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By Lawrence R. Greenwood
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SUMMARY

A research program is being conducted at the NASA Langley Research Center to determine the effects of vacuum exposure on solid rocket fuels. As part of this program the outgassing rates of two solid rocket fuels were measured in vacuum at ambient temperature. The rate of rise method was used to make the outgassing measurements. A series of calibrated leaks were measured to validate the measuring system.

The two fuels investigated were composites having polybutadiene acrylic acid (PBAA) and polyurethane as their binders. The outgassing rates were measured as a function of storage time in vacuum.

The outgassing rate for the polybutadiene acrylic acid fuel varied from 1.15×10^{-6} torr-liters/sec cm^2 after 1 hour of vacuum exposure to 8.32×10^{-9} torr-liters/sec cm^2 after 502 hours. The outgassing rate for the polyurethane fuel varied from 3.02×10^{-7} torr-liters/sec cm^2 after 17 hours of vacuum exposure to 3.17×10^{-8} torr-liters/sec cm^2 after 452 hours. The outgassing rates of both fuels decreased rapidly during the first 100 hours and then decreased more slowly with time. After about 500 hours in vacuum, the outgassing rate of both fuels was continuing to decrease.

INTRODUCTION

The effect of the space environment on solid-propellant rocket motors during space storage has become of concern. Extensive effort is being directed toward determining possible vacuum degradation of the propellant during this storage. A research program is being conducted at the NASA Langley Research Center to determine the effects of

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*The information presented herein is taken in part from a thesis entitled "Determination of the Outgassing Rate of Two Solid Propellant Rocket Fuels in a Vacuum" offered as partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering, Virginia Polytechnic Institute, Blacksburg, Virginia, May 1965.

vacuum exposure on solid rocket fuels. As part of this program, experiments will be conducted in vacuum facilities with samples of solid fuels. In the design of these experiments, the outgassing characteristics of these solid fuels are needed. If the outgassing rates are known, the experimenter may be assured that the desired vacuum level will be attained in the vacuum system.

The rate of pressure rise in a known volume was used to measure the outgassing characteristics of the propellant samples. The fuels investigated were composites with polybutadiene acrylic acid (PBAA) and polyurethane binders. The outgassing rates of these two fuels were measured as a function of storage time in vacuum for periods of 502 hours for PBAA and 452 hours for polyurethane. Two additional samples of polyurethane were also tested over a short storage period to compare measured outgassing rates of different samples of the same propellant from one propellant mix.

SYMBOLS

The units for the physical quantities used herein are presented primarily in the International System of Units (SI). However, in some instances, the units are given in both the SI system and in the U.S. Customary System.

A	area, cm^2
i^-	emission current, milliamperes
p	pressure, torr
Q	outgassing rate, torr-liters/sec
S	pumping speed, liters/sec
t	time, seconds, minutes, or hours
V	volume, liters

METHOD USED TO MEASURE OUTGASSING RATES

The rate of rise method was used to measure the outgassing rates of the propellant samples in vacuum. In this method the rate of pressure rise in a known volume at a given time was measured and, using this measurement, the outgassing rate of the sample at that storage time was calculated. This method has been used by other investigators to measure the outgassing rates of various materials. (See refs. 1, 2, and 3.)

The basic relationship used in the rate of rise method can be obtained by considering the equation for the mass balance in a simple vacuum system. For a vacuum system with a test chamber of volume V and a pump with a speed S, the mass balance equation, at constant temperature, is

$$-V \frac{dp}{dt} = Sp - Q \qquad (1)$$

where p is the pressure in the test chamber at any time t and Q is the total outgassing rate in the test chamber. From equation (1) it can be seen that if at a given time the pumping speed S is reduced to zero by closing a valve placed between the test chamber and the pump, and if the subsequent rate of pressure rise in the test chamber of known volume is measured, then the total outgassing rate at that time can be calculated from the equation

$$Q = V \frac{dp}{dt} \qquad (2)$$

Equation (2) is the basic relationship used in the rate of rise method.

The outgassing rate of a sample at a given time was determined with the rate of rise method by using the following procedure. At a given time, the valve between the test chamber and the pump was closed and the increase of pressure with time was noted. These pressure-time data were used with equation (2) to calculate the outgassing rate. These steps were repeated periodically for the duration of the vacuum exposure, and, in this way, the outgassing rate was determined as a function of vacuum storage time. In these instances the total outgassing rate consists of the outgassing rate of the test chamber and associated appendages. The sample was then placed in the test chamber and the sequence repeated for approximately the same duration of time. In these second instances the total outgassing rate consists of the outgassing rate of the propellant sample and of the test chamber and associated appendages. The difference between these two outgassing rates at a given time is the outgassing rate of the sample alone. The outgassing rate of the sample alone was calculated at each test condition and presented as a function of vacuum storage time. All measurements were made at ambient temperature.

APPARATUS AND TEST PROCEDURE

A sketch of the apparatus used in making these outgassing measurements is shown in figure 1 and a photograph of the apparatus is shown in figure 2. A 4-inch (10.2-cm) oil diffusion pump was used as the main vacuum pump in conjunction with a mechanical roughing pump. Above the diffusion pump were water and liquid nitrogen baffles to reduce backstreaming of oil from the diffusion pump. This particular diffusion pump is

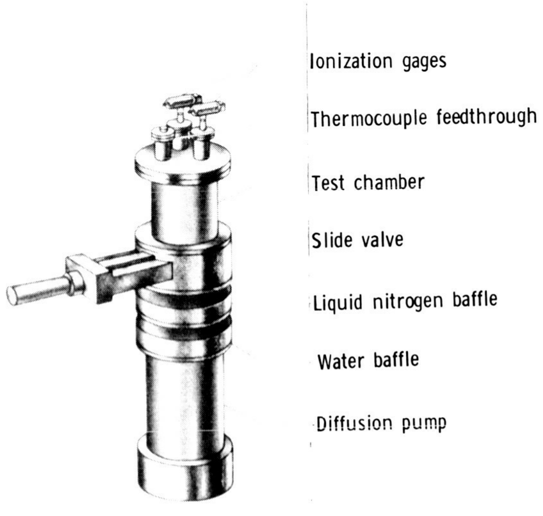

Figure 1.- Sketch of apparatus used to measure outgassing rates.

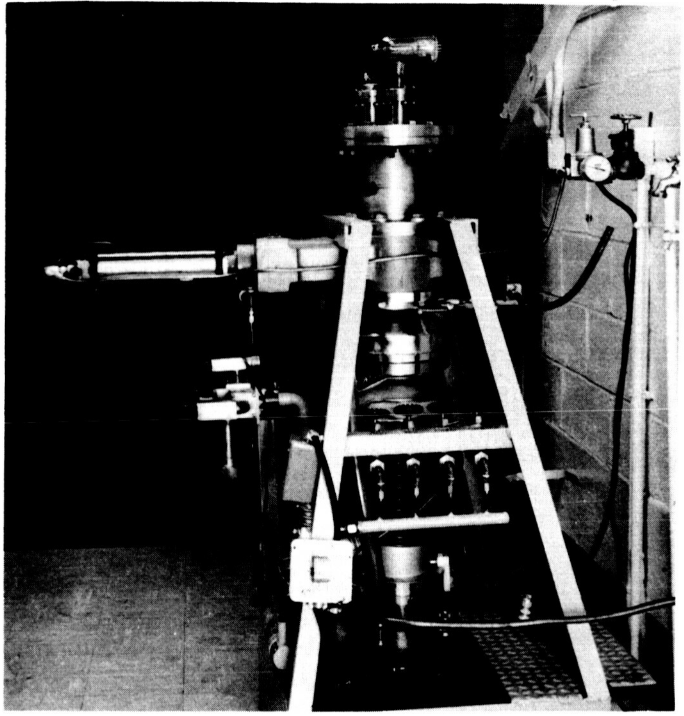

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Figure 2.- Photograph of apparatus to measure outgassing rates.

equipped with a cold cap above the top jet to further reduce backstreaming. The liquid nitrogen level in the liquid nitrogen baffle was maintained by an automatic-level-control device and used a 100-liter liquid nitrogen Dewar flask as a storage supply. The slide valve above the liquid nitrogen baffle was air-operated. The air operation permitted rapid closure of the valve. The valve had a buna-N O-ring on the test chamber side that was compressed to make the vacuum seal when the valve was closed. The test chamber located above the valve had an internal volume of 3.5 liters and was constructed of 347 stainless steel. A sketch showing details of the test chamber is shown in figure 3. A 10-inch-diameter (25.4-cm-diameter) stainless-steel flange with openings for three feedthroughs was sealed to the top of the test chamber by a crushable aluminum seal. On two of the openings on the flange were mounted ionization gages for monitoring the test volume. The gages are the Bayard-Alpert type. While two gages were generally mounted on the system, only one gage, designated the primary gage, was used to record data for the tests. The second gage served as a spare and was occasionally used to provide an independent check on the system behavior. During some tests only the primary gage was mounted on the system. A thermocouple feedthrough was located on the third opening. This thermocouple feedthrough allowed thermocouple wires to be placed on the propellant sample and on the chamber walls. The connections between the 10-inch (25.4-cm) flange and these three feedthroughs were made with 2.75-inch (6.99-cm) flanges. These flanges use a copper gasket to make the vacuum seal. A photograph of the top flange with ionization gages and thermocouples installed is shown in figure 4.

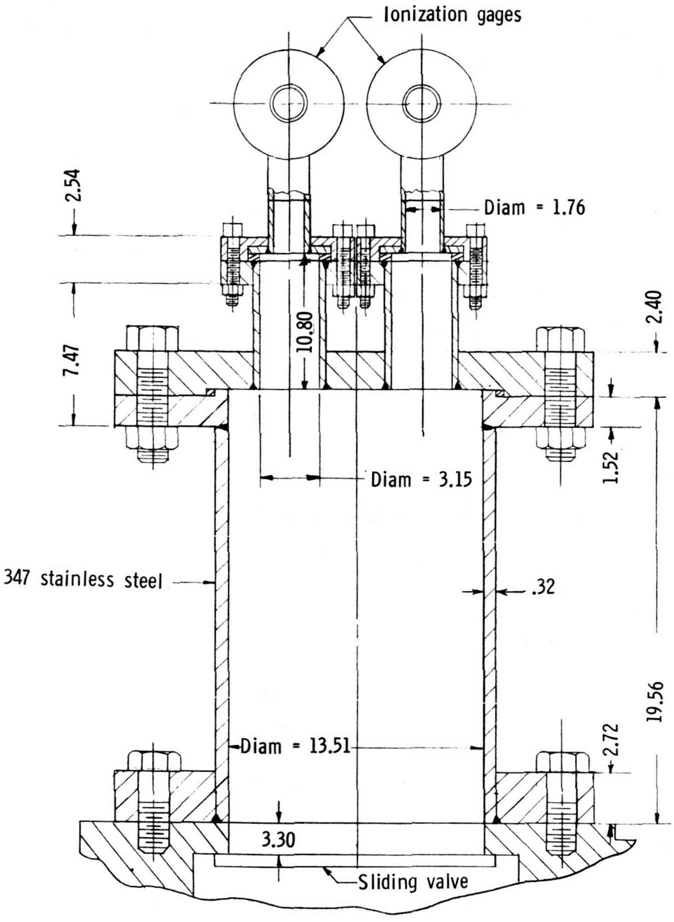

Figure 3.- Details of test chamber. All dimensions are in centimeters.

