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OZONE CONTAMINANT TESTING OF A MOLECULAR SIEVE OXYGEN CONCENTRATOR (MSOC)

91-10029


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ABSTRACT *The purpose of this work was to determine if ozone in concentrations found in the upper atmosphere would penetrate the beds of a molecular sieve oxygen concentrator (MSOC) and enter the product or breathing gas. A three-bed oxygen concentrator was subjected to inlet ozone concentrations over the range of 0-10 ppmv. The concentrator was operated at a simulated aircraft altitude of 40,000 feet and cabin altitude of 8,000 feet, cycle times of 9.6 and 32.4, and product flow rates of 10 and 70 ALPM. During this testing the ozone concentrations in the inlet air, product gas, and exhaust gas were monitored.*

Product ozone concentrations were approximately one thousandth of the inlet concentrations, indicating the concentrator is extremely effective at reducing ozone levels. The primary process responsible for this concentration reduction is an adsorption-decomposition mechanism.

A molecular sieve oxygen concentrator containing either MG3 or 5AMG molecular sieve and subjected to the highest atmospheric ozone concentration will have a product ozone concentration significantly below the Threshold Limit Value of 0.1 ppmv. Hence, aircrews will not experience any adverse physiological effects. Additionally, high levels of ozone had no effect on the oxygen enriching ability of the concentrator.

INTRODUCTION A novel approach for the production of inexhaustible quantities of breathable oxygen aboard aircraft involves the use of pressure swing adsorption (PSA) technology. In the application of this technique engine bleed air pressure is cycled within beds of molecular sieve adsorbent where, the nitrogen component of the air is preferentially adsorbed yielding a nearly pure oxygen product gas suitable for hypoxia protection. In a properly designed system oxygen concentrations up to 95% are possible. This technology is attractive because it lowers the aircraft operating cost, increases the aircraft versatility, and improves safety. Currently, systems of this type are employed on the B-1B strategic bomber and the AV-8B fighter.

A simplified MSOC schematic is shown in

Figure 1. The unit operates by cycling the air pressure within beds A and B. One operating cycle consists of a pressurization/adsorption step and a depressurization/desorption step. In the first half-cycle, engine bleed air is permitted to enter bed A through valve V3; product gas is withdrawn through V5; and bed B is depressurized to the ambient surroundings through valve V1. Valves V2, V4, and V6 remain closed. High purity oxygen is produced at the product side of bed A due to the adsorption of nitrogen from the gas stream onto the molecular sieve adsorbent. A portion of this enriched oxygen is allowed to flow through an orifice and into bed B to purge the desorbed nitrogen gas from the bed. During the second half-cycle beds A and B simply reverse roles. By cycling the pressure in the adsorbent beds, a continuous stream of enriched

oxygen may be withdrawn from the product end of the unit.

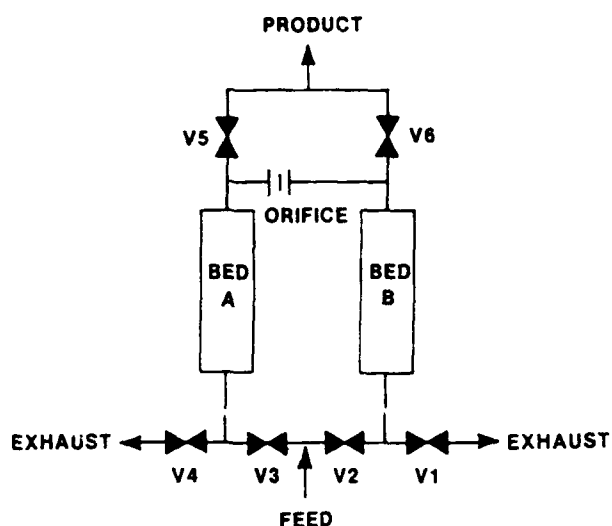


Figure 1 - Schematic of a simple two-bed MSOC

Because these systems used compressed ambient air as their source, the possibility of chemical contamination of the product gas exists. The identification of possible contaminants and the degree these contaminants can penetrate the beds of molecular sieve adsorbent are critically important. Additionally, one desires to select a type of molecular sieve capable of minimizing this penetration. The MSOC must protect aviators from any substance which, if inhaled, could adversely affect their health or performance.

