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AFWAL-TR-88-2081

JET FUEL DEOXYGENATION

S. Darrah

Geo-Centers, Inc.  
7 Wells Avenue  
Newton Centre, MA 02159



October 1988

Interim Report for Period March 1987 - July 1988

AD-A205 006

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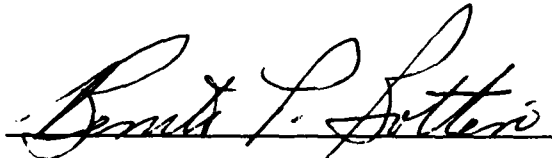
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\_\_\_\_\_  
CHARLES R. MARTEL  
Project Engineer  
Fuels Branch

  
\_\_\_\_\_  
CHARLES L. DELANEY, Chief  
Fuels Branch  
Fuels and Lubrication Division

FOR THE COMMANDER

  
\_\_\_\_\_  
BENITO P. BOTTERI, Assistant Chief  
Fuels and Lubrication Division  
Aero Propulsion & Power Laboratory

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE		4. PERFORMING ORGANIZATION REPORT NUMBER(S) GC-TR-88-1416-013	
4. PERFORMING ORGANIZATION REPORT NUMBER(S) GC-TR-88-1416-013		5. MONITORING ORGANIZATION REPORT NUMBER(S) AFWAL-TR-88-2081	
6a. NAME OF PERFORMING ORGANIZATION Geo-Centers, Inc.	6b. OFFICE SYMBOL (if applicable)	7a. NAME OF MONITORING ORGANIZATION Aero Propulsion Laboratory (POSF) Air Force Wright Aeronautical Labs, AFSC	
6c. ADDRESS (City, State, and ZIP Code) 7 Wells Avenue Newton Centre MA 02159		7b. ADDRESS (City, State, and ZIP Code) Wright-Patterson AFB OH 45433-6563	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION Aero Propulsion Laboratory	8b. OFFICE SYMBOL (if applicable) AFWAL/POSF	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F33615-84-C-2412	
8c. ADDRESS (City, State, and ZIP Code) Air Force Wright Aeronautical Laboratories Wright-Patterson AFB OH 45433-6563		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO. 61101F	PROJECT NO. 3048
		TASK NO. 05	WORK UNIT ACCESSION NO. 46
11. TITLE (Include Security Classification) Jet Fuel Deoxygenation			
12. PERSONAL AUTHOR(S) S / Darrah			
13a. TYPE OF REPORT Interim	13b. TIME COVERED FROM Mar 87 to Jul 88	14. DATE OF REPORT (Year, Month, Day) 1988 October	15. PAGE COUNT 26
16. SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
21	04		
21	05		
		Jet Fuels; Deoxygenation • (mgm) ←	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) This program was initiated to identify and characterize methods of deoxygenating quantities of jet fuel to improve fuel thermal stability. Three methods, chemical getters, molecular sieves and nitrogen sparging, were evaluated in our laboratory. In the case of nitrogen sparging, additional results were obtained by comparing laboratory experimental results with the output from ULLAGE, a computer based mathematical model. Each method was shown to reduce the oxygen content of jet fuel. Economic and system considerations favor nitrogen sparging for large quantities of fuel.			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL CHARLES R. MARTEL		22b. TELEPHONE (Include Area Code) 513-255-7431	22c. OFFICE SYMBOL AFWAL/POSE

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FOREWORD

This report documents work conducted under contract F33615-84-C-2412, Task No. 13, with Geo-Centers Inc. This contract is documented under project 3048, task 05, work unit 46. Funding for Task 13 was provided by Independent Laboratory In House Research funds (ILIR), work unit ILIRP705, program element 61101F. Dr Shirley Darrah, Geo-Centers Inc., was the principal investigator, and Mr Charles R. Martel, AFWAL/POSF, was the program manager.

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## ABSTRACT

This program was initiated to identify and characterize methods of deoxygenating quantities of jet fuel to improve fuel thermal stability. Three methods, chemical getters, molecular sieves and nitrogen sparging, were evaluated in our laboratory.

In the case of nitrogen sparging additional results were obtained by comparing laboratory experimental results with the output from ULLAGE, a computer based mathematical model. Each method was shown to reduce the oxygen content of jet fuel.

Economic and system considerations favor nitrogen sparging for large quantities of fuel.

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## 1.0 INTRODUCTION

The thermal stability of jet fuels has been a problem of increasing importance to both commercial and military users for many years. With the development of higher speed aircraft, which have higher temperature engines, the thermal stability of fuel is becoming increasingly critical. Thermal instability refers to the propensity of the fuel to form carbonaceous deposits on hot metal surfaces. This effect is a function of fuel components (especially trace contaminants), the composition of the metal surface and temperature.

In aircraft, fuel thermal instability can cause problems in three major areas. The first two, deposits on fuel injection nozzles and metering valves, directly and perhaps catastrophically affect engine performance. The third, deposits on walls of heat exchangers, reduces the efficiency of heat transfer to fuel which is used as a coolant. The latter effect is becoming more important as higher Mach number aircraft are developed, which increasingly rely on fuel as a coolant of airframe and engine components.

A major contributor to the thermal decomposition of fuels is dissolved molecular oxygen. When heated, oxygen readily forms free radicals which initiate and propagate autoxidation reactions of hydrocarbons. When the concentration of dissolved molecular oxygen is reduced to levels of 1ppm, this mechanism of decomposition becomes unimportant, and the pyrolysis of the fuel itself is the dominant mechanism. Pyrolysis, however, occurs at much higher temperatures than autoxidation. Thus, by decreasing the oxygen concentration to 1ppm, the fuel can be subjected to higher temperatures before thermal decomposition occurs.



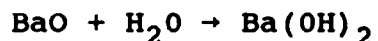
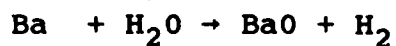
GEO-CENTERS, INC. has conducted a program to identify and evaluate methods of deoxygenating jet fuels. These methods were considered for potential application to either a ground-based, fuel preconditioning and servicing system or to an on-board, aircraft fuel system.

In evaluating methods of deoxygenation, several technical factors must be considered. Kinetics and thermodynamics must strongly favor oxygen removal to affect a low oxygen concentration in a short time. It is important that no new contaminants be introduced into the fuel and that no beneficial components be removed. In addition, safety considerations involving the fire hazard of exothermic reactions, toxicity of materials including the input stream and waste products, and the disposal of waste products must eventually be addressed. Economic factors, including the costs of materials, equipment and manpower, will eventually determine the viability of building large scale deoxygenation facilities. While we are mindful of safety and economic implications of each method examined, the thrust of this effort has been directed to technical feasibility issues.