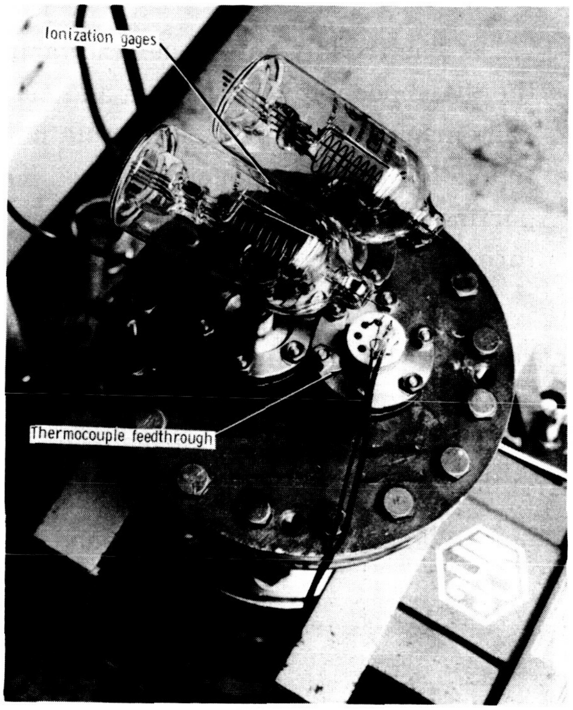

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Figure 4.- Photograph of top flange with ionization gages and thermocouple feedthrough installed.

The primary measurements made during the investigation were test-chamber pressure, rate of pressure rise in the test chamber after closing valve, sample and test-chamber wall temperature, and vacuum exposure time. The test-chamber pressure was measured by using the primary ionization gage and the mating gage controller. The rate of pressure rise in the test chamber was obtained by putting the output of the primary ionization gage into a recorder which produced a trace of the variation of pressure with time after the valve was closed. The temperatures of the sample and of the test-chamber walls were measured with thermocouples. Vacuum exposure time was measured with an electric clock.

To establish confidence in the rate of rise technique for measuring outgassing rates, calibrated leaks were installed on the 2.75-inch (6.99-cm) opening that normally had the thermocouple feedthrough attached. The three calibrated leaks were measured which had leak rates of 2.52×10^{-4}, 8.74×10^{-5}, and 2.28×10^{-5} torr-liters/sec. These leaks were calibrated by a method suggested by Albert Nerken in reference 4.

Once the system was closed, it was important to ascertain that no leaks were present. To check the system for leaks, a helium-type leak detector was used. This leak detector uses helium as a tracer gas to find very small leaks and has a sensitivity of 2×10^{-10} torr-liters/sec.

The freshly cut propellant sample was placed into the test chamber by removing the thermocouple feedthrough and suspending the sample by thermocouple wires. A photograph of a propellant sample mounted on the thermocouple wires is shown in figure 5. The thermocouple wires measure the temperature of the sample while supporting it in the test chamber. After installation of the sample, the thermocouple feedthrough was placed on the 10-inch (25.4-cm) flange and the system resealed.

The roughing pump was then started and when a system pressure of 50 microns was reached, the diffusion pump was also started. At this time, and before the diffusion pump had time to warm up, the water baffle and liquid nitrogen baffle were filled to reduce backstreaming of oil from the diffusion pump. As soon as the pressure reached 10^{-5} torr in the chamber, a leak check was made of the system.

When it had been established that the system did not have leaks the test program was begun. During the tests the primary ionization gage was used to measure the pressure in the test volume. The method used for cleaning the gage in preparation for a pressure measurement was electron bombardment. Electron bombardment of the gage for 15 minutes proved to be a satisfactory method of cleaning. After the gage was bombarded, 20 minutes were allowed for the gage to cool and return to equilibrium before a pressure measurement was made. At 20 minutes after terminating electron bombardment of the ionization gage, the test-chamber pressure was noted, the air-operated slide valve was closed, and the pressure rise in the test chamber was recorded. The gage controller, gage emission current, and recorder were adjusted to record the variation of pressure with time over the pressure range from about 1×10^{-5} to 10×10^{-5} torr. When the pressure reached about 10×10^{-5} torr, the slide valve was opened and the system pumped back down. The gage-cleaning

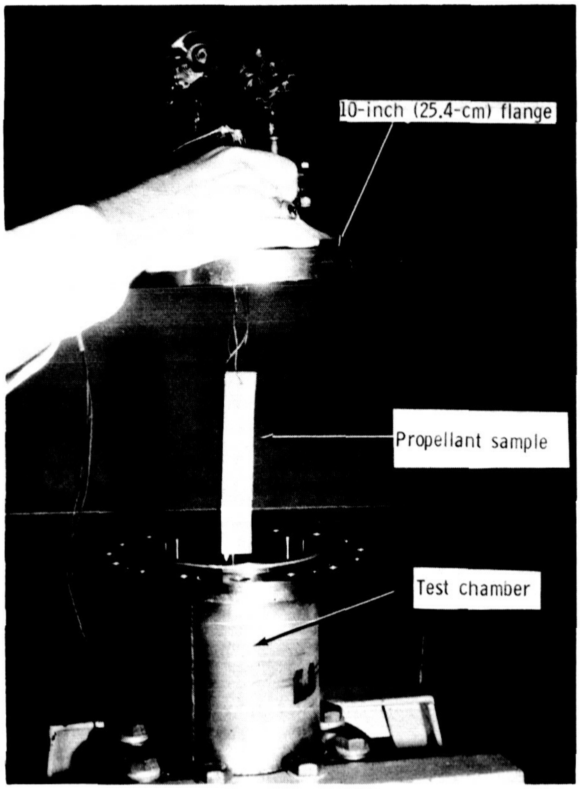

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Figure 5.- Photograph of a propellant sample mounted on thermocouple wires.

procedure and test procedure were then repeated at the next test condition. Generally, data were recorded at intervals of 1 to 3 hours during periods when the system was attended. During unattended periods, the vacuum conditions were maintained and the liquid nitrogen and water baffles were kept filled. By using these procedures, data were obtained over the entire duration of vacuum exposure of about 450 to 500 hours. For the duration of the test, the apparatus was at ambient temperature and the sample and test-chamber-wall temperatures were recorded.

To obtain the outgassing rate at a particular time, the pressure-time records and equation (2) were used. Thus, the test volume and the slope of the pressure-time trace at each test condition must be obtained. The test volume is fixed for a particular series of tests and is equal to the sum of test-chamber volume and the volume of the ionization gages minus the volume of the sample. The slope of the pressure-time trace was obtained directly from the recorded data. A typical pressure-time trace for the system with a propellant sample is shown in figure 6. (The early part of the trace shows the transient condition created when the valve was closed.) Near the end of the trace is the sudden drop in pressure caused by the opening of the valve. The intermediate portion of the trace was generally linear and the slope was taken over this region.

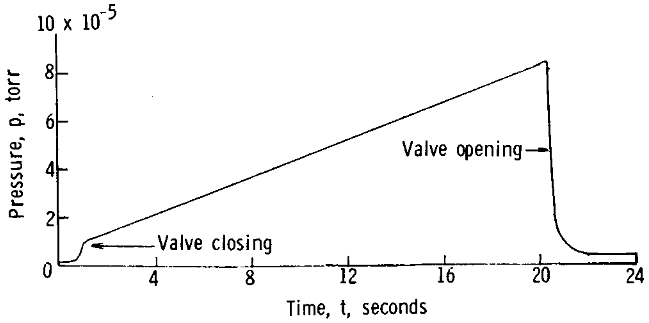

Figure 6.- Typical trace of variation of pressure with time for system with propellant.

In addition to the outgassing measurements made for the system with a propellant sample, measurements were also made by using the same procedures for the empty test chamber. For these tests the intermediate portion of the pressure-time record was not linear as in the tests with a propellant sample. A typical pressure-time trace for the empty system is shown in figure 7. In these instances the slope was taken over the later portion of the trace where it becomes more nearly linear. A possible reason for deviations from linearity for the empty-system traces and the reasons for selecting the later portion over which to take the slope will be discussed in the section entitled "Accuracy and Errors."

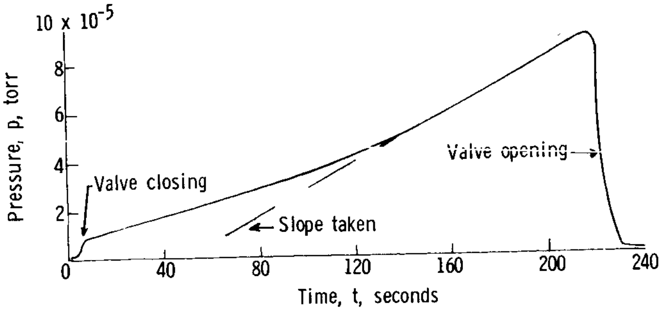

Figure 7.- Typical trace of variation of pressure with time for empty system.

The outgassing rate of a propellant sample alone at a given test condition was obtained by subtracting the outgassing rate for the empty system from the outgassing rate for the system with a propellant sample.

It should be noted that the pressure indicated by an ionization gage is dependent upon the composition of the gas being measured. Since the composition of the gas in the test chamber during these measurements was not known, the pressures and outgassing rates are reported in equivalent nitrogen units except for the measurements of the calibrated air leaks where the pressures and outgassing rates are reported in true air pressure.

PROPELLANT SAMPLES

The two propellants investigated were composites having polybutadiene acrylic acid (PBAA) and polyurethane as their binders. The oxidizer used in both of these fuels is ammonium perchlorate. The propellant samples were mixed and cast at the Langley Research Center. The propellant was deaerated after mixing and prior to casting and curing. After casting and prior to testing, the samples were stored at approximately 50-percent relative humidity and 75° F (297° K) which is in accord with the recommended practices in reference 5.

