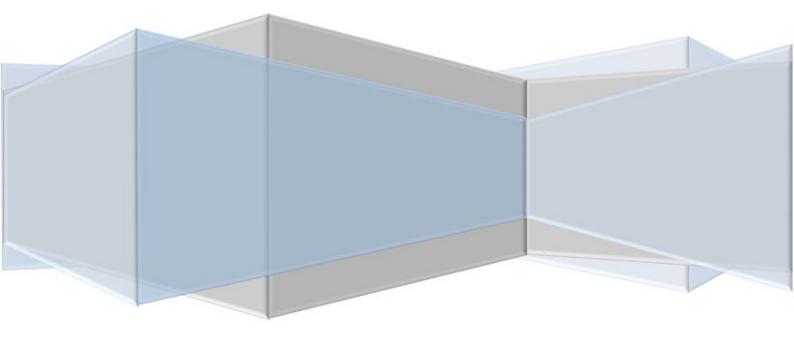
Deakin University School of Engineering

# Ozone Treatment System Operating Manual

Leanne Farago



# **Table of Contents**

Safety	2
Compatible materials	3
Injection of Ozone into water	4
Ozone formulas	4
Advantages of ozone	4
Disadvantages of Ozone	5
Half -life of ozone	5
System Schematic	5
Start up/Operation	6
Sampling	7
Shut down	7
Ozone production vs. flow rate	8
Test procedures	9
Equipment	9
Solutions	9
Risk Assessments	9
Waste Disposal	9
Determining the output of the ozone generator	10
Determining the ozone demand of the sample	10
Determining the Ozone Requirement	11
Ozone Residual 0.05 – 4mg/L	11
Risk Assessments	11
Waste Disposal	11
Appendix 1: MSDS	12
Ozone	12
2N sulphuric acid	12
0.1N Sodium Thiosulphate	12
2% Potassium lodide	12
Merck ozone reagent kit	12
Appendix 2: Risk Assessments	13
2N sulphuric acid	13
Merck ozone reagent kit	13

Ozone	13
Appendix 3: Equipment Specifications	14
Oxygen concentrator	14
Ozone generator	16
Ozone destruct unit	17
Filter column	
Air stone	19
Flow meter on ozone generator	20
Manual for Ozone Destruct Unit	21
Guide to understanding Ozone	21

# Safety

Ozone is a strong oxidiser that is generally not harmful to mammals at low concentrations, however it can be harmful if not handled properly. Ozone may increase sensitivity in individuals with asthma.

If you can smell it levels are above what you should be working with.

**Inhalation** of Ozone will cause dry mouth, coughing, irritation of the nose, throat and chest. It may cause laboured breathing, headaches and fatigue. If affected move to fresh air, loosen tight clothing and seek medical attention if required.

Ozone in **contact with eyes** could cause irritation and minor inflammation. Ensure you *Wear safety glasses.* If required flush eyes with large amounts of water for 15 minutes.

### Ensure you wash your hands before leaving the lab to avoid ingestion or skin contact.

Make sure you are wearing a lab coat as any spills of ozonated water will damage clothing.

Refer to Appendix 1 for MSDS and Appendix 2 for risk assessments.

