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LIQUID OXYGEN/METAL GELLED MONOPROPELLANTS

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ABSTRACT

The objectives of this program were to establish the feasibility of metallized/liquid oxygen monopropellants and select the best monopropellant formulation for continued study. The metal powders mixed with the liquid oxygen were aluminum/magnesium (80/20), silicon and iron (Iron was only tested for burning properties). The formulations were first evaluated on whether they detonated when ignited or burned. The formulations only burned when ignited. The viscosity for the formulations ranged from 900 cps to 100 cps at shear rates up to 300 seconds⁻¹. Two percent (by weight) of Cab-O-Sil was added to the aluminum and aluminum/magnesium formulations for gelling while the silicon formulation used three percent. Within a seven hour period, settling was suggested only in the 29% aluminum and 29% aluminum/magnesium formulations.

The monopropellants were burned in a cylinder submerged in a liquid nitrogen bath. Experimental data at ambient pressure indicated that the monopropellants were extinguished when the flame front reached regions submerged under the liquid nitrogen. The burn rate increased dramatically when burned in a cylinder enclosure with less heat sink available to the monopropellant. The test results were inconclusive as to whether the increased burn rate was due to the lower heat sink capacity or the small amount of pressure (2 psi) generated during the burning of the monopropellant.

The burning of the aluminum and aluminum/magnesium resulted in a brilliant white flame similar to that of an arc welder. These monopropellants burned in a pulsating manner with the aluminum/magnesium appearing to have less pulsating combustion. The silicon monopropellant burned with an orange glow. No sparks or energetic burning was apparent as with the aluminum or aluminum/magnesium.

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1.0 INTRODUCTION

Early in the next century, the United States will begin construction of a permanent manned lunar base. This lunar base will initially serve as a scientific outpost and eventually become a major launch point for unmanned and manned missions to the planets due to the moon's low gravity. The base will strive to be self sufficient by producing oxygen, electrical power, building materials and eventually food.

A key part of making the lunar base successful is a source of rocket propellant on the moon. Liquid oxygen can be produced from the various oxides in the lunar soil and is an excellent oxidizer for a rocket engine. The challenge is to identify and be able to use fuels from the lunar soil. While there has been considerable speculation about frozen water in lunar canyons and craters, the conservative approach is to only consider fuels known to exist on the moon. This was the approach used in this study. The analysis of the soil samples returned during the Apollo and the Soviet Luna missions were used as the basis for selecting potential fuels for a lunar-based rocket engine.

Initial fuel candidates were identified and performance calculated by Wickman, Oberth and Mockenhaupt (Reference 1) in 1986. The major fuel candidates identified were aluminum, silicon, magnesium, titanium, iron, sulfur and phosphorus. With the exception of sulfur and phosphorus, the major difficulty in making a working engine was finding a way to get the metal

powders from the tank into the rocket combustion chamber in a controlled manner. That initial study was continued by Wickman Spacecraft & Propulsion Company and research work was later started by the NASA Lewis Research Center on the same subject matter.

One approach of introducing metal powder into the combustion chamber is to blow the metal powder into the chamber using an inert carrier gas. This method has been pursued by NASA Lewis Research Center. A second method is to mix the metal powder with the liquid oxygen (LOX) to form a monopropellant. However, the risk in this approach is the potential for explosive burning of the monopropellant, propagation of the flame front up the propellant feed tube, propellant shock sensitivity and settling of the metal powder in the propellant tank. The issue of shock sensitivity has been addressed with experiments by NASA White Sands (Ref. 2).

The Liquid Oxygen Gelled Formulation program funded by the NASA Lewis Research Center addresses the remaining issues. The metal fuels selected for study were aluminum, silicon, titanium and iron. To accomplish this, the program was organized into two parts, Task I and II.

The objective of Task I was to calculate performance of the candidate monopropellants, design test equipment and develop a test plan for Task II. The purpose of Task II was the collection of experimental data with respect to the monopropellants

viscosity, metal powder settling and burning rates at various pressures. During Task II, the special test equipment required for this unique propellant was built and tested.

It should be noted that when this program began, it was believed by many chemists that the simple mixing of liquid oxygen and metal powders would result in a spontaneous explosion. It was also believed that if a spontaneous explosion did not result, then the monopropellant would explode upon ignition. The results of this program have shown these beliefs to be incorrect for the tested metal powders. However, it was in the environment of uncertainty that the program began. Due to the strong possibility of explosion or fire, test equipment was designed and built to operate remotely and to be low cost or sacrificial.

2.0 TASK I

Task I had two primary objectives. The first objective was to investigate methods of formulating the candidate monopropellants. This included the use of different gelling agents with the metal fuels. The key consideration in formulating the monopropellant was that all ingredients used should be available from the moon without Earth resupply. To evaluate the formulations the theoretical performance and sensitivity to mixture ratio was calculated using the Performance Evaluation Program (PEP) developed by Naval Weapons Center at China Lake, California (Ref. 3).

The final objective was to generate and submit a Task II formulation and characterization test plan. The test plan would include all required test equipment to measure viscosity, fuel settling and monopropellant burn rate. As part of this task, special test equipment was designed and analyzed for measuring all these parameters.

2.1 Potential Monopropellant Formulations

The candidate metal fuels were aluminum, silicon, iron and titanium. All of these metals can be obtained from processed lunar soil. Methods of processing lunar soil are being investigated by other contractors and was not addressed in this program. Based on the Apollo and Soviet Luna soil samples returned from the moon, the amount of each metal in the lunar soil ranges as follows (Ref. 4):

Aluminum:	5.46 - 14.38%	(Weight Percent)
Silicon:	18.63 - 22.46%	
Iron:	4.03 - 15.35%	
Titanium:	0.29 - 5.65%	

Fuels Without Gelling Agents

A major challenge in using metal powder as fuel is to get it from the tank into the combustion chamber in a controlled and measured manner. One formulation approach is to provide just enough LOX to fluidize the metal powder so that it can be

injected into the rocket chamber. The remaining LOX required to support combustion would be injected separately. These formulations do not require any gelling since the metal/LOX mixture does not contain any excess LOX. Therefore, by definition the powder cannot settle and metal powder distribution should be uniform.

In order for the formulation to work, the metal fuel particle diameter must not be too small or the particles will tend to clump in the mixture forming a sort of paste. Looking at dry particles with diameters of less than 60 microns shows that when the particles are poured, clumping is observed. This clumping is absent when the particle size is increased to about 100 microns.

Fuels With Gelling Agents

Propellants formulated in this category have all the required LOX for combustion and performance. The metal powder only takes a small amount of the total monopropellant volume. Since the metal powder settles almost immediately to the bottom of the mixture, a gelling agent must be added or the propellant must constantly be agitated to prevent settling. It is important to prevent settling to maintain a constant mixture ratio during injection of the monopropellant into the combustion chamber.

The gelling agent can be almost any material, but in our study we restricted ourselves to materials that could be found on the moon. The particle size for the gelling agent

should be about 0.02 microns, if spherical. For particle sizes this small, the gelling material should be inert in the LOX as fuel particles would most likely give the mixture explosive burning properties.

Potential lunar gelling agents are silicon dioxide (Cab-O-Sil tradename), iron oxide, titanium oxide, aluminum oxide, calcium oxide and magnesium oxide. A search of commercial sources yielded silicon dioxide and aluminum oxide as two materials available in particle sizes suitable for gelling the LOX.

Gases which freeze at cryogenic temperatures have been successfully used in the past to gel cryogenic liquids. These gelling agents are normally gaseous at room temperature. Sulfur and phosphorus were found in small amounts in the lunar soil. The sulfur could be combined with lunar oxygen to form sulfur dioxide gas. Sulfur dioxide (SO_2) is a gas at room temperature and would solidify at -72.7°C . The monopropellant temperature is below -183°C . To gel the monopropellant, sulfur dioxide would be injected in the monopropellant in the form of small bubbles which would solidify into submicron particles on the order of 0.02 microns.

Phosphorus pentoxide (P_2O_5) is available directly on the moon in amounts ranging from 0.1 to 0.5% by weight of soil content. It could be heated to its sublimation temperature (300°C) and injected into the LOX as a gas. Again, the bubbler

would be designed to form 0.02 micron solid particles to gel the monopropellant.

Some elements found on the moon can be combined to form compounds which have melting temperatures below 500° C. However, many of these substances are flammable and introducing them to LOX would be very dangerous. A preliminary examination of possible candidates has not yielded any compounds which would prove useful for gelling the LOX.

Another method of gelling the monopropellant is to use non-spherical particles such as platelets. They can safely be made of the fuel material since they are not submicron in size. This method of gelling eliminates inerts in the propellant and raises specific impulse over values for monopropellants using inert gelling agents.

Multiple Fuels With/Without Gelling Agents

The combustion of fuels such as aluminum and silicon may be poor without the addition of more easily combustible materials. Experimental studies in the solid rocket and metalized liquid rocket engine field have shown that obtaining high combustion efficiencies with aluminum powder is not always easy. This, of course, is dependent on the propellant formulation and rocket chamber residence time of the aluminum particles. Silicon combustion, based on our experimental work, appears to burn poorly and not in an energetic fashion.

One method of helping this situation is to alloy the fuel with better burning metals such as magnesium. Magnesium is easily ignited and burns vigorously in an oxygen environment. The drawback to this approach is that the alloy may be more shock sensitive than the original fuel or the alloy may burn explosively in LOX. To investigate this possibility, an alloy of 20% magnesium with 80% aluminum was selected for evaluation.

Encapsulation of Fuels

Encapsulation of the fuels with an inert barrier would provide a monopropellant that was safe with no explosive danger. An oxide layer is always present around each fuel particle in the monopropellant, but the oxide layer is not always sufficient to prevent spontaneous combustion of the metal particles from shock or another energy source. Two examples of this are aluminum and titanium. The aluminum oxide layer on aluminum protects the aluminum from rapid reaction with an oxidizer while the oxide layer on titanium provides virtually no protection. Titanium particles are extremely shock sensitive in LOX and the mixture burns explosively upon impact. In contrast, aluminum particles in LOX showed no shock sensitivity during testing by NASA White Sands (Ref. 2).

2.2 Theoretical Performance

The theoretical specific impulse was calculated for each of the metal fuels including the 80/20 aluminum-magnesium alloy. The Propellant Evaluation Program (PEP) developed by the Naval Weapons Center at China Lake, California was used for all the calculations. Two phase flow losses due to the condensed metal oxides in the exhaust are not included in the performance values. The nozzle geometry would have to be known for this loss to be calculated as it is a function of throat diameter and contour. The expansion ratio was set to 50 for all cases.

Aluminum powder is the highest performing fuel. Maximum specific impulse of 282.7 seconds is obtained at a metal content of about 33% (mass of fuel/total propellant mass). Figure 1 shows the specific impulse is relatively flat between 25 and 40% metal fuel content. To ensure sufficient working fluid and minimum solid products, the metal content should be the minimum to achieve maximum specific impulse.

Silicon powder is the next highest performing fuel. It has a maximum specific impulse of 272.2 seconds at a fuel content of 30%. Figure 2 shows that the specific impulse is relatively flat between fuel contents of 20 and 30%, but drops rapidly at fuel contents above 30%. This suggests that the monopropellant fuel content should be kept close to 20%. Any error should be on the high side of 20% to minimize specific impulse loss.

ALUMINUM/LOX MONOPROPELLANT PERFORMANCE

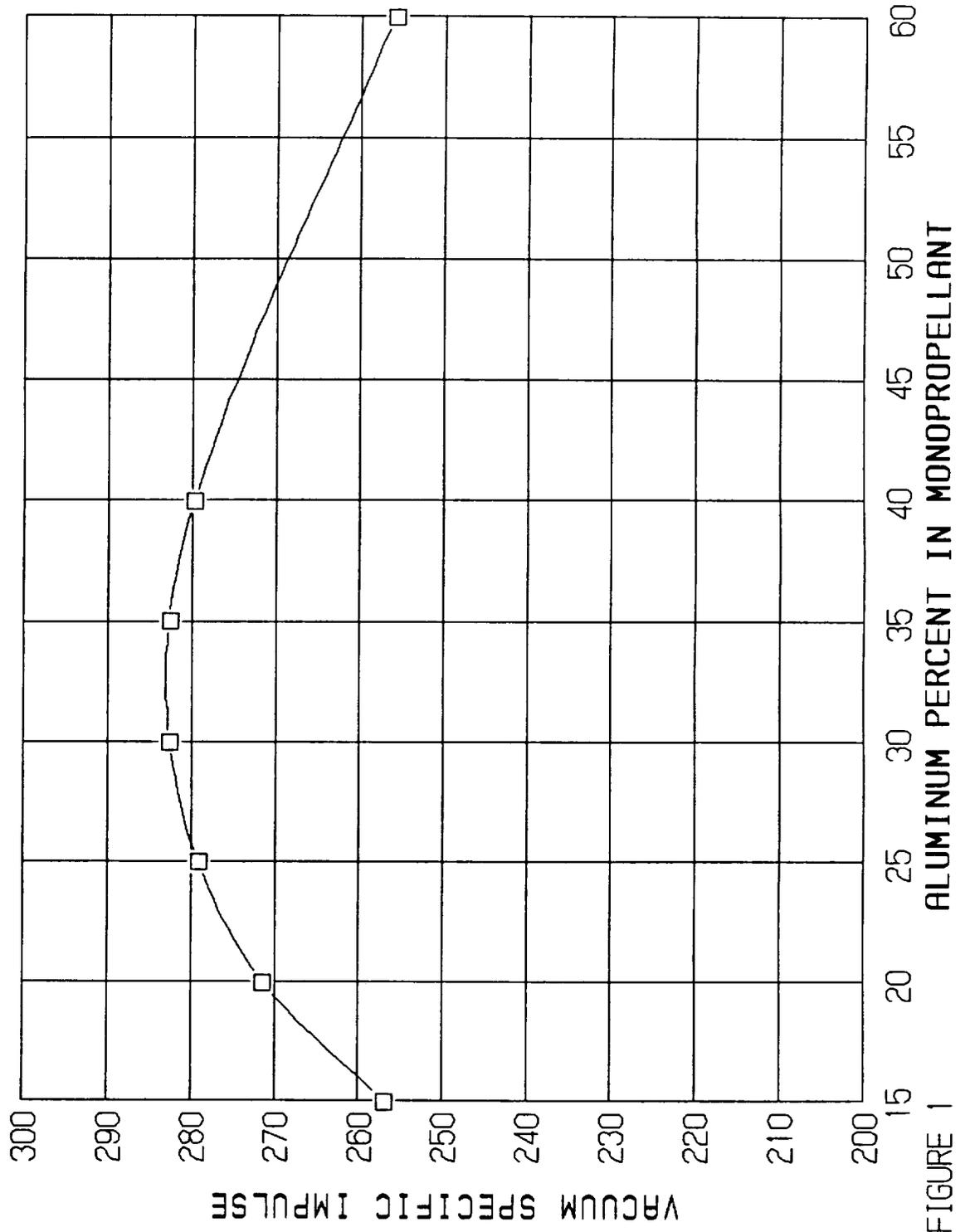


FIGURE 1

SILICON/LOX MONOPROPELLANT PERFORMANCE

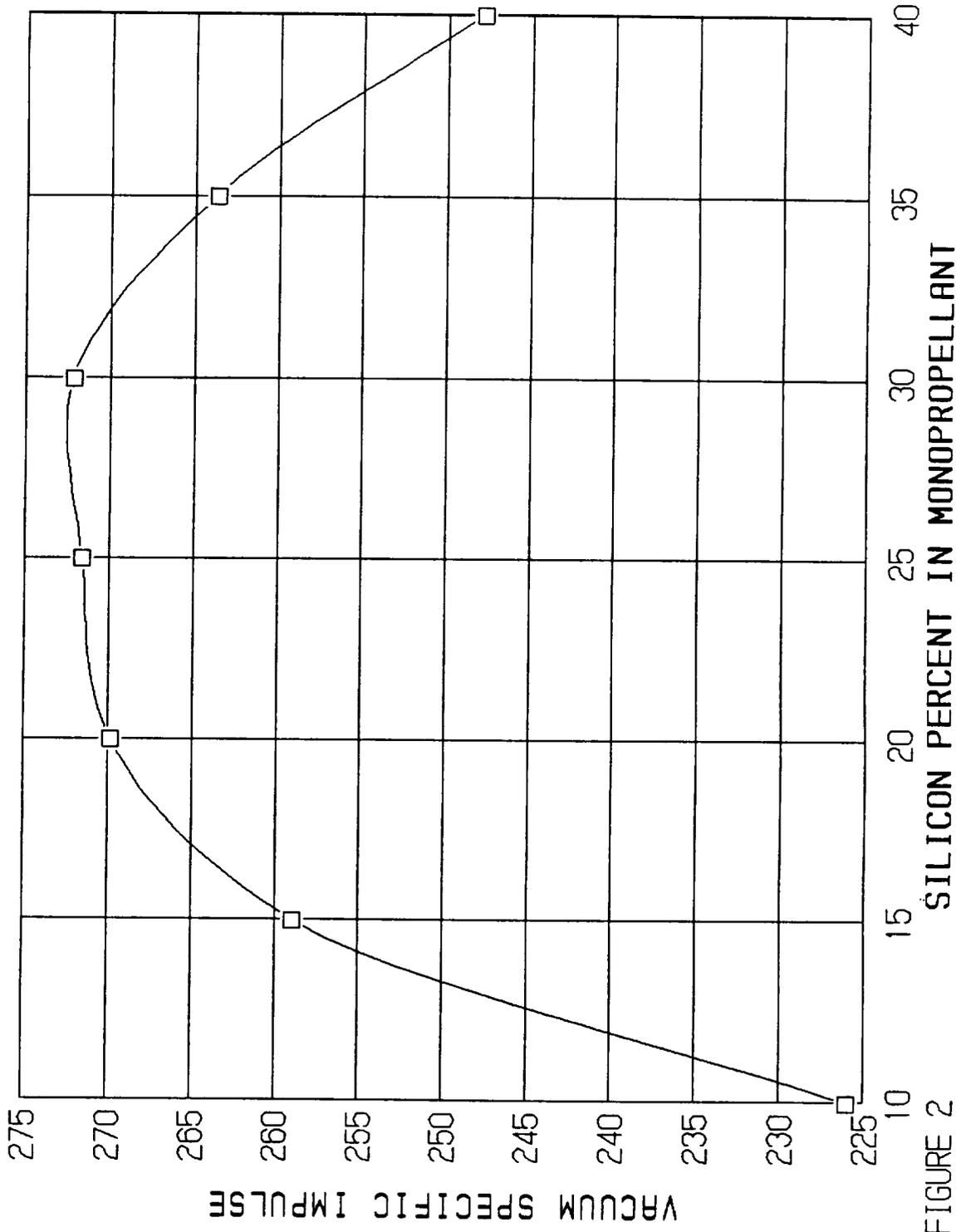


FIGURE 2

Titanium and oxygen have a maximum specific impulse of 255.3 seconds at a metal content of 35%. The variance of specific impulse with metal content is very small between 30 and 40%. As with the other metal fuels, the metal content can vary significantly without a large effect on specific impulse (Figure 3).

Iron is the worst performing metal powder. Its specific impulse only reaches a maximum of 183.1 seconds at a metal content of 35% (Figure 4). It does not have a range of mixture ratios where the specific impulse is relatively constant. Since its performance is almost a full 100 seconds below the best performing metal powder, aluminum, it would seem that iron is not worth pursuing as a monopropellant fuel.

The last metal fuel evaluated was an aluminum/magnesium alloy consisting of 80% aluminum and 20% magnesium by weight. The magnesium in the alloy lowers the energy output of the monopropellant resulting in a slightly lower specific impulse by about 3 seconds. Figure 5 shows a comparison of specific impulse between the pure aluminum and the aluminum/magnesium alloy. The maximum specific impulse of 281.1 seconds for the alloy is reached at a metal content of 40%. The specific impulse is relatively constant between 35 and 45% metal content.