The samples used were cut from a propellant block. The samples were freshly cut on all surfaces prior to testing. The PBAA sample was a parallelepiped with dimensions of approximately 3.65 cm by 0.65 cm by 8 cm with a volume of 0.019 liter and surface area of 73.5 cm^2. The polyurethane samples were parallelepipeds with dimensions of sample 1 being approximately 15 cm by 3 cm by 1.5 cm with a volume of 0.0675 liter and surface area of 144 cm^2. The surface area of polyurethane samples 2 and 3 was essentially the same as that for sample 1. The density of both propellant types is approximately 1.72 $grams/cm^3$. A photograph of a typical polyurethane sample is shown in figure 8.

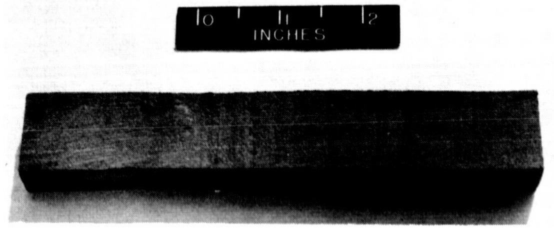

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Figure 8.- Photograph of typical polyurethane propellant sample.

Normally, solid rocket fuel is mixed in large batches. The propellant samples used for the present investigation were taken from batches which were mixed in 50- to 100-pound (21.68- to 45.36-kg) quantities. In these batches there is some concern that the mixture might not be homogeneous and thus the outgassing properties of a single sample might not be representative of the batch. In order to determine the extent of variations in outgassing rate among samples from the same propellant batch, two additional polyurethane samples were taken from different locations in the batch from which polyurethane sample 1 was taken.

Since safety precautions precluded the propellant samples from being stored immediately adjacent to the test apparatus, the samples were exposed to ambient temperature and humidity for approximately 1 hour during transfer from the storage site to the testing apparatus.

ACCURACY AND ERRORS

Ionization Gages

The primary ionization gage was used to make all pressure measurements reported herein. The ionization gage and controller used for measurement of the calibrated leaks was calibrated for nitrogen gas with an orifice calibration system similar to that described in reference 6. It is estimated that this calibration system is accurate to within 8 percent. For the ionization gage and controller used for all other measurements, the manufacturer's calibration was used and an independent calibration was not conducted. It is estimated that this gage indicates true equivalent nitrogen pressure to within 20 percent.

Recorder

The output of the ionization gage was plotted as a function of time by a direct writing instrument which uses a D'Arsonval galvanometer. The pressure indication of the recorder was calibrated frequently against the calibrated ionization gage and controller. The timing marks on the pressure-time record could be read to 0.1 second.

Volume

The volume of the test chamber was calculated from micrometer measurements and found to be 3.500 liters. The volume of the ionization gages was found by measuring the amount of fluid required to fill the gages and was found to be 0.233 ± 0.001 liter per gage. The volume of the propellant samples was calculated from their measured dimensions and is estimated to be accurate to within ±0.005 liter.

Combined Effect

The maximum possible error in the measured quantities indicates that the accuracy of the outgassing rates presented herein are accurate within the limits shown in table I.

Other Errors

When equation (2) is used to calculate outgassing rates from the measured data, two assumptions are implicit:

(1) When the slide valve is closed, the pumping speed in the test volume is zero, and

(2) The only gas entering the test volume after the slide valve is closed is from outgassing and not from leaks.

Deviations from these assumptions could introduce errors into the rate of rise technique.

In the first assumption, there are several possibilities which could result in a nonzero pumping speed. One possible method of pumping is by a leak between the test volume and the diffusion pump through the slide valve. To insure that a leak did not exist, the valve was checked with a leak detector. When the leak detector was attached to the foreline so that all the exhaust from the diffusion pump was passing through it, no leak was detected. The leak detector was calibrated and found to have a sensitivity of 2×10^{-10} torr-liters/sec. If leaks had been present, the aggregate must have been smaller than 2×10^{-10} torr-liters/sec and leaks of this magnitude would not contribute a measurable error when measuring outgassing rates of 10^{-6} torr-liters/sec and higher. This test was repeated at regular intervals during the test program to insure that the valve had not developed a leak during normal use. These procedures assured that, during this investigation, leaks through the slide valve could not result in spurious pumping in the test volume.

There are two other processes that could remove gas molecules from the test chamber and thus cause the pumping speed in the test chamber to be nonzero. The first of these processes is the pumping of gas molecules by the ionization gage. Pumping action of the ionization gage may be ionic, as well as chemical. In reference 7, Alpert points out that measured pumping rates for ionization gages vary from a few liters per minute for inert gases to greater values for the more active gases. Since the composition of the gases being measured was not known, the magnitude of this error could not be evaluated for the present tests. It is of interest to note, however, that the ionic pumping by the ionization gage depends on the number of molecules ionized, which is a direct function of the emission current. Thus, Alpert and others have concluded that the ionic pumping action of the gage is a function of the emission current. (See refs. 1 and 7.)

The second method of pumping gas molecules from the test chamber, and in so doing be a source of error, is adsorption pumping by the walls of the test chamber and by the envelope of the ionization gage. Adsorption pumping may take place as a result of the rapid change in pressure due to the slide valve closing. Prior to the valve closing, the system is at pressure of 10^{-8} to 10^{-9} torr. The internal surfaces are in equilibrium at this lower pressure so that the gas desorption rate is equal to the adsorption rate. When the valve is closed the pressure increases to about 1×10^{-5} torr in less than 1 second. With this rapid increase in pressure, the rate of adsorption could increase and disturb the equilibrium which previously existed. Thus some period of time would be required before the rate of adsorption was again equal to the rate of desorption and equilibrium was reestablished. During this time, more gas could be adsorbed than desorbed and produce a net removal of gas molecules from the system. Efforts were made to determine the rate at which the adsorption takes place in systems such as the one used in this investigation with the hope of evaluating the magnitude of the error introduced by adsorption pumping. However, very little has been reported on adsorption of gases on stainless steel at low pressures. This lack of adsorption data, coupled with the fact the composition of the gas in the test chamber, which would be adsorbing on the internal surfaces, was unknown, prevented a definitive evaluation of the magnitude of the error introduced by adsorption pumping. Adsorption effects have been noted in other experimental investigations. For example, in reference 1, Markley concluded that a pressure-time trace with a characteristic shape, as shown in figure 7, is a result of adsorption pumping. It should be noted that if no gas were being removed from the system, the pressure-time trace should be linear. In the case shown in figure 7 the trace is not linear, and it is suggested that gas molecules are being removed from the system by adsorption. This nonlinearity, thought to be characteristic of adsorption pumping, was found only with the lower starting pressures and is consistent with the fact that a more pronounced effect of adsorption would be expected for lower starting pressures. Thus, while there is evidence that adsorption was taking place during some of the measurements made during this investigation, the magnitude of the error introduced by these effects could not be ascertained.

Thus the errors introduced in the rate of rise technique by ionization gage pumping and by adsorption pumping could not be ascertained for the present investigation by considering these pumping mechanisms individually. Because of this uncertainty, and to insure that other factors which could produce errors were not overlooked, it was decided to calibrate the rate of rise measuring technique. This calibration consisted of using the rate of rise technique to measure a series of known outgassing rates (calibrated leaks) and comparing the known rates with the measured rates. This calibration is discussed in the next section of this report.

Considering the second assumption, it is necessary to insure that gas was not leaking into the test chamber from spurious sources. To do this, the leak detector was again used, but, in this instance, the exterior of the system was blanketed with helium gas. Since no leaks were detected, the aggregate of all leaks present must have been less than 2×10^{-10} torr-liters/sec and leaks of this magnitude would not contribute a measurable error when measuring outgassing rates of 10^{-6} torr-liters/sec and higher. These leak checks were repeated at regular intervals during the test program to insure that leaks did not develop. These procedures assured that, during this investigation, gas was not leaking into the test chamber from spurious sources.

It should be pointed out that pressure-time traces with nonlinear intermediate portions, such as the one shown in figure 7, can introduce errors into the measured outgassing rates for the empty system because the region over which the slope should be taken cannot be determined with certainty. For these traces it was thought that adsorption pumping was responsible for the nonlinearity and that the adsorption effects would be expected to diminish with time. For this reason the slope was taken over the latter part of the trace where the trace becomes more nearly linear. During the latter part of the trace the pressure is higher and the walls of the test chamber and envelope of the ionization gage have had more of an opportunity to come into equilibrium. It should be emphasized, however, that errors induced in the outgassing measurements of the empty system are not necessarily reflected in a substantial error in the outgassing rates of the propellant sample. For instance, the empty-system contribution is always lower than the system-propellant combination and, as long as it is one-half the system-propellant value or less, a given percentage error in the system outgassing rate results in a lesser percentage of error in the propellant-sample outgassing rate. In this program, the empty-system rate was less than one-half the outgassing rate of the system-propellant combination except for a few measurements with the PBAA sample between 450 and 500 hours. Thus, errors in measurement of the empty-system outgassing rates due to deviations of the pressure-time trace from linearity tend to have only small effects on the outgassing rate of the propellant samples. For example, for polyurethane at 239 hours, a 10-percent error in measurement of the empty-system outgassing rate would cause an error of only 2 percent in the calculation of the outgassing rate of the propellant sample.

Corrections

No corrections have been made to any of the data presented herein for the errors discussed in the preceding sections.