The purpose of this study was to determine the effects of ozone on the ability of an oxygen concentrator to produce an enriched oxygen product, and further, to determine the degree this contaminant can penetrate the beds of the molecular sieve adsorbent and enter the product gas. Aircraft operating at high altitudes will encounter ozone in relatively high concentrations. Further, this contaminant will be present in the engine bleed air supplied to the aircraft MSOC. Another concern was whether these high levels of ozone in the inlet air would affect the concentrator's operation. Molecular sieves 5AMG and MG3 were compared in this study because both of these adsorbents are presently employed in aircraft MSOCs.

A three-bed oxygen concentrator manufactured by Normalair-Garrett Limited (NGL) of Yeovil, Somerset, England was subjected to inlet ozone concentrations of 0-10 ppmv for a total operational period of 272 h. During this testing ozone concentrations in the inlet air, product gas, and exhaust gas of the concentrator were monitored. Data were collected at a simulated aircraft altitude of 40,000 feet and a cabin altitude of 8,000 feet. Additionally, the concentrator was operated at cycle times of 9.6 and 32.4s, product gas flow rates of 10 and 70 ALPM, and a temperature of 297K.

APPARATUS The major components of the ozone contaminant test apparatus were a three-bed NGL molecular sieve oxygen concentrator, an ozone analyzer, an ozone generator, and a venturi injector (Figure 2). Other components of the apparatus were a Perkin-Elmer medical gas analyzer Model MGA-1100, two Technology, Inc. Model LFC-20 mass flowmeters, one LFC-10 mass flowmeter, and a 270 L inlet plenum and mixing chamber. All piping with gas streams containing ozone were constructed of stainless steel to minimize ozone decomposition.

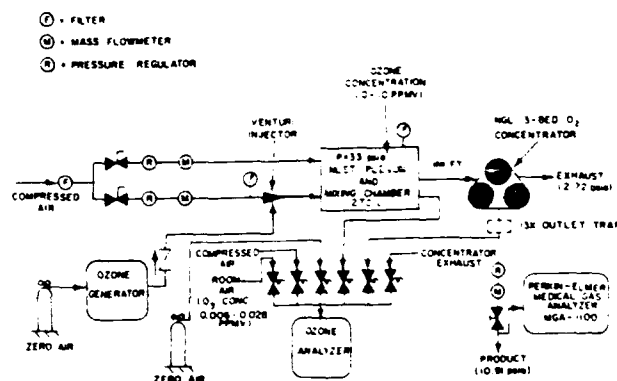


Figure 2 - Simplified schematic diagram of the apparatus used for the ozone contaminant testing of a MSOC

The overall dimensions and weight of the NGL oxygen concentrator (S/N 787 035-2) were 31.0 x 29.9 x 33.0 cm and 18.6 kg, respectively. The unit operated on 28 VDC and consumed approximately 20W. The concentrator was capable of

operating at two selectable cycle times, 9.6 and 32.4s. Each bed of the concentrator held 2.90 kg of MG3 (Lot No. 9366-8501-0188) or 2.96 kg of 5AMG (Lot No. 9355-8408-0220) reactivated molecular sieve. Reactivation was accomplished at atmospheric pressure by heating the molecular sieve pellets to 623K and passing a heated nitrogen purge gas through the material for a period of at least 8 h. Both molecular sieves were manufactured by Union Carbide Corporation, Tarrytown, New York, U.S.A.

The PCI Corporation, Model LC-1, ozone analyzer employed an ultraviolet photometric method of measuring ozone. The concentration of ozone is determined by the ratio of the intensity of the ultraviolet light traversing the optics tube with and without ozone present. Because ozone adsorbs ultraviolet light the intensity will be lower with ozone present. An ozone scrubber containing copper screens coated with manganese dioxide decomposed the ozone in the sample flow to diatomic oxygen. This sample gas without ozone served as the analyzer's internal reference gas for the measurement. The calculation of the ozone concentration was based on the Beer-Lambert Law.