TITANIUM/LOX MONOPROPELLANT PERFORMANCE

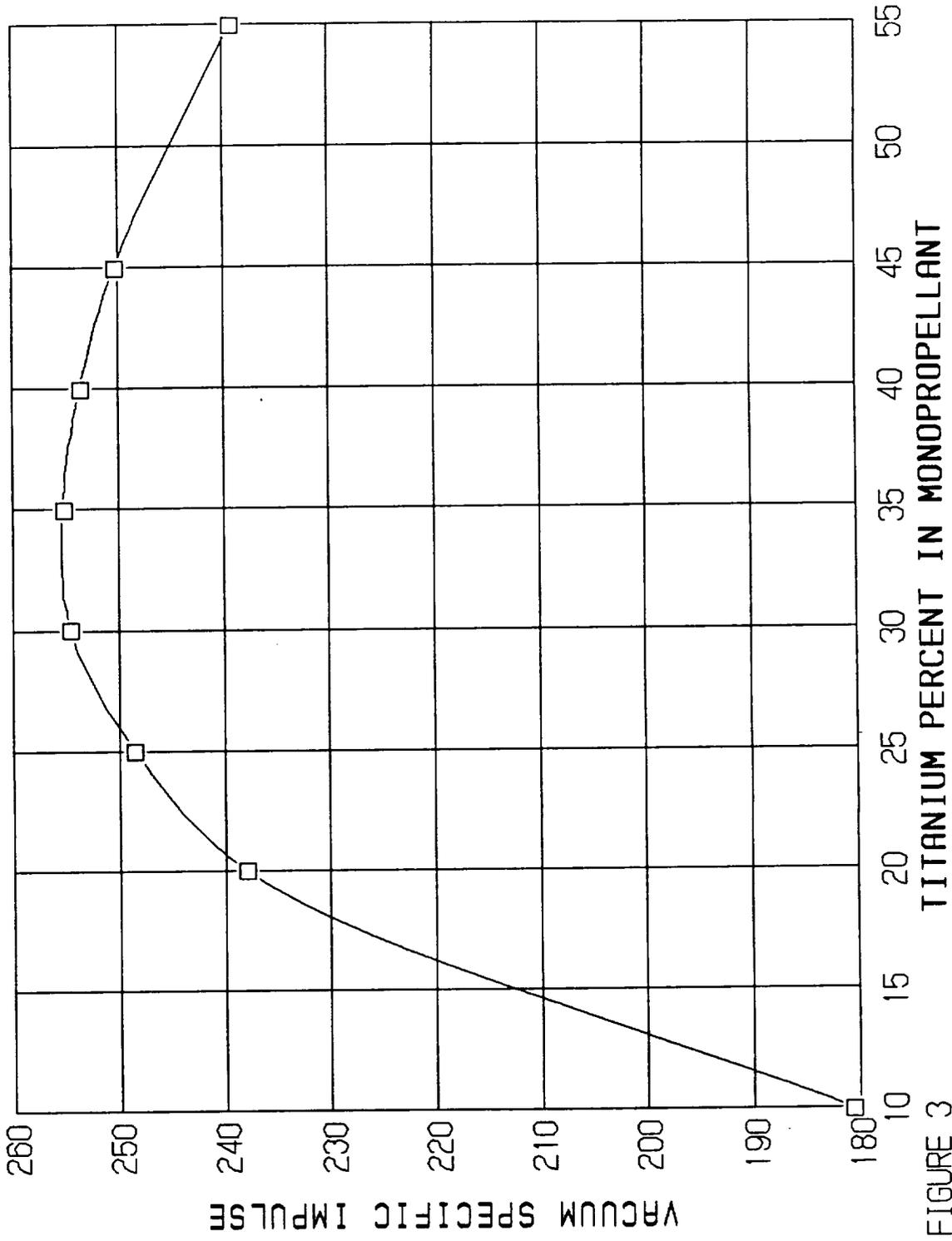


FIGURE 3

IRON/LOX MONOPROPELLANT PERFORMANCE

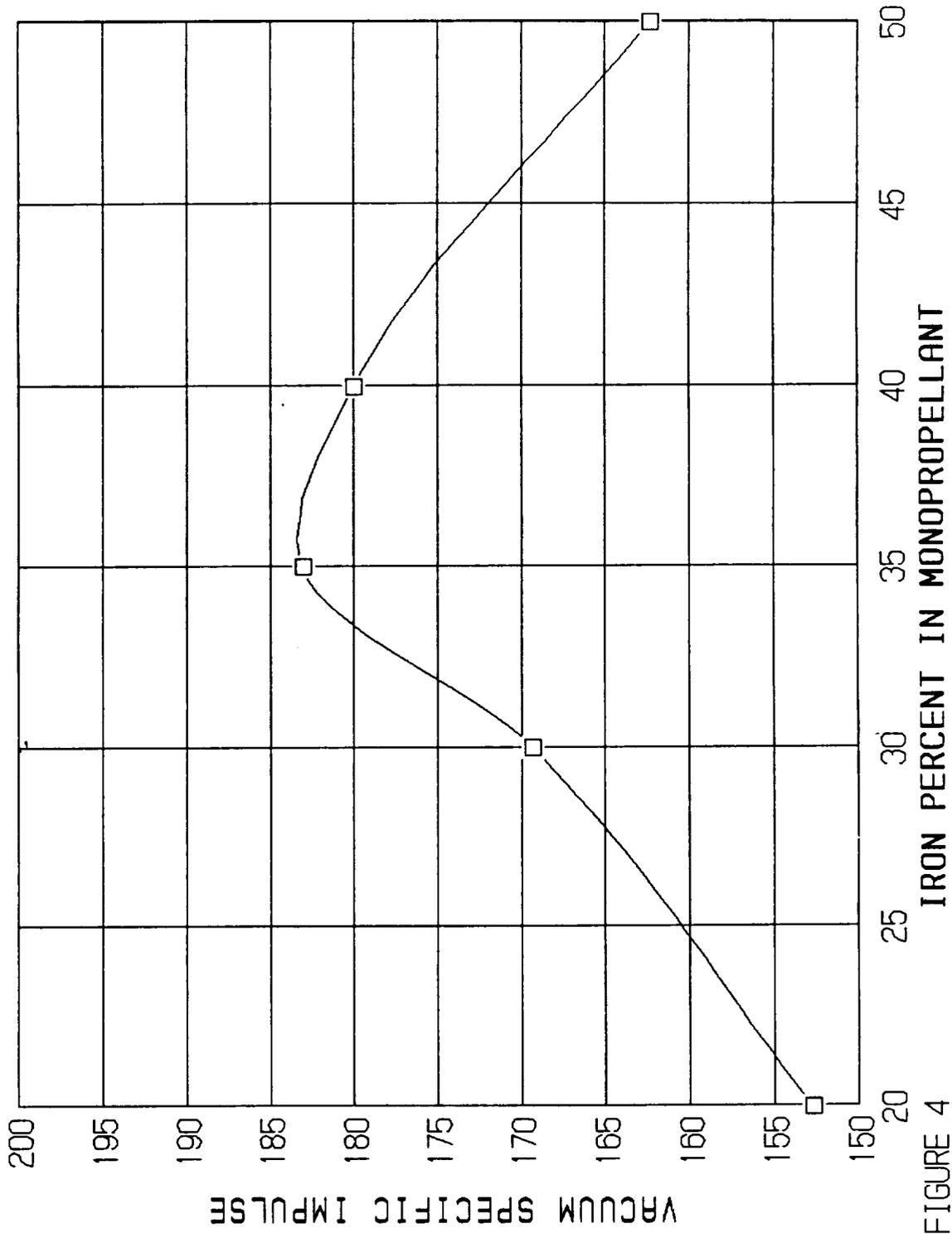


FIGURE 4

AL & AL-MG/LOX MONOPROPELLANT PERFORMANCE

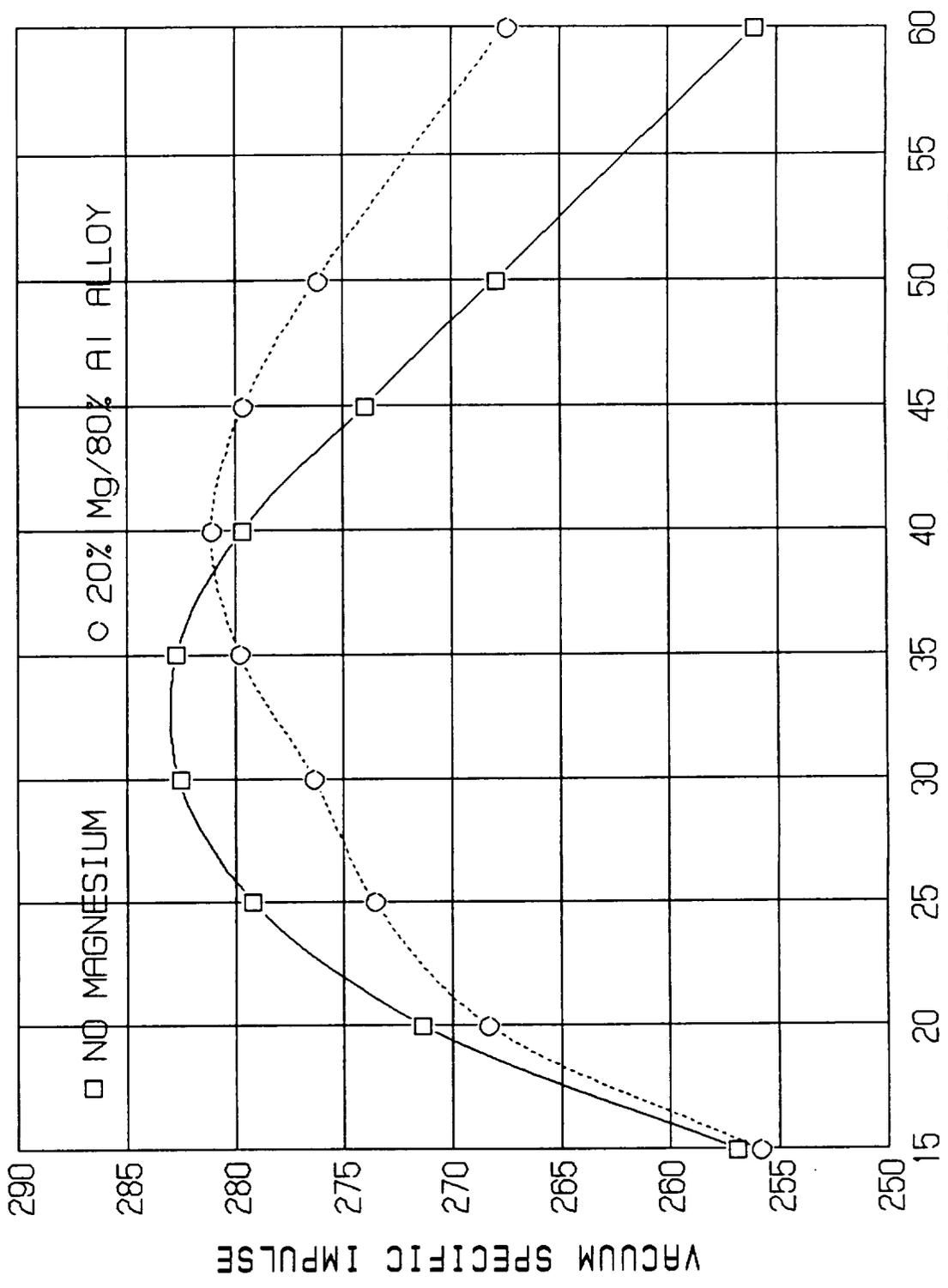


FIGURE 5

2.3 Monopropellant Formulations for Task II Testing

Based on Task I results, iron was eliminated from the list due to its low performance. Shock sensitivity tests by NASA White Sands eliminated titanium from the test matrix due to its extreme sensitivity to any impact. A weight from a height of 6 inches consistently caused a reaction 100% of the time.

The aluminum and silicon formulations did not react when subjected to the maximum shock by tests at White Sands. This showed that these formulations were safe to handle in small quantities. The aluminum/magnesium formulation did react 50% of the time when subjected to the weight drop test from a height of 20 inches. The addition of 20% magnesium to the aluminum increased the shock sensitivity from the pure aluminum formulation. However, the aluminum/magnesium formulation was safe enough to be tested in Phase II using the proper safety precautions.

3.0 TASK II

The candidate monopropellants were mixed and tested during Task II of the program. The experiments focused on the measurement of viscosity, burn rate and metal powder settling for the monopropellant formulations. There were four basic experiments performed in Task II.

The first test was a simple burn test to see if the metal powder would detonate when ignited in liquid oxygen. All monopropellants that detonated upon ignition would be eliminated from the test matrix. The second test series measured the viscosity of the monopropellant as a function of shear rate. The third series of tests measured the settling of the fuel powder in the liquid oxygen over a period of several hours. The last test series was the ambient and high pressure burn rate tests. These tests sought to measure the monopropellant burn rate at a variety of pressures.

3.1 Monopropellant Testing Sequence and Logic Diagram

Each fuel powder and liquid oxygen formulation was initially screened by detonation tests. If the monopropellant only burned and did not explode, propellant formulation work continued for that fuel loading.

The monopropellant formulation began with the viscosimeter tests (Figure 6). Each monopropellant candidate

PROPELLANT FORMULATION FLOW CHART

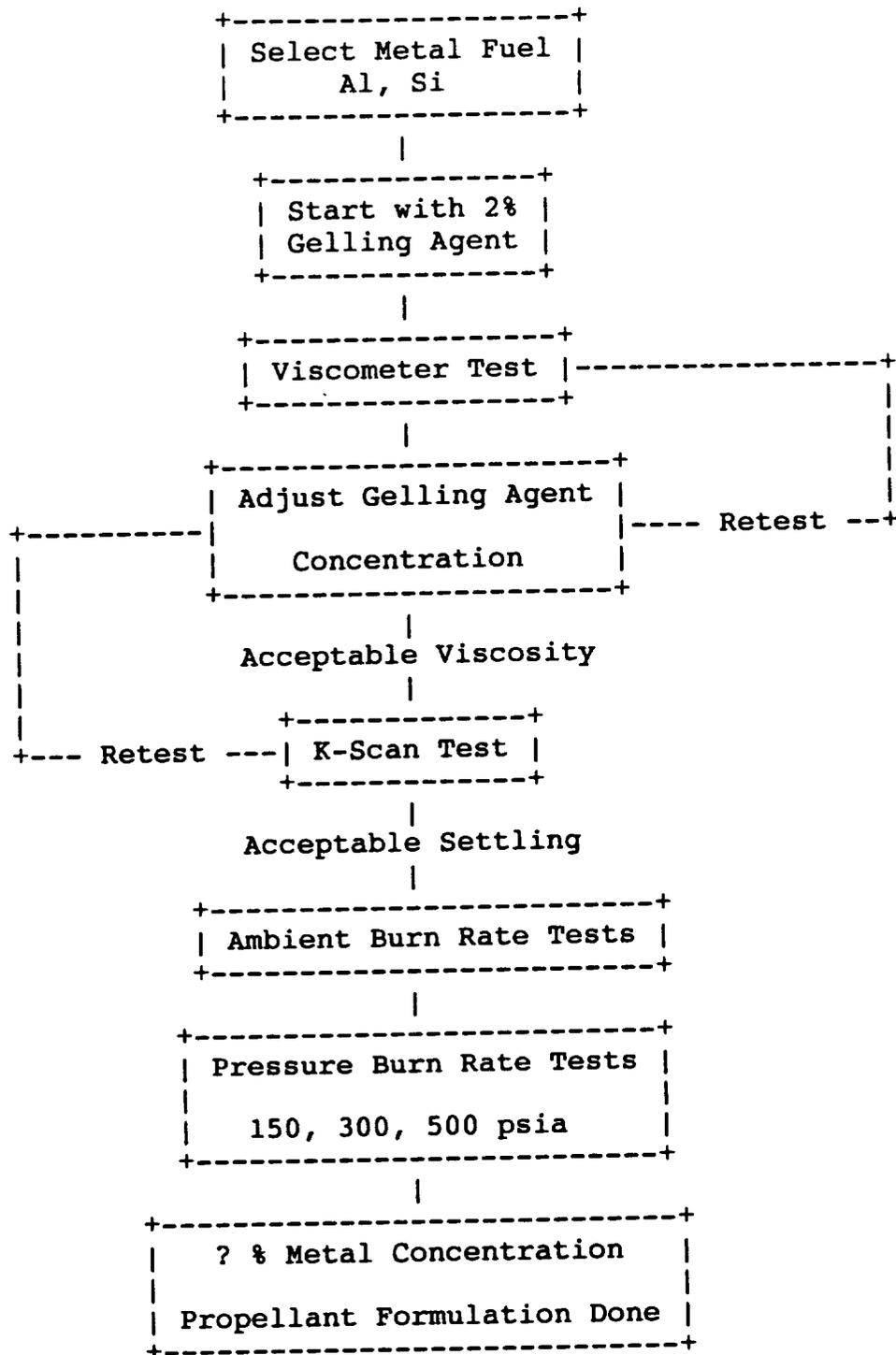


Figure 6

started with the silicon dioxide (Cab-O-Sil) level set at 1% by weight. Based on the measured viscosity, the amount of Cab-o-sil was increased or decreased. Once the viscosity was within acceptable limits, the fuel powder settling was measured using settling tubes. If the settling was outside of the acceptable range, then the amount of Cab-o-sil was increased to reduce the settling. The formulation was retested in the viscosimeter and then retested for settling. After successfully completing these tests, the ambient burn rate of the formulation was measured. At the end of this test, the propellant formulation was complete for that metal and concentration level.

3.2 Test Equipment

The test equipment used was specially built for this program. Since the monopropellants could potentially ignite during testing, it was decided to build low cost, sacrificial equipment for the measurement of viscosity. The other equipment had to be built since there was no off-the-shelve equipment for measuring the burn rate of a cryogenic monopropellant. While commercial equipment did exist for measuring settling, it used nuclear materials and was very expensive. Consequently, equipment was also built to determine the settling of the fuel powder in the monopropellant.

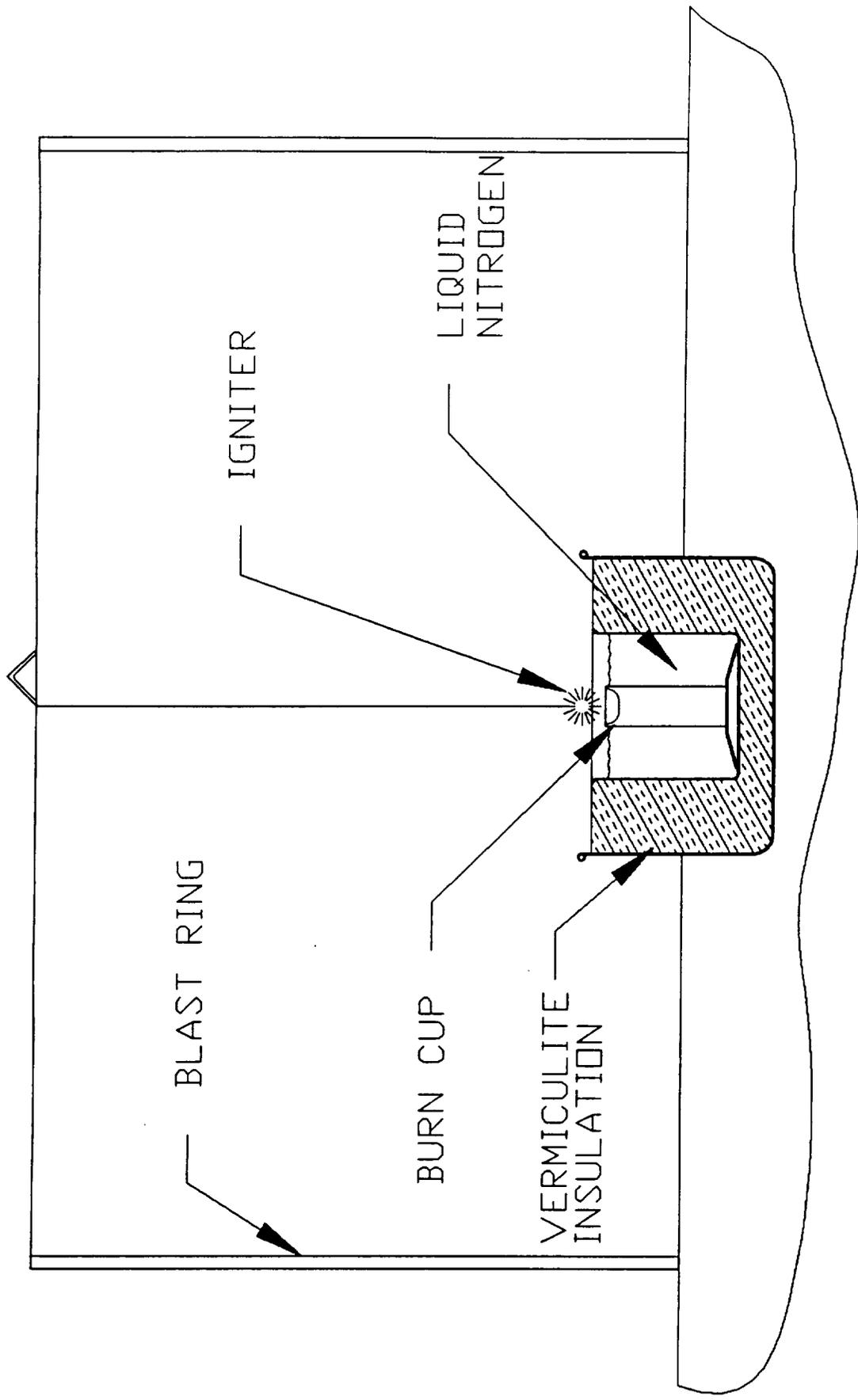
3.2.1 Burn Rate Tests

Burn rate tests were conducted to measure the speed at which the propellants burn. This data was necessary for engine design and to see if combustion was likely to propagate up the monopropellant feed line and into the storage tank. If the burn rate was found to be too high, the monopropellant could not be injected fast enough to prevent burning in the feed line.