## 2.0 CHEMICAL GETTERS

Chemical methods were the first approach evaluated during this program. Among the chemical methods, "getters", metals or alloys with chemically active surfaces, appeared attractive because they react with oxygen to form insoluble oxides. They have been widely used in the semiconductor and nuclear fuels industries. Many of these are transition metals which were deemed unacceptable for this application because of the potential for catalyzing reactions in fuel. Barium metal, however, is a commonly used getter and looked

attractive. Barium reacts readily with oxygen and water, but the reaction is not as exothermic as the reaction of other metals such as sodium. The relevant reactions of barium are:



Laboratory experiments were performed to determine the effectiveness of this method. The initial experiments were conducted with JP-5 in a threeneck round bottom flask. Two of the necks contained stopcocks attached to ground glass joints. One stopcock was used to admit nitrogen gas while the other was used to vent the flask. The third neck held the probe for a Yellow Springs Instrument International, Inc. model 58 dissolved oxygen meter. One liter of JP-5 fuel was used. The barium metal was purchased in 6 mm diameter sticks (granulated barium metal is not currently available from laboratory suppliers). These sticks were cut into pieces approximately 6 mm in length. The barium was stored in oil under a nitrogen atmosphere in an attempt to prevent its surface from oxidizing. The JP-5 fuel was saturated with air prior to the experiment by bubbling air through it.

In the first experiment, fuel and barium were stirred together in the flask under a nitrogen atmosphere. After 30 minutes, the oxygen concentration had decreased to 50% of the initial concentration. It was observed, however, that this result could be obtained simply by stirring the fuel under a nitrogen atmosphere without the barium metal. The presence of white solid did indicate that some barium had reacted. Subsequent experiments were performed with air initially present and no nitrogen purging of the flask.

In order to increase the exposed surface area of barium, the threenecked flask was replaced with a blender. The lid was sealed to the container to prevent air leakage after the deoxygenation process began. The system was simplified by using cyclohexane rather than JP-5. The cyclohexane was mixed with the pieces of barium in the blender until the barium was broken into very small particles. After 30 minutes, the oxygen concentration was 4% of the initial concentration. The absolute concentration of oxygen in the cyclohexane could not be determined by our measuring instrumentation which gives a relative indication, however, assuming the air saturated concentration to be 50 ppm then 4% would be 2 ppm. This value may well reflect the air-tightness of the blender rather than the limit of the deoxygenation capability of the barium. A white solid was observed in the liquid. At the conclusion of the experiment, solid barium metal remained in the container. The white solid was filtered from the liquid with Whatman No. 2V filter paper. This paper retains solids larger than 8 microns in size. The filtered solution was observed to be clear and did not scatter light, thus indicating that all solid by-products had been removed by the filter. It was also observed that a coating of white solid remained on the walls of the container.

Calculations of the quantity of barium metal required to stoichiometrically react with 10,000 gallons of fuel with 50 ppm oxygen yield a value of 35 lbs of barium. Additionally, twenty-two lbs of barium react with 70 ppm of water in 10,000 gallons of fuel. The actual quantity of barium metal required to react with both the oxygen and water is less than the total of these two values because barium oxide itself reacts with water. Barium sells commercially for approximately \$20/lb which is in the range of economically feasibility. However, barium is costly enough that recycling technique should be explored for large throughput operations.

### 3.0 MOLECULAR SIEVES

The use of molecular sieves as deoxygenating agents was also investigated in this program. They are commonly used in industrial processes to remove water and contaminant gases. Molecular sieves have advantages over chemical means of deoxygenation in that they are nontoxic, nonflammable, and can be regenerated by physical means, thus avoiding disposal problems.

Zeolite molecular sieves have pores of a uniform size which can adsorb small molecules such as oxygen, while excluding the larger molecules present in jet fuel. These materials have a high internal surface area available for adsorption due to the channels or pores which uniformly penetrate the entire volume of the solid. The available surface area is very much larger than the external surface of the adsorbent particles, which contributes only a small percentage of the total area. The particular sieves to be used are chosen by matching the size of the molecules to be occluded with the diameter of the channels.

Initially, Type 3A sieves in 1/16 inch pellets were chosen. These were chosen because they occlude only molecules which are 3 angstroms in size or smaller. Thus they should occlude oxygen in the channels but exclude the larger fuel molecules. The

experiments were performed in a one liter round bottom flask. The sieves were placed in the flask and activated by heating them while the flask was being evacuated. JP-5 jet fuel was then allowed into the flask. The small volume in the flask which was not occupied by the 1 liter of fuel was allowed to fill with air so that the system was under atmospheric pressure. The probe for the oxygen meter which had previously been calibrated on air-saturated fuel was inserted into the flask. The mixture was stirred until equilibrium was attained (the meter readings were constant).

The results of these experiments are shown in Figure 1. Deoxygenation does occur and is a function of the quantity of sieves used. The efficiency of these particular sieves, however, is not sufficient for fuel deoxygenation because even at a ratio of 150g sieves/liter fuel a reduction of oxygen concentration by only 27% was obtained.

Because oxygen molecules are near the limit of the Type 3A channels in size, experiments were also performed with Type 4A sieves in the form of 8-12 mesh beads. Although the larger size of the channels allow slightly larger molecules such as CO<sub>2</sub> to be occluded, it was hoped that they would be more efficient deoxygenating agents. As in the previous experiments, a 1 liter, three necked flask was used. The beads were activated by heating them under vacuum. After cooling, fuel was admitted to the flask and the mixture was stirred until equilibrium was obtained. The concentration of oxygen in the fuel relative to the concentration of oxygen in air saturated fuel was measured. The concentrations of oxygen in the gas space above the fuel and in the sieves were calculated.