# **Compatible materials**

Material	Rating		Rating	Description		
ABS plastic	В					
Acetal (Delrin®)	С	A	Excellent	Ozone has no effect on these materials. They will last		
Acrylic (Perspex®)	В			indefinitely.		
luminum	C (Wet Ozone)					
	B (Dry Ozone)	в	Good	Ozone has minor effect on these materials. Prolonged use with high concentrations of ozone will break down or corrode these materials beyond usefulness.		
rass	В					
ronze	В					
una-N (Nitrate)	D	_				
utyl	Α	C	Fair	Ozone will break down these materials within weeks of		
ast Iron	C			use. Prolonged use with any ozone concentration will		
hemraz	A			break down or corrode these materials beyond usefulness.		
lopper	В					
PVC	A - Does get brittle					
ross-Linked Polyethylene (PEX)		D	Poor	Ozone will break down these materials within days or even hours of use. These materials are not		
urachlor-51	A	100				
PDM	B (Dry Ozone)			recommendeed for any use with ozone.		
	C (Wet Ozono)	-				
PR	A					
hylene-Propylene	A	25				
ber Reinforced Plastics (FRD)	D					
exelene	B					
uorosilicone	A					
alvanized Steel	C			Electron microscope image of a		
ass	A		and the second	nitrile butadiene rubber diaphragm		
astelloy-C®	A		1000	seal after exposure to ozone. Note the		
DPE	A	-		cracks are formed at sharp corners in		
ypalon®	C	100		the seal.		
vtrel	C					
conel	A	100				
alrez	A	100				
el-F® (PCTFE)	A	1000	North Company			
DPE	B	5001	Najo Del WD 1 201 CSE 0.2	0 A Terr		
agnesium	D					
onel	C	100	CONC.	Contract of the local distance of the local		
atural Rubber	D	1150	Ser.			
eoprene	C		1. 1. C.	State		
vlon	D	100	1 Charles	Ozone cracking in natural rubber		
EEK	A	12	Alter Labor	tubing,		
olyacrylate	B	1		thong,		
ilyamide (PA)	C	1	/			
lycarbonate	A	1				
lyethelyne	B	/				
lypropylene	C	6				
lysulfide	B		and the store of the	and the second		
lyurethane, Millable	A			and the second sec		
ryurethane, miliable /C	A (Ozone in water)	200				
(Mar)	Does get brittle	-				
	B (Ozone in air) - Does get brittle	all's	SKIL TO	1112		
/DF (Kynar®)	A	200		A CHE STORE		
ntoprene	A	23.		Close-up of ozone cracking on nitrile		
icone	A		Contraction of the	butadiene rubber, taken with an		
inless Steel - 304/316	A			electron microscope.		
inless Steel - other grades	B			The Children of the second sec		
eel (Mild)	D					
TFE	A		and Manage and series	1 300 m		
tanium	A	KV 5	o 167x GSE 9.3	0.4 Terr 200 μm		
gon	B					
imac	A					
ton	A	S	ource: "Th	e definitive guide to understanding Ozone", Ozon		
	D					

# **Injection of Ozone into water**

As Ozone is a gas correct contact measures are critical to system design. Bubble diffusers are a popular and inexpensive method for injecting ozone into small volumes of water. The gas transfer occurs immediately at the interface between the bubble surface and surrounding water. A diffuser creates bubbles in the water similar to a fish tank air-stone. **The smaller the bubble the better the ozone transfer.** 

For larger volumes of water a venturi outlet is the best way to inject oxygen. Venturi injectors work by forcing water through a conical body, this creates a pressure differential between the inlet and outlet ports, creating a vacuum inside the injector body. This creates a suction port where the ozone gas is sucked into the water. This method is an extremely efficient way of injecting ozone and required little maintenance, however it is not appropriate for small volumes of water.

Source: "The definitive guide to understanding Ozone", Ozone solutions.

# **Ozone formulas**

### Ozone dosage in water =

Water flow rate (L/min) x ozone dosage(mg/L) = required ozone production (mg/min)

Ozone dosage is the quantity of ozone applied to the water. This will exceed the amount of ozone absorbed into solution. Ozone not absorbed into the water must be off-gassed through an ozone destruct unit, converting it back to harmless oxygen.

### Calculate the output of an ozone generator =

Flow rate (L/min) x ozone concentration (g/m<sup>3</sup>) x ( $1m^3/1000L$ ) = ozone production (g/min)

Source: *"The definitive guide to understanding Ozone"*, Ozone solutions.

# **Advantages of ozone**

- Ozone is the most powerful oxidant for disinfecting water and sanitising surfaces
- Ozone can kill pathogens in seconds versus minutes for other agents.
- Ozone decomposes in oxygen, leaving no harmful by-products.
- Ozone by itself does not alter pH.
- Ozone enhances the flocculation and coagulation of organic material there by improving filtration
- Ozone can be effective in partially oxidizing organics in the water to biodegradable compounds that can be removed by biological filtration.

