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# DIFFUSION OF TRACE GASES FOR LEAK DETECTION IN AEROSPACE SYSTEMS

By James L. Brown Quality and Reliability Assurance Laboratory

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# DIFFUSION OF TRACE GASES FOR LEAK DETECTION IN AEROSPACE SYSTEMS

By

James L. Brown

## George C. Marshall Space Flight Center Huntsville, Alabama

#### ABSTRACT

This report presents the results of injecting trace gases into systems by various methods. The primary objective was to measure and evaluate the dispersion and diffusion of the trace gases (freon and helium) in various systems.

One series of tests utilized a freon/air mixture of 1 percent by volume. Slug injection and the freon/air premix injector were the insertion techniques utilized. The freon diffusion tests utilized a nonspecific system that consisted of spheres and tubing with sampling ports at five locations. Results of the tests indicated that the various slug injection modes did not produce the desired uniform mixture of freon and air; however, the freon/air premix injector did produce suitable mixing and distribution. The second series of tests utilized a helium/air mixture of 10 percent by volume. The helium slug injection technique was tested on the same nonspecific system and found incapable of producing uniform mixtures. The third series of tests utilized a helium/air mixture of 10 percent by volume in a large cylinder. The helium slug injection technique was tested and found acceptable when injected at the beginning of the pressurization cycle.

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# DIFFUSION OF TRACE GASES FOR LEAK DETECTION IN AEROSPACE SYSTEMS

By

James L. Brown

TEST RESEARCH SECTION APPLIED TECHNOLOGY BRANCH ANALYTICAL OPERATIONS DIVISION QUALITY AND RELIABILITY ASSURANCE LABORATORY

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#### TECHNICAL MEMORANDUM TM X-53742

# DIFFUSION OF TRACE GASES FOR LEAK DETECTION IN AEROSPACE SYSTEMS

#### SUMMARY

Various leak detection techniques are used when searching for leaks in aerospace systems. Slug injection and the freon/air premix injector techniques were the insertion techniques utilized for freon; the slug injection technique was utilized for helium. Measurements were taken of the actual dispersion and diffusion of freon and helium in the various systems.

One series of tests utilized a freon and air mixture of 1 percent by volume in a nonspecific system that consisted of spheres and tubing with sampling ports at five locations. Slug injection and the freon/air premix injector were the insertion techniques utilized. Results of these tests indicated that the various slug injection modes did not produce the desired uniform mixture of freon and air; however, the freon/air premix injector technique did produce suitable mixing and distribution.

The second series of tests utilized a helium and air mixture of 10 percent by volume in the same nonspecific system utilized in the freon tests. The helium slug injection technique was used and found incapable of producing uniform mixtures.

The third series of tests utilized a helium and air mixture of 10 percent by volume in a large cylinder. The helium slug injection technique was tested and found acceptable when injected at the beginning of the pressurization cycle.

The results of these tests indicate that the diffusion of freon and helium does not occur as predicted. The sphere test results indicated that the diffusion of helium or freon in the slug injection mode cannot be relied upon to obtain a satisfactory uniform distribution of the leak detection media in a system consisting of tubing and volumes; however, a proper leak detection media can be obtained by premixing the trace gas and air prior to insertion in the system. In large open systems, such as a booster lox tank, the required freon/air or helium/air mixture can be obtained satisfactorily either by careful slug injection or by premixing the gases.

# SECTION I. INTRODUCTION

Various leak detection techniques are used when searching for leaks in aerospace pneumatic systems. One common technique is the injection of Freon-22 (CHC1F<sub>2</sub>) or helium (U.S. Bureau of Mines, Grade A) into the system to be tested and location of leaks with detection devices such as the General Electric H-2 halogen detector which senses freon, the Uson leak detector which senses various trace gases but is most efficient with helium, or a helium mass spectrometer. Pure freon or helium is seldom used due to the cost unless maximum sensitivity is required. Good practice in leak detection would normally specify freon or helium concentrations in air or  $GN_2$  ranging from 1 to 10 percent by volume. The method of inserting the trace gas into a system under test is usually slug injection, i.e., at some point or points during the pressurization cycle, a specific amount or slug of pure trace gas is inserted in the system. Theoretically, the trace gas diffuses throughout the system, resulting in a uniform mixture of known (by calculation) concentration.

This program was an experimental measurement of the actual dispersion and diffusion of helium or freon in various systems. The results are reported separately with Section II covering the freon diffusion; Section III, the helium diffusion; and Section IV presenting general conclusions.

