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PURITY OF BREATHING OXYGEN GENERATED BY FLUOMINE

SCHOOL OF AEROSPACE MEDICINE
BROOKS AIR FORCE BASE, TEXAS

DECEMBER 1976

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ABSTRACT (Continued)

carbon dioxide, and nitrogen dioxide did not accumulate in the oxygen product. Water concentrated to 5%. The organic contaminants accumulated up to 8 times their concentration in the supply air. Nitrogen dioxide was the only contaminant irreversibly absorbed by the fluomine. Odor was present in the oxygen product when odorous organic contaminants were present in the supply air. We concluded that the fluomine beds need protection from excessive water, N-oxide and organic contamination to avoid oxygen of unacceptable quality and possible accelerated fluomine degradation.

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EFFECTS OF CONTAMINATED SUPPLY AIR ON PURITY OF BREATHING OXYGEN GENERATED BY FLUOMINE

INTRODUCTION

This report expands our previous studies (1, 2) concerned with the purity of oxygen generated by an Open Loop Oxygen Generating System (OLOGS) primarily using an uncontaminated source of process air. Air used to supply oxygen to the fluomine [bis (3-fluorosalicylaldehyde) ethylenediamine cobalt II] chemical beds of a model Open Loop Oxygen Generating System was adulterated with various contaminants to study their effect on performance of the fluomine chemical and purity of the oxygen product. The contaminants investigated were water, carbon monoxide, carbon dioxide, benzene, n-heptane, ethanol, acetone, acetaldehyde, Freon 12, and nitrogen dioxide. Contaminants were chosen either because they are commonly found in the atmosphere or are representatives of classes of compounds that could be expected in engine bleed air due to combusted lubricants, runway contamination, and other sources. For example, benzene and n-heptane are aromatic and aliphatic hydrocarbons, respectively, used to simulate constituents in jet fuel and jet engine exhaust.

EXPERIMENTAL

The test apparatus used for this study is shown schematically in Figure 1. Apparatus parts and operation are described in detail in reference 2.

Cycling sequence with applicable times and a description of valve functions is presented in Figure 2. Note that the sorption part of a sorption-desorption cycle is shorter (3.5 min) than the desorption (9 min) contrary to equal times planned for the full-scale double-bed aircraft system. The unequal cycling times were mandated because of the poorer heat exchange characteristics of the model system. Removal of residual air after sorption took 1 min and precooling 1.5 min to complete the four operations necessary for a complete cycle. Table 1 lists the general experimental conditions in effect during cycling of the OLOGS.

1. Luskus, L. J., et al. Breathing oxygen systems: Contaminants in oxygen desorbed from fluomine. SAM-TR-73-37, Nov 1973.

2. Luskus, L. J., and H. J. Kilian. Breathing oxygen: Purity of oxygen generated by a fluomine-based system. SAM-TR-76-25, Sept 1976.

TABLE 1. OLOGS CYCLING CONDITIONS

Pressure	
Sorption	1800 mmHg (35 psia)
Desorption	155 mmHg (3 psia)
Temperature	
Sorption	20°C
Desorption	104°C
Supply gas flow rate	650 cc/min
Weight of fluomine in bed	10 gm
Bed size	10 cm x 1.1 cm ID

Generated oxygen was continuously cycled over a 24-hr period and collected in a low-pressure tank (type D2 with 7-liter capacity). The composite oxygen sample collected was then analyzed as is or diluted with aviator's breathing oxygen, if a diluent gas was needed to facilitate analysis.

ANALYSIS

Accumulated oxygen samples were analyzed for contaminants by gas chromatography (gc), infrared spectroscopy (ir), and/or chemiluminescence. GC analyses were made on a dual-column instrument (Tracor Model MT-150G) equipped with dual ultrasonic detectors. The columns used were a molecular sieve to quantify oxygen, nitrogen, and CO and a "Porapak Q" to quantify CO₂ and water. GC was used to verify analyses done by ir and to monitor the nitrogen content of the product oxygen. Nitrogen content was used as a measure of system integrity (freedom from leaks) and residual supply gas volume.