Source: "The definitive guide to understanding Ozone", Ozone solutions.

# **Disadvantages of Ozone**

- Ozone is unstable and quickly converts back to oxygen meaning it can't be stored. Half-life is about 20-60 minutes depending on temperature, pH and water quality.
- Requires feed gas and ozone generating equipment to be available on site.

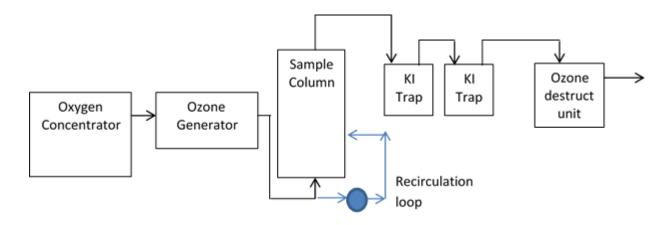
Source: *"The definitive guide to understanding Ozone"*, Ozone solutions.

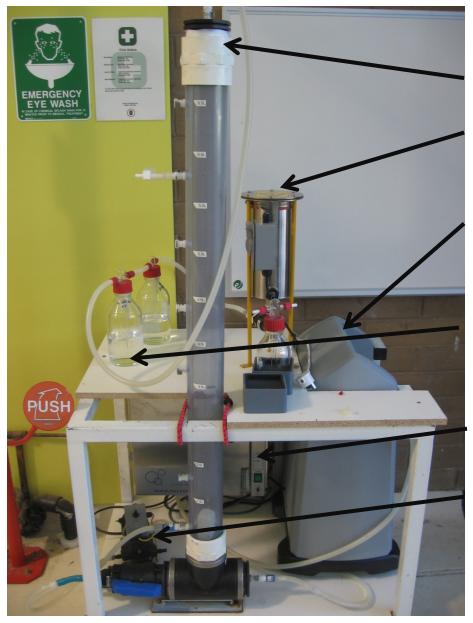
# Half -life of ozone

Temperature (°C)	Half- life (mins)
15	30
20	20
25	15
30	12
35	8

Source: "The definitive guide to understanding Ozone", Ozone solutions.

# **System Schematic**





Refer to Appendix 3 for specifications on all components.

Sample Column for treating sample .

**Ozone destructor** is used to convert any residual ozone back to oxygen and thus rendering it harmless.

**Oxygen concentrator** is used to concentrate oxygen in air to 90% pure with little moisture allowing for greater, more pure ozone production

**KI traps** are used to trap ozone escaping the system and allow for ozone consumption calculations to be completed.

**Ozone generator** uses the corona discharge method to produce ozone.

**Recirculation loop** has been installed to ensure mixing throughout column.

# Start up/Operation

Plug in trolley power and turn on.

Start Ozone destruct unit 30 mins prior to commencing experiments.

Turn switch to ON.

**CAUTION:** top of unit gets very hot.

# **Start Oxygen concentrator 5 minutes prior to commencing experiments** to allow air flow to stabilise and output to become pure.

Make sure column inlet hose is not connected to column.

Turn switch to ON position.

Adjust flow rate to desired level.

### Fill column with sample.

Check valve at base of column is closed.

Disconnect column outlet hose

Gently unscrew cap from top of column.

Pour sample into column and replace cap.

### Start recycling pump.

If required fill KI traps with 2% KI,

### Ensure they are connected correctly and in series. Note labels on caps

Ensure the outlet of the final trap is connected to the ozone destruct unit.

Turn on ozone generator, adjust oxygen generator flow rate until desired flow rate of ozone is reached.

**Connect** ozone inlet tube to base of column, treatment will begin immediately.

Run ozone generator until desired time/dose is reached.

# Sampling

To sample from column place sampling tube in desired location.

Open valve on sample tube and collect sample. Make sure that you have a sample collection vessel held under sample tube outlet.