Detonation Tests

The detonation tests were done first to determine whether the oxygen/metal powder mixtures would explode when ignited. The results of this test were used to determine whether the particular monopropellant formulation merited continued testing.

The detonation test was a simple setup (Figure 7). A solid aluminum bar with a depression machined into one end was mounted to a base plate. The entire unit was set into an aluminum beaker filled with liquid nitrogen. The beaker was set inside an aluminum pot packed with vermiculite insulation. Liquid oxygen was then poured into the cup and an igniter suspended above it. The igniter was a small piece of solid propellant with an electrical squib inside it. After the area was cleared, the igniter squib was energized remotely with the results recorded by a video camera. The detonation tests were conducted inside a 42 inch diameter steel ring with 1/2 inch



DETONATION TEST

FIGURE 7

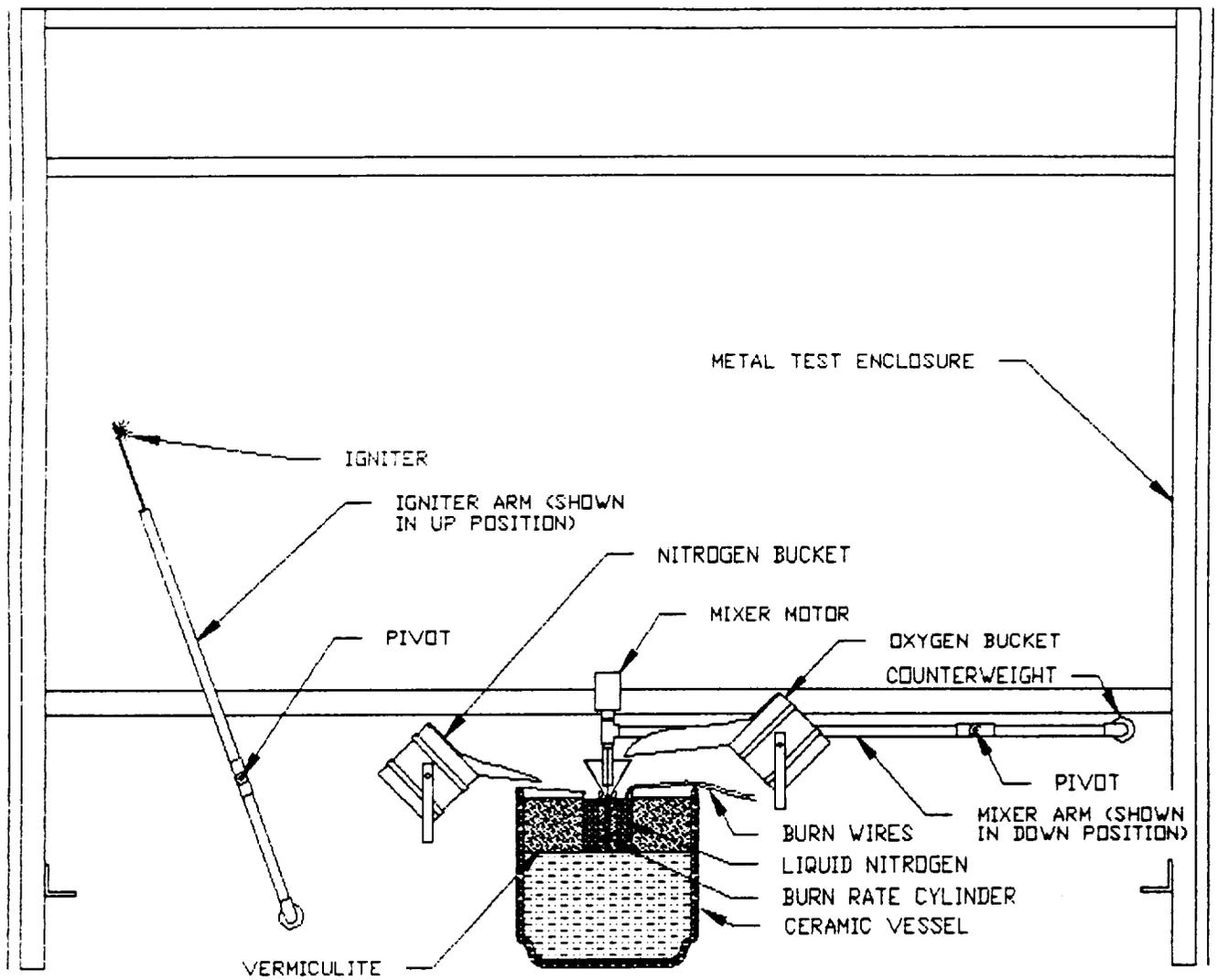
thick walls for blast protection. The entire apparatus was sited in an open field, well clear of structures.

Ambient Burn Rate Tests

Once detonation tests determined that the mixtures were not explosive, additional burning rate tests were done with the monopropellants. Figure 8 shows the system used to make these tests. Two wires 2 inches apart were installed in the burn chamber. As the mixture burned, first one wire burned through and then the other. A computer monitored the voltage in the wires. When a break occurred in the wires, it recorded the time. The distance between the wires divided by the time interval between wire breaks yielded the burn rate.

Like the detonation tests, these tests were done remotely. The equipment in this test series was required to have many more functions than before and every function was to be performed remotely. The monopropellant was mixed while the nitrogen bath level to maintain the monopropellant was monitored and filled. After mixing, the mixing equipment had to be retracted out of the way so that an igniter could be placed over the opening of the burn rate chamber. Finally, the monopropellant was ignited and the burn rate measured.

The heart of the system was the burn rate chamber. This was a 1-1/2" diameter aluminum rod with a 1/2" diameter hole bored four inches deep into one end. There were



BURN RATE TEST

FIGURE 8

two fittings in the side of the cylinder that contained special wires that melted apart when exposed to the burning propellant. The propellant was mixed in the chamber which was kept cold by being immersed in a liquid nitrogen bath. The bath was contained in an aluminum beaker. The beaker was set in a larger ceramic pot and packed in vermiculite insulation. Two aluminum pouring buckets, one for liquid nitrogen and one for liquid oxygen, were mounted in yokes that were tipped by DC motors. The yokes were on an adjustable mount and fastened to the enclosure frame.

Since the nitrogen was constantly boiling away, the level was maintained by a bucket that poured more nitrogen into the container surrounding the burn rate chamber. This was done automatically by measuring the nitrogen level with a float activated switch. When the switch was opened by the falling float, the test computer detected the break in the circuit and signaled the bucket to fill the nitrogen chamber until the float again closed the switch.

The metal fuel powder was placed in the burn rate chamber by hand during set up. Liquid oxygen was poured into the burn rate chamber by a remotely operated bucket similar to the nitrogen bucket. The oxygen bucket was activated by the computer based on key board commands given by the operator. By observing the pouring process via the video system, commands were given to pour the oxygen until the burn rate chamber was full.

The igniter was mounted on a wire with the wire attached to an arm. The wire could be bent to position the igniter directly over the burn rate chamber. The arm was lowered to the burn rate chamber, energized, and retracted by the computer.

The burn rate tests were conducted in a test enclosure consisting of a steel frame with corrugated steel sides set in an open field. The metal sides would contain any blast and direct the energy up through the open roof and away from nearby objects. The burn rate chamber was located in the center with the pouring buckets, mixer and igniter arms mounted to the frame. One video camera was also mounted on the frame for viewing the test operation.

Setting up the burn rate tests involved hooking up all electrical connections and video cables. The igniter and burn wires were checked for continuity and proper sequence. The cameras were positioned followed by the burn rate chamber being cooled by filling the liquid nitrogen bath.

For the initial burn rate test, the adding of liquid oxygen, mixing and ignition of the propellant were done remotely. After liquid nitrogen was added to the beaker surrounding the burn rate chamber, the liquid nitrogen pouring bucket and liquid oxygen pouring buckets were filled to their proper levels. The required amount of fuel powder had already been added to the burn rate cylinder before placing the cylinder

in the liquid nitrogen beaker. At this point, the test area was cleared of all personnel.

The computer software for the test was started which monitored the liquid nitrogen bath and automatically kept it at the proper level. The operator poured liquid oxygen remotely into the cylinder by entering in a pour duration into the computer which in turn operated the bucket. When the liquid oxygen reached the top of the cylinder, the computer returned the pouring bucket to its full upright position. The operator then turned on the mixing motor to mix the LOX and fuel powder. When mixing was complete, the operator entered into the computer the firing command which started the final sequence. The computer commanded the mixing arm and blade to be retracted while simultaneously lowering the arm with the igniter attached to it. When the igniter reached its position over the burn rate cylinder, the igniter was turned on. At this point, the computer monitored the burn rate wires to record their time of burn-through. During the entire test sequence, the operation was monitored by the test operator and could be manually overridden at any time.

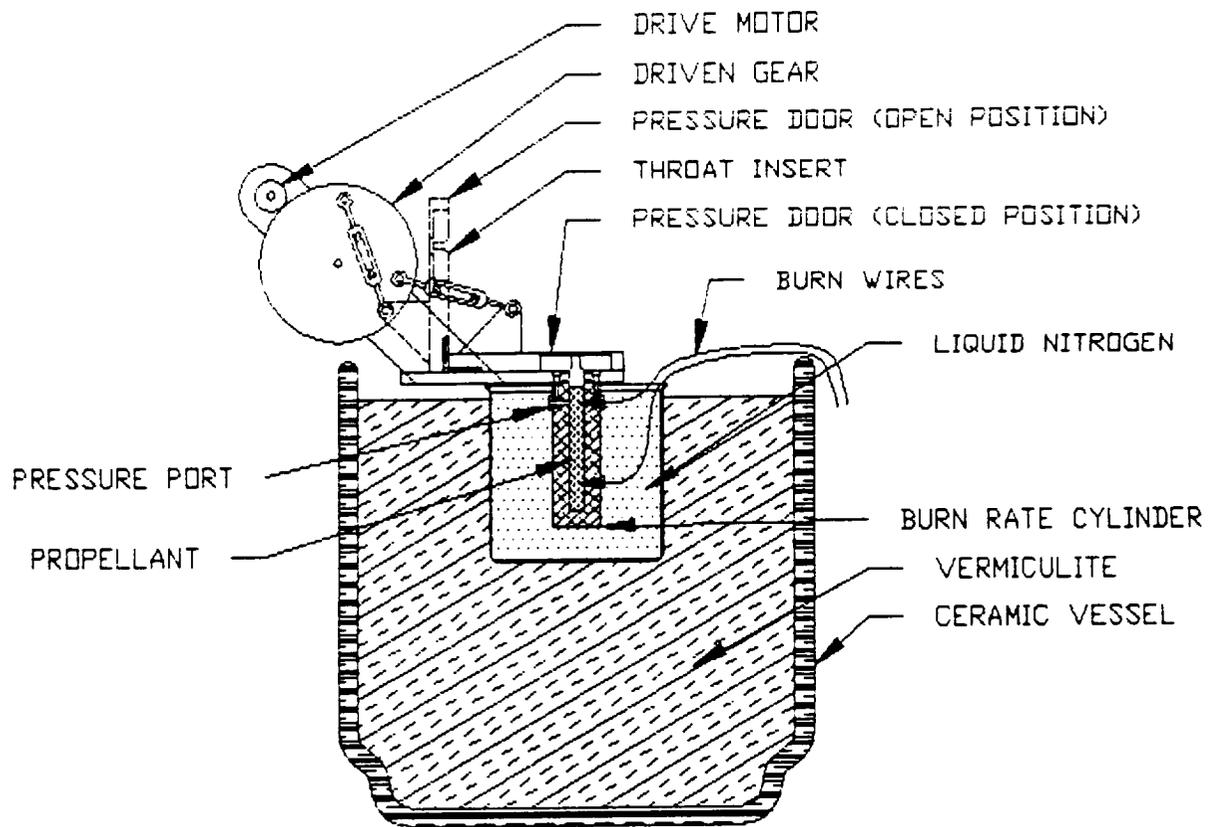
After this first test, it was apparent from the NASA White Sands results and the burn rate test results, that the monopropellants of aluminum/LOX and silicon/LOX could be mixed with a hand mixer. Using this new procedure, the monopropellant was mixed and brought out to the test area where it was loaded into the cylinder. The new procedure was used for the remaining tests.

When the area was safely cleared, the igniter circuit was armed and the computerized sequence begun. The computer lowered the igniter arm and fired the igniter. During ignition, the computer monitored the burn rate wires and simultaneously took periodic still photographs. When the igniter finished burning, the igniter arm was raised out of the way. The computer continued recording data until the number two burn wire circuit was broken. When the burning stopped, the igniter circuit was disarmed and power to the system was turned off. The video tape and computer data files were then examined for test results.

Pressurized Burn Rate Tests

To explore the effects of combustion chamber pressure on the burn rate, a separate series of tests were conducted. These tests shared most of the equipment with the ambient tests. The differences were in the burn rate chambers and the software. The special pressurized burn rate chamber is depicted in Figure 9. The test procedure for the pressurized burn rate test was essentially the same as for the ambient pressure burn rate test.

Since the burn chamber needed to be pressurized, a throat was added to the exit plane of the burn rate cylinder. The throat diameter controlled the burn rate cylinder pressure. A large diameter was tested first and the throat size was then



PRESSURIZED BURN RATE TEST

FIGURE 9

adjusted for a different pressure. The throat insert was mounted in a door that could be opened and closed remotely from the control panel. The reason for the door was so that the propellant could more easily be loaded into the burn rate chamber. A pressure port in the burn chamber lead through a line to a pressure transducer.

Mixing

The chemical stability of the oxygen and fuel powder mixtures were unknown at the beginning of the project. There was a possibility that the propellant could detonate during testing. With that in mind, much effort was invested in developing remote operation capabilities. Stirring the oxygen/metal powder mixture was done by a specially designed wire whip which was activated remotely from the control panel. Experiments using a propellant simulant and a transparent cylinder were conducted to arrive at the best whip design.

The mixing mechanism was mounted on a counter-balanced arm made of copper tubing. A DC motor rotated the arm up and down. The mixer itself consisted of a funnel for pouring the liquid oxygen into the burn rate chamber and a wire whip driven by an electric motor. After the stirring operation, the entire mixing arm was retracted by the computer prior to ignition.

As testing progressed, it became apparent that as long as the propellant was kept below the boiling point of oxygen and away from an ignition source, it could be handled safely. This greatly simplified the burn rate testing. All the complicated mixing equipment was eliminated. The mixing was done in a separate mixer before being loaded into the burn rate chamber. The new mixer used the same type of motorized wire whip, but the mixing was done in a glass beaker set in a liquid nitrogen bath. Since less time was needed between mixing and ignition, the remote filling of the nitrogen bath was also eliminated. The nitrogen was topped off just before the area was cleared for ignition.

Control System

The control system consisted of the control panel and the computer (Figure 10). The panel allowed the operator to begin or interrupt automated sequences, control manual functions, and monitor the test apparatus. It consisted of an array of power switches, indicator lights, and video monitors. The computer operated in conjunction with the control panel and handled the automated sequences and collected test results.

Three video cameras and a still camera observed the operations. One video camera covered the test area. A second camera observed the operations of the various working systems. The third video camera recorded the reaction close-up

and in color. The still camera recorded essentially the same scene as the close-up video camera.

3.2.2 Viscosimeter

To measure propellant viscosities, a rotating viscosimeter was built. Due to the potential for explosion, the rotating design was chosen over other types as it required a smaller fluid volume and did not confine the sample. Like the burn rate equipment, the viscosimeter was considered to be sacrificial and had to be designed accordingly. Though the propellant was not ignited in this test, it was unknown whether the high shear forces could ignite the mixture. For safety, the instrument was operated remotely.

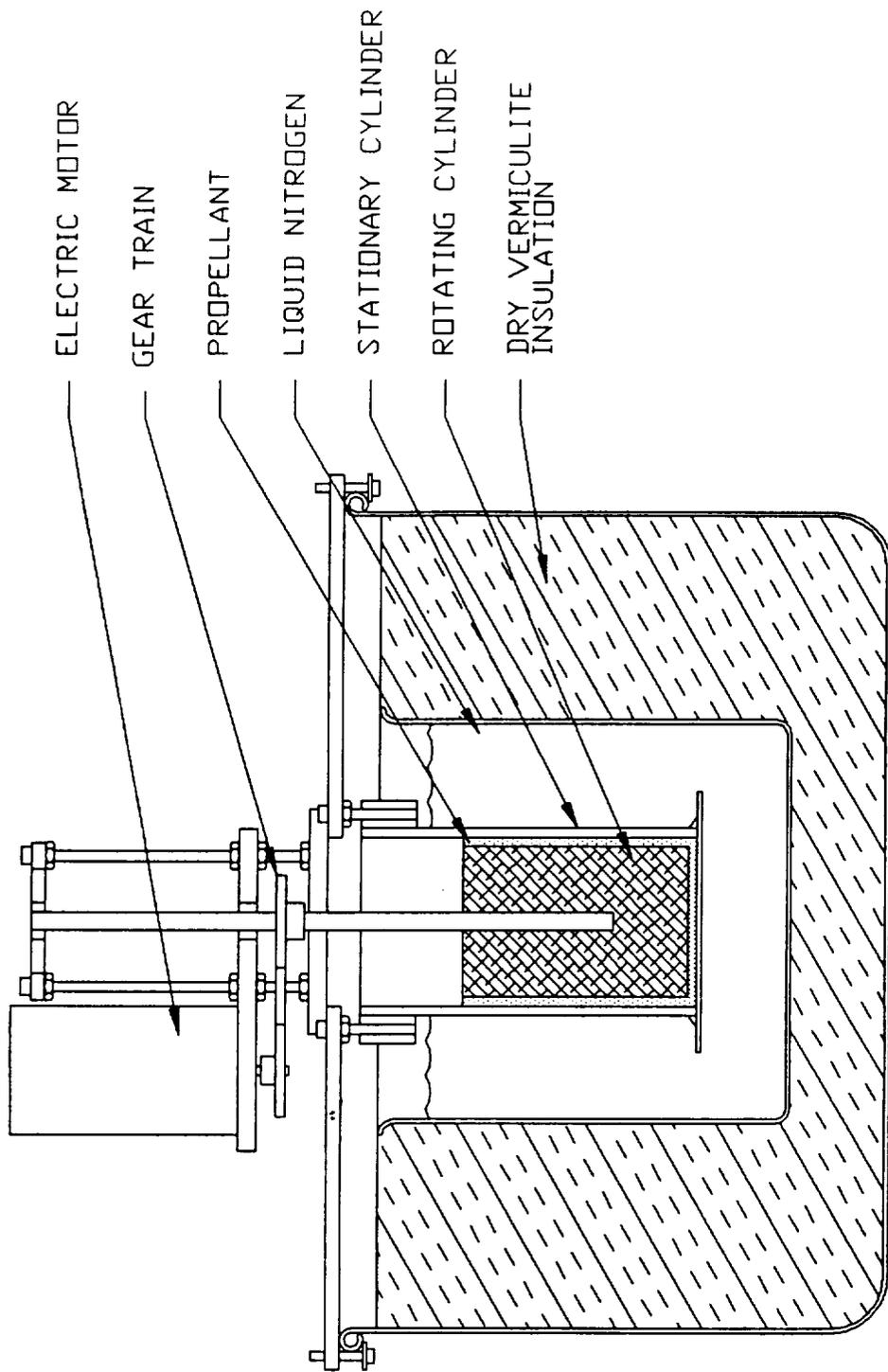
The viscosimeter was required to maintain the sample at cryogenic temperature. To accomplish this, the entire propellant sample holder was immersed in a liquid nitrogen bath. An optical tachometer measured the rotor speed while the shaft torque was determined by measuring the input motor voltage. Knowing the dimensions of the viscosimeter, the input torque, and the rotation speed, the viscosity was calculated. Calibration was done using fluids with a known viscosity (i.e. olive oil and castor oil.)