The ozone analyzer had a range of 0-10 ppmv, an accuracy of 3%, and an incremental sensitivity of 0.001 ppmv. The concentrator inlet air and product gas ozone concentrations were determined at an analyzer pressure of 795 Torr and a sample gas flow rate of 1.2 SLPM. The exhaust gas ozone concentration was determined at an analyzer pressure of 141 Torr and sample gas flow rate of 0.44 SLPM. Because the analyzer was designed for operation near atmospheric pressure, a calibration at 141 Torr was required to ensure accurate measurements at the pressure of the concentrator exhaust gas. The results of this calibration are shown in Figure 3 and expressed in the equation shown below:

$$\text{Actual O}_3 \text{ Conc.} = 1.284 \times \text{Measured O}_3 \text{ Conc.}$$

(Analyzer Pressure = 141 Torr)

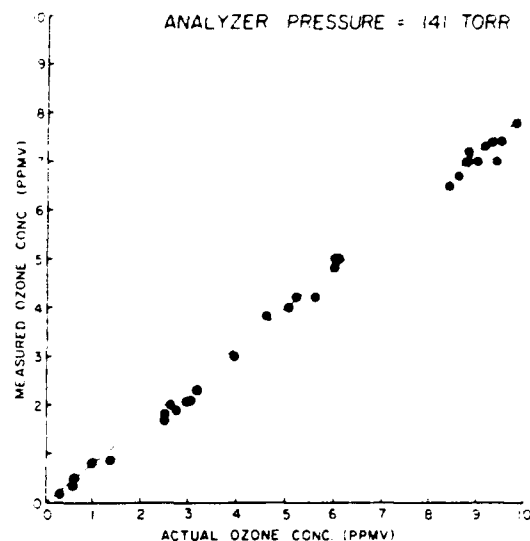


Figure 3 - Calibration of the ozone analyzer at an inlet pressure of 141 Torr

The Ozone Research and Equipment Corporation, Model 03V5-0, ozone generator had a rated ozone output of 2.5 g/h at an inlet air pressure of 9 psig and current setting of 0.8 amp. Ozone was generated by silent arc discharge. Using this method, oxygen is passed between two high voltage electrodes separated by a dielectric. The excited molecular oxygen (O_2) disassociates into atomic oxygen which recombines to form ozone (O_3). The feed gas for the generator was zero air. Dilution of the ozone rich output was accomplished in the 270 L mixing chamber (Figure 2).

PROCEDURE The three-bed NGL concentrator was placed inside an altitude chamber maintained at 40,000 feet (141 Torr). The concentrator exhaust gas flowed to a 160 L exhaust plenum maintained at 40,000 feet before entering the primary volume of the chamber. The product gas was piped to an external plenum maintained at 8,000 feet (564 Torr). During testing the concentrator was fed dry air at a pressure of 33 psia. All components of the system were at room temperature ($\sim 297K$).

Parameters adjusted before each test were the MSOC inlet ozone concentration, cycle time, and product flow. The amount of ozone entering the mixing plenum was controlled by adjusting

the ozone generator outlet flow and power settings. Nominal settings were 1 SLPM flow with a power setting between 55 and 65% depending on the desired inlet ozone concentration (0-10 ppmv). Cycle time (9.6 or 32.4s) was selected by a switch connected to the concentrator electronics control unit. The product flow from the concentrator was set to 10 or 70 ALPM by adjustment of a valve in the product flow line. During testing the concentrator inlet air mass flow rate, product oxygen concentration, and product gas mass flow rate were continuously monitored.

Ozone concentration analysis was initiated by opening the appropriate sampling valve at the inlet to the analyzer (Figure 2). The ambient temperature and inlet pressure were input into the instrument using the front panel keyboard. Because the analyzer operates on 20s cycles (alternating between the sample gas flow and the ozone-free sample flow) a minimum of 5-7 min were required for a stable reading. The zero air and compressed air source were used as additional reference gases (Figure 2). In all cases the zero air and compressed air source gave zero baseline (no ozone present) readings.