Ir absorption spectra of the product oxygen were obtained with a high-resolution spectrophotometer (Beckmann IR-9) fitted with a variable 10-m gas cell. All spectra were recorded at a sample gas cell pressure of 1550 mmHg (30 psia). The range of 400 to 4000 cm⁻¹ (25 to 2.5 μm) was scanned and absorption intensities were measured at appropriate wavelengths (Table 2).

NO₂ was measured using a Thermo Electron Model 12A NO_x chemiluminescent analyzer.

Calibration curves were obtained for all contaminants, both those added to the supply air and those produced via fluomine degradation. Using standard gas dilution techniques, calibration gas mixes were prepared in zero grade air and/or aviator's breathing oxygen, depending on the gas to be analyzed.

TABLE 2. INFRARED ABSORPTION BANDS USED TO QUANTIFY CONTAMINANTS IN OLOGS SUPPLY AIR, EFFLUENT AIR, AND ACCUMULATED OXYGEN

Contaminant	Wavelength (cm ⁻¹)
H ₂ O	1540
CO	2160
CO ₂	719
Benzene	675
Heptane	2950
Ethanol	1065
Acetone	1219
Acetaldehyde	2730
Freon 12	922

RESULTS AND DISCUSSION

Fluomine used for this study was batch 23 material prepared and activated by Olin Corporation, New Haven, Connecticut. Baseline performance of the chemical was obtained under ideal operating conditions with "pure" unadulterated high-pressure cylinder air (<1000 ppm H₂O, <5 ppm CO₂, <1 ppm CO and <1 ppm total hydrocarbon) as the simulated source of bleed air. Normal fluomine off-gassing and degradation as a function of number of cycles of system operation are illustrated in Figures 3 and 4, respectively. These results are typical of those we observed in our earlier studies using different batches of Olin and Airesearch fluomine.

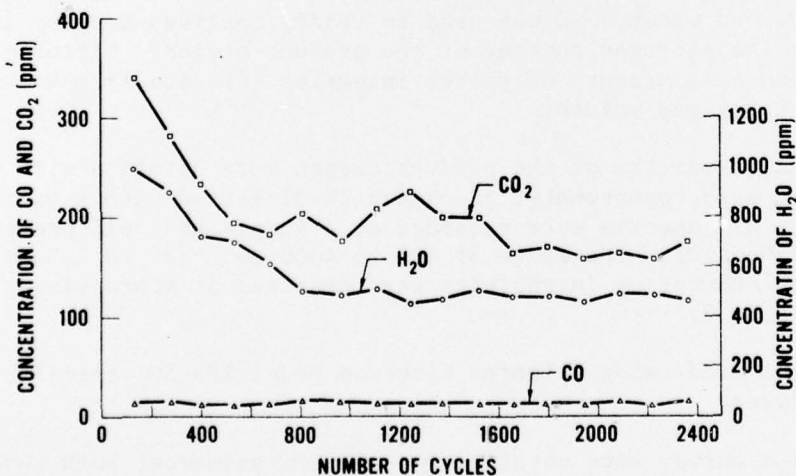


Figure 3. Concentration profiles for major contaminants found in oxygen produced by batch 23 fluomine under baseline conditions.