# Shut down

Turn off ozone generator at end of exposure time, continue to have oxygen run through system.

Disconnect KI traps from column outlet.

Connect outlet tube to ozone destruct unit. This will help get rid of any excess ozone leaving the column.

Continue to run oxygen through column and solution for 5-10 minutes to ensure all excess ozone is forced from system and through destruct unit. If this is not done there will be a high level of ozone present when the cap is removed from the column.

After 5-10 minutes disconnect ozone inlet from base of column.

If further experiments are to be run leave oxygen concentrator & ozone destruct unit turned on, turn off if experiments are to stop.



Ensure column outlet is directly over drain or has a container underneath it.

Close tap and remove recirculation hose at joiner.

Open tap and drain column.

Open column securing point and tip column towards end of trolley until all liquid has been drained out.

Replace column in upright position and refasten column securing device.

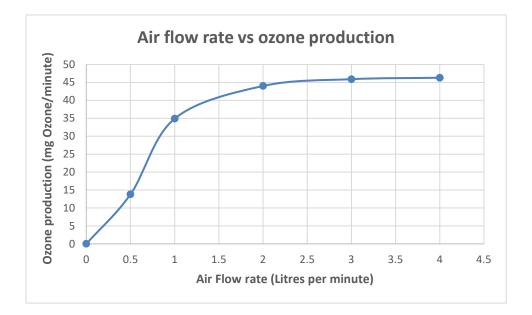
Ensure outlet tap has been closed.

# **Ozone production vs. flow rate**

At an oxygen flow rate of 2 litres per minute, 69.12 mg/min of ozone is produced using this equipment.

Flow rate of oxygen generator

Oxygen flow rate (Lpm)	Ozone produced (mg/min O₃)
0.5	13.8
1	34.9
2	44
3	45.9
4	46.3



# **Test procedures**

#### From standard methods 20<sup>th</sup> edition 1998.

This method involves the determination of ozone demand with the continuous addition of gaseous ozone to a batch reactor. The results obtained in this method depend on the mass transfer characteristics of the reactor. In addition, some compounds that consume ozone may volatilize during the test.

# Equipment

- 250ml gas washing bottles
- Buret
- Beaker 400ml
- Measuring cylinder 250ml
- Wash bottle
- Magnetic stirrer
- Ozone generator: capable of providing 5% ozone in gas phase at a flow rate of 1L/min
- Solutions as listed below
- Equipment for measuring ozone residual: Merck test kit 1.00607 and spectrophotometer

## **Solutions**

### Ozone free water

Ozonate reagent grade water for 1 hour then purge with nitrogen gas for 1 hour

### Sulfuric Acid 2N

Add 56ml concentrated sulphuric acid to 800ml of ozone free water, make up to the mark

### Potassium Iodide 2%

Dissolve 20g Potassium lodide in 1 litre of ozone free water

### Sodium Thiosulphate Titrant 0.005N

Add 50ml 0.1N sodium thiosulphate to 1 litre of ozone free water

### Starch indicator

Dissolve 5g starch in cold water until runny paste Add to 1litre boiling ozone free water Settle overnight – transfer supernatant to storage bottle and add 4g Zinc Chloride.

### **Risk Assessments**

Refer to Appendix 1 for MSDS and Appendix 2 for risk assessments

### **Waste Disposal**

All solutions can be flushed down the sink with plenty of water.

# Determining the output of the ozone generator

- Fill two gas washing bottles with at least 200ml of 2% KI solution (Trap A & B).
- Connect bottles in series to the outlet of the ozone generator.
- Turn on ozone generator and pass the gas through the bottles for 10 minutes.

For best results keep gas flow below 1L/min.

- Quantitatively transfer the contents of each bottle into separate beakers.
- Add 10ml of 2N Sulphuric acid to each beaker.
- Titrate with 0.005N Sodium Thiosulphate until the yellow Iodine colour almost disappears.
- Add 1-2ml Starch Indicator and continue titrating until the blue colour disappears.