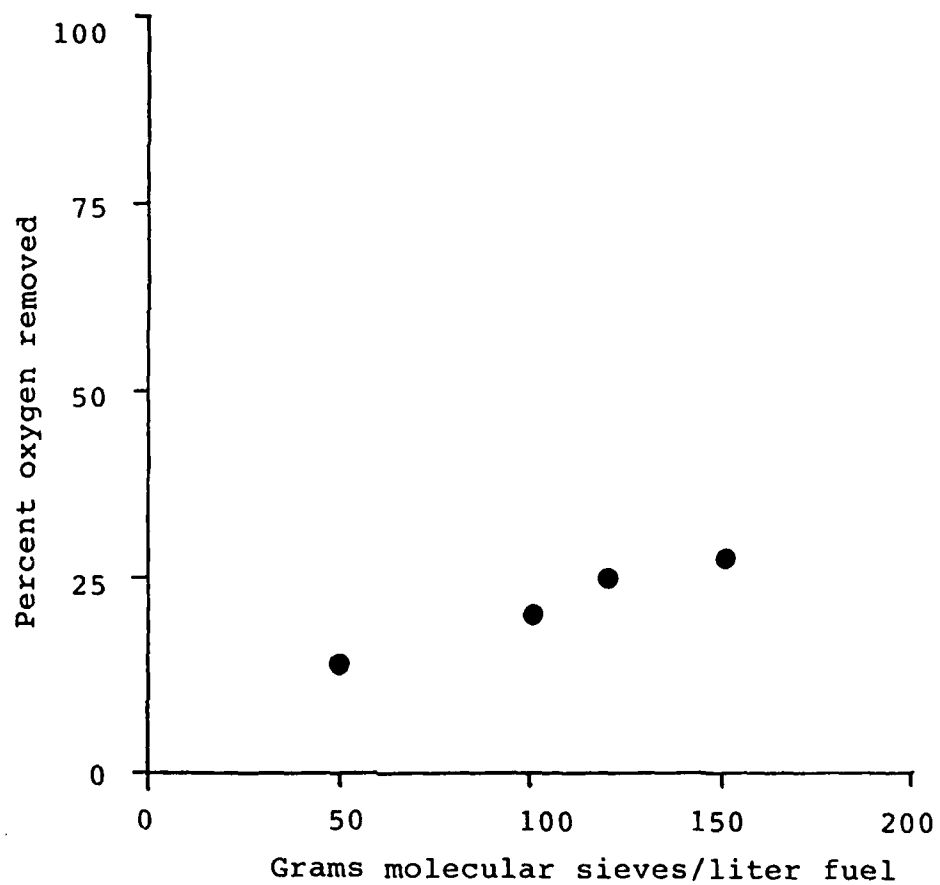


Figure 1. Percent oxygen removed from JP-5 with Type 3A molecular sieves

Since the flask is under vacuum when the fuel is admitted to it, gases boil out of the air saturated fuel as the flask is being filled. Therefore, the flask can not be completely filled with fuel. In order to evaluate the effect of this phenomenon, experiments were first performed simply with an evacuated flask and no sieves present. The total volume of the flask was actually 1.215 liter. With no sieves present, the fuel occupied a volume of 1.15 liter. Assuming a concentration of 40 ppm in air saturated fuel, the fuel contained 46.1 mg of oxygen. When the flask was filled with fuel, 10 ml of air at atmospheric pressure which was in the tubing was also admitted into the Figure 1 flask. It contained 2.8 mg of oxygen. Thus, the total oxygen content of the flask was 48.9 mg. After equilibrium, the oxygen concentration in the flask was 31.0 ppm or a total content of 35.5 mg. From Henry's law and the ideal gas law, the gas phase contained 13.9 mg of oxygen. The total oxygen in the flask was 49.4 mg which agrees well with the calculation of the quantity of oxygen initially admitted to the flask.

For the experiments with the sieves, the initial and final concentrations of oxygen in the fuel were measured with the oxygen meter. The oxygen contents of the gaseous and liquid phases were calculated by the same procedure used when no sieves were present. The oxygen content of the sieves was the difference between the initial and final contents of the gaseous and liquid phases. The results of the measurements and calculations are given in Table 1. Although the sieves do remove oxygen from the fuel, their efficiency is not good and in practice massive quantities would be required.

TABLE 1. Results of Jet Fuel Deoxygenation Experiments  
Using Type 4A Molecular Sieves

Wt. of sieves (g)	0	50	100	150
Vol. of sieves (ml)	0	30	60	90
Vol. of fuel (ml)	1150	1110	1075	1035
Vol. of gas (ml)	65	75	80	90
Initial O <sub>2</sub> in fuel (mg)	46.1	44.5	42.9	41.3
Initial total O <sub>2</sub> (mg)	48.9	47.4	45.8	44.2
Final O <sub>2</sub> in fuel (mg)	35.5	30.2	24.0	17.8
Final O <sub>2</sub> in gas (mg)	13.9	14.1	12.3	10.7
Final O <sub>2</sub> in sieves	0	3.1	9.5	15.7



#### 4.0 NITROGEN SPARGING

The deoxygenation of fuel by nitrogen sparging has also been evaluated during this program. Sparging is the method which has been used in laboratory studies on thermal stability of deoxygenated fuel. It has the advantage of not introducing possible contaminants or reactive materials to the fuel and also poses no disposal problem.

Sparging is a physical process in which the composition of the atmosphere which is in contact with the fuel is changed. As the composition of the atmosphere changes, then the composition of the fuel must also change in order to maintain equilibrium. The relationship between the concentrations of oxygen in the two phases is give by Henry's Law:

$$p_j = x_j k_j$$

where  $p_j$  is the partial pressure of the solute (oxygen) in the vapor above the solution,  $x_j$  is the mole fraction of the solute in the fuel and  $k_j$  is a constant. Calculations using Henry's law show that fuel which contains 40 ppm oxygen when air saturated should contain 1 ppm oxygen when in equilibrium with an atmosphere which has no more than 0.5% oxygen.

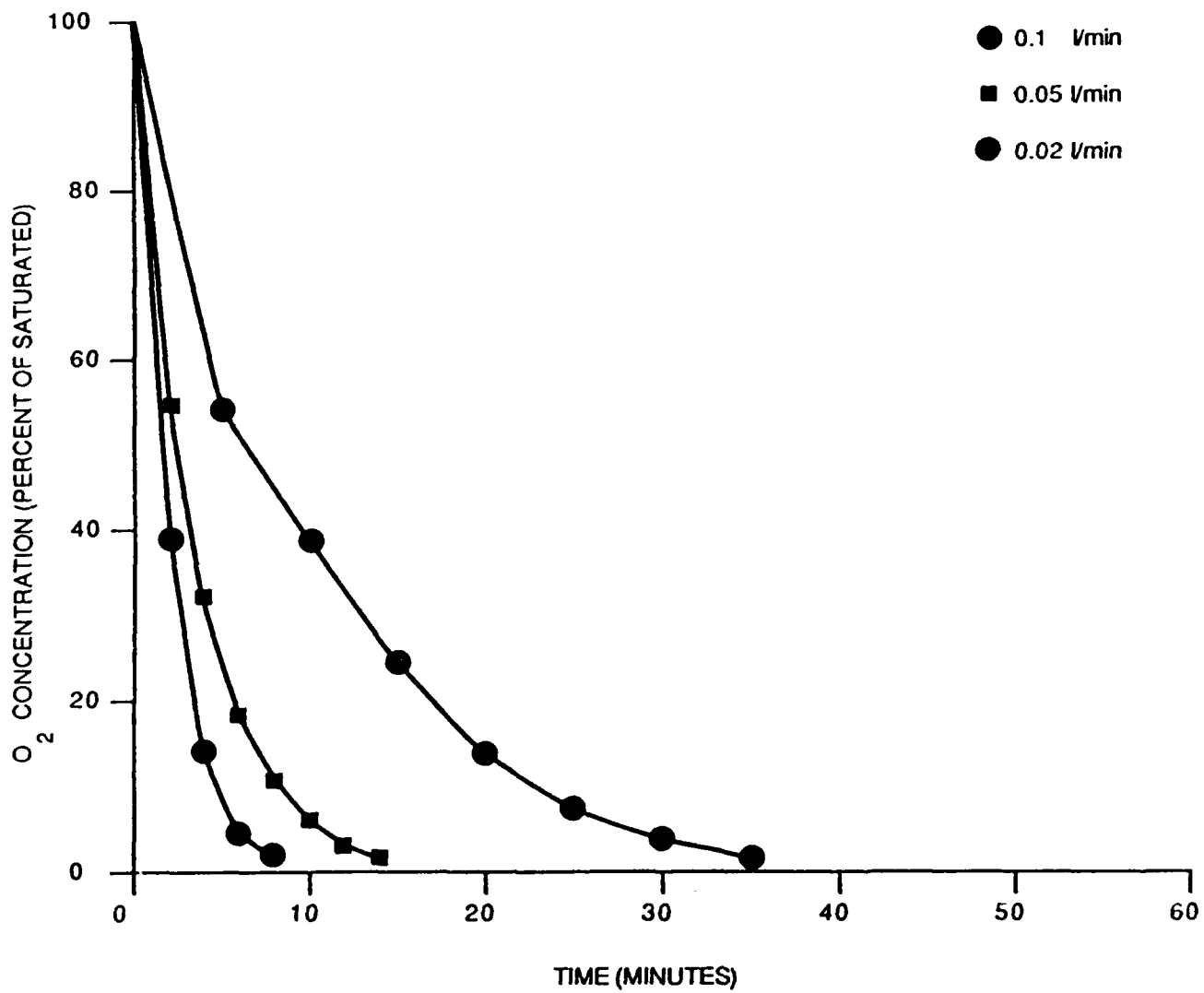
Experiments were performed to study the effect of fuel temperature and sparging gas concentration and flow rate on deoxygenation rate and final oxygen concentration. A three-neck round bottom flask containing 1 liter of JP-5 fuel was used. The fuel was first saturated with air and then deoxygenated by bubbling the sparging gas through the fuel with a glass tube with a fritted