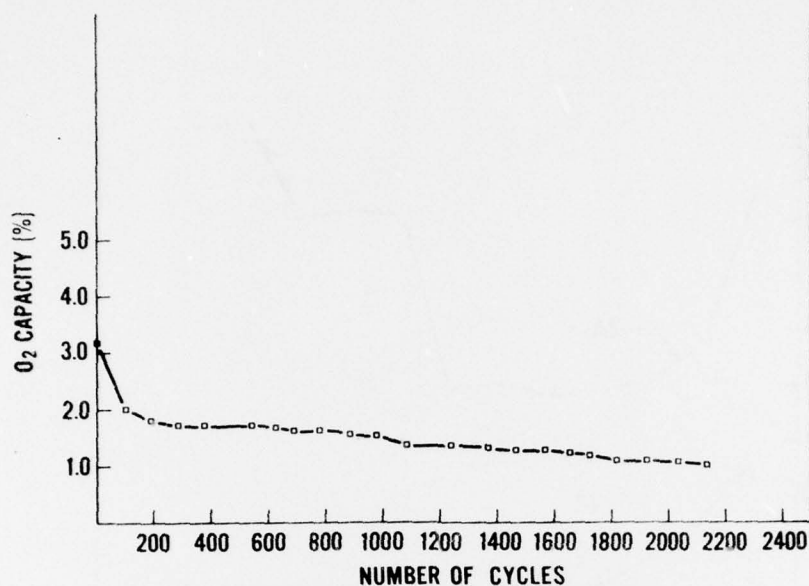


Figure 4. Baseline degradation of batch 23 fluomine as a function of use.

As illustrated in Figure 3, fluomine normally produces CO, CO₂, and water as degradation products during oxygen generation. These products are considered harmless at the concentrations observed in a 95% oxygen product. During testing with contaminated supply air, an increase in the level of these oxidation products and the rate of decrease in fluomine oxygen capacity (Fig. 4) were used to estimate the extent of abnormal degradation of the fluomine chemical.

CO, CO₂, and water are products of oxidative degradation of fluomine (2) with, however, most of the water observed in the oxygen product during tests coming from a second source: cosorption - desorption with oxygen. In review (2), Figure 5 demonstrates the water cosorption effect. Curve 1A is a time profile of water concentrations found in product oxygen samples obtained by continuous cycling with relatively dry air (<150 ppm). The constant water level of about 500 ppm was reached and maintained during most of the 1A study. Initial higher water levels observed at the start of OLOGS operation are due to residual cosorbed water being removed from fluomine and possibly some accelerated oxidation at the beginning of the run (suggested by the similar shape of the degradation curve). When humidified air is used (curve 2A), water levels in the product oxygen are much higher. Supply air used in this case was 1500 ppm water through cycle number 900. At cycle number 624 the flow rate of supply air was increased by about 50%, bringing a larger total mass of water in contact with the fluomine and producing a corresponding higher water concentration in the oxygen product. A further increase in the supply air water level to 8000 ppm without changing flow rate produced a sustained level of about 50,000 ppm (5%) or better for water in the product oxygen. These studies were

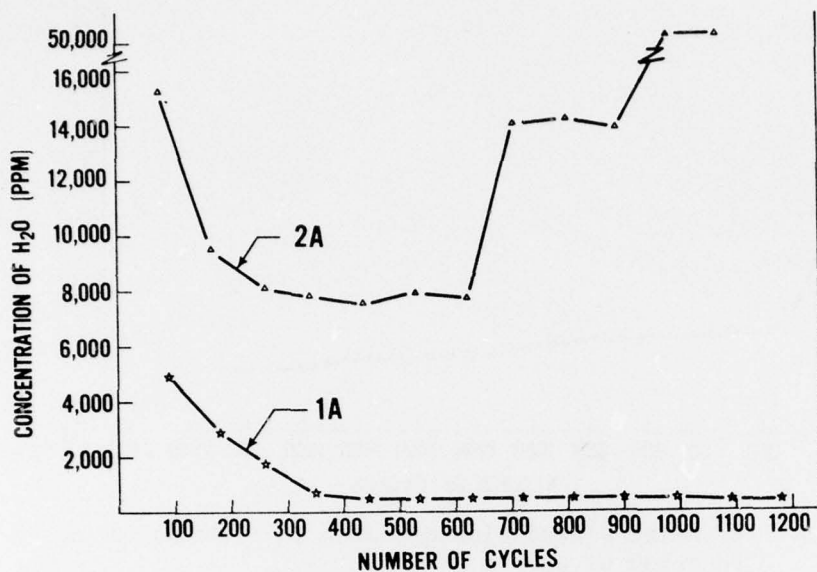


Figure 5. Water concentrations found in oxygen generated during two separate experiments. Curve 1A is a result of using 20 ppm water in the supply air. Curve 2A is a result of stepwise increases in quantity of water seen by the fluomine beds; initially 1500 ppm H₂O, with a first step caused by a 50% increase in supply air flow rate and a second step by an increase to 8000 ppm H₂O.

done at a somewhat lower bed sorption temperature than will be seen by the B-1 OLOGS but are indicative of the water problem. We expect less cosorption at higher operating temperatures but how much less remains to be determined.