The first design used a direct drive motor. This proved to be unusable because of dynamic unbalances and an underpowered motor. The final design was driven through a gear

train by a larger motor (Figure 11). Since the rotor had to spin at speeds up to 3000 Rpm, dynamic balancing became a primary concern. Ordinary ball bearings could not be used as the lubricants became solid at cryogenic temperatures and were dangerous to use near liquid oxygen. Polyethylene journal bearings were used instead. The rotor spun inside an open topped cylinder that was suspended in an aluminum vessel filled with liquid nitrogen. The nitrogen container was packed in vermiculite insulation.

The viscosity test was set up by first adding liquid nitrogen to the cooling chamber. The rotor was cooled in a separate bath. When all equipment had cooled to cryogenic temperature, premixed propellant was added to the viscosimeter. The rotor was then carefully inserted into the viscosimeter and all wires hooked up. Any ice that had formed on the rotor shaft was brushed off so that the mark on the shaft could be seen by the optical tachometer.

The viscosimeter was operated using a variable DC power supply to control the rotor speed. The torque supplied by the motor is proportional to the current, so at various rotor speeds the current was recorded. Data points were taken at increasing speeds up to the maximum and then again at decreasing speeds. When all data points were taken, the wires were disconnected, and the viscosimeter disassembled and allowed to reach room temperature.



VISCOMETER

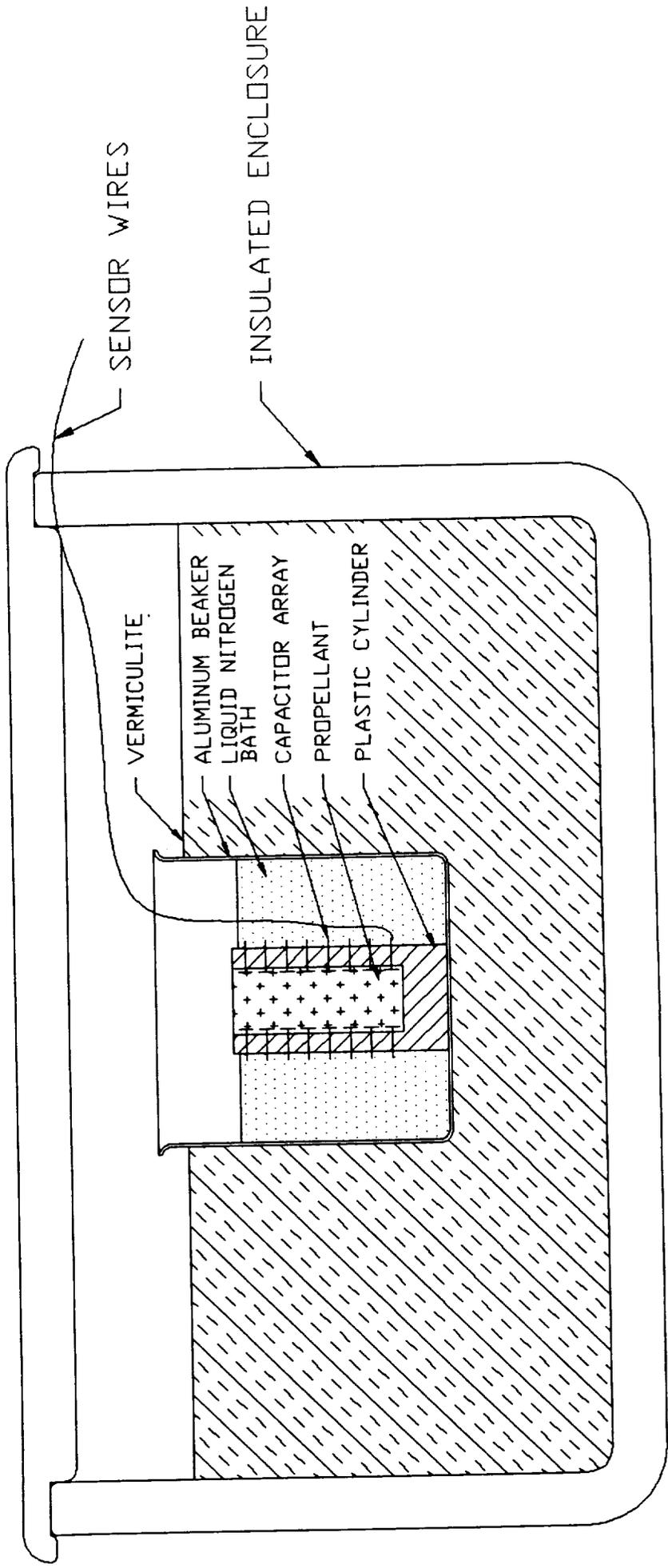
FIGURE 11

3.2.3 Settling Test

The monopropellants are powder in liquid oxygen suspended in a Cab-o-Sil matrix. Since the fuel particles have greater density than the oxygen, over time the powder will try to settle to the bottom of the mixture. The physical and chemical properties of the fuel will change as the metal/oxygen proportion changes. Consequently, it was attempted to measure the settling of the powder with time.

Initially, standard methods that were non-intrusive and non-destructive were used to measure settling, i.e, visual inspection and K-scanning. Visual inspection experiments proved to be unreliable as the reflectivity of the metal powders is so great that it was not possible to visually discern even large variations of density. Clear layers of liquid could be seen, but 20% metal powder looked the same as 80%. K-scanners measure density changes by measuring the change in electrical capacitance of the mixture as it settles (Figure 12). This device worked with a water simulant at room temperature, but the cryogenic temperatures affected the measurements to the point that it was not possible to distinguish real changes from sensor noise.

Since the non-intrusive and non-destructive methods had failed, it was decided to try an intrusive, but non-destructive method. The method chosen to measure settling in the monopropellant was by measuring the rate at which a probe falls



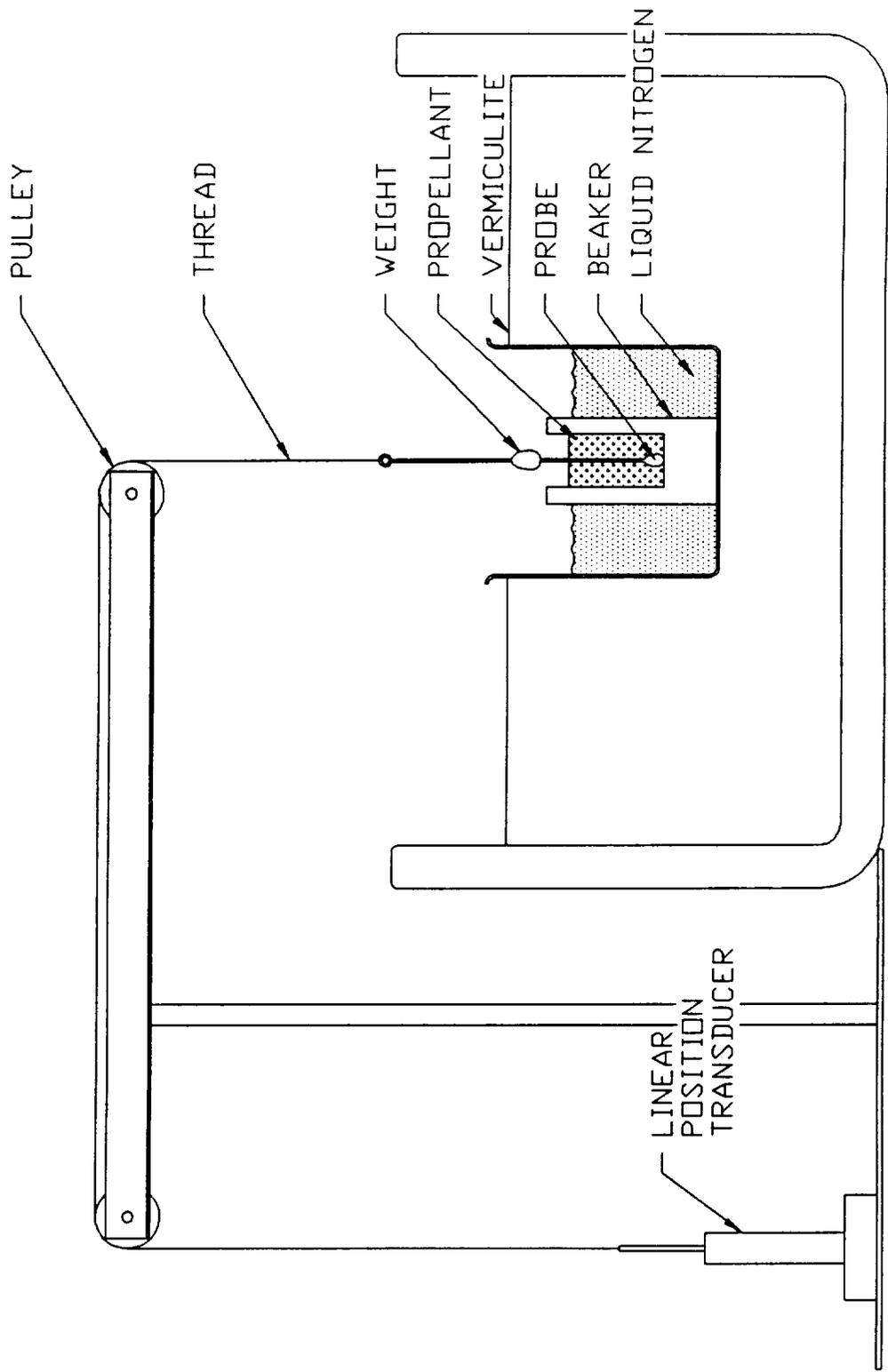
K-SCANNER

FIGURE 12

through the fluid. The rate would be directly proportional to the viscosity of the monopropellant. By assuming that the viscosity is proportional to the metal content, high rates of descent would indicate regions of lower metal content where as slow rates would show regions of higher metal content.

To measure the acceleration of the probe, a thread attached to the probe pulled open a shutter covering a photocell. The output of the photocell was linearized by adding a resistor to the circuit, but the exposure of the cell by the shutter was not easily made linear with distance traveled by the probe. The photocell was then replaced by a linear position transducer which measured the movement accurately (Figure 13). Unfortunately, the falling probe method did not give usable data as the mixture viscosity was affected by factors other than settling. The viscosity was low when subjected to high shear rates and dropped when the shear rate was increased. Some probes would not penetrate at all if lightly placed on the surface while others would fall through without measuring any changes. Experimentation with different probe shapes, sizes and weights gave no usable results.

Since measuring viscosity changes did not work out for measuring settling, the possibility of measuring density changes was considered. One idea was to measure changes in density at different levels by measuring buoyancy of probes with



FALLING PROBE

FIGURE 13

known densities. This method would be intrusive, but non-destructive. However, it was rejected since it would be too difficult to make the buoyancy measurement accurately.

The final method was the least desirable since it was intrusive and destructive. The procedure was to remove layers of propellant from cylindrical samples at measured depths, allow the oxygen to evaporate and weigh the remaining powder. Since the volume of the layer was known, the change in weight of the powder per given volume indicated changes in density. By taking samples at different times, settling could be observed. This method introduced a new source of error since it did not measure the same sample at different times, but measured different samples taken from the same batch.

A sample container was made that could be immersed in a nitrogen bath, would not tip over during sampling, and would not float after the sample was removed. A set of depth gauges and a special scoop were made for this test. Since the test had to be done over a long period of time (several hours), maintaining cryogenic temperature was important. This was done by heavily insulating the nitrogen bath and carefully maintaining the nitrogen level.

The procedure was to load the premixed monopropellant into an array of sample containers to a prescribed depth. When all the sample containers had been loaded they were stirred. At periodic intervals, a monopropellant sample was

removed in four layers and placed into previously weighed beakers. A depth gauge was used to measure and partition the sample into four layers. Each layer was allowed to evaporate and warm to room temperature in a separate beaker leaving only the metal powder and gelling agent. The beaker was then carefully weighed using an analytical balance to determine the remaining weight of powder and gelling agent. The difficulty in this method was in accurately removing a layer without disturbing the remainder of the sample. A special scooping spoon that could fit easily inside the sample cylinders was made to solve this problem.

4.0 EXPERIMENTAL RESULTS AND DISCUSSION

The changes made to the test equipment during the experimental phase of this program are documented in the Section 3.0. This part of the report focuses on the results of the experiments using the final configuration for test apparatus.

The overall objective of the experimental work was to arrive at a monopropellant formulation suitable for further evaluation during Option 2 of this program. Titanium as a fuel was not tested due to its shock sensitivity which made it unsuitable for launch vehicles and spacecraft applications. Iron was also eliminated from the program since its theoretical performance was too low. In place of the iron, an 80/20 aluminum-magnesium alloy was added to the list of candidate fuels. The remaining fuels, aluminum and silicon were tested in monopropellant formulations.

The fuel powder loadings were determined by the NASA program manger based on theoretical performance calculations. They were as follows:

Aluminum:	29 and 35 percent
Silicon:	33 percent
Aluminum-Magnesium (80/20):	29 percent

The logic of the experimental program was to determine the amount of silicon dioxide, in this case the product Cab-o-Sil, to add to the mixture of liquid oxygen and metal powder to form a gel with

a viscosity within the guidelines set by the NASA. When the viscosity goal was reached, the monopropellant was tested in the ambient burn rate equipment and finally in the settling test equipment.

After testing all the metal powders, the NASA program manager selected one formulation to be tested in the high pressure burn rate equipment. The selection made was a 35% loading of aluminum with 2% Cab-o-Sil gelling agent.

4.1 Viscosity Tests

The viscosity of the monopropellant was measured as a function of shear rate. The viscosity goal is shown in Figure 14. The samples were tested up to shear rates of 200 seconds⁻¹ by using a rotating viscosimeter. The monopropellant was mixed in a beaker submerged in a liquid nitrogen bath and then added to the viscosimeter test container.

The rotating cylinder was maintained in a liquid nitrogen bath so that when it was finally added to the monopropellant container boil off of the monopropellant did not occur. The rotating cylinder was placed into the test container by hand and rotated slowly to ensure that the monopropellant was uniformly spread along the sides of the walls. The cylinder was also turned by hand after installation to remove ice which usually formed in the gears leading to the drive motor.

MONOPROPELLANT VISCOSITY GOAL

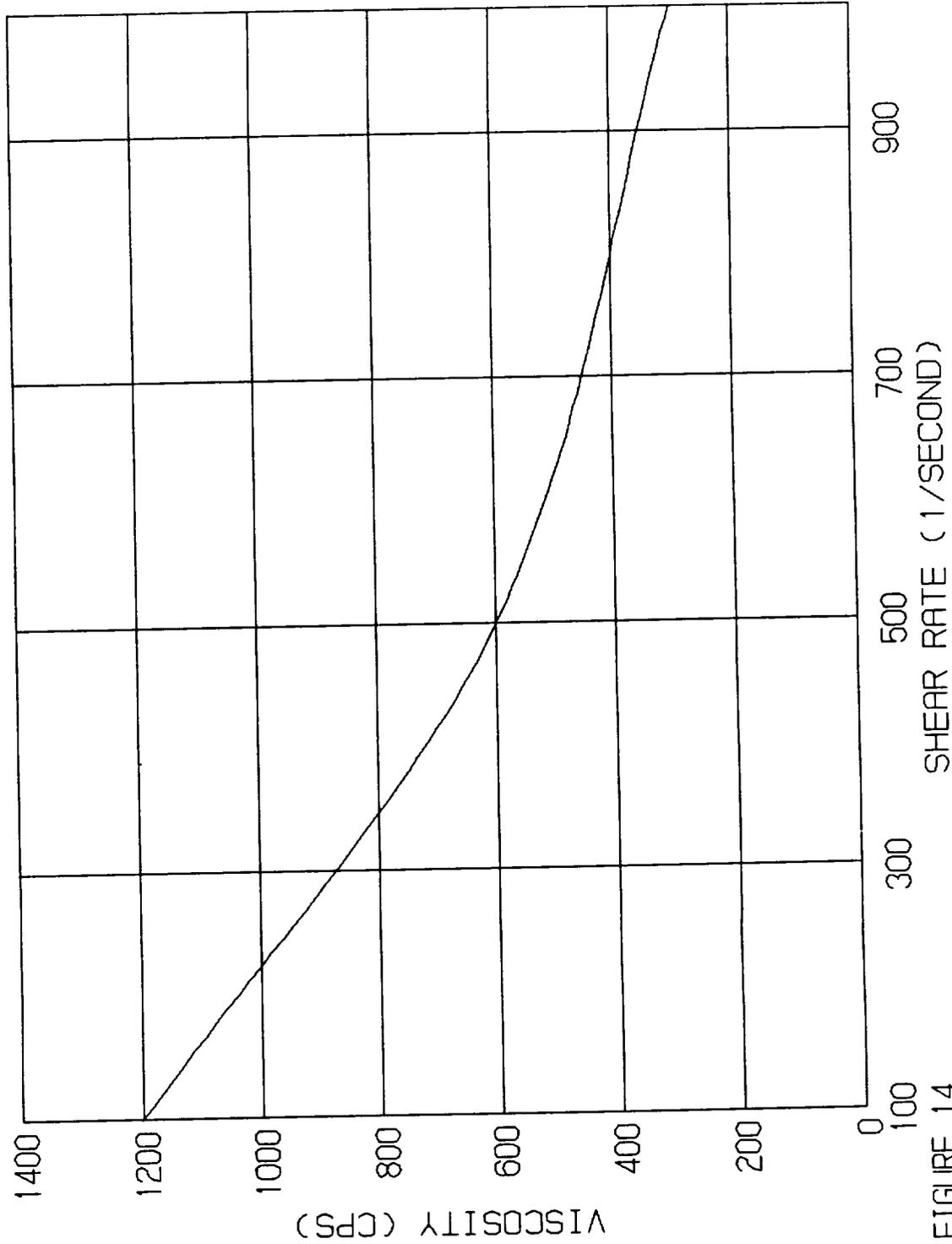


FIGURE 14

The torque on the rotating cylinder was measured by a multimeter measuring the current going to the motor. The torque of the motor was directly proportional to the current drawn by the motor. The revolutions per minute of the viscosimeter were measured directly by an optical tachometer providing a DC voltage output directly proportional to RPM.

The viscosimeter was calibrated using olive oil and castor oil since their viscosities bracketed the range of interest. The results of the calibration are shown in Figures 15 and 16. The values obtained compare closely with those listed in the CRC Handbook of Chemistry and Physics.

35% Aluminum - Liquid Oxygen Monopropellant

The first formulation run in the viscosimeter was the 35% aluminum monopropellant. Initially, a 1% Cab-o-Sil level was tried, but the resulting monopropellant was very "soupy" with a very low viscosity (Figure 17). Notice the sudden drop in viscosity around 100 seconds^{-1} most likely due to the particles being thrown away from the rotating cylinder leaving liquid oxygen behind.