cylinder. The fuel was stirred during deoxygenation in order to maximize the efficiency of the deoxygenation and also to ensure proper response of the oxygen meter. The two different gases used for sparging were a special mixture containing 99.5% N<sub>2</sub> and 0.5% O<sub>2</sub> and "ultrapure" nitrogen, containing 99.999% N<sub>2</sub>.

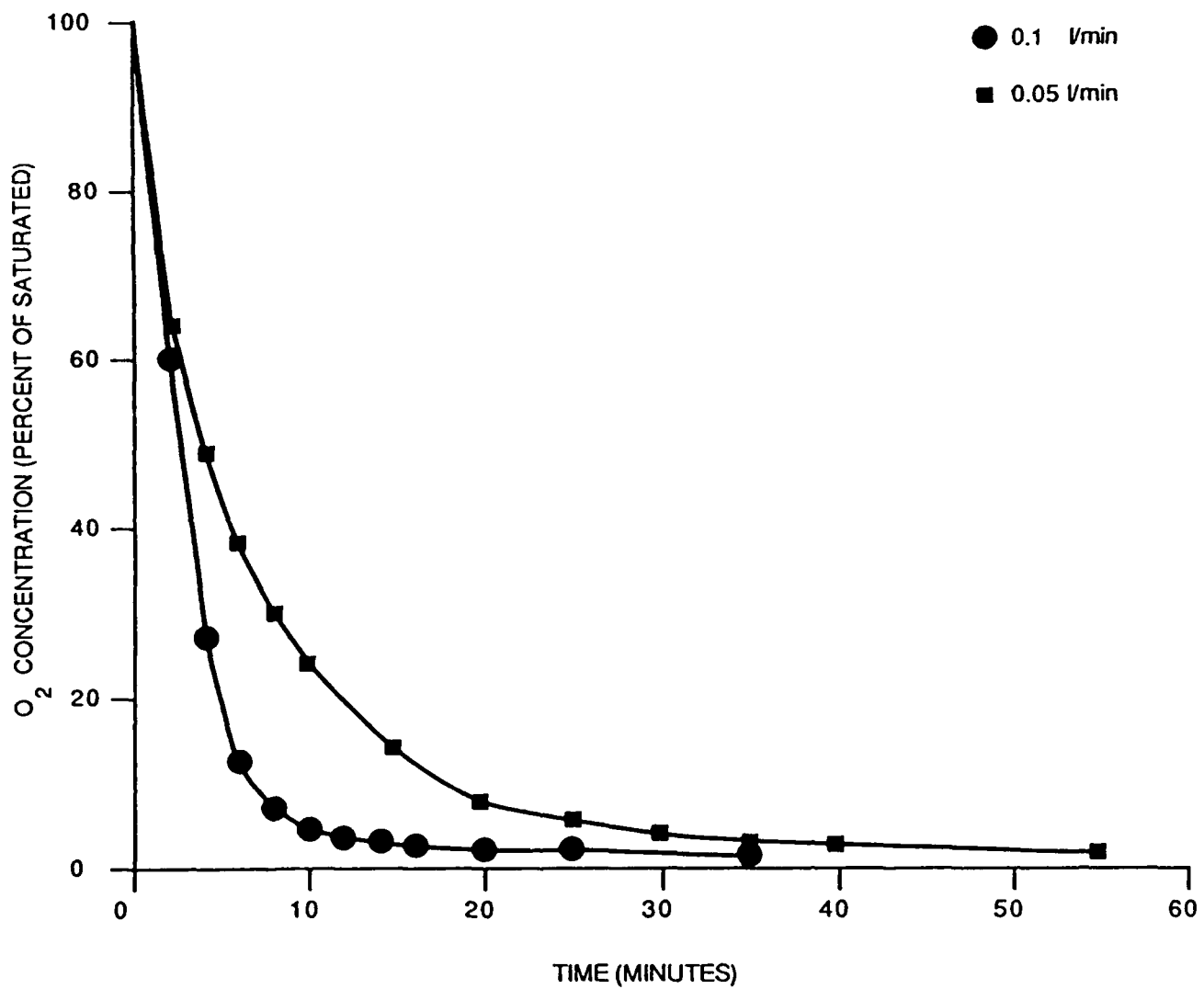
With the gas containing 99.5% N<sub>2</sub>, a final concentration of 2.5% of the air saturated oxygen concentration was obtained. Assuming the air saturated JP-5 contains 40 ppm of O<sub>2</sub> then 2.5% of this value is 1 ppm. Figures 2 and 3 show graphically deoxygenation experiments with "ultrapure" and 99.5% N<sub>2</sub>. Although, a 1 ppm level can be attained with either gas, the time of deoxygenation and volume of sparging gas required were significantly less for "ultrapure" N<sub>2</sub> rather than the 99.5% mixture. Thus, the 99.5% mixture is adequate to maintain fuel at the 1 ppm dissolved oxygen concentration, but higher purity nitrogen is more efficient for the initial deoxygenation process. In actual practice, different methods may be advantageous for purifying the nitrogen gas used at different steps of the fuel handling and storage process.