The major results of the study described herein were obtained using one background and one test sample (because of limited sample availability) each cycled about 2300 times. Unadulterated and contaminated supply air were used alternately during the test run. The contaminated supply air was introduced during the middle of an accumulation without interrupting the cycling sequence. For example, product oxygen from cycles number 541 through 613 was collected with unadulterated supply air used for cycle numbers 541 through 570, supply air containing 20 ppm hydrocarbons (10 ppm benzene and 10 ppm n-heptane) used for cycle numbers 571 through 598, and unadulterated supply air used for the remaining 15 cycles. All contaminant concentrations in the product oxygen given in this report are adjusted to account for the dilution. Fluomine degradation, due to the presence of these hydrocarbons and other contaminants tested, was estimated by comparing the degradation curve plotted in Figure 6 with that of the background curve in Figure 4. No observed differences in

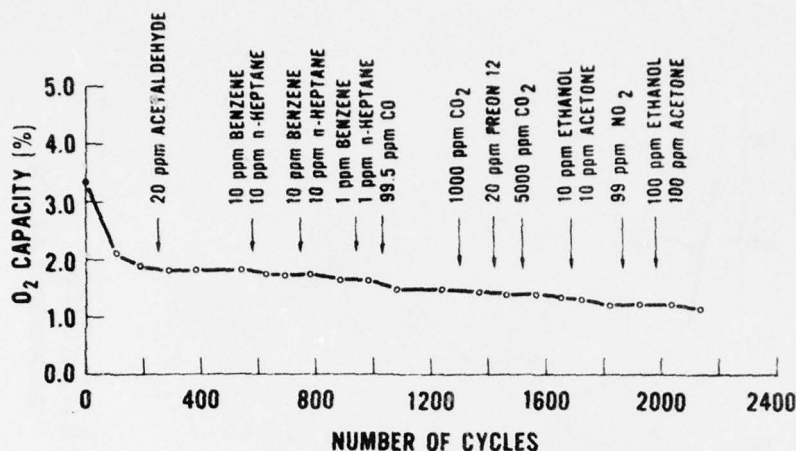


Figure 6. Degradation of fluomine as a function of use during the addition of contaminated supply air.

the slope of the two curves indicated no accelerated degradation occurred during the contamination testing. Figure 6 identifies the contaminants and the approximate cycle numbers where they were introduced to the OLOGS.

The effects of supply air contaminants on normal fluomine off-gassing are illustrated in Figure 7. No dramatic differences in output of CO, CO₂, and water were observed when compared to background (Fig. 3).

Table 3 lists the contaminants added to the supply air and the extent of their accumulation in the product oxygen. With the exception of CO, CO₂, and NO₂, the contaminants were found at higher levels in the product oxygen than in the supply air. A CO concentration of 99.5 ppm did not raise the CO level above that found in the background study (Fig. 3). A concentration of 1000 ppm CO₂ had little or no effect on fluomine off-gassing. Raising the concentration to 5000 ppm increased CO₂ levels in the product oxygen by about 50% (180 to 275 ppm) but not to the extent that the CO₂ created a purity or safety problem. CO₂ in ambient air is approximately 300 ppm. In the case of NO₂ introduction into the OLOGS, Table 3 shows little NO₂ in the oxygen product. NO₂ accumulation is small; however, a check of the nitrogen-rich air exhausted during the sorption cycle also indicated little if any NO₂. Most of the NO₂ disappeared into the fluomine bed. While no significant degradation or accumulation occurred in this limited exposure, extensive irreversible reaction of NO₂ with the fluomine chemical could create problems. The NO₂ results verify Sieckhaus and coworkers' (3) demonstration of the NO₂ reaction with fluomine and stress the need for OLOGS bed protection if significant exposure to the N-oxides is anticipated.