CALIBRATION OF MEASURING TECHNIQUE

Calibration tests were performed with the following objectives:

(1) To determine the test conditions most conducive to accurate outgassing measurements

(2) To determine the acceptability of the rate of rise method for measuring outgassing rates over a limited outgassing range

In normal operation of the ionization gage, the components of the gage become contaminated and require cleaning before use. During preliminary tests it was observed that the time elapsed after cleaning the ionization gage had a measurable effect on the measured outgassing rate. In order to establish the magnitude of this effect and to develop test procedures, a series of preliminary tests were conducted. Outgassing measurements of the empty system and with PBAA and polyurethane samples were made at various times after the ionization gage had been cleaned by electron bombardment for 15 minutes. For these preliminary tests, the propellant sample sizes were about the same as those for the final tests. These tests were conducted with two gages, the primary and a second gage, mounted on the system and operating at 1-mA emission current for PBAA and the empty system. With the polyurethane sample difficulty was experienced in cleaning the ionization gages, and therefore the gages were operated at 10-mA emission current. The higher emission current assisted in keeping the gages clean. The results of these measurements are presented in figure 9 and table II. For the polyurethane sample, there was a slight variation in the measured outgassing rate during the first 10 minutes after cleaning the gage; subsequently, there was little variation with increased time. The measured outgassing rate of the PBAA sample increased during the first 15 minutes after degassing the gage and then remained relatively constant for the next 20 minutes. For the empty system the measured outgassing rates remained constant for about 10 minutes, increased for the following 10 minutes, and then remained relatively constant for the next 30 minutes. It is believed that the variations in the measured outgassing rates evident in figure 9 during the first 20 minutes after cleaning the gage are due to transient conditions within the gage. Thus the gage-cleaning technique has no further influence on the measured outgassing rates after 20 minutes following completion of electron bombardment. For this reason a test procedure was established wherein the ionization gage was cleaned by electron bombardment for 15 minutes and the outgassing measurements

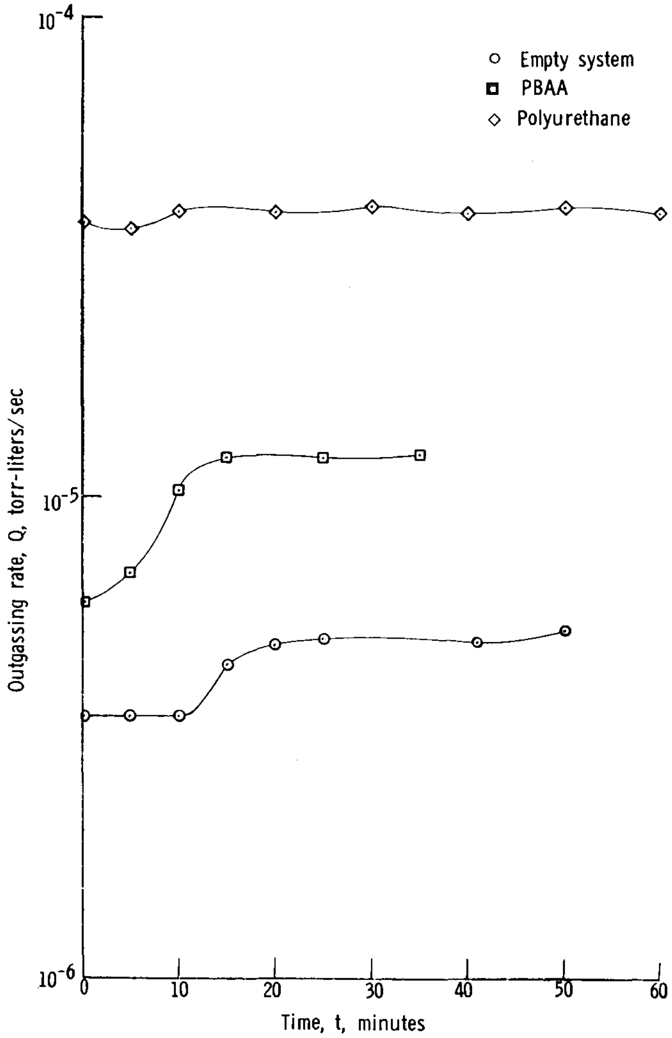

Figure 9.- Variation of outgassing measurements with time after degassing the ionization gage.

were made 20 minutes after completion of the electron bombardment. Except as noted, this test procedure was used for all subsequent measurements presented herein.

The calibration of the measuring technique was performed by measuring known leak rates. These known leak rates were calibrated leaks which let air into the test chamber at a known rate. Air leaks were used because they were readily available and permitted system evaluation with a mixture containing both inert and active gases. An evaluation of the measuring technique was then possible by comparing the measured leak rate with the known calibrated leak rate.

As has been discussed previously, the response of an ionization gage is dependent upon the composition of the gases being measured. In the calibration tests the known leaks were calibrated for air and thus air was used as the test gas. In order to obtain air-leak rates from the measurements, it was necessary to adjust the measured ionization gage equivalent nitrogen pressures to true air pressures. The ionization gage used for these calibration tests was calibrated for nitrogen and the sensitivity was adjusted for air by using a ratio of nitrogen sensitivity to air sensitivity of 0.85. (See ref. 8.)

The calibration tests then were measurements where a known leak rate of air was being measured by a calibrated ionization gage the sensitivity of which had been adjusted for air. The results of these tests would be a true assessment of the ability of the overall system and technique to measure leak rates of air over the range of leak rates covered by the calibration. The calibrated air leaks measured cover a range from 2.52×10^{-4} to 2.28×10^{-5} torr-liters/sec, which is the range where a substantial number of outgassing measurements on the solid fuels were made. It would be of interest to extend the comparison of calibrated and measured leak rates to 1×10^{-6} torr-liters/sec to cover the complete range of outgassing measurements made; however, calibrated leaks could not be obtained which had leak rates below a value of 2.28×10^{-5} torr-liters/sec. It should be noted that during these tests all the inherent errors and inaccuracies of the system and measuring technique were present. Even though it would be difficult to evaluate individually the errors discussed previously, the magnitude of the aggregate of these errors should be evident in the results of the calibration measurements.

The calibrated air leaks were installed in the system, and leak rates were measured as a function of the time after degassing the calibrated ionization gage at several emission currents. The empty-system outgassing rate was measured and subtracted from the measured leak rate for each calibrated leak to obtain the values for the calibrated leak alone. The empty-system outgassing rate was 3.0×10^{-6} torr-liters/sec for the calibrated leaks of 2.52×10^{-4} torr-liters/sec and 8.74×10^{-5} torr-liters/sec, and was 2.0×10^{-6} torr-liters/sec for the calibrated leak of 2.28×10^{-5} torr-liters/sec. During the measurements made with the calibrated leak of 2.28×10^{-5} torr-liters/sec, two gages, the calibrated primary gage and a second gage, were mounted on the system; however, only the calibrated primary gage was operated during the tests. During the measurements made with the other two calibrated leaks, only the calibrated primary gage was mounted on the system operated.

The results of the calibrated air-leak measurements are presented in figure 10 and table III. The dashed horizontal lines in figure 10 indicate the values of the three calibrated leaks. For the calibrated leak of 2.28×10^{-5} torr-liters/sec at 10-mA emission current, the measured leak rate increased from 9.10×10^{-6} torr-liters/sec immediately after cleaning the gage to 1.58×10^{-5} torr-liters/sec 55 minutes after degassing the gage. At 5-mA emission current, the measured leak rate of the same calibrated leak increased from 7.32×10^{-6} torr-liters/sec immediately after cleaning the gage to 2.15×10^{-5} torr-liters/sec after 55 minutes. When the leak was measured at 1-mA emission current, the variation in the measured leak rate with time after degassing was from 1.98×10^{-5} torr-liters/sec immediately after gage cleaning to 2.27×10^{-5} torr-liters/sec after 55 minutes. The measurements of the other two calibrated leaks generally followed similar trends with increase in time after cleaning the ionization gage.

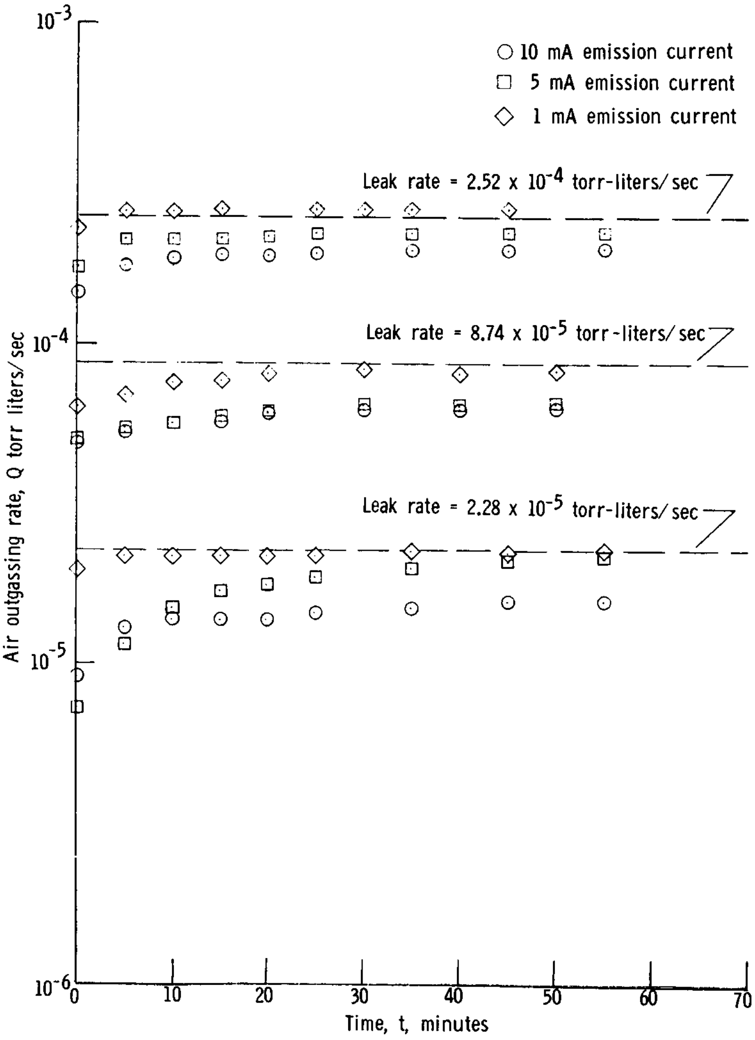

Figure 10.- Variation of outgassing measurements of three calibrated air leaks with time after degassing the ionization gage.

It can be seen in figure 10 that the measured air-leak rates are in generally good agreement with the calibrated rates at 1-mA emission current after 20 minutes following degassing of the gage. With increases in emission current to 5 and 10 mA, the measured leak rates were generally successively less than the calibrated leak rates. These results are consistent with the conclusions of reference 7, already noted, which indicate that the ionic component of gage pumping should increase with increases in emission current. Thus it would be expected that increases in emission current would result in measured leak rates which are successively less than the calibrated rates. Since the measurements using a 1-mA emission current were in good agreement with the calibrated leaks, it was determined that 1-mA emission current would be used in the program for pressure measurement whenever possible. As noted previously, 1-mA emission current was used for the PBAA and empty-system tests but 10-mA emission current was required for the polyurethane tests. The data in figure 10 can serve to provide an assessment of the ability of the overall system and technique to measure the leak rates for air at the two emission currents used during the tests. It should be noted that the data in figure 10 are in agreement with the findings from figure 9 in that in most instances the gage has come into equilibrium by about 20 minutes after degassing. Thus the calibration data in figure 10 tend to substantiate that the gage-cleaning technique has very little or no influence on the measured leak rates 20 minutes after degassing. With this in mind, a comparison of the measured and calibrated leak rates was made 20 minutes after degassing by using the data from figure 10. At 1-mA emission current, the calibrated leak rate of 2.52×10^{-4} torr-liters/sec was measured to within about +6 percent; the calibrated leak rate of 8.74×10^{-5} torr-liters/sec was measured to within about -7 percent; and the calibrated leak rate of 2.28×10^{-5} torr-liters/sec was measured to within about -4 percent. At 10-mA emission current, the calibrated leak rate of 2.52×10^{-4} torr-liters/sec was measured to within about -24 percent; the calibrated leak rate of 8.74×10^{-5} torr-liters/sec was measured to within about -30 percent; and the calibrated leak rate of 2.28×10^{-5} torr-liters/sec was measured to within about -39 percent.