In Figure 4, the effect of temperature is illustrated. The deoxygenation process is as expected from kinetics arguments slower at lower temperatures. Therefore, the range of temperatures to which the fuel could be subjected in the deoxygenation process must be considered in setting the requirements for the sparging system.

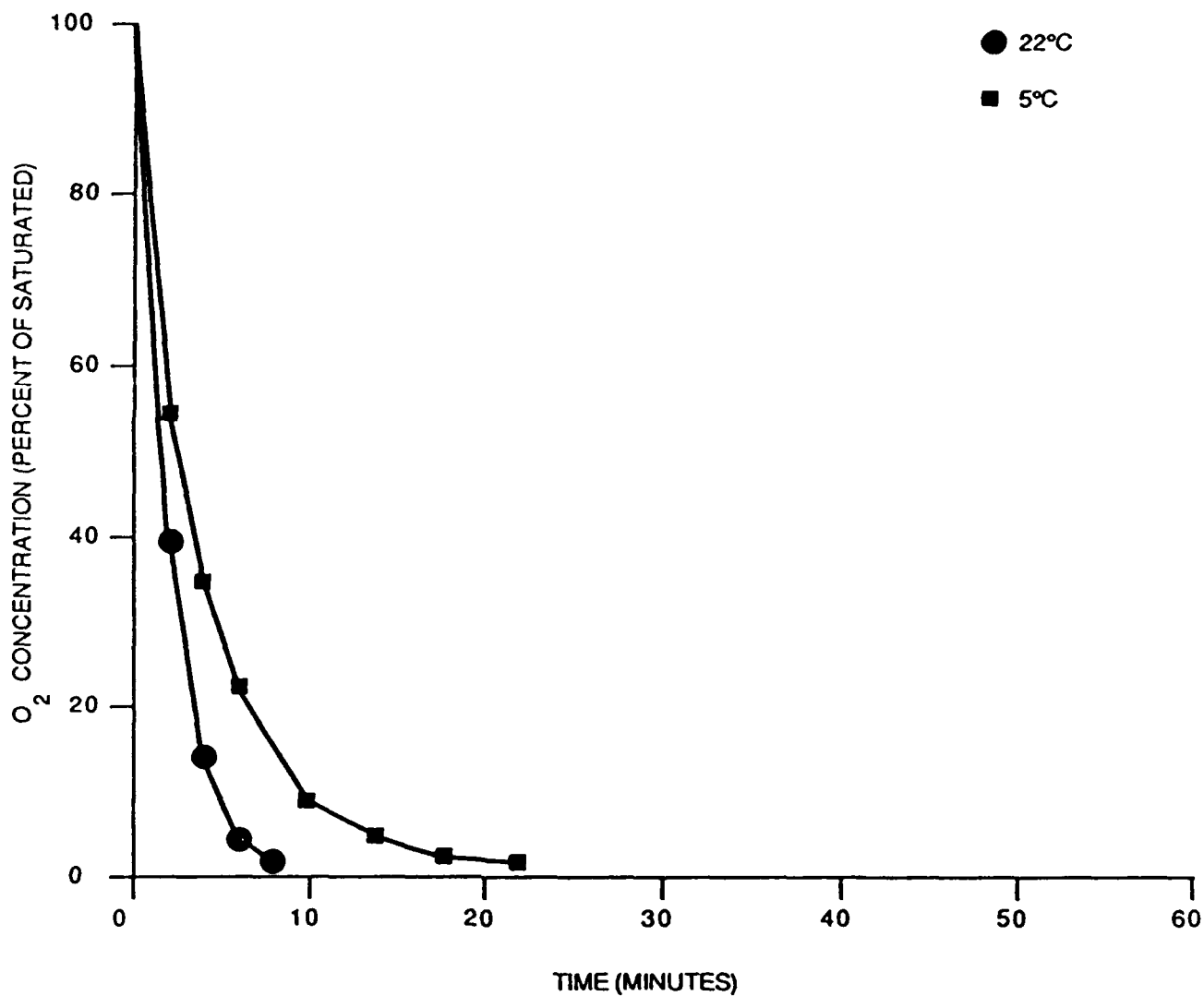
Following these laboratory experiments, the application of nitrogen sparging to deoxygenation of full-scale systems was studied by performing calculations with the computer based mathematical model, ULLAGE. This model is described in the report



**Figure 2. Deoxygenation of JP-5 with 99.999% Sparging Gas at Different Flow Rates.**



**Figure 3. Deoxygenation of JP-5 with 99.5% N<sub>2</sub> Sparging Gas at Different Flow Rates.**



**Figure 4. Deoxygenation of JP-5 with 0.1 l/min of 99.999% N<sub>2</sub> Sparging Gas at Different Temperatures.**

AFWAL-TR-87-2060, "Volume I - Airplane Fuel Tank Ullage Computer Model". In this model, aircraft fuel tanks are simulated and conditions such as temperature and flow rates can be varied. The software was modified to calculate the concentration of oxygen dissolved in the fuel in ppm (mg oxygen/liter fuel) and to output this value as a function of time.

Initially, the model was used to simulate the conditions which had been used in previous laboratory experiments. These conditions were 1 liter of JP-5 at 72°F in a volume of 1.2 liter with a surface area of 0.102 ft.<sup>2</sup> Calculations were performed for 99.999% N<sub>2</sub> at flow rates of .01 liter/min, 0.05 liter/min and 0.02 liter/min with an efficiency of 0.96. Comparing Figure 2 with Figure 5 shows there is good agreement between the experimental and calculated results.

Calculations were also performed to show the effect of temperature on deoxygenation. These results did not agree well with experiments. The calculated rates of deoxygenation showed an insignificant change with change in temperature unlike the laboratory experiments. The reason for this discrepancy is not understood at this time. However, the laboratory results shows a consistent temperature effect which leads us to believe that the computer model should be examined with respect to its handling of temperature parameters.

The deoxygenation of fuel in storage tanks was next simulated. For the calculations, 10,000 gallons of fuel in a 1500 ft<sup>3</sup> tank with 113 ft<sup>2</sup> surface area was used. The calculations were performed for a variety of conditions including an empty tank being filled with fuel at various flow rates and a previously filled tank. The fuel and upper skin temperatures were varied as were the scrub flow rate, scrub gas concentration and scrub efficiency.