RESULTS The product and exhaust gas ozone concentrations for the MG3 filled concentrator are shown in Figures 4 and 5. Product ozone concentrations are generally three orders of magnitude below inlet concentrations indicating the oxygen concentrator is extremely effective at reducing these high inlet O_3 concentrations. For an inlet concentration of 10 ppmv and cycle times of 9.6 and 32.4s the ozone concentrations in the product were 0.008 and 0.022 ppmv, respectively. Ozone concentrations in the product and exhaust taken at a product flow of 10 ALPM were found nearly identical to those observed at 70 ALPM. The higher product concentrations at a cycle time of 32.4s are a result of the higher bed feed air consumption, Ω (Table 1) ³. While operating at an inlet concentration of 10 ppmv the percentage of feed ozone decomposed in the oxygen concentrator was approximately 74-84% (Table 1).

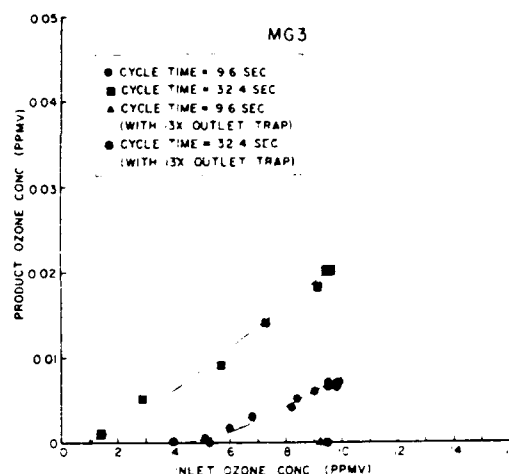


Figure 4 - Product ozone concentrations for the oxygen concentrator filled with MG3 molecular sieve (product flow = 70 ALPM).

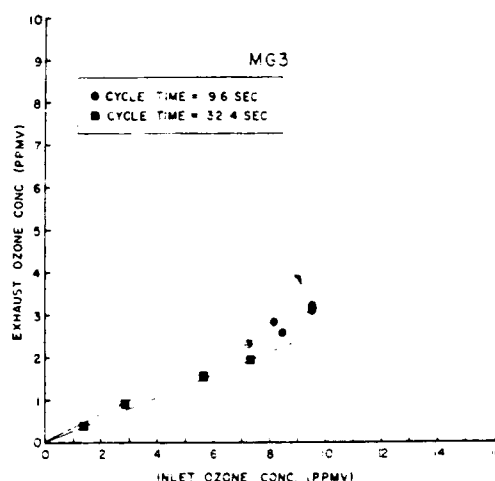


Figure 5 - Exhaust ozone concentrations for the oxygen concentrator filled with MG3 molecular sieve (product flow = 70 ALPM).

In an attempt to further reduce the product concentrations an outlet trap was installed on the product line of the concentrator (Figure 2). The trap was constructed of a plenum chamber with an internal volume of approximately 1 L which held 830 g of 16x40 mesh 13X molecular sieve. With the concentrator inlet ozone concentration of approximately 10 ppmv maintained for a period of 8 h, no detectable amount of ozone was found in the product flow downstream of the 13X outlet trap (Figure 4).

The product and exhaust ozone concentrations for the 5AMG filled concentrator are shown in Figures 6 and 7. For a cycle time of 9.6s no detectable amount of ozone was measured in the product stream over the inlet concentration range of this work. However, it is very likely the product ozone concentration will rise, in a manner similar to that observed for the MG3 product concentration (Figure 4), if inlet concentrations of 10 to 16 ppmv are applied. This prediction is based on the similar adsorption characteristics of MG3 and 5AMG molecular sieves and the fast cycle (9.6s) exhaust data of Table 1. The data suggest 5AMG has a slightly greater ozone adsorption capacity. Hence, ozone breakthrough will occur at slightly higher inlet concentrations. When the 5AMG filled unit was operated at slow cycle (32.4s) the ozone concentrations in the product and exhaust were nearly identical to those observed for MG3.