### Ozone Dose:

### (ozone being produced by ozone generator at set flow rate)

Т

Where:

Ozone dose (mg/min) =  $(A + B) \times N \times 24$ 

A = ml titrant for trap A B = ml titrant for trap B N = normality of Sodium Thiosulphate T = ozonation time

# Determining the ozone demand of the sample

- Place at least 200ml of sample into gas washing bottle (label as sample).
- Connect a KI trap to the sample bottle outlet (Label as Trap C)
- Connect the bottle containing sample to the ozone generator outlet.
- Turn on ozone generator for a given time.
- Turn ozone generator off at the end of the contact time.
- Quantitatively transfer the contents of Trap C into a beaker.
- Add 10ml of 2N Sulphuric acid to beaker.
- Titrate with 0.005N Sodium Thiosulphate until the yellow lodine colour almost disappears.
- Add 1-2ml Starch Indicator and continue titrating until the blue colour disappears.

Ozone Demand:	Where:
Ozone demand (mg/min) = ozone dose – <u>C x N x 24</u>	C = ml titrant for trap C
Т	N = normality of Sodium Thiosulphate
	T = ozonation time

Report sample and blank ozone demand, ozone dose, ozonation time, sample temperature, pH & volume, vessel volume & type, gas flow rate, as well as analytical method used. This is because ozone transfer rate is highly dependent on experimental conditions

# **Determining the Ozone Requirement**

Ozone requirement is the ozone dose required (mg/min) to obtain the target residual at the end of the contact time. When reporting include target residual as well as experimental characteristics as listed in ozone demand.

Using the sample treated in ozone demand test, remove a volume of sample and test for ozone residual using Merck test method 1.00607

# Ozone Residual 0.05 – 4mg/L

Using Merck reagent test kit 1.00607. Analyse immediately as ozone residuals change dramatically with time.

## Equipment

- 10ml pipette and filler
- Spectrophotometer
- Empty round cells
- 1cm square spectrophotometer cells
- Merck reagent test kit 1.00607

### Method

Turbid samples must be filtered. Turbid samples will yield false high results.

Check pH of sample. pH must be between 4.4-5.5. Adjust with sodium hydroxide or sulphuric acid if required.

- Using pipette add 10ml sample to empty round cell.
- Add 2 drops reagent O<sub>3</sub>-1.
- Cap and mix.
- Add 1 level micro-spoon of reagent O<sub>3</sub>-2.
- Shake vigorously until reagent is dissolved.
- Wait 1 minute.
- Insert auto-selector into round hole on spectrophotometer.
- Transfer reacted sample to 1cm square cell.
- Insert into cell holder and wait for result.
  - Results are stable within 30 minutes of reaction.

### **Risk Assessments**

This product can damage fertility or the unborn child, cause serious eye damage and irritation. **Ensure a fume** cupboard and nitrile gloves, safety glasses and lab coats are used.

Refer to Appendix 1 for MSDS and Appendix 2 for full risk assessment.

### Waste Disposal

All solutions can be flushed down the sink with plenty of water.

# **Appendix 1: MSDS**

Ozone

2N sulphuric acid

**0.1N Sodium Thiosulphate** 

2% Potassium Iodide

Merck ozone reagent kit

# **Appendix 2: Risk Assessments**

2N sulphuric acid

Merck ozone reagent kit

Ozone

# Appendix 3: Equipment Specifications

# **Oxygen concentrator**

6lpm 90% oxygen



- Advanced engineering
- Low operating cost
- Transportable with rollerbase design
- Easy to operate and maintain
- Time-proven reliability
- Thousands in use around the world

VPSA Plants

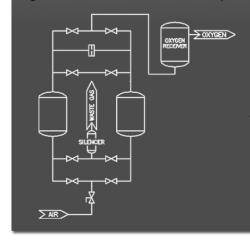


# How Do AirSep PSA Systems Work?

Air contains 21% oxygen, 78% nitrogen, 0.9% argon, and 0.1% other gases. AirSep Oxygen Generating Systems separate oxygen from compressed air through a unique Pressure Swing Adsorption process. The PSA process uses molecular sieve (a synthetic zeolite), which attracts (adsorbs) nitrogen from air at high pressure and releases (desorbs) it at a low pressure.