Thus these calibration tests have demonstrated that, for air over a range of leak rates from 2.52×10^{-4} to 2.28×10^{-5} torr-liters/sec, this overall system and measuring technique measures the correct leak rates to within +6 percent and -7 percent when 1-mA emission current is used in the gage; and to within -39 percent when 10-mA emission current is used. The errors associated with measurements of the calibrated air leaks cannot be directly related to the errors anticipated in making outgassing measurements of the solid rocket fuels. In the latter case, the composition of the outgassing products is not known and, if different from air, could cause changes in the adsorption and gage pumping which could affect the errors involved. On the other hand, the measurements of the calibrated air leaks generally serve to validate the overall system and measuring technique used and give an indication of the order of error that might be expected. In using these results to get an indication of the order of error which might be expected in the measurement of outgassing rates, it should be noted that these results were obtained from a single operating ionization gage. If two ionization gages are operated in making the measurement, as is the case for measurements presented herein, the error introduced by gage pumping would be expected to be twice as large as the errors noted above for a single operating gage.

TESTS

The outgassing rate of the empty system was measured as a function of time in vacuum for a period of 438.8 hours. During this time the temperature of the walls of the system was $76^{\circ} \pm 1^{\circ}$ F ($297.8^{\circ} \pm 0.5^{\circ}$ K). The pressure in the test chamber during these measurements varied from 7.8×10^{-8} torr at 5.58 hours to 9.5×10^{-9} torr at 438.8 hours. The measurements were made with two gages, the primary gage and second gage, mounted on the system and operating at an emission current of 1 mA.

Outgassing measurements were made with a PBAA propellant in the test chamber as a function of time in vacuum for a period of 502 hours. During these measurements the temperature of the propellant sample was $80^{\circ} \pm 4^{\circ}$ F ($299.9^{\circ} \pm 2^{\circ}$ K) and the temperature of the walls of the system was $77^{\circ} \pm 5^{\circ}$ F ($298.3^{\circ} \pm 2.5^{\circ}$ K). The pressure in the test chamber during these measurements varied from 2.8×10^{-7} torr at 1 hour to 7.8×10^{-9} torr at 502 hours. The measurements were made with two gages, the primary gage and second gage, mounted on the system and operating at an emission current of 1 mA.

For the polyurethane propellant, outgassing measurements were made with three separate samples taken from the same propellant batch. Outgassing measurements were made with sample 1 in the test chamber for 452 hours. During these measurements, the temperature of propellant sample 1 was $75^{\circ} \pm 5^{\circ}$ F ($297.2^{\circ} \pm 2.5^{\circ}$ K) and the temperature of the walls of the system was $80^{\circ} \pm 5^{\circ}$ F ($299.9^{\circ} \pm 2.5^{\circ}$ K). The pressure in the test chamber during these measurements varied from 1.6×10^{-7} torr at 17 hours to 1.0×10^{-8} torr at 452 hours. Outgassing measurements were made with sample 2 in the test chamber for 47.66 hours. During these measurements, the temperature of propellant sample 2 was $83^{\circ} \pm 4^{\circ}$ F ($301.6^{\circ} \pm 2^{\circ}$ K) and the temperature of the walls of the system was $81^{\circ} \pm 2^{\circ}$ F ($300.6^{\circ} \pm 1^{\circ}$ K). The pressure in the test chamber during these measurements varied from 2.1×10^{-7} torr at 22.25 hours to 1.1×10^{-7} torr at 47.66 hours. Outgassing measurements were made with sample 3 in the test chamber for 77 hours. During these measurements, the temperature of propellant sample 3 was $80^{\circ} \pm 2^{\circ}$ F ($299.9^{\circ} \pm 1^{\circ}$ K) and the temperature of the walls of the system was $82^{\circ} \pm 4^{\circ}$ F ($301.1^{\circ} \pm 2^{\circ}$ K). The pressure in the test chamber during these measurements varied from 4.1×10^{-7} torr at 3.83 hours to 4.2×10^{-8} torr at 77 hours. Measurements for all three of the polyurethane samples were made with two gages, the primary gage and a second gage, mounted on the system and operating at an emission current of 10 mA. At lower emission currents the gage became contaminated very rapidly and was not usable.

RESULTS AND DISCUSSION

Empty-System Measurements

The results of the measurements of the outgassing rate of the empty system are presented in figure 11 as the variation of outgassing rate with vacuum exposure time. The empty-system outgassing rate varies from about 4.90 × 10^{-6} torr-liters/sec at 5.58 hours to 1.61 × 10^{-6} torr-liters/sec at 438.83 hours. The outgassing rate decreases rapidly during the first 100 hours and then remains relatively constant during the remainder of the exposure time. Some data scatter is evident after about 200 hours. Empty-system outgassing values are required at times intermediate to those at which empty-system data are recorded. Also a single empty-system value is needed in those instances where several measurements at about the same time indicate a range of outgassing rates. To obtain a single value of empty-system outgassing rate at any time during the test, a curve was faired through the data and the faired value was used. From 5.58 hours to 150 hours the curve was faired by eye and a least-squares straight line was fitted to the data between 150 hours and 438 hours. Since empty-system data were required for about 502 hours, the least-squares straight line was extrapolated from 438 hours to 502 hours.

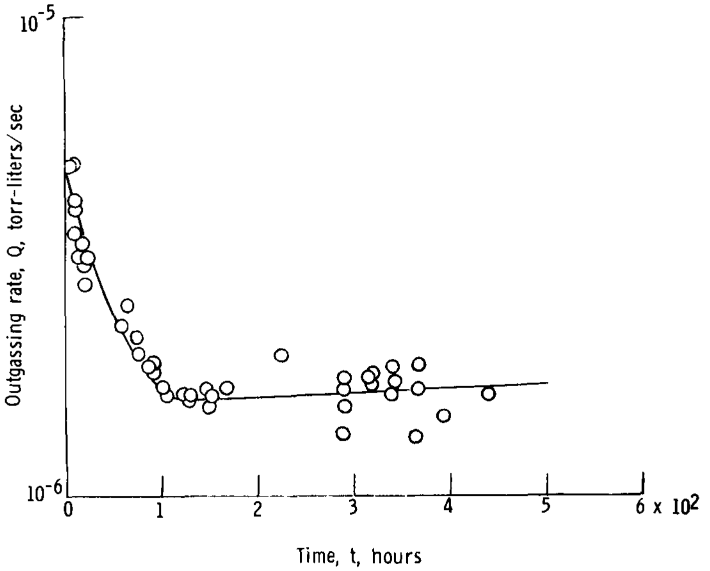

Figure 11.- Variation of outgassing rate with time for empty system.

It is of interest to examine the outgassing rate of the empty system on the basis of unit surface area so it can be compared with existing outgassing data for stainless steel. The outgassing rate per unit area for the empty system is presented in figure 12 as a function of vacuum exposure time. The outgassing-rate measurements and the outgassing rate per unit area are shown in table IV. It should be noted that these are composite outgassing rates with contributions from all the various materials used in construction of the test chamber. The internal surface area of the test chamber contained about 72.5-percent stainless steel (chamber and flange), 27-percent glass (ionization gages), and 0.5-percent buna-N rubber (valve 0-rings). Das in reference 9 presents outgassing rates for stainless steel, with pretreatment similar to that received by the test chamber in this program, of 1.5 × 10^{-10} and 1.1 × 10^{-10} torr-liters/sec-cm^2 at 23 hours and

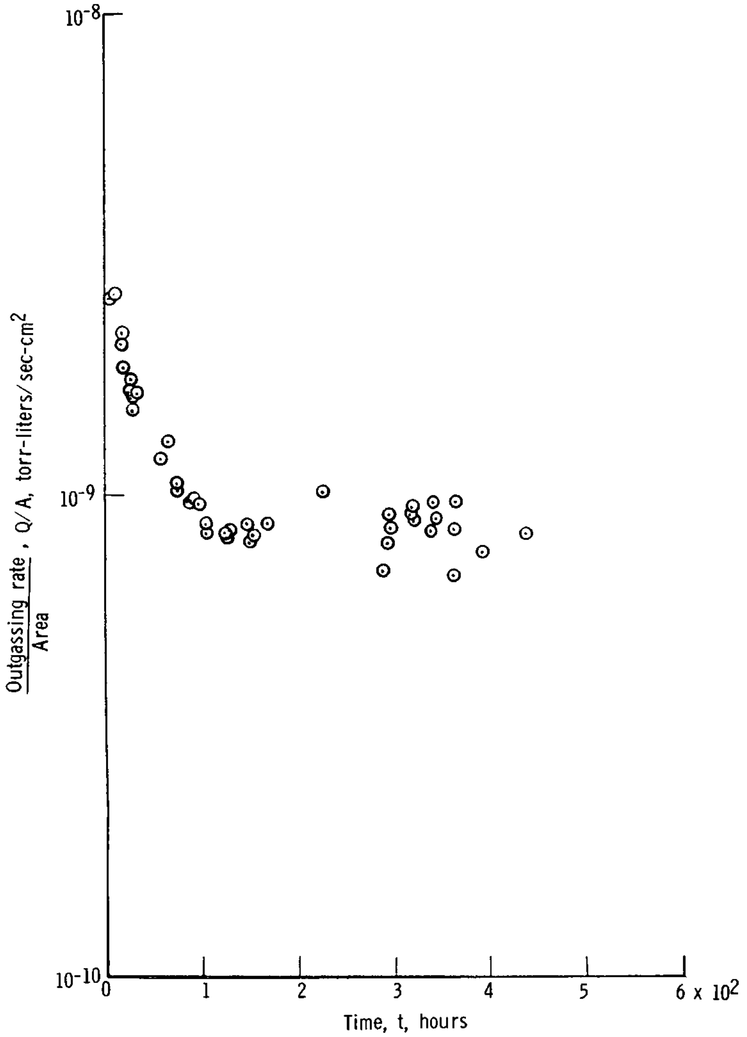

Figure 12.- Variation of outgassing rate per unit area with time for empty system.

167 hours, respectively. These values are in contrast to composite outgassing rates for the empty system of about 1.7×10^{-9} and about 8.1×10^{-10} torr-liters/sec-cm^2 at 23 hours and 167 hours, respectively. Since glass and rubber have higher outgassing rates than stainless steel, the empty-system data presented herein are consistent with those presented by Das (ref. 9).