CYCLE TIME (S)	m ₁ (NLPM)	n (NL/ BED CYCLE Kg ADSORBENT)	%O ₂	O ₃ FEED RATE (GMOL/ MIN x 10 ⁵)	%FEED O ₃ IN EXH	%FEED O ₃ IN PROD	%FEED O ₃ DECOMP
MG3							
9.6	232	6.4	64	9.61	25.65	0.02	74.33
32.4	125	11.6	44	5.18	15.93	0.09	83.98
5AMG							
9.6	285	7.8	69	11.97	33.85	0	66.14
32.4	170	15.5	46	7.04	14.98	0.07	84.95

Table 1 - Some Results of the Ozone Contaminant Tests. (Inlet O₃ conc. = 10 ppmv; Product flow - 70 ALPM)

In general, the mechanism producing the significant ozone concentration reduction between the concentrator inlet and product involves two processes: adsorption-desorption and adsorption-decomposition. Clearly, from Table 1 the adsorption-decomposition process is the most dominate. When operating at the shorter cycle time (9.6s) more ozone is desorbed (exhausted) implying less time is available for the adsorption-decomposition process. This fact is evident by the higher percentage of feed ozone in the exhaust (Table 1). Comparison of the bed feed air consumption, and the percentages of feed ozone in the exhaust

and product indicates 5AMG is slightly more effective at reducing ozone levels (Table 1). The product ozone concentrations can be expected to increase with an increase in the bed feed air consumption.

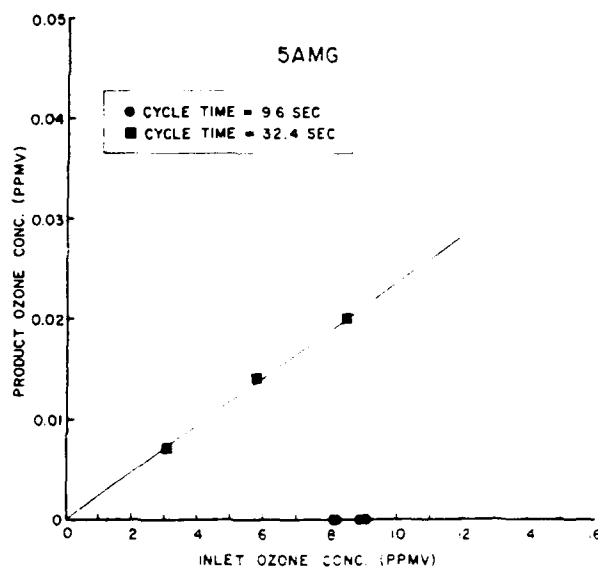


Figure 6 - Product ozone concentrations for the oxygen concentrator filled with 5AMG molecular sieve (product flow - 70 ALPM).

The finding that ozone concentration levels in the product stream were nearly identical regardless of the product flow rate (10 or 70 ALPM), indicates the product flow rate has only a minor effect on the product ozone concentration. Also, product ozone concentration data taken at higher aircraft altitudes would not be expected to differ appreciably from the data of this work. Throughout the testing inlet ozone concentrations had no effect on the product oxygen concentration.

The inlet ozone concentrations used in this study caused three inlet diaphragm valves on the NGL oxygen concentrator to fail and resulted in the near failure of an additional inlet valve. The failure mechanism appears to begin with a breakdown of the elastomer and/or fiber network supporting the inner ring of the diaphragm. This breakdown eventually produces a hole in the diaphragm. This hole equalizes the pressure across the diaphragm and renders the valve inoperative. Symptoms of a failed dia-

phragm are significant reductions in the concentrator's feed air consumption and product oxygen concentration. The three valve failures occurred at 168, 240 and 272 h into the testing. The concentrator was subjected to a total of 272 h of ozone contaminant testing. Other components of the oxygen concentrator, including the molecular sieve, appeared not to have been affected by the ozone.