A 2% Cab-o-Sil formulation was tried next and it resulted in a much thicker monopropellant. Its viscosity as a function of shear rate is shown in Figure 18 and is compared to the NASA target in Figure 19. The peak viscosity at a shear rate of 50 seconds^{-1} is about 300 cps. At a shear rate of 150 seconds^{-1} ,

OLIVE OIL AT T=70-75 F

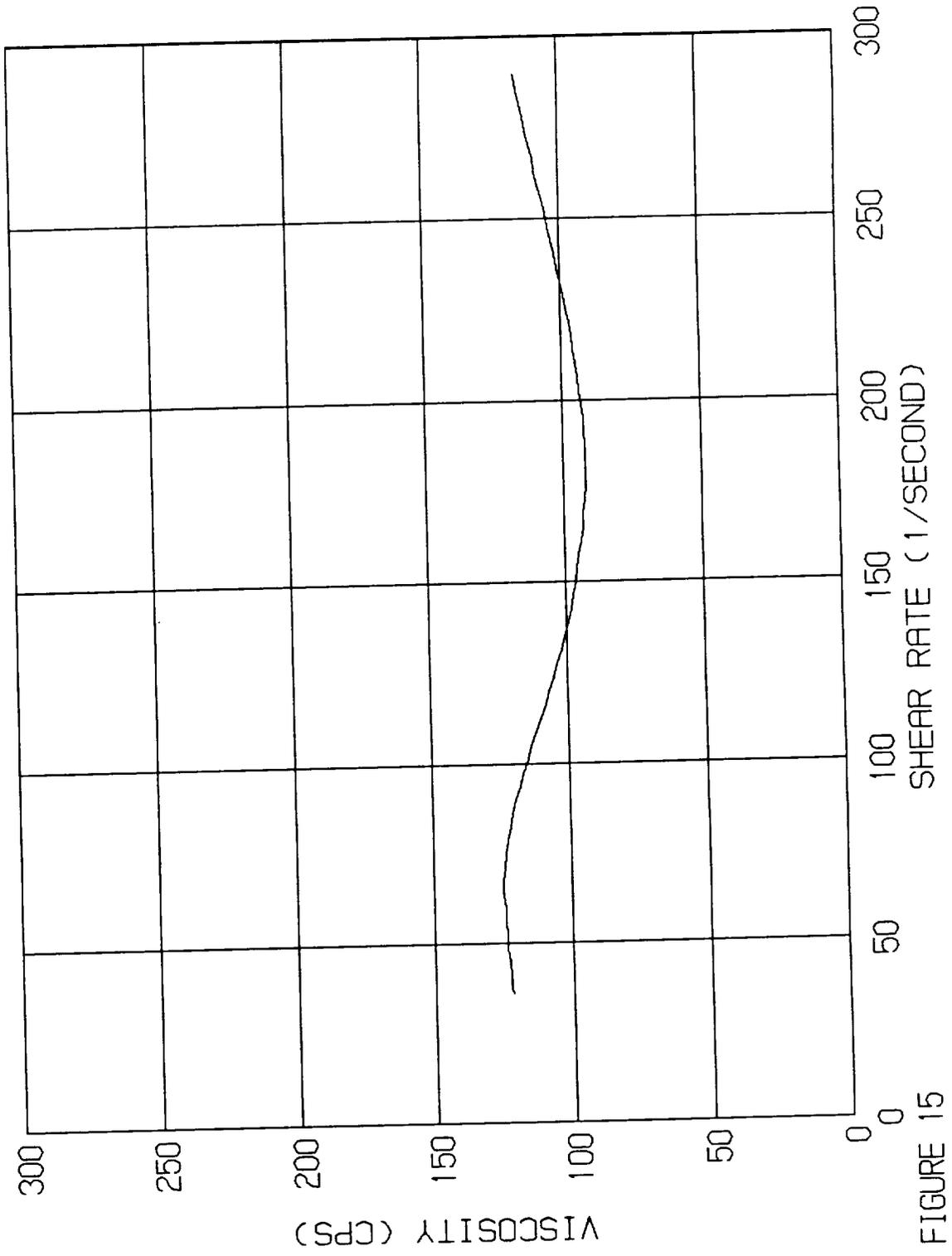


FIGURE 15

CASTER OIL AT T=69-79 F

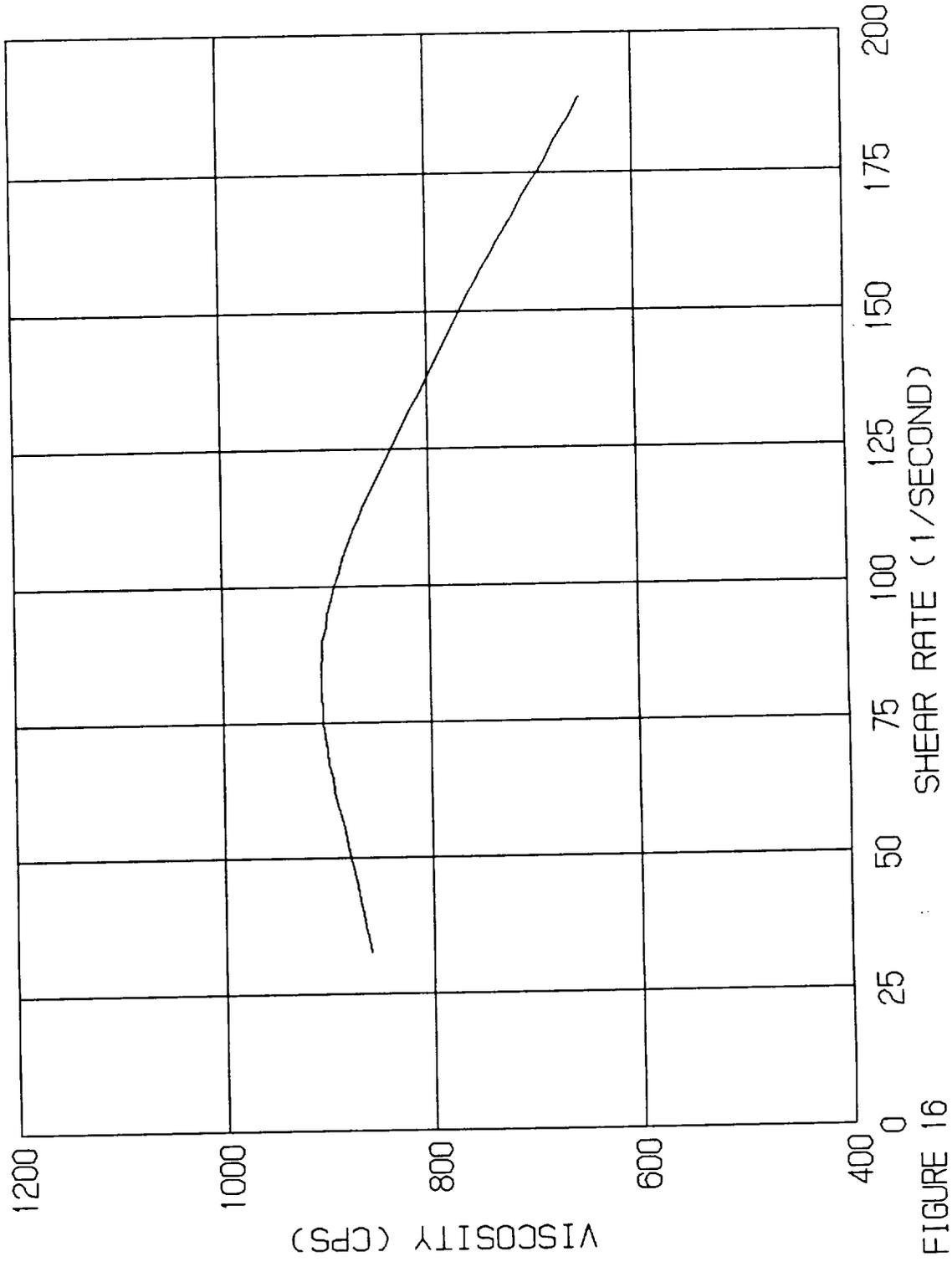


FIGURE 16

35% ALUMINUM/1% CABOSIL

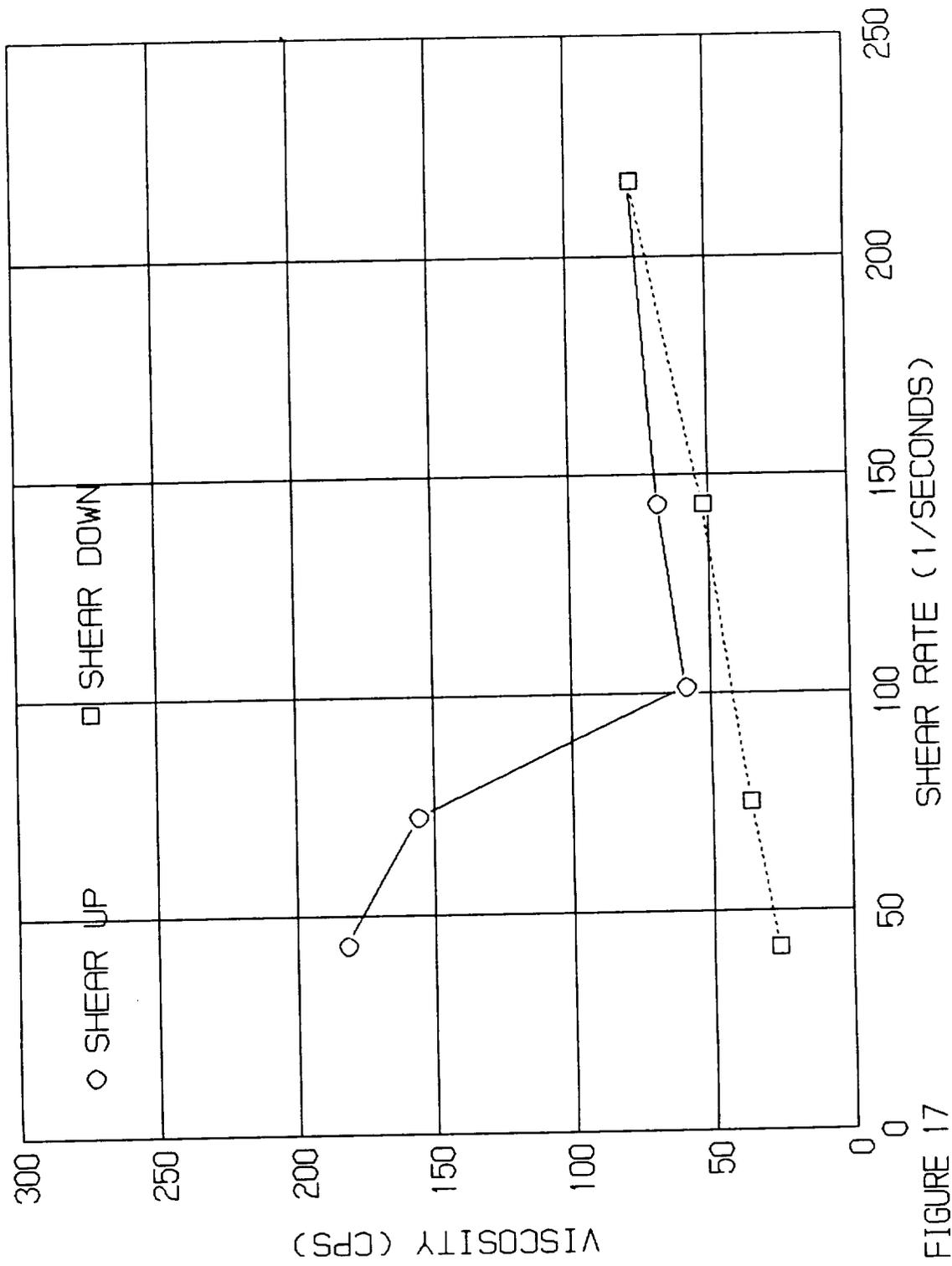


FIGURE 17

35% ALUMINUM/2% CABOSIL

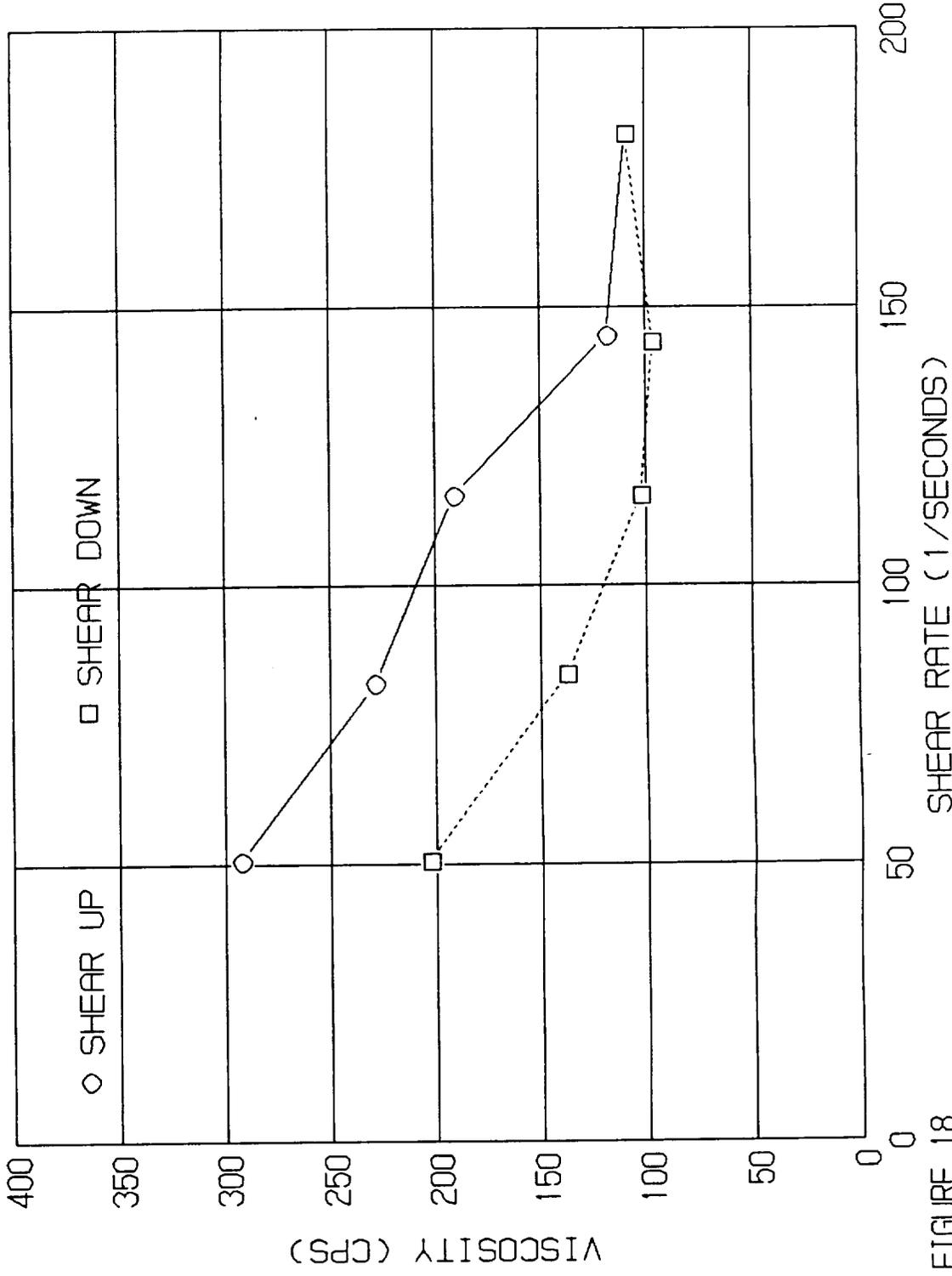


FIGURE 18

35% ALUMINUM/2% CABOSIL

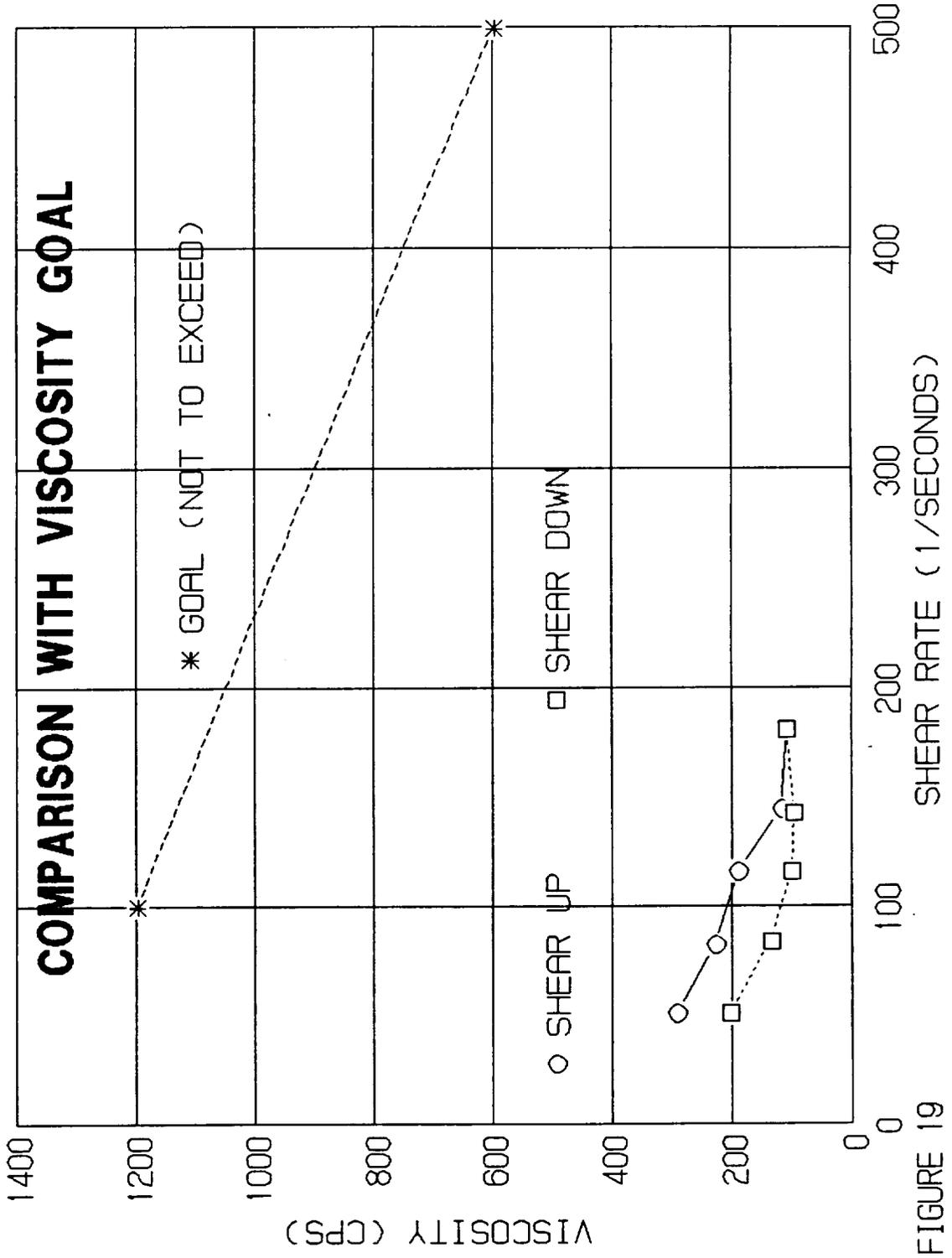


FIGURE 19

the viscosity is down to 100 cps and appears to flatten out with increasing shear rate. As the shear rate is decreased, the viscosity goes up again but at a slightly lower level ending up at 200 cps for a shear rate of 50 seconds⁻¹. Since the viscosity was essentially flat after 150 seconds⁻¹, data beyond this point was not taken. A Cab-o-Sil formulation higher than 2% was not tried since the 2% formulation was already very thick and dry.

While the 2% Cab-o-Sil monopropellant would hold shape, its viscosity was well below the target. Since the target viscosity curve was a not-to-exceed goal, the low viscosity was acceptable. However, it soon was apparent that the aluminum monopropellant had unique physical properties.

The outward appearance and texture of the monopropellant was that of mortar. If a flat trowel precooled to liquid nitrogen temperature was worked across the surface of the monopropellant, liquid oxygen would soon appear at the surface. This is the same effect observed when a trowel is worked across the surface of mortar and water starts to come to the surface. While the monopropellant would retain shape when formed with the trowel, it had very little resistance to movement. This correlated with the low viscosity measurements. In this respect, it has the properties of whipped cream. A very low viscosity, yet the ability to hold a shape.

29% Aluminum - Liquid Oxygen Monopropellant

The 29% aluminum loading formulation was started at the 2% Cab-o-Sil level. This level gave the monopropellant a nice thickness, somewhat less thick in appearance than the 35% aluminum/LOX with 2% Cab-o-Sil. The viscosity of the 29% aluminum loading monopropellant is shown in Figure 20. The viscosity is lower than for the 35% aluminum monopropellant with a leveling off of the curve at about 140 seconds^{-1} shear rate. The hysteresis effect in the viscosity is also more pronounced than for the 35% aluminum formulation.

33% Silicon - Liquid Oxygen Monopropellant

The 33% silicon formulation was initially run with a 2% Cab-o-Sil gelling agent level. This resulted in a "soupy" mixture which had a low viscosity at all shear rates (Figure 21). The gelling agent level was increased to 3%. This gave a much better mixture and higher viscosity levels, although below target values (Figure 22). The mixture also exhibited a "stickiness" to it, almost acting as a mild adhesive. The viscosity as a function of shear rate shows no indication of a sudden drop in viscosity.

