

COMPOSITE SOLID PROPELLANT PREDICTABILITY AND QUALITY ASSURANCE

Kumar Ramohalli

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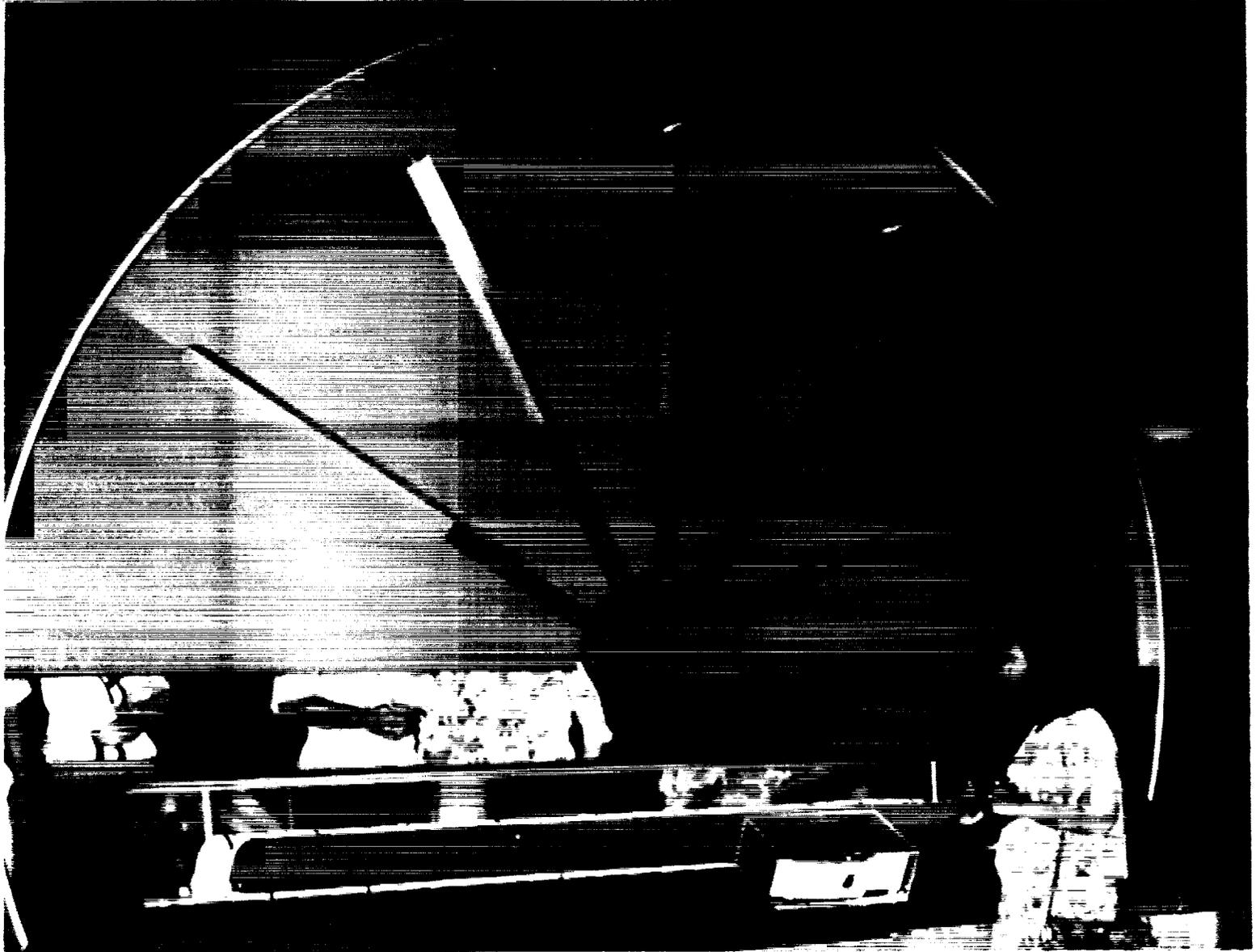