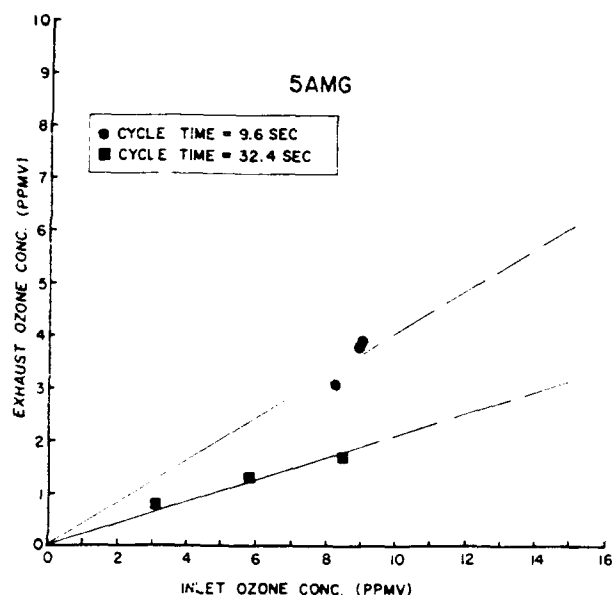


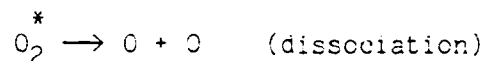
Figure 7 - Exhaust ozone concentrations for the oxygen concentrator filled with 5 AMG molecular sieve (product flow - 70 ALPM).

Room ozone concentrations were measured on several occasions. The range of the room ozone concentration was 0.006 - 0.028 ppmv, although the actual readings were generally closer to the average value of 0.017 ppmv. These data are within the range of published earth surface values (Figure 9).

DISCUSSION In designing an oxygen concentrator for use in the upper atmosphere one must consider three key issues: the maximum inlet ozone concentration, the maximum product ozone concentration, and the resistance of the concentrator's components to ozone attack. In this analysis the maximum inlet ozone concentration was assumed equal to the maximum atmospheric ozone

concentration. Although this assumption is not completely valid, it does provide a starting point for a worst case analysis. In actuality some ozone decomposition can be expected to occur in the aircraft engine compressors, although this amount would depend on the specific characteristics of the engine. For a Boeing 747SP the bleed air ozone concentration was generally 15% below the ambient ozone concentration.⁴ In general, an increase in ozone pressure, velocity, or temperature will increase its decomposition rate. Secondly, a successful design must not allow the ozone product concentration to subject the aircrew to any physiological effects or undesirable odor. Finally, all components of the concentrator, especially elastomers, in contact with the inlet air stream must be resistant to ozone attack. Ozone is a powerful oxidizing agent.

Ozone is formed in the atmosphere about 25 km by the interaction of oxygen with short wavelength ultraviolet radiation from the sun. The reaction occurs as shown below:



Ozone concentration, altitude, and latitude are shown in Figure 8.¹ From this plot the maximum atmospheric ozone concentration of 16 ppmv occurs at an altitude of approximately 30 km over the equatorial regions. Also, ozone concentrations increase from 10-15 km to a maximum at 25-30 km and then decrease. The ozone layer is thinnest over the equator and thickest at the polar regions. Hence, over the tropics the ozone layer is first encountered at 17 km, but over the poles it may be found at an altitude of 10 km. In the subsequent analysis an ozone concentration of 16 ppmv was assumed as the maximum inlet ozone concentration.