#### SECTION II. FREON DIFFUSION

#### A. INTRODUCTION

A freen concentration in air of 1 percent by volume was used in this project since this concentration is widely used in leak detection. Two methods of freen insertion were utilized, i.e., slug injection and premixing freen and air. The test utilized a nonspecific system that consisted of spheres and tubing (approximately 12 cubic feet) with sampling ports at five locations (figure 1). After filling this system, samples were drawn from all ports at regular intervals and analyzed with a mass spectrometer to determine the concentration of freen at each location.

#### B. DESCRIPTION OF TEST SETUP

A residual gas analyzer, C.E.C. Model 21-614, with a capillary continuous inlet system, was used to determine the percent of freon in samples drawn from the system. The sensitivity was approximately



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Figure 1. Sphere Test Setup - Freon Diffusion

200 chart divisions per percent freon. Five minute samples were taken at all five ports, 1 through 5 in succession, at 0, 1, 2, 4, and 24 hours after pressurizing the system. The sample lines were purged with missile grade air for 5 minutes after each sampling period. The sensitivity of the residual gas analyzer was determined before and after each sample group was taken.

The premixing of the freon and air was accomplished with the Freon Injector which was developed by Astro-Space Laboratories on a contract from the Methods Research Section, R-QUAL-ATR (evaluated in Internal Note, IN-R-QUAL-66-52).

The complete test setup shown in figure 1 is not a simulation of a specific flight system, but is a nonspecific model consisting of moderately large volumes connected by tubing, i.e., four sets of three interconnected spheres for a total volume of 12 cubic feet with five sampling positions or ports. This setup is representative of aerospace systems with respect to leak detection requirements.

# C. DATA FOR VARIOUS INJECTION MODES

Figure 2 shows the freon distribution as percent freon versus port number (1 through 5) of a freon/air mixture. This mixture was obtained by starting with a purged system at one atmosphere, pressurizing to 1.4 psig with 100 percent freon, and finally pressurizing to 125 psig with missile grade air. If uniformly mixed, this would produce a 1 percent freon/air mixture as shown by partial pressures. The theoretical cylinder shown beneath the graph shows the location of the freon slug if no diffusion or mixing occurs. The curves show the actual distributions measured at intervals of 1, 2, 4, and 24 hours after filling. The distribution at 1 and 2 hours after fill shows that no trace gas was present at ports 1, 2, 3, and 4.

Figure 3 shows the freon distribution in a freon/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 54.0 psig with missile grade air, adding 1.4 psid of 100 percent freon, and finally pressurizing to 125 psig with missile grade air. If uniformly mixed, this would produce a 1 percent freon/air mixture. The theoretical cylinder beneath the graph shows the location of the freon slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution at 0, 1, 2, and 4 hours after fill shows that very little trace gas was present at ports 1 and 5.

N ŝ က AIR 2 0 ŝ FREON 14.7 PSIA AIR + 1.4 PSID FREON, THEN PRESSURIZE TO 125 PSIG W/AIR AIR σ **D** \* LEGEND 24 HOURS **2 HOURS** 4 HOURS 1 HOUR THEORETICAL CYLINDER FREON INSERTION PORT NUMBER ო 1% FREON, 125 PSIG 2 0 DERCENT FREON

DERCENT FREON

Test Results - Freon Slug Inserted at Beginning of Pressurization Cycle Figure 2.



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Figure 4 shows the freon distribution in a freon/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 123.6 psig with missile grade air, and finally pressurizing to 125 psig with 1.4 psid of 100 percent freon. This would produce a 1 percent freon/air mixture if uniformly mixed. The theoretical cylinder shows the location of the freon slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution at 1, 2, 4, and 24 hours after fill shows that no trace gas was present at ports 3, 4, and 5.

Figure 5 shows the freon distribution in a freon/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 0.7 psig with 100 percent freon, pressurizing to 124.3 psig with missile grade air, and finally pressurizing to 125 psig with 0.7 psid of 100 percent freon. This would produce a 1 percent freon/air mixture if uniformly mixed. The theoretical cylinder shows the location of the freon slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution on all the curves shows that very little trace gas was present at ports 2, 3, and 4.

Figure 6 shows the freon distribution in a freon/air mixture obtained by starting with a purged system at one atmosphere and then pressurizing to 125 psig with a premixed 1 percent freon/air mixture supplied by the Freon Injector. The theoretical cylinder shows the location of the premixed freon/air. The curves show the actual distributions measured. The distribution on all the curves shows that significant amounts of trace gas were present at all ports. A flat curve at the selected freon/air percentage, with equal readings from all ports, could be obtained by venting the dead end of the test system until completely purged by the 1 percent mixture coming from the injector. However, this mixture must be vented outside the test area to prevent contamination of the test area atmosphere with resultant loss of detector sensitivity.