3. Sieckhaus, J. F. Definition and optimization of fluomine. Final report, Olin Corporation, Chemicals Group, New Haven, Conn. 06504, July 1974.

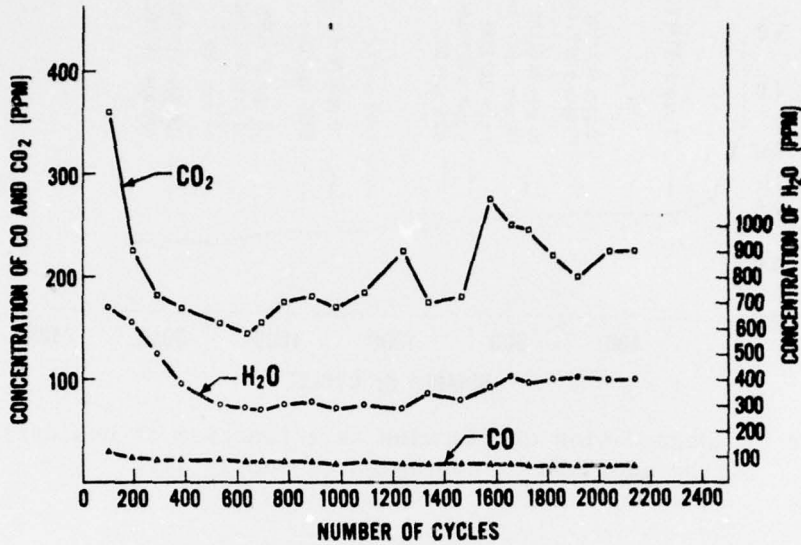


Figure 7. Normal off-gas contaminants found in fluomine-generated oxygen during the addition of contaminated supply air.

TABLE 3. ACCUMULATION OF SUPPLY AIR CONTAMINANTS IN OXYGEN PRODUCED BY THE FLUOMINE OLOGS

<u>Contaminant</u>	<u>Concentration (ppm)</u>	
	<u>Supply air</u>	<u>Oxygen product</u>
Acetaldehyde	20	94
Benzene	1	9
	10	80
Heptane	1	9
	10	80
CO	99.5	18
CO ₂	1000	180
	5000	275
Freon 12	20	28
Acetone	10	43
	100	611
Ethanol	10	28
	100	329
NO ₂	99	0.23

The oxygen product had an odor in those instances where contaminants had accumulated. Oxygen produced during background and CO₂ contaminant testing was odor free.

CONCLUSIONS AND RECOMMENDATIONS

The purity of oxygen generated by a model OLOGS was affected to various extent by different contaminants present in the supply air:

1. Carbon monoxide, carbon dioxide, and nitrogen dioxide showed no significant accumulation.
2. Water concentrated in the oxygen product to 5%.
3. All the organic contaminants (benzene, n-heptane, ethanol, acetone, acetaldehyde, and Freon 12) accumulated in the oxygen product up to 8 times their concentration in the supply air.
4. Odor was present in oxygen generated from supply air contaminated with organic compounds.
5. NO₂ was irreversibly sorbed on fluomine which could lead to degradation if exposure to large concentrations is not avoided.

From these preliminary results we conclude that the fluomine beds must be protected from water and organic contamination to prevent chemical degradation, oxygen of unacceptable quality, and problems with safe operation of the OLOGS and life support accessories. Protection could involve supply air and product oxygen scrubbing, variation of system parameters such as cycling times and sorption temperatures, and/or minimum operation of OLOGS at ground and lower altitudes where water and contaminant concentrations are highest.

We recommend the supply air contaminant problem be further investigated using prototype systems as they become available and the requirements be examined for extensive OLOGS operation at ground level and during low-altitude runs. In collaboration with the 412A Life Support System Program Office and Airesearch Manufacturing Company, we plan to investigate supply air contamination effects on the B-1 OLOGS prototype during the USAFSAM man-rating effort scheduled for early 1977.