29% Aluminum/Magnesium - Liquid Oxygen Monopropellant

The final monopropellant tested was the 29% aluminum/magnesium (80/20) with a 2% Cab-o-Sil level. The

29% ALUMINUM/2% CABOSIL

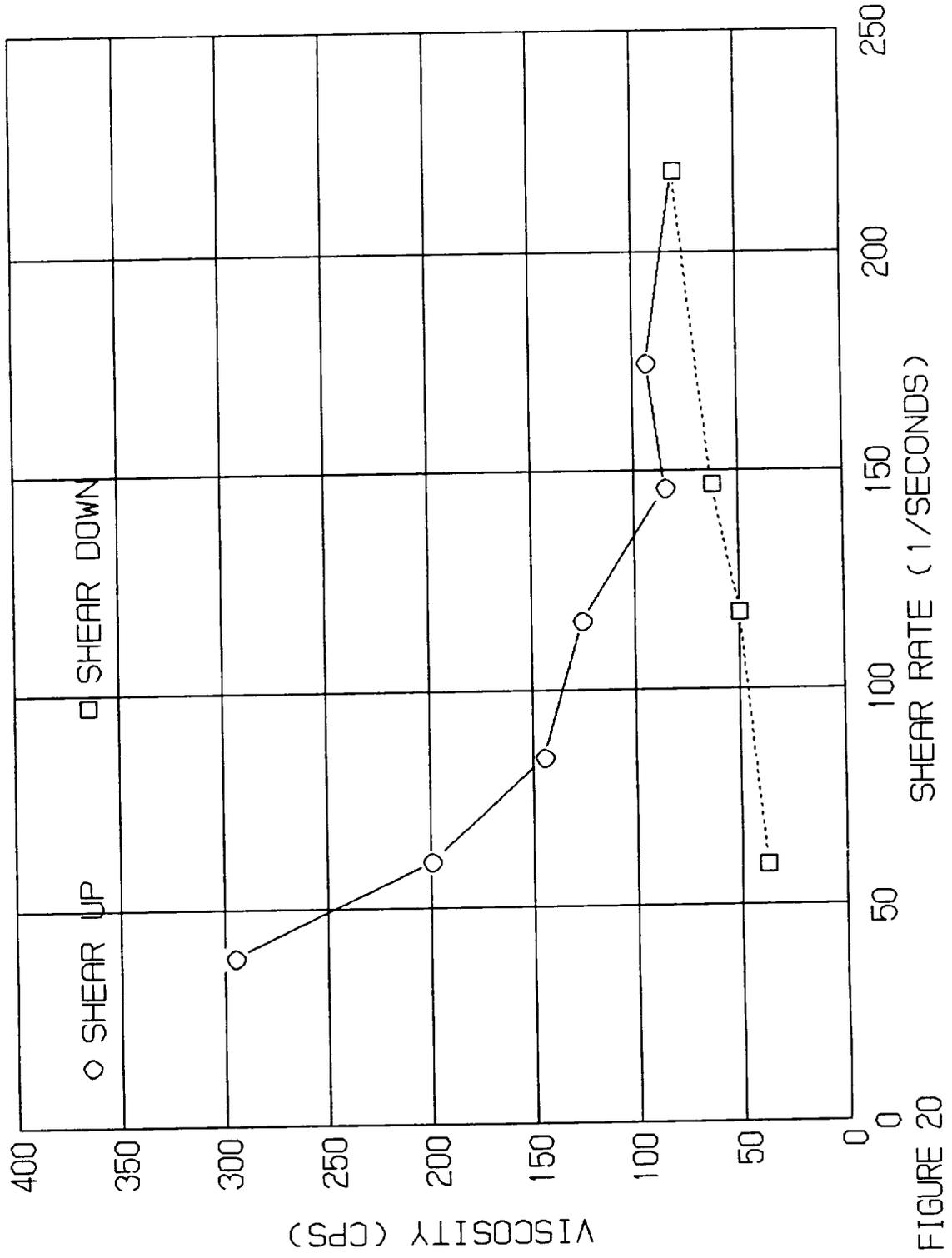


FIGURE 20

33% SILICON/2% CABOSIL

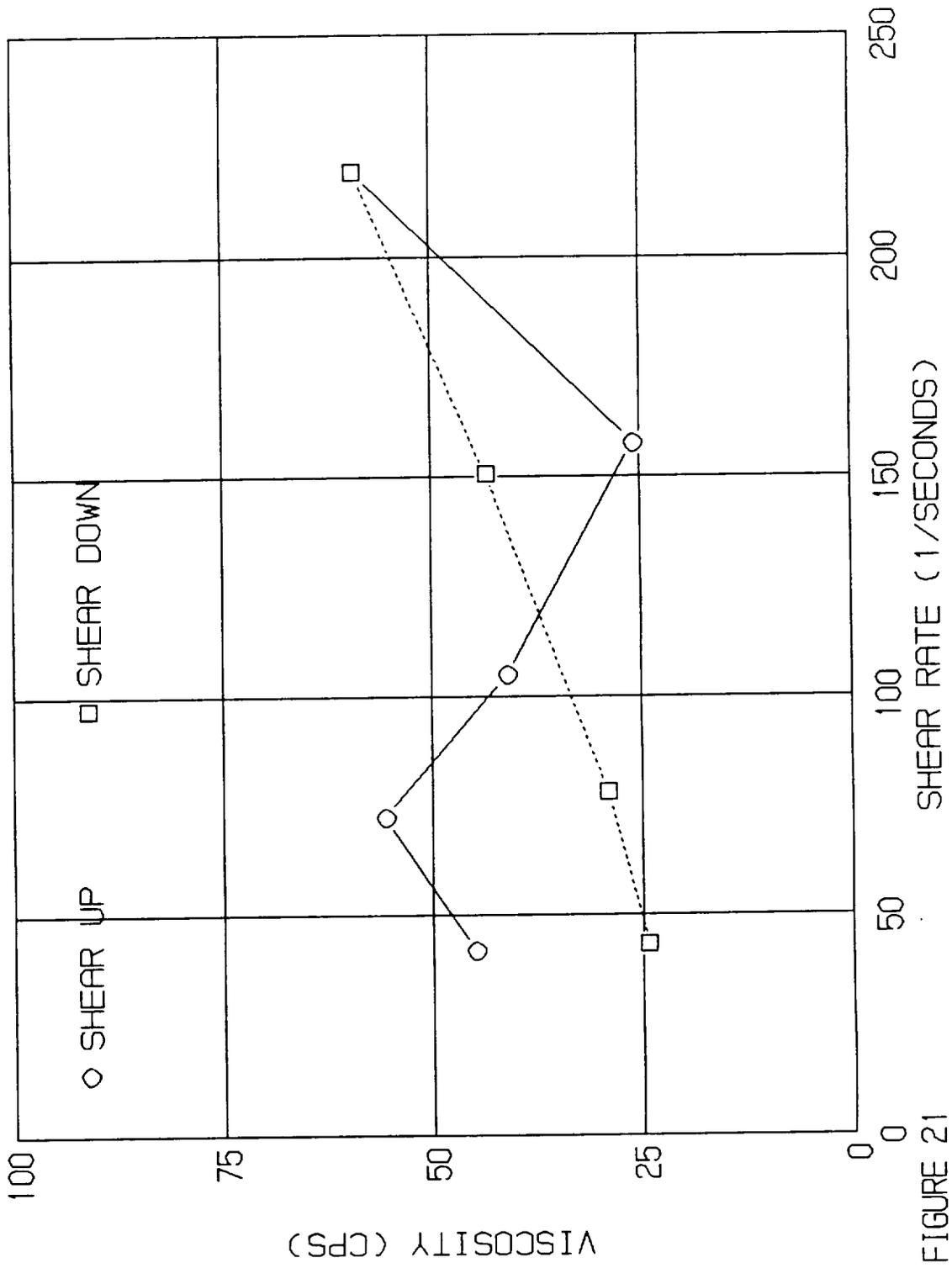


FIGURE 21

33% SILICON/3% CABOSIL

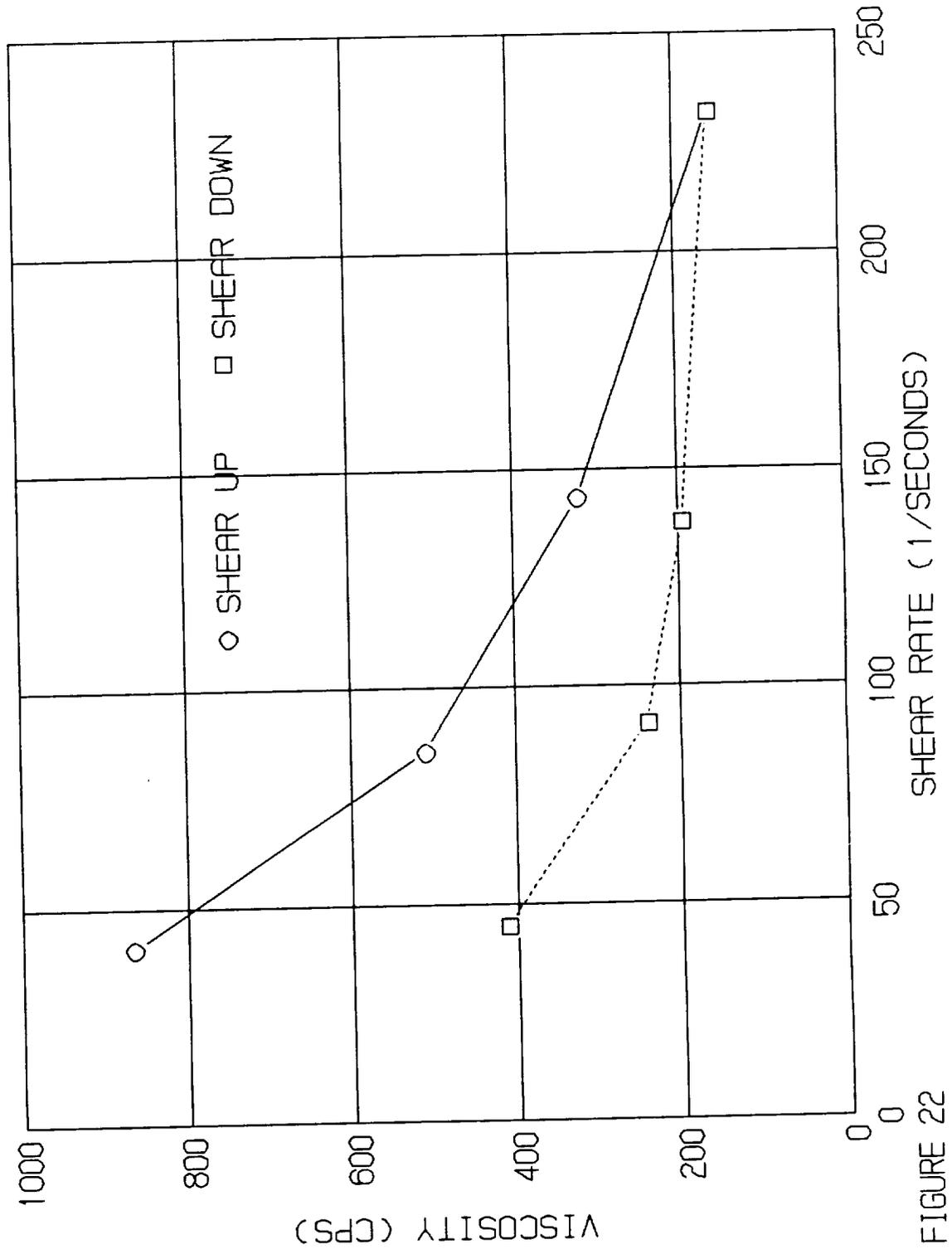


FIGURE 22

formulation had the texture of the 29% aluminum with 2% Cab-o-Sil. Its viscosity yielded a curve with the viscosity becoming flat at about 250 seconds^{-1} (Figure 23). There was no sudden drop in the viscosity as the shear rate increased during the test. However, the viscosity stayed low and did not increase with decreasing shear rate.

4.2 Burn Rate Tests

The burn rate tests were divided into three series of tests. The test series were called detonation, ambient and pressure tests. All of the testing was done remotely for safety.

4.2.1 Detonation Tests

The first series of burn rate tests were called detonation tests. The purpose of these tests was to determine if any of the candidate metal powders would detonate upon ignition when mixed with LOX. About 2 grams of each metal was placed in a small cup machined out of the end of an aluminum rod which was cooled by liquid nitrogen. A small amount of LOX was added to the cup. A small solid propellant charge suspended above the cup was ignited so that its flame was directed into the dish. A TV camera recorded if the mixture detonated or simply burned.

None of the formulations exploded in the detonation tests. The aluminum monopropellant would not sustain combustion once the solid propellant charge stopped burning. The

29% Al/Mg ALLOY/2% CABOSIL

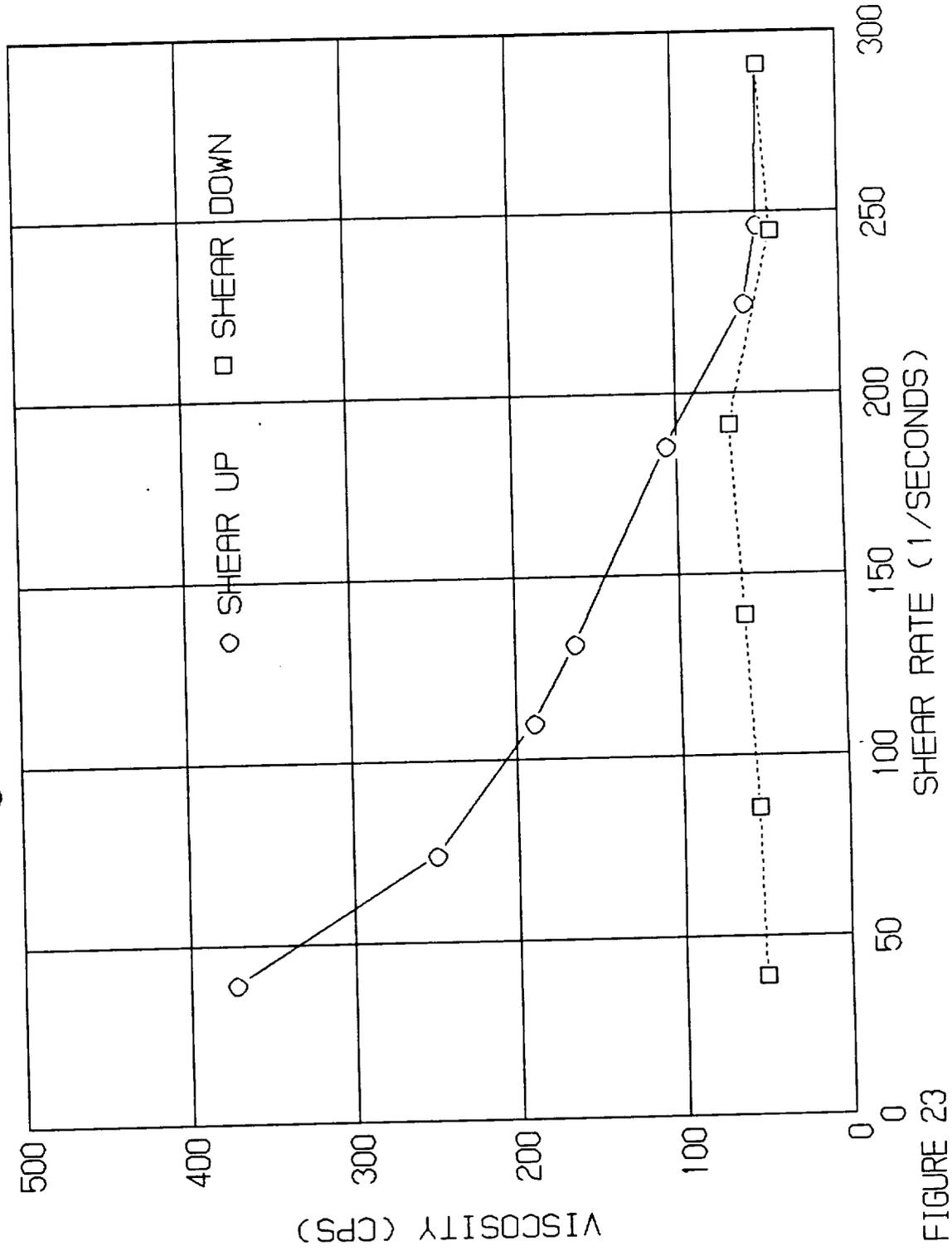


FIGURE 23

silicon formulation gave some indication of continued burning once the solid propellant went out. This was unlike the aluminum which gave off a brilliant white light whenever combustion occurred. Iron appeared to burn by itself once it was ignited by the solid propellant. The aluminum/magnesium alloy was not tested since it was so close to pure aluminum in properties.

4.2.2 Ambient Burn Rate Tests

In this test series, the monopropellant was burned in an aluminum cylinder under ambient pressure. The cylinder is surrounded with liquid nitrogen to keep the monopropellant at cryogenic temperatures. A small amount of solid propellant is deployed over the open end of the cylinder. The solid propellant charge is ignited and burns so that its flame is directed into the open hole of the cylinder to ignite the monopropellant.

Along the axial bore of the cylinder are located two probes with two thin wires connected together and protruding out into the bore. The probes are located 2 inches apart. As the monopropellant burns, it burns out the wires and breaks each circuit. The amount of time between the burnout of each circuit is measured and divided by two inches. This yields the burning rate of the monopropellant at one atmosphere.

35% Aluminum - Liquid Oxygen Monopropellant

The 35% aluminum with 2% Cab-o-Sil showed consistent ignition under ambient pressure. The results of the tests are summarized in Table 1. Unfortunately, in none of the ambient burn rate tests did the monopropellant burn all the way to the second wire. The combustion process would stop as soon as the flame front reached a level slightly below the level of the liquid nitrogen bath.

Ambient Burn Rate Test Results 35% Aluminum/LOX with 2% Cab-o-Sil

Table 1

<u>Test</u>	<u>Description</u>
2/21	Ignited immediately and burned to slightly below the liquid nitrogen bath level. Burned in a sporadic manner and was self sustaining.
5/10	Ignited immediately and burned to slightly below the liquid nitrogen bath level. Burned in a sporadic manner and was self sustaining. Note: Ice on cylinder lip and small amount in bore. Possible spill of liquid nitrogen into bore, but would have been very small amount.

Table 1 - Continued

<u>Test</u>	<u>Description</u>
5/20	Marbles were added to the liquid nitrogen bath to reduce the volume of liquid nitrogen for the test. Monopropellant was loaded in bore and across lip and spilled onto surface of adjacent marbles. Monopropellant only burned on lip and not in bore or on adjacent marbles. Combustion was very unsteady and not sustained after solid propellant went out.

The monopropellant burned in a brilliant white flame with sparks shooting into the air. The intensity of the flame was equivalent to that of an arc welder. The burning of the aluminum monopropellant was erratic with brief periods of almost extinguishment followed by brilliant flare ups of the monopropellant.

The test results showed that for two of the tests, the monopropellant ignited immediately when subjected to the solid propellant flame and burned down into the cylinder bore. The monopropellant stopped burning when it reached cylinder walls at liquid nitrogen temperatures. It was speculated that the heat sink capacity of the cylinder walls in the nitrogen bath was too great to sustain the reaction. The addition of

marbles to the liquid nitrogen bath reduced the nitrogen volume, but added solid mass in contact with the cylinder walls providing even greater heat sink capacity. The result of that test shows that the monopropellant did not burn at all in the cylinder bore, but only the amount of monopropellant on the external lip of the cylinder burned. Combustion at these cold temperatures was very poor and not self sustaining. These results suggest that the monopropellant may be extinguished by exposure to temperatures at or below liquid nitrogen.

29% Aluminum/LOX with 2% Cab-o-Sil

The lower loading of aluminum in this formulation resulted in a monopropellant with noticeably poorer burning characteristics. In none of the tests did this formulation burn after the solid propellant igniter went out, Table 2. When the monopropellant did burn, it burned in the same brilliant fashion as the 35% aluminum formulation with a bright flame similar in intensity to an arc welder's.

33% Silicon/LOX with 3% Cab-o-Sil

The 33% silicon formulation burned immediately when ignited under ambient conditions. However, its combustion was simply a conversion of the monopropellant to a glowing orange slag. No sparks or brilliant flame was observed in any of the tests. The flame front never went into the cylinder bore during the tests, Table 3.