Measurements With a Polybutadiene Acrylic Acid Sample

After completion of the empty-system measurements, a polybutadiene acrylic acid (PBAA) sample was installed in the test chamber and a series of outgassing measurements was made on the combination of propellant sample and system. The results of

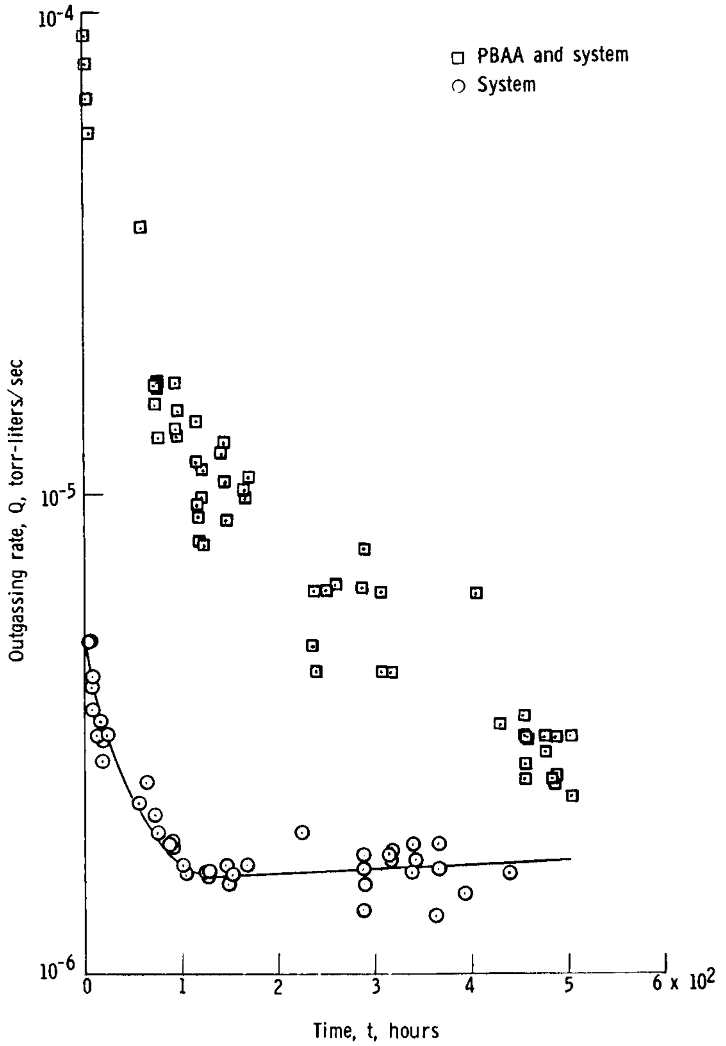

Figure 13.- Variation of outgassing rate with time for polybutadiene acrylic acid (PBAA) propellant sample in system.

these tests are shown in figure 13 where outgassing rate is plotted as a function of storage time in vacuum. Also shown for reference in figure 13 is the outgassing rate of the empty system.

The outgassing rate for the propellant sample alone was found at a given time by subtracting from the measured outgassing rate for the propellant and system the faired value of outgassing rate for the empty system. To insure that the empty-system outgassing rate was not changing during the period in which a propellant sample was located in the system, two tests were conducted. One of these tests was conducted prior to placing the sample in the system, and the second after the sample had been removed. Results of these tests indicated no substantial variations in the empty-system outgassing rate during propellant exposure. The resultant outgassing rate was then divided by the surface area of the sample to obtain the outgassing rate per unit area of the sample. The

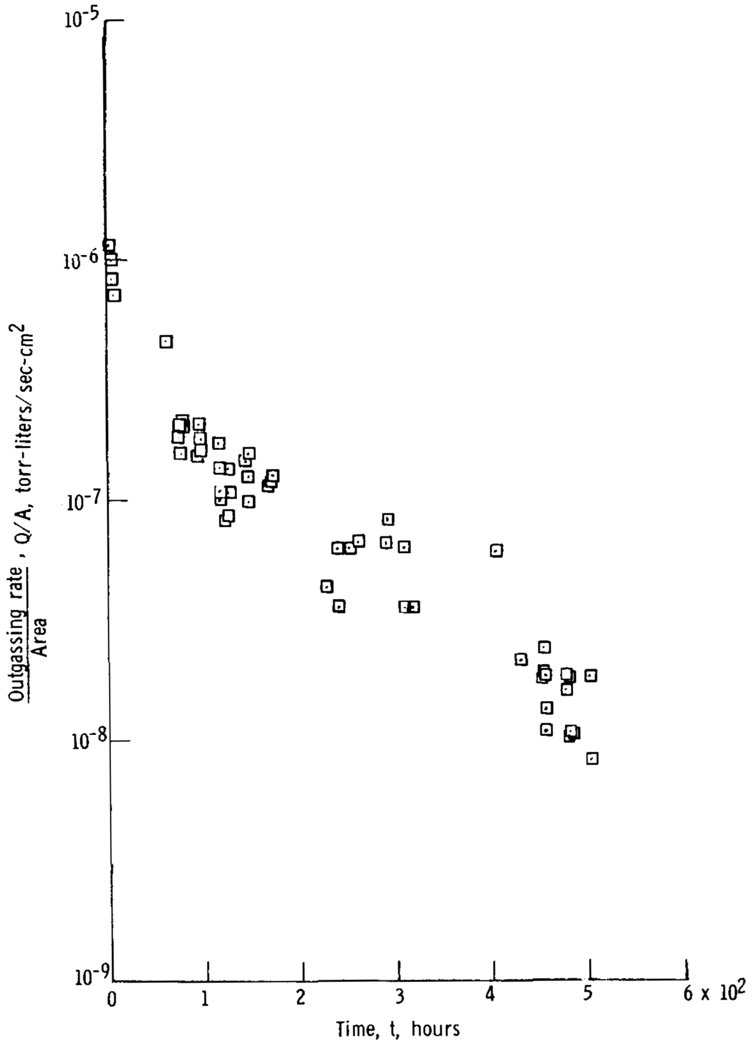

Figure 14.- Variation of outgassing rate per unit area with time for polybutadiene acrylic acid (PBAA) propellant sample.

outgassing rate per unit surface area of the sample for the PBAA is presented in figure 14 as a function of storage time in vacuum. These data are also shown in table V along with the measured outgassing rates for the propellant and system and the faired values of the empty-system outgassing rates used in obtaining the outgassing rates per unit area for the propellant sample alone. In figure 14, the outgassing rate of the PBAA decreases from 1.15×10^{-6} torr-liters/sec-cm^2 after 1 hour to 8.32×10^{-9} torr-liters/sec-cm^2 after 502 hours in vacuum. The outgassing rate decreased rapidly during the first 100 hours and then continued to decrease more slowly. It is also observed that, at the end of the 502 hours, no equilibrium of outgassing rate had been reached and the outgassing rate appeared to be diminishing at a constant rate of a decade about every 350 hours.

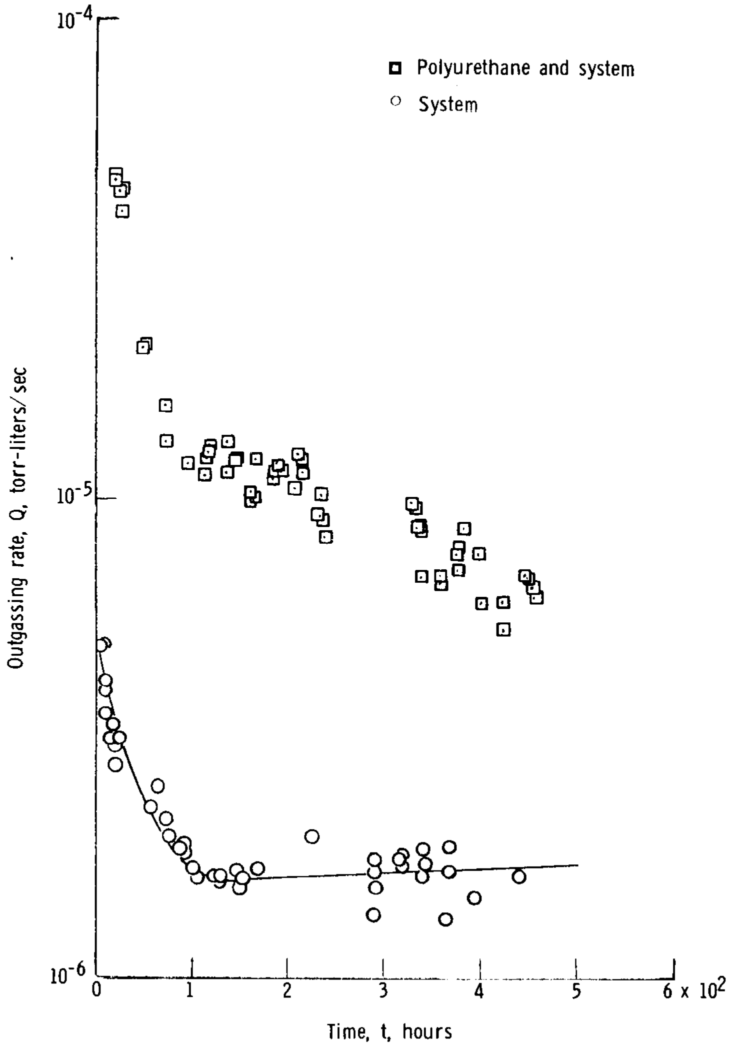

Figure 15.- Variation of outgassing rate with time for polyurethane propellant sample 1.

Considerable scatter was noted in the PBAA outgassing measurements. The reason for this scatter has not been ascertained, but could be caused by: (1) small temperature fluctuations of the sample during the testing period, (2) errors inherent in the measuring technique, or (3) fluctuations in the outgassing rate of the propellant sample during small periods of time.

Variations in the temperature of the sample are not thought to be the reason for the changes in measured outgassing rate. For instance, at 308 hours the measured outgassing rate was 3.55×10^{-8} torr-liters/sec-cm^2 and the temperature of the sample was 84° F (222° K). One hour later the measured outgassing rate was 6.33×10^{-8} torr-liters/sec-cm^2 and the temperature of the sample remained at 84° F (222° K). This difference indicates variations in outgassing rate even when the temperature of the sample is constant over short periods of storage time.

In the establishment of the measuring technique, calibrated air leaks were measured. The results of these measurements are shown in figure 10. The outgassing measurements in this plot indicate very little scattering which indicates that the technique is reliable and that the scattering is not inherent in the measuring technique. It is believed that fluctuations in outgassing rate of the propellant sample occur over short periods of time. The nature of these fluctuations at the present cannot be explained.