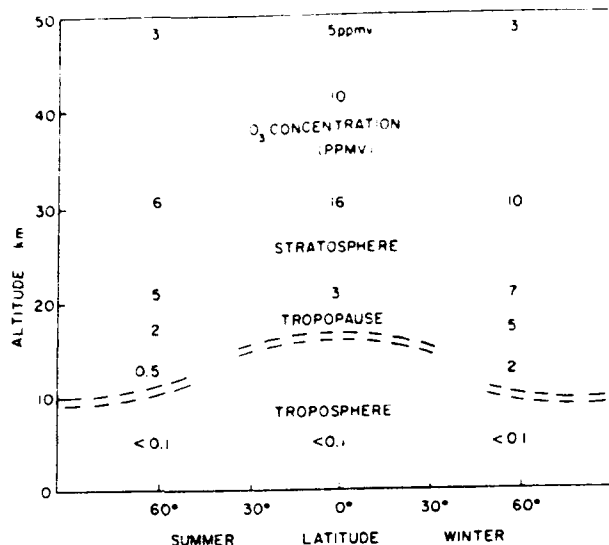


Figure 8 - Ozone concentrations in the atmosphere ¹

Ozone may cause a wide range of symptoms from an unpleasant smell to irreversible poisoning (Figure 9). The toxicity of ozone depends on the concentration and time of exposure. In general, ozone has its greatest effect on the respiratory tract. Severe irritant symptoms are deep coughing, chest pain, difficulty breathing, and throat pain. Less severe irritant symptoms generally involve coughing. Symptoms are termed symptomatic when the first sensation of throat and nose irritation occur. ² The Threshold Limit Value or Threshold Limit of Sensitivity of 0.1 ppmv is the concentration a worker can be exposed to for an 8 h period with no ill effects (Figure 9). Concentrations slightly above 0.1 ppmv cause the mildest symptoms such as headache and dehydration of the throat. Razumovskii and Zaikov suggest that ozone concentration within buildings should not be permitted to exceed 0.047 ppmv. ⁵ Interestingly, concentrations in the 0.02-0.05 ppmv range cause a diminution of the sense of smell which tends to mask other odors. ² The General Threshold of Olfactory Sensation of 0.02 ppmv is the concentration at which the majority of people first perceive the unpleasant odor of ozone. The Threshold Limit of Sensation of 0.01 ppmv is the concentration at which more sensitive in-

dividuals perceive the odor of ozone. To ensure the aircrew experiences no physiological effects or unpleasant odor, I recommend the maximum ozone concentration in the product gas not exceed 0.01 ppmv.

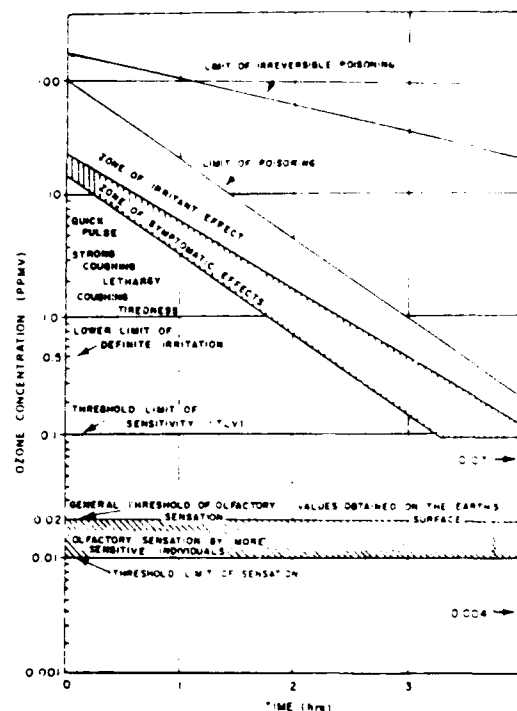


Figure 9 - Ozone threshold values and toxicity ²

An oxygen concentrator for use in the upper atmosphere must be constructed of materials resistant to ozone attack. In general, high ozone concentrations damage rubber components. Particularly sensitive to ozone are rubber components subjected to mechanical stress, such as seals in which cracks have developed, due to embrittlement at ozone concentrations above 1 ppmv; this leads to total disintegration into powder at deformed spots. ² Synthetic rubbers and fluorinated polymers (PTFE) have shown excellent resistance to ozone. Great care must also be exercised to ensure high ozone concentrations do not come into contact with ordinary oil or grease. These materials strongly promote decomposition and may cause an explosion. Fluorinated lubricants should be used instead. ² Although some metals such as iron and zinc are corroded by high ozone concentrations, stainless steel and aluminum are quite resistant.