#### D. FREON DIFFUSION CHARACTERISTICS

The diffusion patterns shown in figures 2 through 5 were affected by the following two factors:

 The first factor is the method of filling. The test spheres were filled through sample port 1. This resulted in some mixing, but heavy concentrations of freon were evidenced in the area of the theoretical slug.



Test Results - Freon Slug Inserted at End of Pressurization Cycle Figure 4.









(2) The second factor is that at  $70^{\circ}$ F and 125 psig the freon is operating near a phase change. A moderate drop in temperature could result in the temporary liquefaction of some freon.

Theoretically, due to the open structure of gas, all constituents of a mixture of gases will fill the total volume independently of the other gases present. Thus, it was expected that the freon would diffuse rapidly throughout the system and a uniform mixture would be present at all the sampling ports, regardless of how, where, or when during the pressurizing cycle the freon was inserted. It is obvious from the previous discussion of the data curves that in this test the trace gas did not follow the prediction.

A good example of the actual characteristics of Freon-22 is shown in figure 2. The 1 hour curve shows a heavy concentration of freon at port 5 but practically no freon at port 4. After 2 hours, the heavy freon concentration at port 5 had dropped without showing an increase at port 4. After 4 hours, the heavy freon concentration at port 5 had dropped even further and the freon at port 4 had increased significantly but port 3 still had practically no freon. After 24 hours, the freon at port 5 was 68 percent of the original heavy concentration. The 24 hour curve also shows a further increase in freon at port 4; however, no practical amount of freon was found at port 3. A close look at figure 2 indicates that the freon was first concentrated in the dead end due to the filling procedure and then diffused throughout the three end spheres during the first few hours. At 4 hours, the freon had begun to diffuse into the next set of three spheres. If the system had remained pressurized for a sufficiently long period, it is expected that the freon would diffuse throughout the system.

#### E. CONCLUSIONS - FREON DIFFUSION

The various slug injection modes, which were tested in this program, do not produce the desired uniform mixture of freon and air. The data clearly shows that the freon did not mix uniformly or disperse throughout the system. Much of the system had little or no freon while other parts had well above the desired concentrations.

Suitable mixing and distribution of the freon was obtained using the Freon Injector. This was to be expected since the injector premixes the freon and air prior to entering the system. Some dilution occurs at the dead end of the system as a result of the air in the system being partially compressed and only partially mixed with the pressurizing mixture. This could be prevented by venting the dead end during fill.

#### SECTION III. HELIUM DIFFUSION

#### A. INTRODUCTION

A helium concentration in air of 10 percent by volume was used in this series of tests since this concentration is widely used in leak detection. The commonly used slug injection technique was utilized in the following two systems:

- A nonspecific system consisting of spheres and tubing (approximately 12 cubic feet) with sampling ports at five locations. (See figure 7.)
- (2) A lox tank (approximately 1250 cubic feet) with sampling ports at six locations. (See figure 8.)

After filling the systems, samples were drawn from all ports at regular intervals and analyzed with a mass spectrometer to determine the concentration of helium at each location.

B. DESCRIPTION OF TEST SETUP

A residual gas analyzer, C. E. C. Model 21-614, with a capillary continuous inlet system, was used to determine the percent of helium in samples drawn from the system. The sensitivity was approximately 20 chart divisions per percent helium. The five minute samples were taken at all five ports, 1 through 5 in succession, at 0, 1, 2, 4, and 24 hours after pressurizing the system. The sample lines were purged with missile grade air for 5 minutes after each sampling period. The sensitivity of the residual gas analyzer was determined before and after each group of samples was taken. The complete test setup shown in figure 7 uses the same basic equipment as the freon test and is not a simulation of a specific flight system.

The test setup shown in figure 8 is an S-I lox tank, 70 inches in diameter by 50 feet long, with a volume of approximately 1250 cubic feet. Six sampling ports are located as shown.