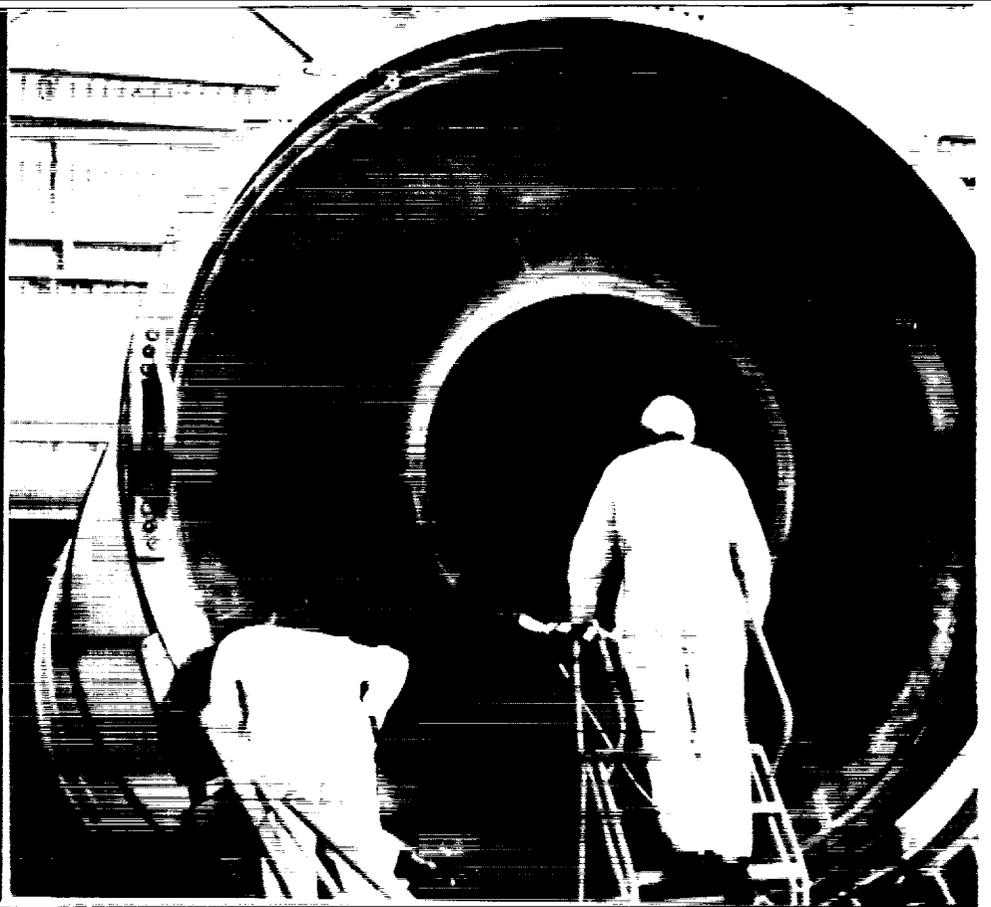
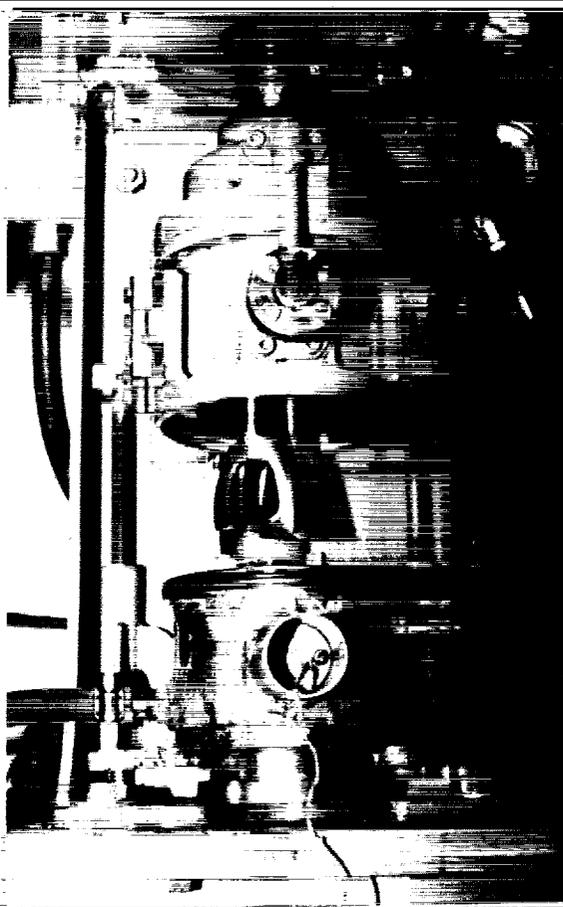
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MEETING REPORT