Measurements With Polyurethane Samples

The measured outgassing rate for polyurethane sample 1 is shown in figure 15 as a function of storage time in vacuum. Also shown in figure 15 for reference is the outgassing rate of the empty system. The procedure for finding the outgassing rate per unit surface area for the polyurethane samples alone was the same as that described for the PBAA sample. The outgassing rate per unit surface area is shown in figure 16 as a function of storage time in vacuum. The outgassing rate for the polyurethane decreased from 3.02×10^{-7} torr-liters/sec-cm^2 after 17 hours to 3.17×10^{-8} torr-liters/sec-cm^2 after 452 hours. As with PBAA, the rate of decrease of outgassing rate is very rapid during the first 100 hours but then decreases more slowly during the last 400 hours. After 452 hours in vacuum, the outgassing rate had not reached a constant value and was still decreasing at a rate of about a decade every 850 hours. Again there is noticeable scatter in the outgassing data. As with the PBAA sample, it is thought that the scatter is due primarily to fluctuations in the outgassing rate which occur over short periods of time.

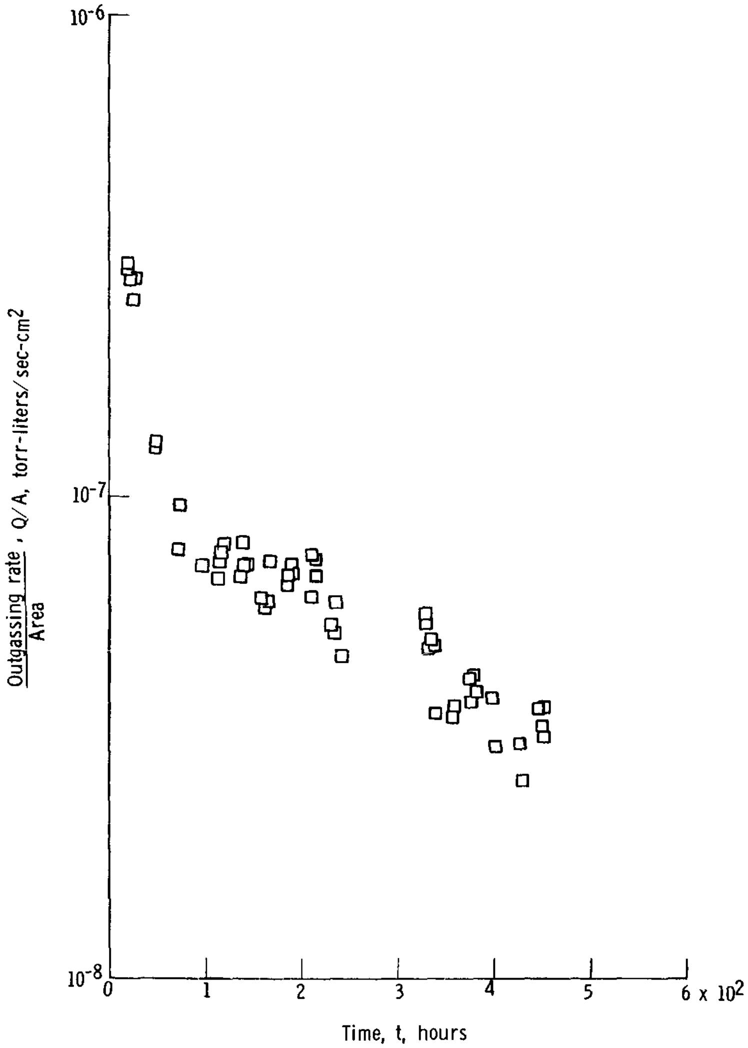

Figure 16.- Variation of outgassing rate per unit area with time for polyurethane propellant sample 1.

In order to determine the extent of variations in outgassing rate among samples from the same propellant batch, two additional polyurethane samples were taken from different locations in the batch used for polyurethane sample 1. These samples, designated polyurethane samples 2 and 3, were tested for shorter periods of time; 47.66 hours for sample 2, and 77 hours for sample 3. The results of these measurements are shown in figure 17 along with data for sample 1 for the first 97 hours. All three samples had approximately the same dimensions and about the same surface area. The agreement among these three samples is generally good with only slight variations in the measured outgassing rates beyond 45 hours of vacuum exposure.

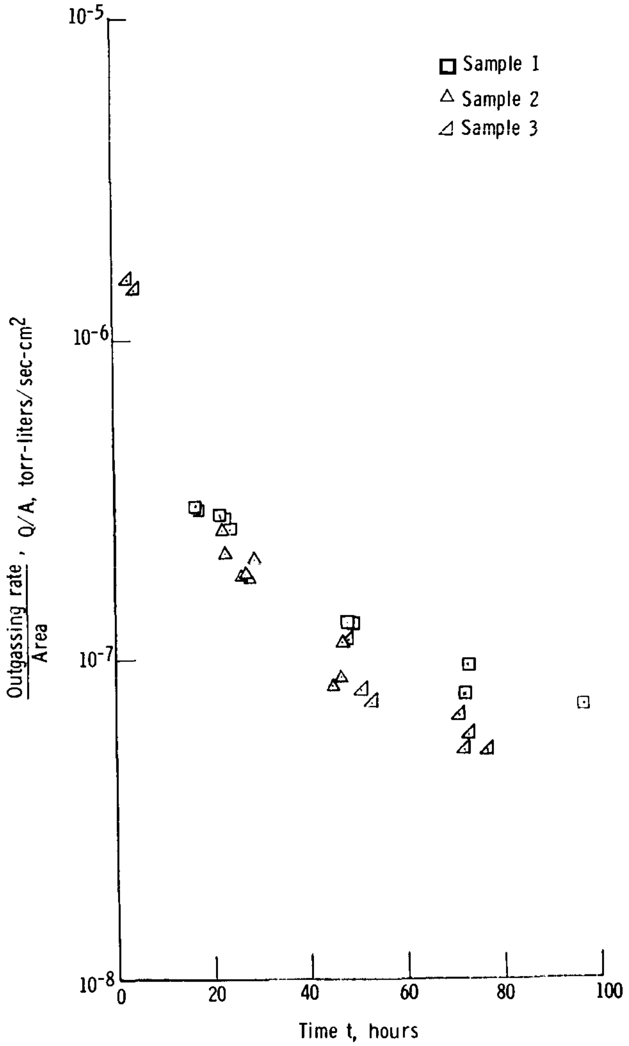

Figure 17.- Outgassing measurements for three different samples of polyurethane rocket propellant.

The data for all three polyurethane samples are presented in table VI along with the faired values of the empty-system outgassing rates used in obtaining the outgassing rates per unit area for the propellant sample alone.

CONCLUSIONS

An investigation has been conducted to measure the outgassing rate of two solid rocket fuels, having polyurethane and polybutadiene acrylic acid (PBAA) as their binders, in a vacuum. The following conclusions were reached:

1. The outgassing rate of PBAA solid rocket fuel in vacuum at ambient temperature decreased from 1.15×10^{-6} torr-liters/sec-cm^2 after 1 hour to 8.32×10^{-9} torr-liters/sec-cm^2 after 502 hours. The outgassing rate decreased rapidly during the first 100 hours and then continued to decrease more slowly.

2. The outgassing rate of polyurethane solid rocket fuel in vacuum at ambient temperature decreased from 3.02×10^{-7} torr-liters/sec-cm^2 after 17 hours to 3.17×10^{-8} torr-liters/sec-cm^2 after 452 hours. The outgassing rate decreased rapidly during the first 100 hours and then decreased more slowly during the last 400 hours.

3. After 502 hours in vacuum, the outgassing rate of the PBAA had not reached a constant value and was still decreasing at a rate of about a decade every 350 hours.

4. After 452 hours in vacuum, the outgassing rate of the polyurethane sample had not reached a constant value and was still decreasing at a rate of about a decade every 850 hours.

5. For the three polyurethane samples tested, there were only slight variations in the measured outgassing rate among samples after 45 hours of vacuum exposure.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., October 3, 1966,
124-09-04-01-23.

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TABLE I.- COMBINED EFFECT OF ERRORS IN OUTGASSING RATES

Test	Measured value, torr-liters/sec	Error range, torr-liters/sec
Empty system	4.90×10^{-6} 1.60×10^{-6}	1.50×10^{-6} to -1.50×10^{-6} 0.50×10^{-6} to -0.50×10^{-6}
PBAA	8.95×10^{-5} 2.34×10^{-6}	4.65×10^{-5} to -3.98×10^{-5} 0.83×10^{-6} to -0.85×10^{-6}
Polyurethane	4.69×10^{-5} 6.25×10^{-6}	1.95×10^{-6} to -1.83×10^{-5} 2.03×10^{-5} to -2.03×10^{-5}

TABLE II.- VALUES OF MEASURED OUTGASSING RATE AT VARIOUS TIMES AFTER DEGASSING IONIZATION GAGE FOR EMPTY SYSTEM AND PROPELLANT SAMPLES

Time after degassing, min	Outgassing rate, Q, torr-liters/sec, for –		
	Empty system ($i^- = 1$ mA)	PBAA sample ($i^- = 1$ mA)	Polyurethane sample ($i^- = 10$ mA)
0	3.50×10^{-6}	6.05×10^{-6}	3.74×10^{-5}
5	3.50	6.98	3.62
10	3.50	1.03×10^{-5}	3.96
15	4.50	1.21	
20	4.90		3.96
25	5.10	1.21	
30			4.07
35		1.23	
40			3.92
41	5.00		
50	5.30		4.02
60			3.96

TABLE III. - VALUES OF MEASURED LEAK RATES AT VARIOUS TIMES AFTER DEGASSING IONIZATION GAGE FOR CALIBRATED AIR LEAKS

Time after degassing, min	Outgassing rate, Q, torr-liters/sec, for –								
	Calibrated air leak = 2.52×10^{-4} torr-liters/sec			Calibrated air leak = 8.74×10^{-5} torr-liters/sec			Calibrated air leak = 2.28×10^{-5} torr-liters/sec		
	$i^- = 1$ mA	$i^- = 5$ mA	$i^- = 10$ mA	$i^- = 1$ mA	$i^- = 5$ mA	$i^- = 10$ mA	$i^- = 1$ mA	$i^- = 5$ mA	$i^- = 10$ mA
0	2.31×10^{-4}	1.84×10^{-4}	1.47×10^{-4}	6.40×10^{-5}	5.14×10^{-5}	4.90×10^{-5}	1.98×10^{-5}	7.32×10^{-6}	9.10×10^{-6}
5	2.62	2.14	1.78	6.95	5.53	5.30	2.18	1.16×10^{-5}	1.30×10^{-5}
10	2.62	2.15	1.87	7.62	5.70		2.18	1.50	1.38
15	2.67	2.17	1.91	7.74	6.00	5.75	2.18	1.70	1.38
20		2.20	1.91	8.13	6.18	6.10	2.18	1.78	1.38
25	2.67	2.25	1.95				2.18	1.88	1.46
30	2.67			8.40	6.58	6.27			
35	2.67	2.25	1.99				2.24	2.00	1.50
40				8.13	6.48	6.27			
45	2.67	2.27	1.99				2.22	2.10	1.58
50				8.27	6.57	6.38			
55		2.28	2.01				2.27	2.15	1.58