AirSep Oxygen Generators use two vessels filled with molecular sieve as adsorbers. As compressed feed air flows through one of the vessels, the molecular sieve adsorbs nitrogen. The remaining oxygen passes through the vessel and exits as the product gas. Before the adsorber becomes saturated with nitrogen, the feed air is diverted to the second vessel. At that point, the sieve in the first vessel regenerates by desorbing the nitrogen through depressurization and purging it with oxygen from the second vessel.

This process is then repeated in the second vessel to complete a cycle that allows the oxygen generator to deliver a constant flow of product oxygen at 90% minimum purity. Under normal operating conditions, the molecular sieve is completely regenerative and will last indefinitely.



MK228-1

01 07/06

# **ONYX**<sup>®</sup>PSA Oxygen Generator

## SPECIFICATIONS

### **Operating Characteristics**

Product Flow: 6 LPM / 0.31 Nm<sup>3</sup>/hr (Onyx): 8 LPM / 0.44 Nm<sup>3</sup>/hr (Onyx Plus) Standard Product Pressure: 9 psig / 62 kPa (Onyx): 20 psig / 138 kPa (Onyx Plus) Product Purity:  $93\% \pm 3\%$ Product Dew Point: -100°F (-73°C) Sound Level: 49 dB

### **Ambient Operating Conditions**

Locate the oxygen generator in a well-ventilated area that is protected from weather elements and remains between 40°F (4°C) and 120°F (44°C)

#### Power Requirements<sup>1</sup>

 $100V{\sim}\pm10\%,$  50/60Hz, Single Phase, 5.5A  $120V{\sim}\pm10\%,$  60Hz, Single Phase, 5.0A  $220V{\sim}\pm10\%,$  50 or 60Hz, Single Phase, 2.5A Typical Power Consumption: 350 W

#### **Physical Connections**

Product Gas Outlet: 1/4" NPT/B size oxygen adapter

#### **Certifications and Approvals** CSA, CE

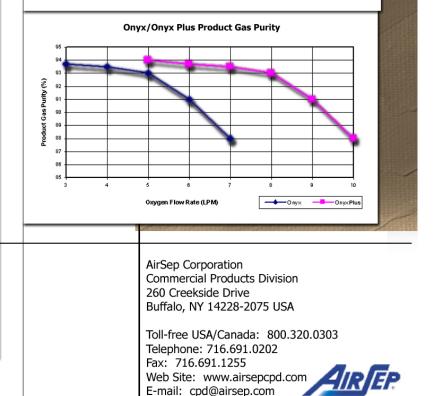
#### Warranty

1-Year Parts and Factory Labor<sup>2</sup>

#### Dimensions

Generator Dimensions: 15.7" x 14.5" x 28.5" (40 x 37 x 73 cm) Shipping Dimensions: 17" x 15.5" x 33.5" (44 x 40 x 85 cm) Weight: 54 lb (25 kg) Net; 65 lb (30 kg) Gross

<sup>1</sup>Specify required voltage and frequency at time of order
<sup>2</sup>An unprotected or inadequately ventilated environment or improper control power may cause damage to the oxygen generator that is not covered under warranty.
\*AirSep Corporation reserves the right to change specifications and/or design without notice.



# **Ozone generator**

Supplied by: Oxyzone Pty Ltd PO Box 518, Woy Woy NSW 2256 Phone: +61 2 4341 5858 www.oxyzone.com.au

 $O_2$  feed 2lpm = 10g/h  $O_3$ As flow increases,  $O_3$  concentration decreases Pressure 40kpa

### T4200 description

The T4200 is the 2 cell version of the T4400 and is built to be the same dependable product as the larger systems.

With its stainless steel construction it is ideal for use in the food industry where it can provide sanitising ozone treatment for automatic equipment or processes.

