.
In the event of the red fault light illuminating during use: SWITCH OFF POWER TO THE OZONE GENERATOR and refer to the user manual.
2. If planning to produce a large quantity of ozone, consider scrubbing exhaust gas through a bubbler of magnesium thiosulfate solution. TAKE SPECIAL CARE TO AVOID BLOCKAGE, confinement of ozone is dangerous.

Figure 1:



Researcher signed

Researcher name

Date

Supervisor signed

Supervisor name

Revised Simon Holden

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