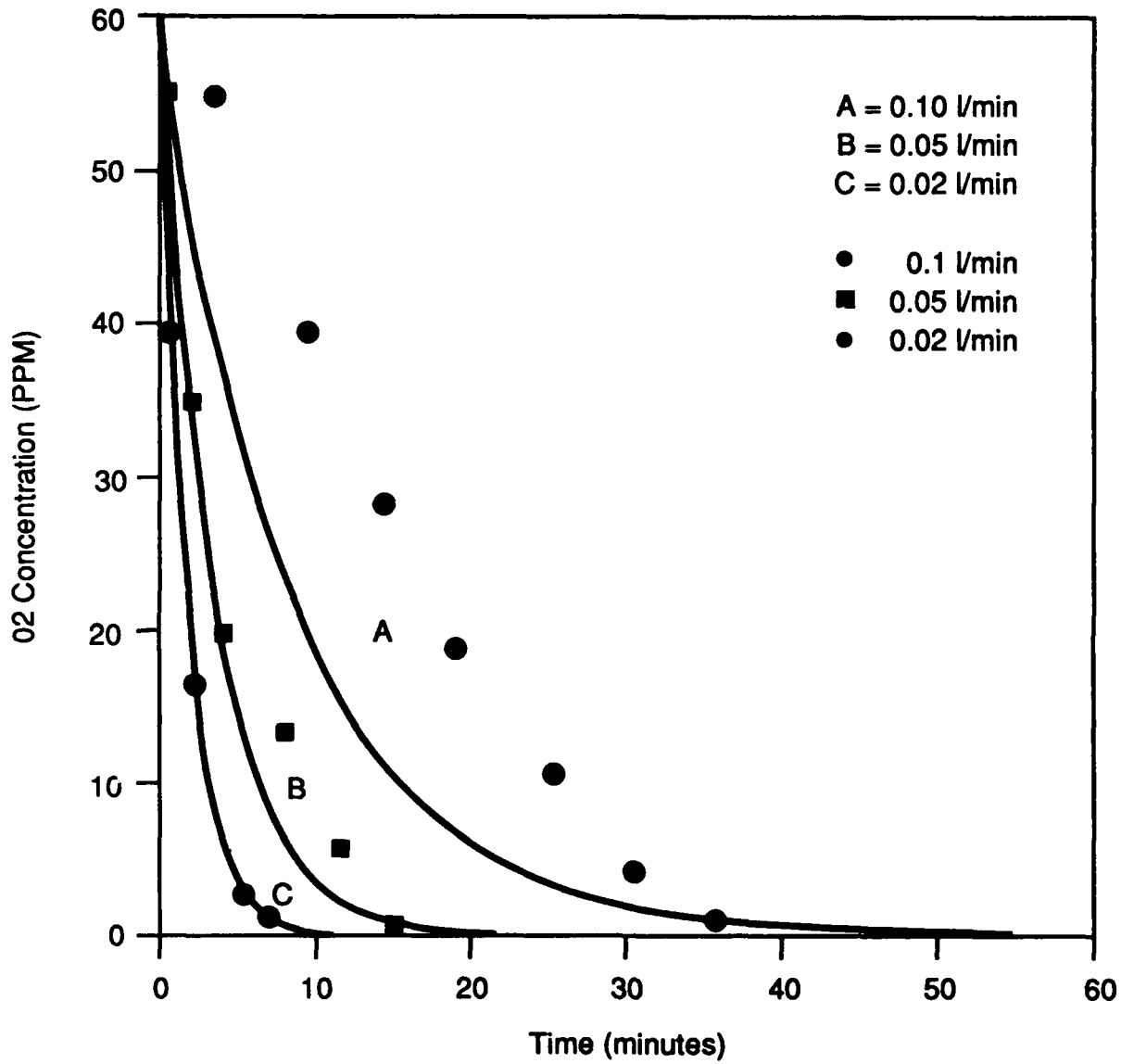


Figure 5. Deoxygenation of 1 l of JP-5 at 22° with 99.999% N<sub>2</sub> at various flow rates and 95% efficiency. Calculations (A,B,C) compared with experimental results (●, ■ ●).

The time and quantity of nitrogen required to deoxygenate a tank of fuel is highly dependent on the fuel flow rate. Figure 6 shows oxygen concentration versus time for several flow rates plus for a previously filled tank. As can be seen, deoxygenating a previously filled tank is the least efficient process. At the given conditions of 60°F, 0.4 lb/min scrub rate, 99.95% N<sub>2</sub> and 95% efficiency, the 250 gal/min flow rate is most efficient because the tank is filled in forty minutes and a 1 ppm O<sub>2</sub> level is reached after 62 minutes. At 100 gal/min the 1 ppm O<sub>2</sub> concentration level is reached in less time, but the tank is not filled until 100 minutes have elapsed. Therefore, more nitrogen is also required to maintain scrub conditions through the longer time interval.

These results show that it is important to optimize the fuel flow and scrub flow rates in order to produce a tank of fuel deoxygenated to the 1 ppm oxygen level most efficiently. Although a fast fuel flow rate will fill the tank more quickly, a slower rate may actually produce deoxygenated fuel more quickly depending on the scrub flow rate. On the other hand, a fast scrub rate may result in excess nitrogen being used if the fuel flow rate is low. Figure 7 illustrates this by showing the deoxygenation of 10,000 gallons of JP-5 with fuel flow rate of 100 gals/min. Scrub flow rates of 0.40 and 0.18 lbs/min were used. Although both produce the desired result in 100 minutes, the time required to fill the tank, the faster scrub flow required an excess of 32 lbs of nitrogen over the slower scrub rate.

Other variables of the system were also tested in these calculations. Figure 8 shows the deoxygenation of JP-4 under the same conditions as the JP-5 shown in Figure 6. Because of the higher initial O<sub>2</sub> concentration, the deoxygenation proceeds more