TABLE IV.- VALUES OF MEASURED OUTGASSING RATE AT VARIOUS TIMES

FOR EMPTY SYSTEM

Time, hr	Outgassing rate, torr-liters/sec	Outgassing rate per unit area, torr-liters/sec-cm^2
5.58	4.90×10^{-6}	2.53×10^{-9}
7.58	4.94	2.55
17.42	3.95	2.04
18.42	4.16	2.14
19.75	3.54	1.83
21.25	3.12	1.61
24.42	3.09	1.59
26.33	3.36	1.73
29.00	2.79	1.49
31.08	3.12	1.61
56.75	2.28	1.18
62.67	2.50	1.29
73.00	2.13	1.05
75.17	1.97	1.01
84.08	1.88	9.70×10^{-10}
86.08	1.88	9.70
96.33	1.86	9.60
100.33	1.69	8.70
101.83	1.61	8.30
122.33	1.62	8.35
124.75	1.58	8.15
127.08	1.63×10^{-6}	8.40×10^{-10}
146.08	1.69	8.70
149.33	1.54	7.95
150.83	1.60	8.25
168.33	1.69	8.70
224.58	1.97	1.01×10^{-9}
289.17	1.35	6.95×10^{-10}
290.58	1.53	7.90
293.83	1.77	9.10
295.25	1.65	8.50
316.83	1.77	9.10
318.25	1.82	9.40
319.33	1.71	8.80
338.33	1.63	8.40
340.33	1.86	9.60
342.92	1.73	8.90
361.08	1.32	6.80
362.58	1.65	8.50
364.33	1.87	9.65
392.33	1.47	7.60
438.83	1.61	8.30

TABLE V.- VALUES OF OUTGASSING RATES AT VARIOUS TIMES FOR POLYBUTADIENE ACRYLIC ACID PROPELLANT SAMPLE

Time, hr	Propellant and system outgassing rate, torr-liters/sec	Empty-system outgassing rate (faired values), torr-liters/sec	Propellant outgassing rate, torr-liters/sec	Propellant outgassing rate per unit area, torr-liters/sec-cm^2
1.0	8.95×10^{-5}	5.00×10^{-6}	8.45×10^{-5}	1.15×10^{-6}
2.0	7.80	4.95	7.31	9.96×10^{-7}
3.0	6.60	4.90	6.11	8.34
5.0	5.60	4.60	5.14	7.01
60.0	3.54	2.19	3.32	4.52
71.5	1.68	2.01	1.48	2.02
72.0	1.68	2.00	1.48	2.02
72.5	1.68	2.00	1.48	2.02
73.0	1.54	1.98	1.34	1.83
77.0	1.31	1.95	1.12	1.53
95.0	1.70	1.73	1.53	2.08
96.0	1.37	1.72	1.20	1.64
97.0	1.34	1.71	1.17	1.60
	1.49	1.70	1.32	1.80
116.0	1.42	1.62	1.26	1.72
116.5	1.16	1.62	1.00	1.36
117.0	9.50×10^{-6}	1.61	7.89×10^{-6}	1.08
118.0	9.00	1.61	7.39	1.01
119.5	7.90	1.60	6.30	8.60×10^{-8}
120.5	9.75	1.60	8.15	1.11×10^{-7}
122.0	7.85	1.60	6.25	8.51×10^{-8}
123.0	1.13×10^{-5}	1.60	9.70	1.32×10^{-7}
141.0	1.21	1.59	1.05×10^{-5}	1.43
144.0	1.29	1.59	1.13	1.54
146.0	1.06	1.59	9.01×10^{-6}	1.23
146.0	8.80×10^{-6}	1.59	7.21	9.82×10^{-8}
166.0	9.80	1.60	8.20	1.12×10^{-7}
167.0	1.01×10^{-5}	1.60	8.50	1.16
170.0	1.08	1.60	9.20	1.25
237.0	4.80×10^{-6}	1.62	3.18	4.33×10^{-8}
239.0	6.23	1.62	4.61	6.30
240.0	4.25	1.62	2.63	3.58
250.0	6.25	1.62	4.63	6.31
260.0	6.45	1.63	4.82	6.56
289.0	6.35	1.64	4.71	6.43
290.0	7.67	1.65	6.02	8.21
308.0	4.25	1.65	2.60	3.55
309.0	6.29	1.65	4.64	6.33
315.0	4.25	1.65	2.60	3.54
406.0	6.20	1.69	4.51	6.15
430.0	3.30	1.70	1.60	2.18
452.0	3.10	1.71	1.39	1.90
453.0	3.10	1.71	1.39	1.90
455.0	3.48	1.71	1.77	2.41
456.0	2.52	1.71	8.10×10^{-7}	1.10
457.0	2.71	1.71	1.00×10^{-6}	1.36
458.0	3.10	1.72	1.38	1.88
476.0	3.10	1.72	1.38	1.88
477.0	2.90	1.72	1.18	1.61
479.0	3.10	1.72	1.38	1.88
480.0	2.52	1.72	8.00×10^{-7}	1.09
481.0	2.52	1.72	8.00	1.09
482.0	2.52	1.72	8.00	1.09
501.0	3.11	1.73	1.38×10^{-6}	1.88
502.0	2.34	1.73	6.10×10^{-7}	8.32×10^{-9}

TABLE VI.- VALUES OF OUTGASSING RATES AT VARIOUS TIMES FOR POLYURETHANE SAMPLES

(a) Polyurethane sample 1

Time, hr	Propellant and system outgassing rate, torr-liters/sec	Empty-system outgassing rate (faired values), torr-liters/sec	Propellant outgassing rate, torr-liters/sec	Propellant outgassing rate per unit area, torr-liters/sec-cm^2
17	4.69×10^{-5}	3.70×10^{-6}	4.32×10^{-5}	3.02×10^{-7}
19	4.56	3.50	4.21	2.94
22	4.33	3.32	4.00	2.80
23	4.35	3.30	4.02	2.81
24	3.95	3.25	3.62	2.54
48	2.04	2.40	1.80	1.26
49	2.08	2.38	1.84	1.29
72	1.31	1.98	1.11	7.77×10^{-8}
73	1.57	2.13	1.36	9.55
97	1.19	1.70	1.02	7.14
112	1.13	1.61	9.70×10^{-6}	6.79
115	1.22	1.60	1.06×10^{-5}	7.42
117	1.25	1.60	1.09	7.63
119	1.29	1.59	1.13	7.92
137	1.13	1.58	9.70×10^{-6}	6.79
139	1.31	1.58	1.15×10^{-5}	8.05
141	1.19	1.58	1.03	7.21
143	1.21	1.58	1.05	7.35
160	1.04	1.59	8.81×10^{-6}	6.16
162	9.96×10^{-6}	1.59	8.37	5.85
166	1.02×10^{-5}	1.59	8.61	6.02
167	1.21	1.59	1.05×10^{-5}	7.35
184	1.10	1.60	9.40×10^{-6}	6.57
186	1.14	1.60	9.80	6.85
189	1.19	1.60	1.03×10^{-5}	7.21
191	1.14	1.60	9.80×10^{-6}	6.85
209	1.05	1.60	8.90	6.22
210	1.24	1.60	1.08×10^{-5}	7.56
213	1.22	1.60	1.06	7.42
215	1.14	1.60	9.80×10^{-6}	6.85
231	9.31×10^{-6}	1.61	7.70	5.39
233	9.05	1.61	7.44	5.20
236	1.02×10^{-5}	1.61	8.59	6.01
239	8.30×10^{-6}	1.62	6.68	4.67
328	9.83	1.66	8.17	5.72
330	9.45	1.66	7.79	5.45
332	8.63	1.66	6.97	4.87
334	8.90	1.66	7.24	5.06
336	8.76	1.66	7.10	4.97
339	6.75	1.67	5.08	3.56
357	6.69	1.68	5.01	3.51
358	6.92	1.68	5.24	3.66
374	7.68	1.69	5.99	4.19
376	7.71	1.69	6.02	4.21
378	7.05	1.69	5.36	3.75
381	8.72	1.69	7.03	4.92
398	7.16	1.70	5.46	3.82
400	6.02	1.70	4.32	3.02
428	6.10	1.70	4.40	3.08
429	5.39	1.71	3.68	2.58
446	6.92	1.72	5.20	3.64
449	6.92	1.72	5.20	3.64
451	6.50	1.72	4.78	3.35
452	6.25	1.72	4.53	3.17

TABLE VI.- VALUES OF OUTGASSING RATES AT VARIOUS TIMES FOR POLYURETHANE SAMPLES – Concluded

(b) Polyurethane sample 2

Time, hr	Propellant and system outgassing rate, torr-liters/sec	Empty-system outgassing rate (faired values), torr-liters/sec	Propellant outgassing rate, torr-liters/sec	Propellant outgassing rate per unit area, torr-liters/sec-cm^2
22.25	4.30×10^{-5}	3.35×10^{-6}	3.97×10^{-5}	2.54×10^{-7}
23.25	3.70	3.30	3.37	2.16
26.25	3.12	3.20	2.80	1.80
27.17	3.12	3.05	2.82	1.81
28.17	3.12	3.00	2.82	1.81
29.08	3.52	3.00	3.22	2.06
45.87	1.52	2.50	1.27	8.15×10^{-8}
46.75	1.61	2.45	1.37	8.75
47.66	1.96	2.38	1.73	1.10×10^{-7}

(c) Polyurethane sample 3

Time, hr	Propellant and system outgassing rate, torr-liters/sec	Empty-system outgassing rate (faired values), torr-liters/sec	Propellant outgassing rate, torr-liters/sec	Propellant outgassing rate per unit area, torr-liters/sec-cm^2
3.83	1.88×10^{-4}	4.80×10^{-6}	1.84×10^{-4}	1.52×10^{-6}
4.25	1.75	4.70	1.71	1.41
48.16	1.59×10^{-5}	2.40	1.35×10^{-5}	1.11×10^{-7}
51.16	1.19	2.32	9.60×10^{-6}	7.90×10^{-8}
53.16	1.00	2.28	7.80	6.40
71.17	1.00	2.00	8.00	6.60
72.66	8.00×10^{-6}	1.95	6.10	5.05
73.16	8.80	1.92	6.90	5.70
77.00	8.00	1.90	6.10	5.05

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