Ambient Burn Rate Test Results
29% Aluminum/LOX with 2% Cab-o-Sil

Table 2

<u>Test</u>	<u>Description</u>
5/7	Did not light immediately and burned in unsteady pulses lasting about 1/30 of a second. About 2 to 3 pulses total of combustion. No measurable burning in cylinder bore.
5/7-A	Top of cylinder bore reloaded with propellant. Igniter failed to light.
5/7-B	Propellant in cylinder refreshed and igniter replaced. Flame from igniter pushed a small amount of monopropellant out of bore onto lip of cylinder. Some of the monopropellant on the lip burned in a continuous manner towards end of solid propellant burn. Unburned monopropellant remained on lip and in bore after the test.

Ambient Burn Rate Test Results

33% Silicon with 3% Cab-o-Sil

Table 3

<u>Test</u>	<u>Description</u>
5/14	Ice on cylinder and monopropellant on cylinder lip. Ignition of monopropellant fairly soon after ignition of solid propellant. No flame, only orange glowing of monopropellant while burning. No sparks during combustion. Monopropellant did not burn into bore.
5/14-A	Repeat of previous test with same results.

29% Aluminum/Magnesium with 2% Cab-o-Sil

The final formulation tested was the 29% aluminum/magnesium with 2% Cab-o-Sil. It exhibited the same burn properties as the 35% aluminum monopropellant of immediate ignition when subjected to the solid propellant flame, Table 4. However, during the initial part of the aluminum/magnesium burn, it showed a more steady combustion. The unsteady and pulsating combustion was not observed until the monopropellant flame front was getting to the cooler parts of the burn rate cylinder. The monopropellant flame had the same appearance as the pure aluminum monopropellant.

Ambient Burn Rate Test Results
29% Aluminum/Magnesium (80/20) with 2% Cab-o-Sil

Table 4

<u>Test</u>	<u>Description</u>
7/16	Igniter failed to come down to surface of monopropellant before igniting. No ignition of monopropellant.
7/16-A	Monopropellant refreshed in upper bore. Indication of monopropellant drying out before new monopropellant added to the bore. Igniter wire came off squib during deployment. No ignition of solid propellant.
7/16-B	Igniter wire reconnected to squib. Less than 10 small ice globs noticed on lip of cylinder. Immediate ignition of monopropellant from solid propellant. Short (1/30 second) explosive burn about 25% into monopropellant burn. Flame extinguished, but monopropellant relit by solid propellant still burning. Periods of fairly steady combustion, but mostly unsteady, pulsating combustion. Brilliant white flame and sparks as with pure aluminum combustion.

Table 4 - Continued

<u>Test</u>	<u>Description</u>
7/16-C	Retest due to explosive burning in previous test. Propellant not brought all the way to top of bore. Propellant starts about 0.25 inch from top. Squib failure
7/16-D	Squib replaced and previous test reran. Immediate ignition of monopropellant from solid propellant. No explosive burning during test. Combustion same as test 7/16-B with monopropellant sustaining combustion for about 0.2 seconds after burning solid propellant removed from entrance of bore.
7/17	Delay in ignition of monopropellant possibly due to flame of solid propellant not directly aligned with bore in cylinder. Small period of fairly steady combustion at beginning. Most of burn was unsteady - same as aluminum only. Burned down into bore about 0.25 inches before flame went out.
7/17-A	Retest of same monopropellant lot as 7/17 test. Placed large amount of monopropellant on lip of cylinder for better ignition. Squib misfire on this test.

Table 4 - Continued

<u>Test</u>	<u>Description</u>
7/17-B	Same test as 7/17-A with replacement of squib. Immediate ignition of monopropellant with steady combustion at beginning of burn. Best seen in test series. Flame went out in middle of burn, but was relit by still burning solid propellant. Pulsating combustion from this point on. Unburned monopropellant blown onto lip by flame from solid propellant.

The aluminum/magnesium monopropellant explosive burn during the first test is most likely an anomaly since this only occurred in one out of four burns of the monopropellant. The test in which it did occur experienced technical difficulties resulting in the monopropellant sitting a long time in the bore.

4.2.3 High Pressure Burn Rate Tests

The last series of burn rate tests measured the burning rate of the monopropellant under pressure. It was suspected that the burning rate would be a function of pressure. To pressurize the burning rate cylinder, a plate was remotely deployed over the opening of the cylinder. A graphite insert with a hole in it was located in the center of the plate and

formed a throat during the burning of the monopropellant. For the initial test, the throat was 0.25 inches in diameter. For the second test, the throat was reduced to 0.175 inches for a 50% reduction in throat area.

It was hoped that the burning rate as a function of pressure would be in the classical manner as solid propellants with an exponent of less than one.

$$\text{Burning rate} = (C) * (\text{Chamber Pressure})^n$$

n = Slope of log-log plot data

With experimental data at several different pressures, the value of c and n could be determined, then the burning rate for any given chamber pressure can be calculated.

The monopropellant selected for this test series was the 35% aluminum with 2% Cab-o-Sil. Before testing had started on this program, the high pressure burn rate equipment had been used for in-house research with the 29% aluminum formulation. In those tests, sustained burning had not been achieved in any of the tests. Since the 29% aluminum under ambient pressure conditions had not burned in a sustained manner, it was believed that a monopropellant which had burned significantly down into the bore under ambient pressure should be the one tested under pressure.

The results from this series of tests are inconclusive. The first test used a 0.25 inch diameter throat. The pressure reached in that test was more than the equipment was designed for which caused the throat plate to blow off the cylinder, Table 5. Approximately 150 lbs of force is required to break the fasteners holding the throat plate to the cylinder. That corresponds to a burn rate chamber pressure of 1020 psia. Unfortunately, the pressure had to be estimated since pressure data was not recorded until the igniter was finished firing. This was changed for the remaining tests so that pressure data was recorded starting slightly before squib ignition.

High Pressure Burn Rate Test Results
35% Aluminum/LOX with 2% Cab-o-Sil

Table 5

<u>Test</u>	<u>Description</u>
7/19	Immediate ignition of monopropellant. Burned for about 1.0 seconds before explosive burn of monopropellant resulting in overpressurization. Throat plate blew off cylinder. Pressure estimated to reach maximum of 1020 psia.

Table 5 - Continued

<u>Test</u>	<u>Description</u>
7/22	<p>Changed height of monopropellant inside bore. Now monopropellant came only up to 1st burn wire leaving about 0.75 inch between surface of monopropellant and throat plane. Squib failed to light.</p>
7/22-A	<p>Rerun of previous test with squib replacement. During replacement process, liquid nitrogen level dropped to between burn wires 1 and 2. Immediate ignition from solid propellant. Brilliant white glow around throat with some sparks around 10 seconds into burn lasting up to 25 seconds into burn. Smoldering of monopropellant until 41 seconds into burn when pressure increased from about ambient to 16.2 psia.</p> <p>Burn time = 41 seconds</p> <p>Burn rate = 0.05 inches/second</p>
7/23	<p>Throat diameter reduced to 0.175 inches to reduce throat flow area by 50% from previous tests. Immediate ignition with no measurable pressure. White glow observed in throat plane with a few sparks initially in burn about 3 inches above the throat plane. Burn rate wires not broken.</p>

Table 5 - Continued

<u>Test</u>	<u>Description</u>
7/23-A	<p>Repeat of previous test with a small peak of monopropellant up to throat to form burn path to main body of monopropellant. Immediate ignition with pulsating burning. Fire balls pulsating out of throat with 1st fireball about 1 foot high, 2nd and 3rd fireballs about 1.5 feet high.</p> <p>Sparks shoot about 2 feet up into air followed by measurable pressure rise and fireball about 3 feet above throat. Monopropellant did not burn down to second wire.</p> <p>Burn rate = 1.6 inches/second</p> <p>Pressure = 15.4 psia</p>

The monopropellant burned for about one second before the pressure increased to about 1020 psia. The monopropellant in the bore was consumed or any remaining was blown out of the hole during the explosive release of gas. While the time of explosive burning can be estimated from the video, it is impossible to know what was the burn length of monopropellant. Rather than guessing, the test was repeated with changes in the software to get the data.

The equipment was repaired to repeat the previous test. Up to this time the monopropellant was loaded to the top of the bore. In this test and in the remaining tests, the monopropellant was loaded only to the top of the first burn wire. An initial free volume was then created in the bore and monopropellant was not forced into the pressure transducer line. Heavy erosion had been observed in this area in previous tests, particularly in the entrance region to the pressure line. This change in loading seemed to reduce the amount of erosion.

The burn rate during this test was not explosive as in the previous test (Figure 24). The burn time was 41 seconds based on the video of the test. An average burn rate of 0.05 inches/second was measured during the test with the pressure barely above ambient until the end of the test. At that point, the pressure rose to 16.2 psia.

After this test, the throat area was reduced by 50 percent to try and get measurable pressure data. The first attempt resulted in the monopropellant only smoldering. This was believed to be due to the smaller throat reducing the solid propellant heat flux getting into the bore. The test was repeated with a "fuse" of monopropellant extending from the main monopropellant bore surface up to the throat.

The results with the smaller throat indicated a slightly higher than ambient pressure of 15.4 psia during the

PRESSURE IN BURN RATE CYLINDER
 35% AL WITH 2% CABOSIL - HPB

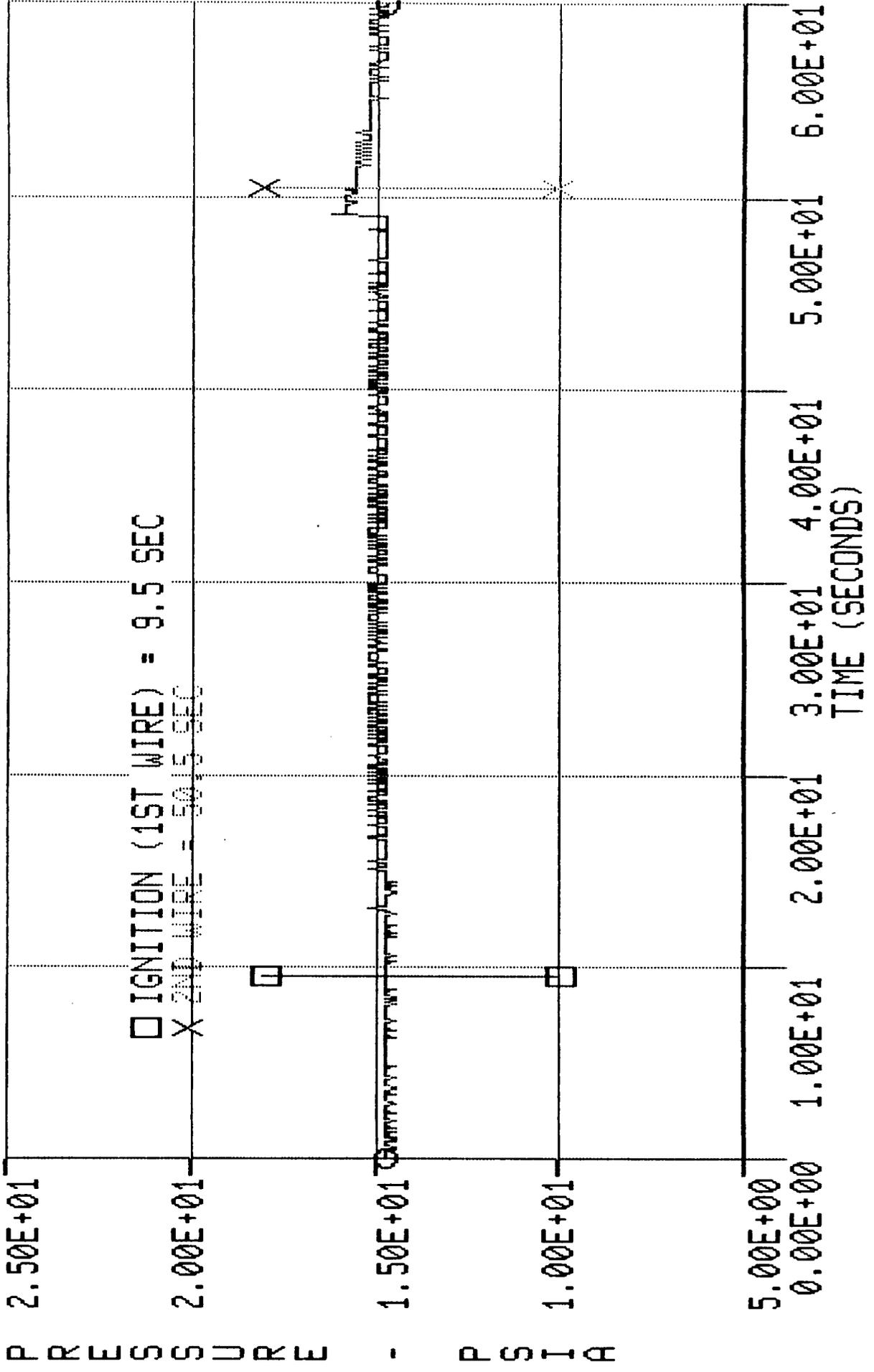


FIGURE 24

burn (Figure 25). Three fireballs of burning monopropellant were seen to fly out the throat during the burn. The fireballs ranged from 1.0 to 1.5 feet in diameter. The monopropellant finished with a very rapid burn at 1 second into the burn. Total burn time was estimated to be about 1.0 seconds based on the pressure curve. The amount of monopropellant burned during the test was measured to be 1.6 inches yielding a burn rate of 1.6 inches/second.

Due to the lack of high pressure burn rate data, the burn rate as a function of pressure could not be calculated. However, the low pressure data suggests a sensitivity to pressure. Low pressures only above ambient greatly increased the burn rate of the monopropellant. While the data is intriguing, more data is needed to determine what, if any, pressure dependency exists.

4.3 Metal Powder Settling Tests

A critical property of the monopropellant is to maintain a uniform distribution of metal powder throughout the LOX with time. To measure this property, monopropellant samples were mixed and poured into specimen holders. The holders were submerged in a liquid nitrogen bath to maintain the monopropellant. Periodically, a sample was removed in quarter segments from the top allowing the liquid oxygen to evaporate. The remaining powder was weighed on an analytical balance and compared with theoretical values.

PRESSURE IN BURN RATE CYCLINDER
35% AL WITH 2% CABOSIL - HP BURN

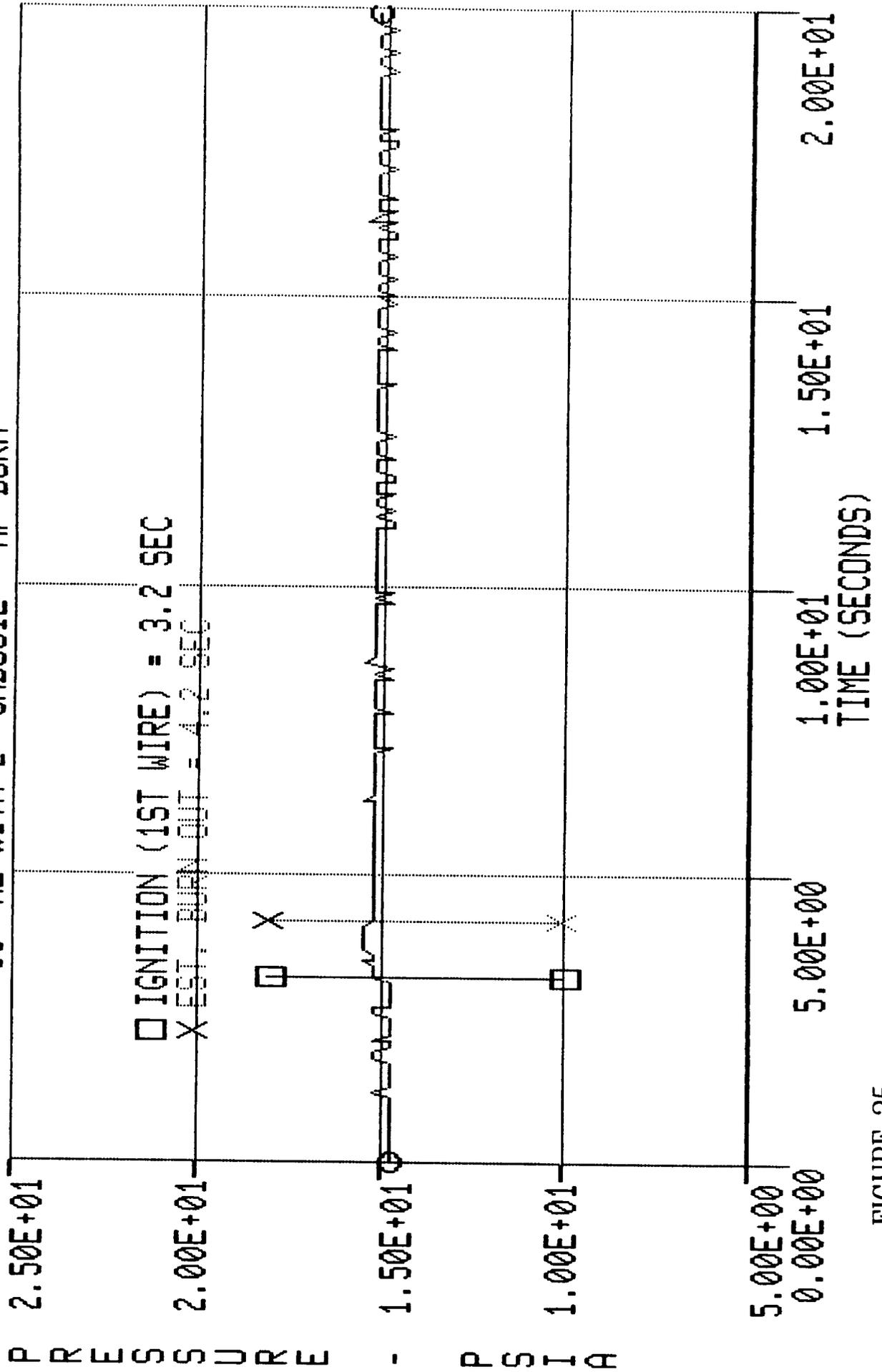


FIGURE 25

The segment volume in each specimen holder was different due to machining of the holder and the variance in the depth gauges. The individual segment volumes were determined by filling each segment with a measured amount of water until the water reached the depth gauge. With this information, the theoretical weight of fuel and Cab-o-Sil was calculated for each segment and compared to the measured values. The results are plotted for each of the formulations as a function of segment and time.

Even with the specially designed scooping tool and the depth gauges for each segment, the volume error in scooping out the material could be as much as 0.75 ml. For the formulations, this translated into an error in the ratio of weights of 0.15 to 0.20. As a result, the data could only show gross settling of the fuel with time. Small amounts of settling would be lost in the experimental error of the measurements.

35% Aluminum/LOX with 2% Cab-o-Sil

There was no apparent settling in any of the four segments during a 7 hour period (Figure 26). In the figure segment 1 is the top and segment 4 is the bottom. The upper three segment weights were consistently below theoretical values, but the bottom segment was very close to the expected weights for the time period. Thus, if settling had occurred, the bottom segment would have been much greater than expected.