It can include an internal 40lpm air pump to direct ozone gas directly to work surfaces.

Offering an ozone output of 10 gm/hr using oxygen or about 5 gm/hr using air, it is a very versatile product with the ability to provide large savings in a food processing environment.

### T4200 and external control:

The T4200 is provided with an XLR 3 pin connector for control by an external ORP or dissolved ozone monitor.

T4200 specification				
2 cell, 10gm/hr Corona Discharge ozone generator with input for ORP control				
Cabinet size	Width x Height x Depth 370mm x 420mm x 210mm			
Weight	20kg			
Cabinet material	Grade 304 Stainless Steel			
Ozone output	10 gm/hr at 20C using PSA oxygen			
Ozone producing cells	2 individual air cooled cells			
Power supplies	2 Individual high voltage; high frequency power supplies with overload protection			
Power requirement	240 vac, 50 Hz, 300W nominal			
Controls and indicators				
Start / Stop	Illuminated mains switch			
Circuit protection	3A thermal circuit breaker			
Safety	Low voltage door interlock switch			
Indicators	2 LED indicators showing that the power supplies are operating correctly			
External control	3 pin XLR connector for external controller			
Flowmeter	Indicates the flow level of the oxygen or air feed:			
	0 to 5LPM for oxygen, 0 to 10LPM or 25LPM for air			



# **Ozone destruct unit**

**INTRODUCTION:** The ODS-H series Ozone Destruct Unit utilizes a thermal-catalytic method to remove excess ozone. The catalyst is a transition metal manganese dioxide copper oxide material. It is not consumed by the ozone and acts as a true catalyst.

**INSTALLATION:** Connect ozone compatible tubing, or equivalent, to the inlet (bottom fitting) on the unit. The outlet (on the side of the unit) may be vented to atmosphere, or may be piped outdoors if desired. *It is recommended to pipe the output of the unit outside the facility in case of destruct media failure.* Plug in the electrical cord to provide power to the unit.

**OPERATION:** Turn the switch to ON position. The heater band will begin to heat the top of the unit.

**IMPORTANT:** The heater band must be turned on 30 minutes prior to flow through the unit to allow adequate time for warm-up. Be careful not to touch to top of the destruct unit as this may get very warm.

Be sure that the ozone flow rate does not exceed the specifications for the unit. If the flow rate is too high, the unit will not be effective. The ODS unit is designed to have less than 0.10 ppm ozone concentration at its exit.

The heater band is incorporated onto the unit to prevent moisture from condensing on the destruct media. In the event that the media becomes wet, such as when process water accidentally flows into the unit, the media must be replaced.

**MAINTENANCE:** The destruct media may become fouled or contaminated over time, and will need replacement periodically depending upon usage and conditions. Replacement destruct media can be obtained from Ozone Solutions.

To replace the destruct media:

- Remove the cover from the top of the unit and completely empty the unit by sucking the media out with a vacuum, or by tipping the unit upside down.
- Clean the inside if necessary. Any build-up due to moisture should be removed. If detergents or solvents are used, rinse the unit thoroughly with water and dry it completely before refilling.
- Dump the new media into the unit. With a wood mallet or similar object, tap the side of the unit while filling it so that the media "settles" towards the bottom.
- Ensure that the gasket for the top cover is in good condition. Clean the mating surfaces and re-install the cover. Do not use sealants, if the gasket cannot be reused then it should be replaced.

# Filter column

Volume: 6.5 litres, marked in 0.5 litre intervals Diameter: 88mm OD, 76mm ID Length: 1410mm Base Volume: 1.5 litres Taping intervals: 150mm



Outlet to KI traps & ozone destruct unit

Movable sampling port

Sampling outlets, every 150mm

Treated sample outlet

# **Air stone** Size of the diffuser is 150mm x 38mm with a 30mm inlet barb



# Flow meter on ozone generator

# **Manual for Ozone Destruct Unit**

# **Guide to understanding Ozone**