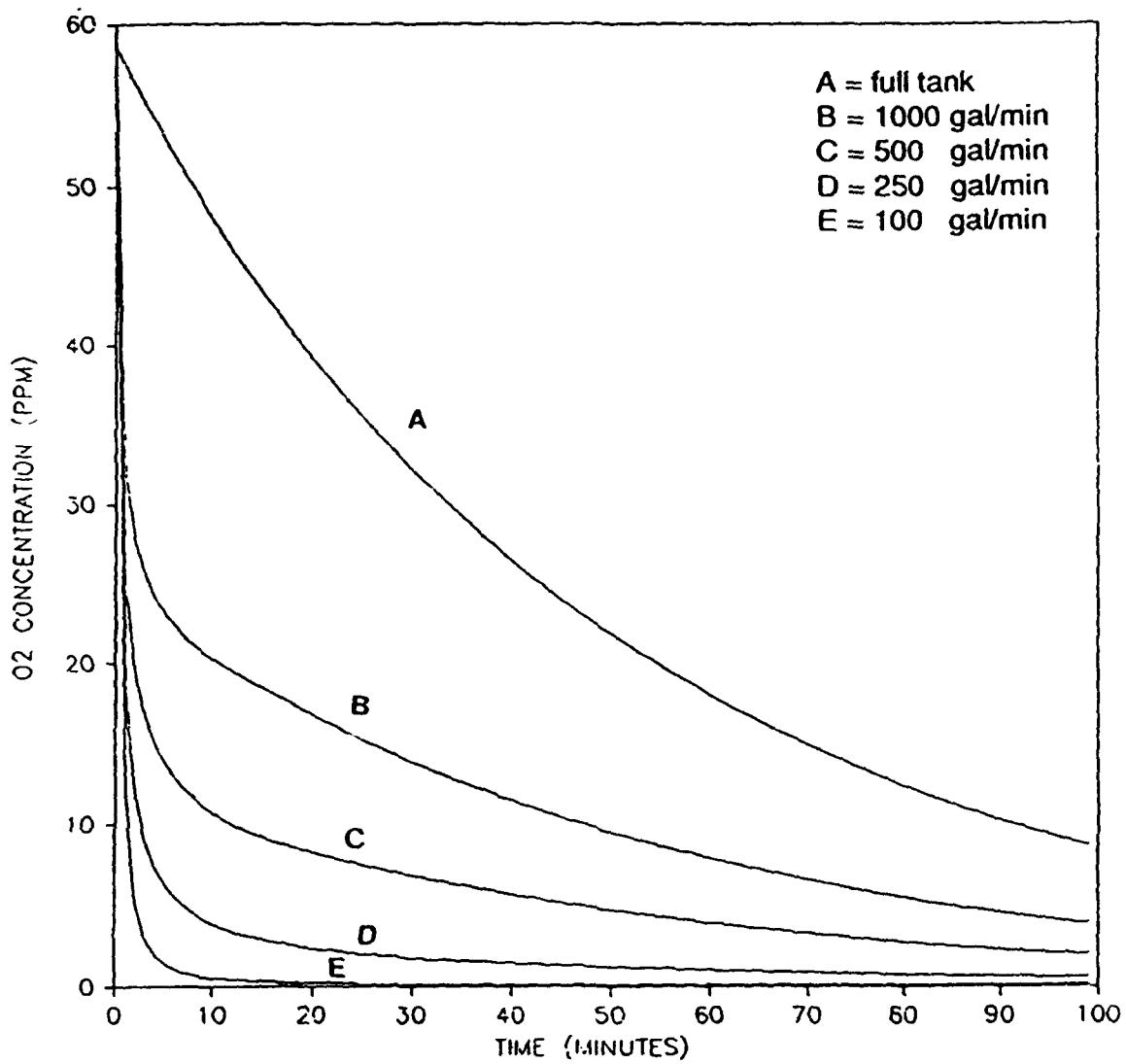


Figure 6. Deoxygenation of 10,000 gals of JP-5 at 60°F and various fuel flow rates. Scrub gas is 99.95% N<sub>2</sub> at 0.4 lb/min and 95% efficiency.

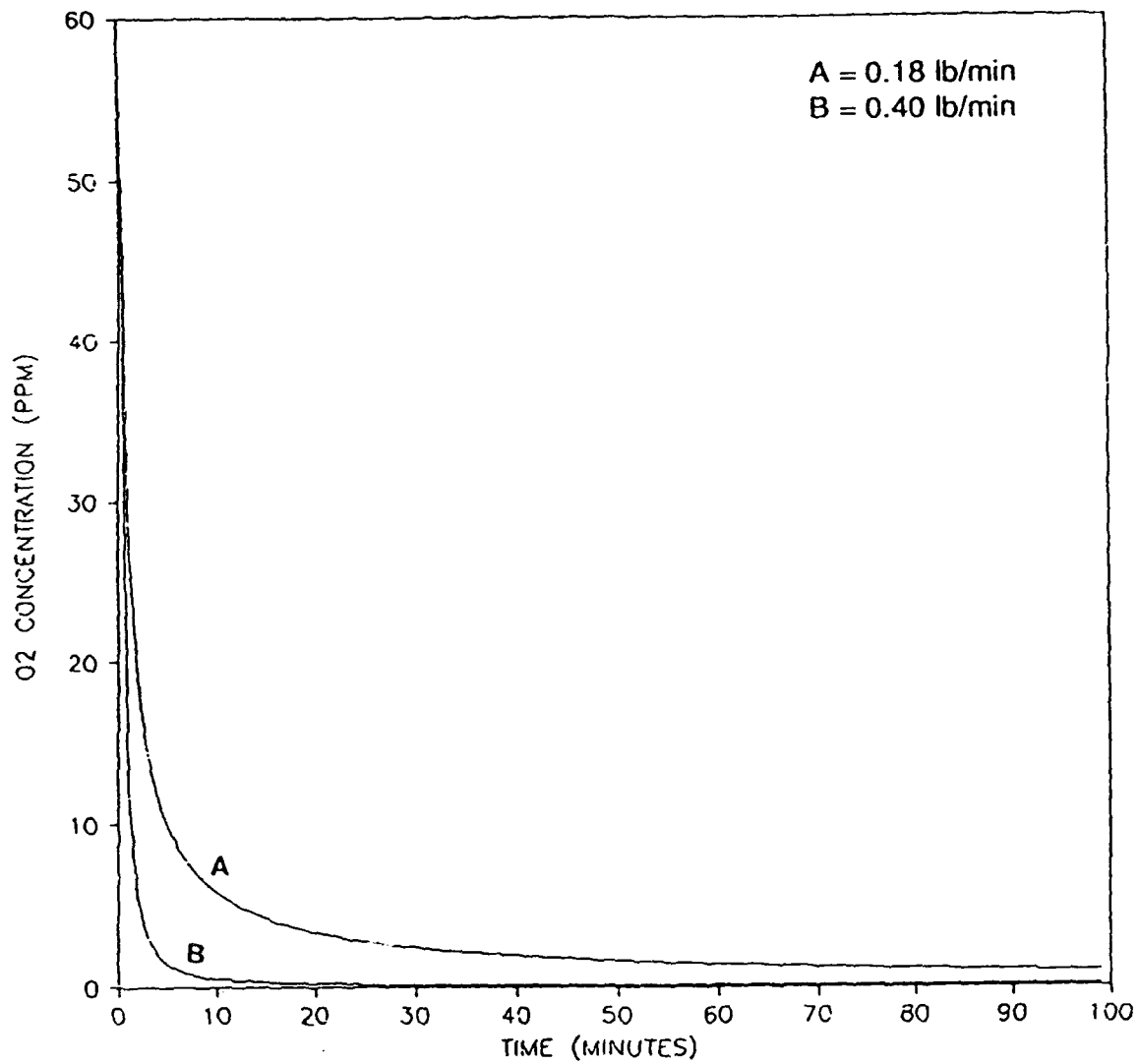


Figure 7. Deoxygenation of 10,000 gals of JP-5 at 60°F and 100 gal/min fuel flow. Scrub gas is 99.95% N<sub>2</sub> at various flow rates and 95% efficiency.