SETTLING TEST DATA

35% ALUMINUM/2% CABOSIL

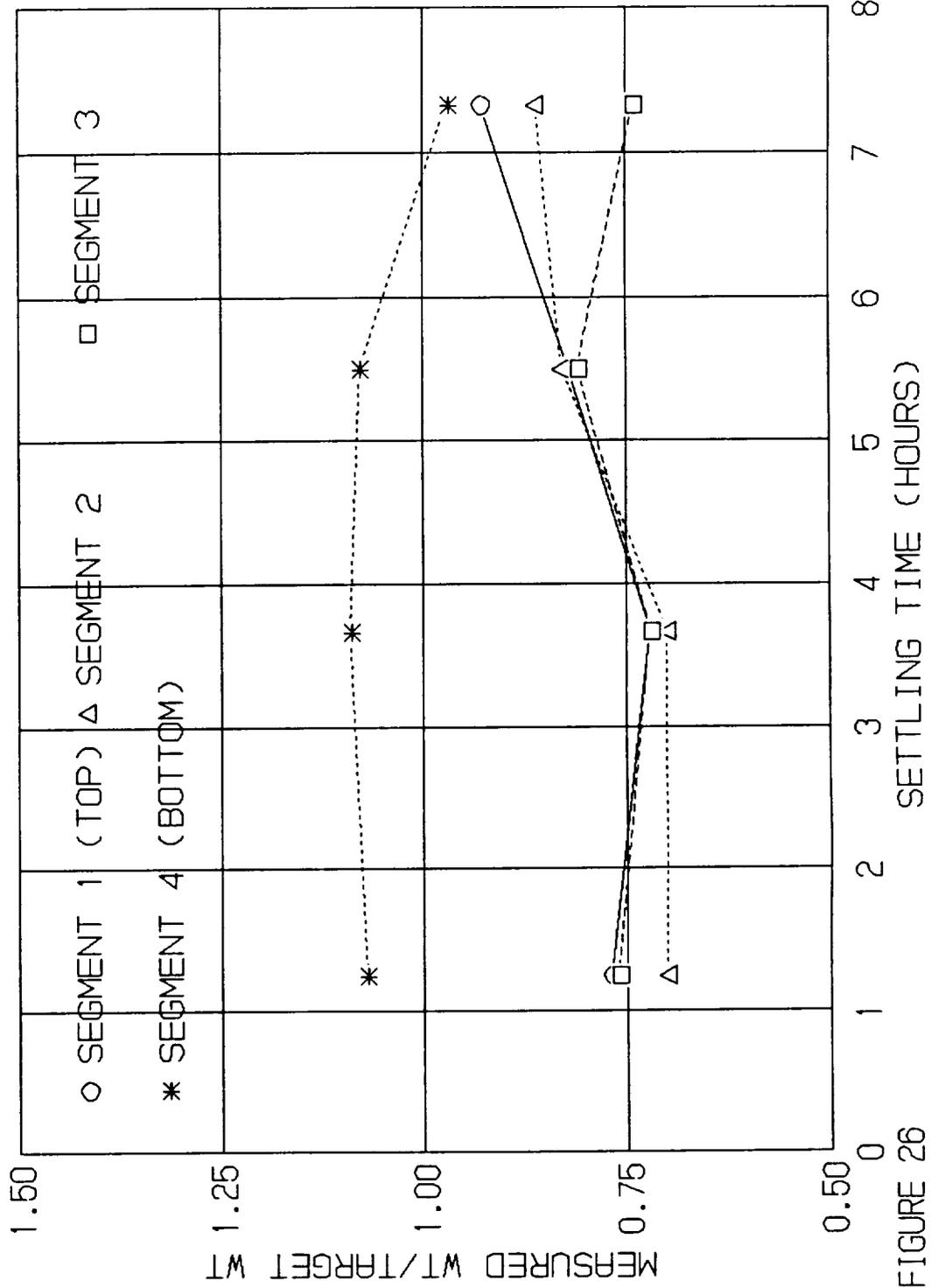


FIGURE 26

29% Aluminum/LOX with 2% Cab-o-Sil

The oscillation in the segment weights when compared to the target values are larger than in the previous case (Figure 27). The only exception is the bottom segment which is fairly constant. There is a slight indication of segment 2 increasing with time, but it is within experimental error. Segment 3 shows a much stronger indication of settling with a fairly steady increase in weight with time.

33% Silicon/LOX with 3% Cab-o-Sil

During a 7 hour standing period, the fuel in the formulation did not appear to settle (Figure 28). The oscillations in the weights is less for all the segments than with the 29% aluminum.

29% Aluminum-Magnesium (80/20)/LOX with 2% Cab-o-Sil

The first three segment increase in weight with time starting after the 3 hour period (Figure 29). The bottom segment appears to stay the same in weight with large oscillations in the measurements. The results could be indicating some settling of the fuel powder, but a more accurate set of measurements will have to be made to be sure.

SETTLING TEST DATA

29% ALUMINUM/2% CABOSIL

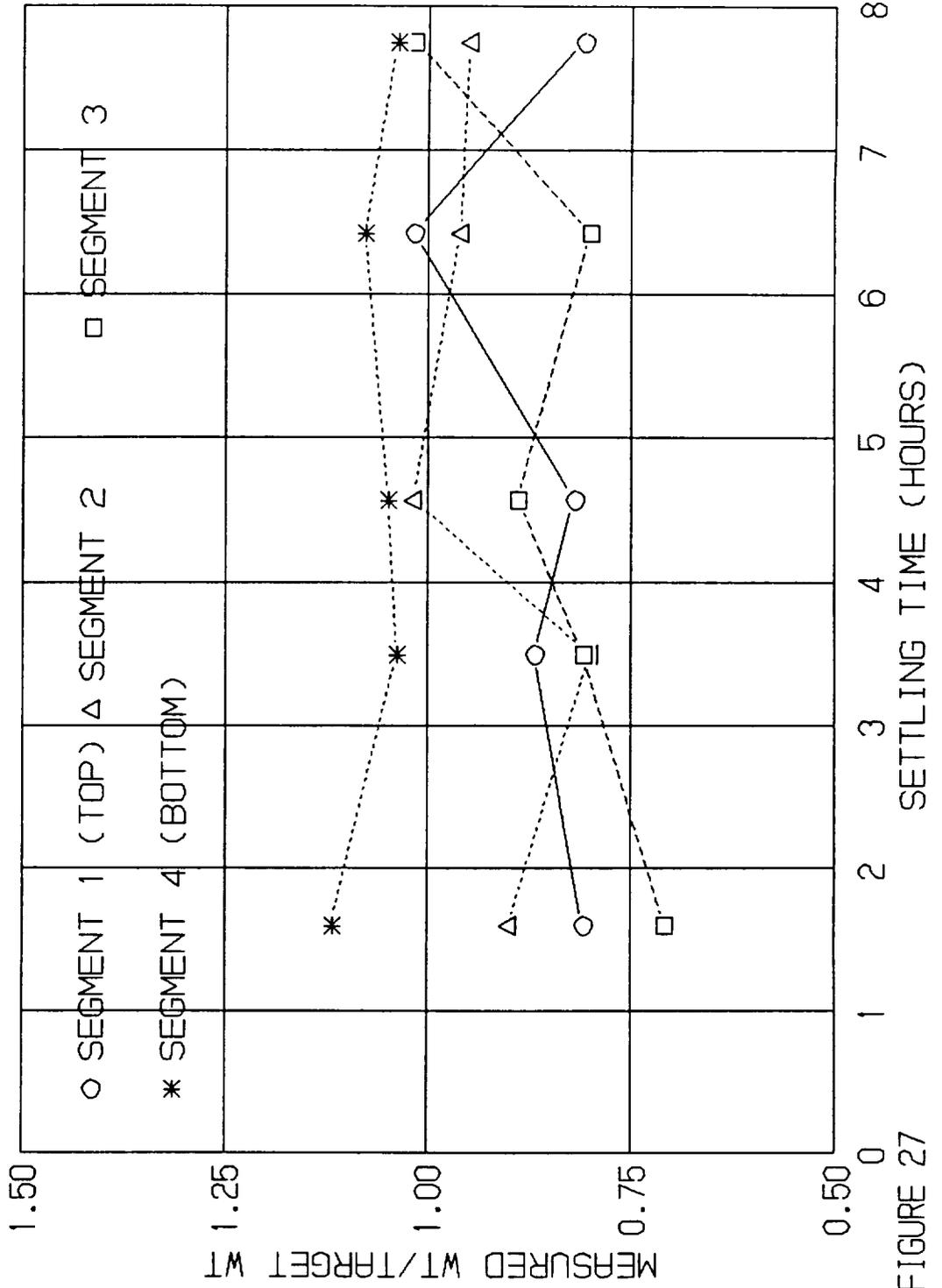


FIGURE 27

SETTLING TEST DATA

33% SILICON/3% CABOSIL

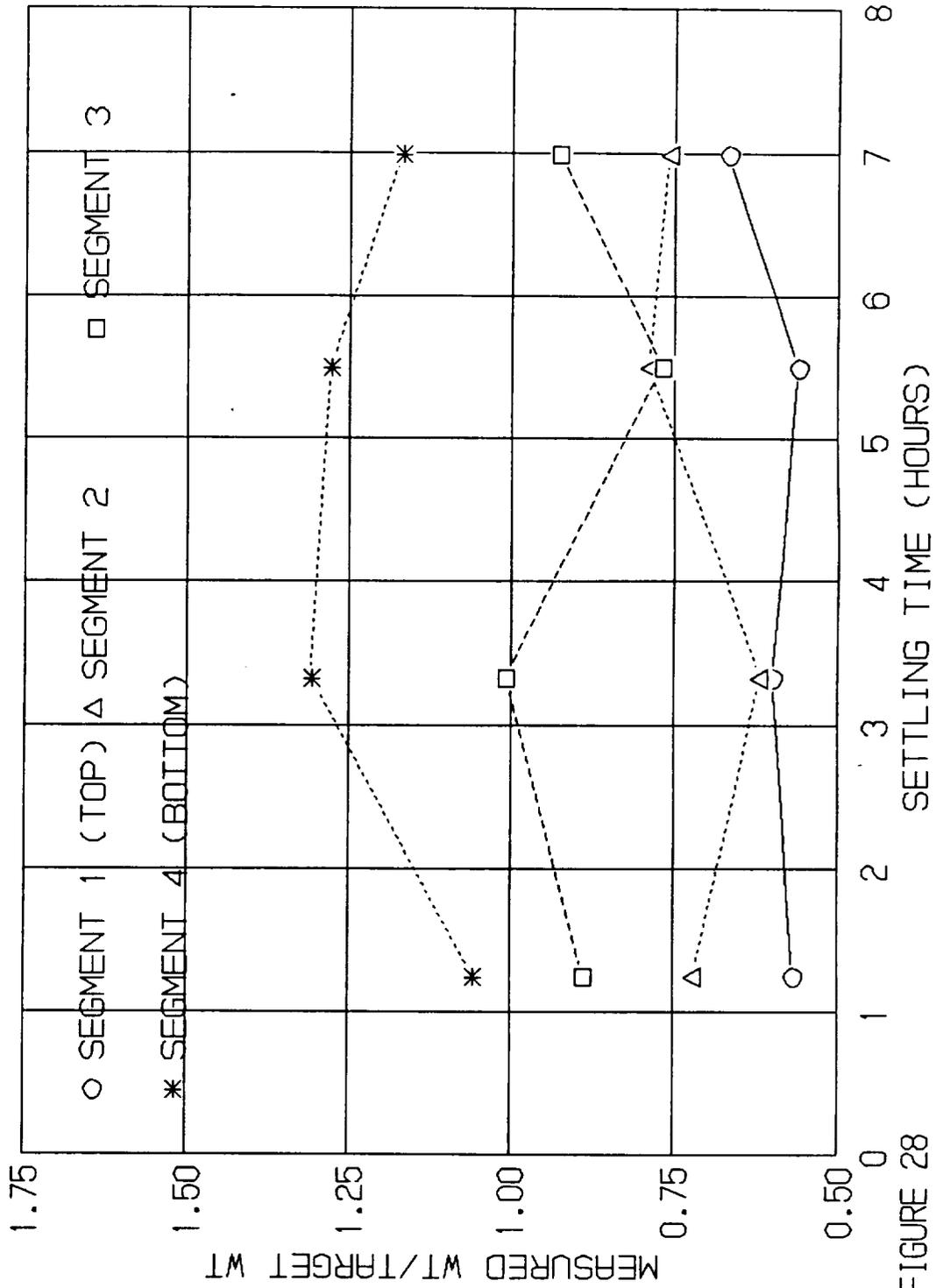


FIGURE 28

SETTLING TEST DATA

29% Al/Mg ALLOY/2% CABOSIL

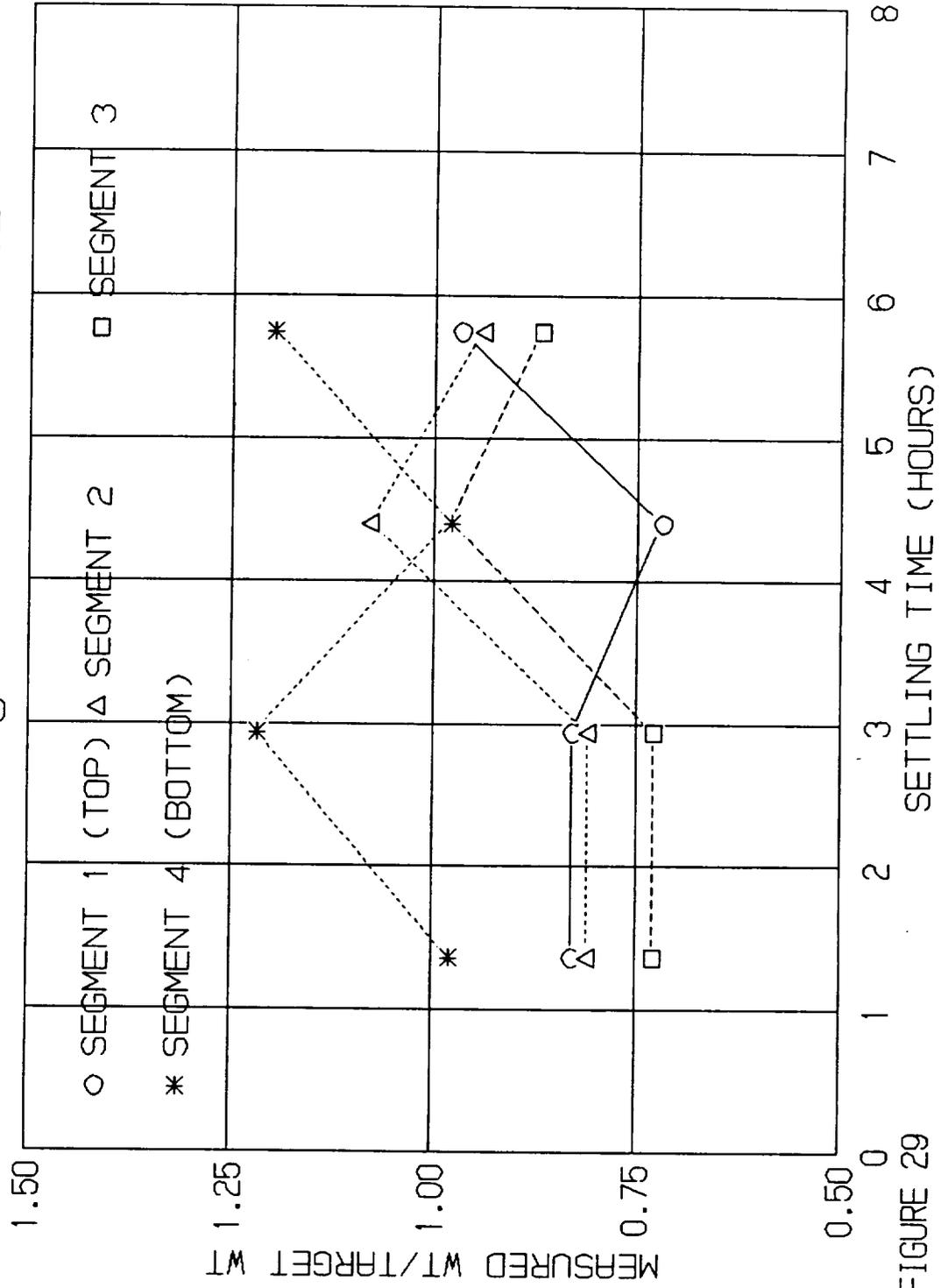


FIGURE 29

5.0 CONCLUSIONS

The theoretical specific impulses for aluminum and silicon have regions where they do not vary significantly with mixture ratio. This suggests that variances in mixture ratio can exist for these formulations without causing a serious deterioration in performance.

The viscosity of all the tested monopropellants with shear rate are low with Cab-o-Sil levels of two and three percent by weight. While the monopropellant can have a texture which is thick enough to hold shape, it has a very low viscosity under shear. The monopropellants act like concrete or mortar in that if a trowel is worked across the surface, a liquid layer appears. In the case of the monopropellants, the liquid layer is liquid oxygen. It is believed that the liquid oxygen layer formed at the moving boundary between the monopropellant and a wall results in low viscosity values.

The combustion of aluminum, silicon and aluminum/magnesium monopropellants appears to be strongly influenced by their temperature. Ambient burn rate tests showed that the monopropellants will not burn if they are kept at or below liquid nitrogen temperatures. If the monopropellant is already burning, the flame will go out when it reaches a region at those temperatures. The 35% aluminum loading was able to sustain itself longer in a liquid nitrogen environment than the 29% aluminum formulation. This is probably due to the higher heat output of the 35%

aluminum formulation. In this case, it took a greater heat sink in the burn rate equipment to extinguish the flame.

The appearance of the combustion process is different between the various fuels. The aluminum formulations burn very energetically with a brilliant white flame and sparks. The silicon turns a glowing orange in color with no sparks or flame. The silicon powder simply undergoes a change to its oxide form giving the appearance of molten glass in the process. The aluminum/magnesium formulation burns in appearance identical to pure aluminum.

The aluminum formulations burned in a pulsating manner. This was particularly true as the flame front approached regions in the monopropellant that were at liquid nitrogen temperatures. The aluminum/magnesium formulation burned more smoothly than the aluminum formulations except when the flame front reached areas at liquid nitrogen temperature. Then it too, pulsated in its burning similar to pure aluminum formulations.

Only the 35% aluminum formulation was tested for burn rates under pressure. The test results indicated that the burn rate of the monopropellant could increase significantly under pressure. With a pressure of about 16 psia, the burn rate was measured at 1.6 inches/second. Repeated tests showed little consistency of the monopropellant burn rate with pressure. One test resulted in a burn rate of 0.05 inches/second at a pressure slightly above ambient. A test with the same equipment and setup resulted in a

very high burn rate resulting in pressures on the order of 1020 psia.

It is possible that thermal effects more accurately reflect the burn rate data than pressure. The enclosed bore of the high pressure burn rate equipment results in more heat being driven into the monopropellant. This raises the monopropellant above the threshold combustion temperature resulting in further heat being generated and more propellant starting to burn. This bootstrap heating of the monopropellant to a bulk temperature where very fast burning can take place could explain the inconsistencies of the results.

Yet another explanation is possible which is dependent on pressure. As the pressure in the bore increases during burning of the monopropellant, the flame is forced down between the bore walls and the monopropellant. As the flame moves down the wall, more monopropellant surface area is exposed resulting in more monopropellant burning and still higher pressures. The higher pressures cause the flame to shoot even further and faster down the walls. A similar phenomena can happen in solid rocket motors. Insulation is bonded to the propellant to keep the flame from propellant areas where burning is not desired. If the insulation is not bonded properly to the propellant, it comes away from the propellant increasing the burning surface area resulting in overpressurization of the motor.

It is impossible to reach any kind of conclusion on the burn rate as a function of pressure and/or temperature without more data points. However, it can be concluded that the monopropellant will not burn at liquid nitrogen temperatures under ambient pressure conditions.

A major concern at the beginning of this program was the settling of the fuel powder in liquid oxygen. Without a gelling agent, the powder settles to the bottom in a few seconds. Only a small amount of gellant, 1.0% by weight, is sufficient to keep the powder from immediately settling. Only two of the formulations showed some indication of settling during a 6 to 7 hour period. The 29% aluminum-magnesium and 29% aluminum show some indication of settling. The 33% silicon and 35% aluminum showed no signs of settling within the accuracy of the measurement.

6.0 RECOMMENDATIONS FOR FUTURE TESTING AND EXPERIMENTS

Another approach to measuring viscosity should be investigated rather than using a rotating viscosimeter. The problem with the rotating viscosimeter is that it may not yield a true measure of viscosity for the monopropellant. A liquid layer appears to form on the outer surface of the rotating cylinder so that the viscosimeter mostly measures the viscosity of this liquid layer. A better method would be to force the monopropellant through a long tube and measure the flow rate and pressure drop. This method was considered at the beginning of this program, but was rejected for safety considerations. The propellant has now been found to be safe enough to use this method in future research.

A different method for measuring burn rate needs to be used in the future. Temperature control appears to be a critical factor in the test. With the present design, there is a large heat sink capacity for the amount of fuel present. The heat generated during burning is absorbed by the liquid nitrogen at a rate faster than is being generated by the monopropellant combustion. There is not enough heat left to continue the combustion process.

In future equipment, the monopropellant temperature should be controlled or at least monitored as a function of time and position so that it is known. Determining if the burn rate is a function of pressure is important. It is recommended that future

testing be done with equipment that pressurizes the monopropellant before ignition to a desired pressure. This could be done by enclosing the burn rate cylinder inside of a larger chamber which is pressurized with nitrogen gas. This is similar to the method used to obtain strand burn rate data for solid rocket propellant formulations. Instrumentation should be along the walls of the bore to determine if the flame is shooting down the walls under pressure or the bulk of the monopropellant is burning due to pressure effects.

After several attempts, the settling tests were done using a scoop method. The K-scan method might work in the future with more sensitive equipment. This might be worth pursuing in the future. Probably, the most accurate method would be to use a radioactive source and measure the penetrating radiation through the monopropellant. Since the radiation passing through the monopropellant to a receiver is directly proportional to the monopropellant density, it would give a direct reading of density. Such equipment is commercially available, but expensive to buy and operate.

7.0 REFERENCES

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