# C. DATA FOR VARIOUS INJECTION MODES

1. Fiberglass Sphere System. (See figure 7.)

a. Figure 9 shows the distribution as percent helium versus port number (1 through 5) of a helium/air mixture. This mixture was



Figure 7. Sphere Test Setup - Helium Diffusion



Figure 8. Lox Tank Test Setup - Helium Diffusion



obtained by starting with a purged system at one atmosphere, pressurizing to 14 psig with 100 percent helium, and finally pressurizing to 125 psig with missile grade air. If uniformly mixed, this would produce a 10 percent helium/air mixture as shown by partial pressure. The theoretical cylinder shown beneath the graph shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured at intervals of 0, 1, 2, 4, and 24 hours after filling. The distribution at 0, 1, 2, and 4 hours after fill shows that no trace gas was present at ports 1 and 2.

b. Figure 10 shows the helium distribution in a helium/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 48 psig with missile grade air, adding 14 psid of 100 percent helium, and finally pressurizing to 125 psig with missile grade air. If uniformly mixed, this would produce a 10 percent helium/air mixture. The theoretical cylinder beneath the graph shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution at 0, 1, 2, and 4 hours after fill shows that no trace gas was present at ports 1 and 5.

c. Figure 11 shows the helium distribution in a helium/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 111 psig with missile grade air, and finally pressurizing to 125 psig with 14 psid of 100 percent helium. If uniformly mixed, this would produce a 10 percent helium/air mixture. The theoretical cylinder shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution at 0, 1, 2, 4, and 24 hours after fill shows that no trace gas was present at ports 3, 4, and 5.

d. Figure 12 shows the helium distribution in a helium/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 7 psig with 100 percent helium, pressurizing to 118 psig with missile grade air, and finally pressurizing to 125 psig with 7 psid of 100 percent helium. If uniformly mixed, this would produce a 10 percent helium/air mixture. The theoretical cylinder shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution on all the curves shows that very little trace gas was present at ports 2 and 3.



Test Results - Helium Slug Inserted at Midpoint of Pressurization Cycle Figure 10.



DERCENT HELIUM





# 2. Lox Tank System. (See figure 8.)

a. Figure 13 shows the helium distribution in the lox tank as percent helium versus port number (A through E) of a helium/ air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 2.47 psig with 100 percent helium, and then pressurizing to 10 psig with missile grade air. If uniformly mixed, this would produce a 10 percent helium/air mixture. The theoretical cylinder shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured at intervals of 0, 1, 2, and 4 hours after filling. The distribution on all the curves shows that a uniform helium/ air mixture was present at all ports.

b. Figure 14 shows the helium distribution in the lox tank as percent helium versus port number (A through E) of a helium/air mixture obtained by starting with a purged system at one atmosphere, pressurizing to 7.53 psig with missile grade air, and then pressurizing to 10 psig with 100 percent helium. If uniformly mixed, this would produce a 10 percent helium/air mixture. The theoretical cylinder shows the location of the helium slug if no diffusion or mixing occurs. The curves show the actual distributions measured. The distribution at 1, 2, and 4 hours after fill shows that trace gas was present at all ports though not uniformly mixed.

## D. HELIUM DIFFUSION CHARACTERISTICS

Helium should follow general diffusion theory, i.e., the helium should rapidly diffuse throughout the system. The diffusion patterns shown in figures 9 through 14 were affected mainly by the method of filling. The test spheres were filled through sampling port 1, and the lox tank was filled through sampling port E. This resulted in some mixing, but heavy concentrations of helium were evidenced in the area of the theoretical slug.

A good example of the actual diffusion characteristics of helium in the test spheres is shown in figure 11. The 0-hour curve shows a heavy helium concentration at port 1, a small amount at port 2, and no helium at ports 3, 4, and 5. After 1 hour, the heavy helium concentration at port 1 had dropped and the helium concentration at port 2 had increased, but there was still no evidence of helium at ports 3, 4, and 5. After 2 hours, the heavy helium concentration at port 1 had decreased further, while the helium concentrations at ports 2 and 3 increased slightly. Ports 4 and 5 still evidenced no helium.







Figure 14. Test Results - Helium Slug Inserted at End of Pressurization Cycle

PERCENT HELIUM

The trends established after the 1 and 2 hour periods continued for the 24 hour period at which time the helium concentration at port 1 had dropped to a value of 53 percent of the original heavy concentration. At port 2, the helium concentration had increased to a total of 14 percent, but at ports 3, 4, and 5, no practical change was detected. Analysis of figure 11 indicated that the helium was first concentrated near the filling port due to the filling procedure. Then, the helium diffused throughout the first three spheres during the first 2 hours. At this time, the helium had begun to diffuse into the next set of three spheres. An exceedingly long period of time would be required for the helium to diffuse throughout the system since after 24 hours no detectable dispersion had occurred past the second set of spheres.