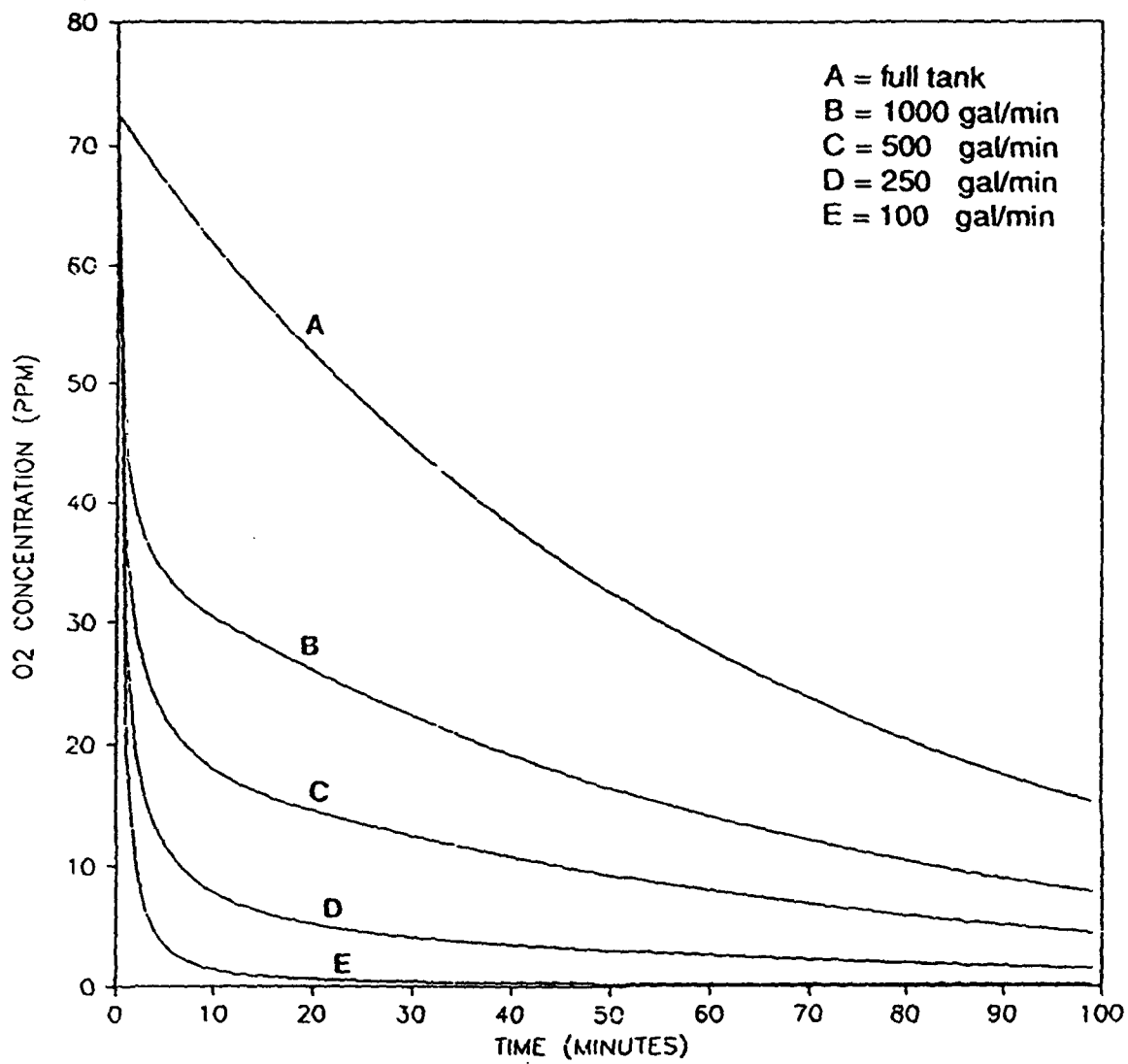


Figure 8. Deoxygenation of 10,000 gals of JP-4 at 60°F and various fuel flow rates. Scrub gas is 99.95% N<sub>2</sub> at 0.4 lb/min and 95% efficiency.

slowly. Simulations in which the temperature of the fuel or upper skin were varied showed a very slight, less than 1 percent, change in deoxygenation rate. As discussed above this conflicts with laboratory results. Also, simulations were performed with the vent valve set at an overpressure. In these cases, the deoxygenation was delayed until sufficient scrub gas was added to equal the overpressure and then the deoxygenation proceeded normally for the given conditions.

## 5.0 CONCLUSIONS

Several methods of deoxygenating jet fuel have been considered and evaluated. These methods were based on both the chemical and physical properties of oxygen. The chemical methods suffer from several disadvantages. The best reducing agents are difficult to handle because they must be protected from air and often react so exothermically as to be a fire hazard. Also, good reducing agents are not easily regenerated after exposure to oxygen. The large volumes of fuel which must be deoxygenated then result in large quantities of used agents which must either be subjected to costly regeneration processes or disposed of in a safe manner.

Molecular sieves do not suffer from these disadvantages because they are inert, nontoxic, nonflammable materials. Also, because they function by the physical process of adsorption rather than by chemical reaction, they are more easily regenerated. The adsorption of gases is an equilibrium process and by placing the sieves under vacuum, the gases can be removed. The disadvantage of sieves is that massive quantities of sieves will be required in order to deoxygenate jet fuel to the low oxygen concentrations required to improve thermal stability.

Sparging fuel with nitrogen gas is also a physical process of removing oxygen involving an equilibrium between the fuel and the atmosphere surrounding it. As the oxygen concentration of the atmosphere is decreased, the oxygen concentration in the fuel also decreases in order to maintain the equilibrium. In this study, it was found that deoxygenation is best achieved by using high purity nitrogen and deoxygenating as fuel flows into a storage container. The fuel flow rate should be adjusted according to the scrub gas flow rate so that a 1 ppm oxygen level is attained in the time that it takes to fill the tank. It is very important that the proper relationship be maintained between the two flow rates in order to most efficiently obtain a full tank of deoxygenated fuel. It may even be more efficient to use a slower fuel flow rate in order to achieve this goal. The scrub gas should also be dispersed into small bubbles in order to present the maximum surface area to the fuel, resulting in maximum efficiency.

Although high purity nitrogen is best for the deoxygenation process, gas which is 99.5% nitrogen is sufficient to maintain the deoxygenated fuel. This number is significant because it is achievable by processing air with hollow fiber membrane filters. Thus, these filters could be used to supply the atmosphere in storage tanks as well as to provide demand gas on the aircraft. This eliminates the need to provide prepurified nitrogen on the aircraft. Such systems are available commercially from Dow Chemical under the trademark GENERON or Permea Inc., a Monsanto Company under the trade name Prism Alpha Nitrogen Systems.