Based on the information presented previously one can analyze the experimental data on the basis of a worst case set of conditions. If we assume an aircraft altitude of 25-30 km, a concentrator inlet ozone concentration of 16 ppmv, and a 32.4s operating cycle, the maximum ozone concentration expected in the product gas based on the conditions of this work is 0.038 ppmv (Figure 6). This concentration is well below the Threshold Limit Value, hence, no physiological effects will occur. However, at this concentration some individuals may perceive an undesirable odor. Therefore, I recommend the use of an outlet trap between the concentrator and the aircrew to ensure the breathing gas ozone concentration never exceeds 0.01 ppmv.

CONCLUSIONS An oxygen concentrator filled with either MG3 or 5AMG molecular sieve and subjected to the highest atmospheric ozone concentration will have a product ozone concentration significantly below the Threshold Limit Value (0.1 ppmv) (Figures 4 and 6). Hence, no physiological effects will occur.

An oxygen concentrator filled with either MG3 or 5AMG molecular sieve and subjected to the highest atmospheric ozone concentration will have a product ozone concentration above the Threshold Limit of Sensation (0.01 ppmv). No physiological effects will occur but some individuals will perceive an undesirable odor.

High inlet ozone concentrations have no effect on the concentrator's ability to enrich oxygen. In addition, the ozone concentrations used in this study had no effect on either molecular sieve adsorbent.

The failure of four inlet diaphragm valves in this study was the direct result of ozone attack. Hence, diaphragm inlet valves and other components of the concentrator in contact with high ozone concentrations must be constructed of ozone resistant materials.

RECOMMENDATIONS A molecular sieve oxygen generation system (MSOGS) designed for use in the upper atmosphere should have a maximum product gas ozone concentration specification of 0.01 ppmv. This specification will eliminate the occurrence of any physiological effects or undesirable odor.

Use of a properly sized outlet trap (secondary ozone scrubber) on the concentrator product stream will ensure the concentration of ozone in the breathing gas does not exceed 0.01 ppmv. Based on the tests performed with the 13X outlet trap, I would speculate the internal volume of this unit could be significantly less than the 1 L volume used in this study. Also, adsorbents such as, activated carbon, silica gel, and activated alumina are effective for ozone decomposition.² This secondary ozone scrubber must be designed to minimize pressure drop.

Diaphragm inlet valves and other components (particularly elastomers) in contact with high ozone concentrations must be tested to ensure they are resistant to ozone attack and possess the necessary life expectancy.

NOMENCLATURE

ALPM	=	L/min ref. to ambient pressure and temperature
km	=	kilometer (1 km = 3,280 feet)
$\dot{m}_i$	=	integrated inlet air flow rate
NLPM	=	normal L/min ref. to 1 atm and 294K
ppmv	=	parts per million by volume
SLPM	=	standard L/min ref. to 1 atm and 273K
Greek symbols		
α	=	bed feed air consumption (NL/bed-cycle/kg adsorbent)

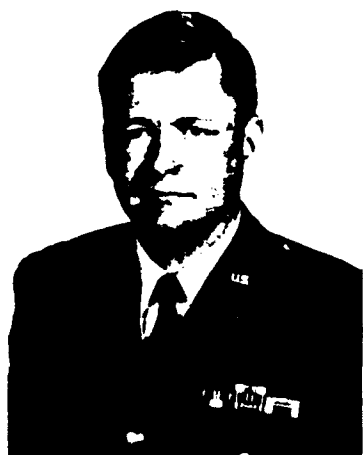
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BIOGRAPHY

Captain George W. Miller is a Scientific Officer assigned to the Crew Technology Division of the USAF School of Aerospace Medicine. He has performed research related to aircraft onboard oxygen generating systems for three years. Captain Miller holds a B.S. in Chemical Engineering from Auburn University, Auburn, Alabama and an M.S. in Chemical Engineering from the Ohio State University, Columbus, Ohio.



Captain George W. Miller