A good example of the diffusion characteristics of helium in the lox tank is shown in figure 14. The 0-hour curve shows less than the required 10 percent of helium at port E and even less helium at ports C and D, but shows approximately 10 percent helium at ports A, B, and F. After 1 hour, the percentage of helium at ports E, C, and D had increased; however, the percentage of helium at ports A, B, and F had remained relatively constant. After 2 hours, the percentage of helium at ports E, C, and D continued to increase with very little change at ports F, A, and B. After 4 hours, all ports evidenced approximately 10 percent helium. A close look at figure 14 indicates that the helium was dispersed to some extent by the filling procedure but was not uniformly mixed. Then, the helium diffused throughout the unrestricted volume until the entire lox tank contained the 10 percent helium/air mixture.

#### E. CONCLUSIONS - HELIUM DIFFUSION

The various slug injection modes which were tested in this program utilizing the fiberglass spheres do not produce the desired uniform mixture of helium and air. The data clearly shows that the helium did not mix uniformly, diffuse, or disperse throughout the system. Much of the system had little or no helium while other parts had well above the desired concentrations.

The slug injection modes which were tested in this program utilizing the lox tank produced varying results. With the helium slug injected at the beginning of the pressurization cycle, a 10 percent helium and air mixture was obtained immediately throughout the lox tank. However, with the helium slug injected at the end of the pressurization cycle, some ports had less than the required amount while other ports had the required 10 percent mixture. After a period of 4 hours, the helium did diffuse throughout the lox tank and a 10 percent mixture was obtained in all areas.

The tests utilizing the lox tank resulted in better mixtures of helium and air than the fiberglass spheres due to the lox tank being one large open volume with no restrictions to inhibit diffusion and dispersion. The fiberglass sphere system consisted of volumes connected by lines that acted as orifices, decreasing the rate of dispersion and diffusion. The slug injection technique is satisfactory for large, open systems such as the lox tank. especially if the slug is injected in the middle of the pressurization cycle. However, the slug injection technique is unsatisfactory for use in systems such as the fiberglass spheres, regardless of when the slug is injected during the pressurizing cycle. The 10 percent helium and air mixture for this type system must be obtained by premixing the gases. This premixing can be accomplished by utilizing a pressure vessel with sufficient capacity to pressurize the system under test. The helium slug should be inserted into this vessel in three parts; at start of pressurization, at midpoint, and at the end of the pressurizing cycle. The gas mixture should be allowed to diffuse for a minimum of 4 hours; then the high pressure bottle can be used to pressurize the system under test with results similar to those given in figure 6, i.e., a relatively constant percentage of helium. The dead ends of the system should be vented during fill to eliminate the dilution of the mixture depicted in figure 6.

## SECTION IV. GENERAL CONCLUSIONS

The results of this project indicate that the diffusion of freon and helium does not occur as predicted. According to Graham's law concerning theoretical gas mixtures, helium should diffuse in air 4.7 times as fast as Freon-22. However, in the fiberglass sphere system the helium diffusion rate was approximately equal to that of freon. Therefore, in this and similar type systems, speed of diffusion is not a valid basis for selection of a trace gas.

In any system to be leak checked by the use of trace gases, the validity of the test is predicated on the uniform distribution of the trace gas throughout the system. Since the leak detection devices only react to the presence of the trace gas, a leak in a portion of the system not containing the trace gas would go undetected. The sphere test results indicated that the diffusion of helium/air or freon/air in the slug injection mode cannot be relied upon to obtain a satisfactory uniform distribution of the leak detection media in a system consisting of tubing and volumes. A proper leak detection media can only be obtained by premixing the trace gas and air prior to insertion in the system. As mentioned in both the helium conclusions and the freon conclusions, the premixing is even more effective if the dead ends of the system are vented, purged with the premixed test gas, and then pressurized to the required test pressure.

In large open systems, such as the lox tank, the required freon/air or helium/air mixture can be obtained satisfactorily either by careful slug injection or by premixing the gases.

This series of tests has shown that the uniform mixing of the trace gas does not always occur and that some portions of the system are likely to be completely void of the trace gas. Thus it is mandatory that not only the method of trace gas insertion be analyzed to ensure uniform distribution but also the system under test be evaluated to determine the optimum fill location(s) and the required vent points. June 10, 1968

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#### APPROVAL

## DIFFUSION OF TRACE GASES FOR LEAK DETECTION IN AEROSPACE SYSTEMS

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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