21 APRIL 1989

UNIVERSITY OF ARIZONA
TUCSON, ARIZONA



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**Meeting Report
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ACROYNMS

| | |
|-----------|---|
| AIAA | - American Institute of Aeronautics and Astronautics |
| AN | - Ammonium Nitrate |
| AP | - Ammonium Perchlorate |
| DoD (DOD) | - Department of Defense |
| HTPB | - Hydroxyl-Terminated Polybutadiene |
| JANNAF | - Joint Army-Navy-NASA-Air Force Interagency Propulsion Committee |
| JPL | - Jet Propulsion Laboratory |
| JSC | - Johnson Space Center |
| MSFC | - Marshall Space Flight Center |
| NASA | - National Aeronautics and Space Administration |
| NOTS | - Naval Ordnance Test Station |
| NWC | - Naval Weapons Center, China Lake, California |
| PBAN | - Polybutyadiene-Acrylonitrile-Acrylic Acid |
| SAIC | - Science Applications International Corporation |
| SEM | - Scanning Electron Microscope |
| SPIP | - Solid Propulsion Integrity Program |

A NOTE ON THIS MEETING

Solid propellant rockets have been in use for almost a thousand years since their first use by the Chinese. Their remarkable simplicity (no moving parts), readiness, and excellent payload ratios have sometimes been overshadowed by unfortunate malfunctions. Most of these can be traced to our lack of understanding of the fundamentals of propellant manufacture and end use (combustion). With several recent developments providing strong motivations (detailed in this report), it was felt worthwhile to introduce scientific rigor to this field still dominated by experience, educated guesses, and some analyses. In order to make significant advances from this legacy of "black art," we need a definite commitment and recognition that the constituent processes that finally result in solid propellant combustion (providing thrust) should be amenable to scientific scrutiny. We are especially fortunate that Norm Schulze at NASA Headquarters has championed this cause and provided sponsorship in the general area of solid propellant predictability, quality, reliability, and safety. Researchers at NASA Marshall Space Flight Center, Richard Brown and Theodore Kublin, have been providing the primary technical direction.

Fundamental to the issue at hand is the translation of years of expert experience into a reliable and scientific data base. For this purpose, a meeting was held at the University of Arizona on April 21, 1989. The long-range, mid-range, and immediate aims of this meeting are outlined in this report. The primary aim was to assemble the top experts in the nation and learn from their experience in order to evolve a general consensus and to identify the most promising avenues toward the ultimate goal of predictable and reliable solid rocket motors (see Appendix A).

The meeting had an excellent representation of the foremost authorities. Professor Summerfield was at GALCIT when the composite propellant was invented (by Parsons); he has worked extensively in composites since the 1940s. Professor Edward Price has also worked (with DoD) on composite propellants since the 1940s; he is also a member of the SRB redesign team for the Shuttle. The DoD agency for solid rocketry research (Air Force Astronautics Lab.) was represented by Robert Geisler and Captain Mark Husband. In addition, the NASA Center representative (Theodore Kublin) had worked at AFAL before moving to MSFC. One of the leading authorities on composite propellant combustion modeling (with remarkable insight) has been Professor Clarke Hermance, and his presence was most valuable. Warren Dowler, Marshall Humphrey, and Dr. Richard McKay represented practically all of Aerojet's and JPL's experience in propellants. We were fortunate to have Joe Barry present at the meeting; he is one of the very few who have spent a number of years actually mixing and making composite propellants in the scale of 1 pint to 150 gallons. His hands-on-slurry experience is hard to match. In addition, many University of Arizona researchers were present and contributed to aspects ranging from mathematical modeling to actual mixing and combustion. Industry representation was through Professor Edward Price, who was identified by Morton-Thiokol as their consultant.

It is hoped that this report will serve a worthwhile cause--the improvement of quality, reliability, and safety of composite solid propellants.

ACKNOWLEDGMENTS

The University of Arizona research discussed at this meeting is sponsored by NASA through Grant NAG8-757 from the Marshall Space Flight Center. Technical direction is provided by Theodore Kublin and Richard Brown. The funding is from Code Q at NASA Headquarters, with Norman Schulze as the manager. It is a pleasure to acknowledge the help of the following individuals who contributed so much toward the success of the meeting: All the attendees, many of whom came at short notice, and Daniel Perez, Paul Schallhorn, and Arthur Mazer, who handled the details so well. Josie Tanner deserves special thanks for taking care of all the logistics, besides taking notes during the meeting. Eileen Walsh (NASA, JSC) has been extraordinarily helpful in supplying valuable photographs and data.

Several others must be acknowledged for their help during the last two years leading to this meeting. Marion Kitchens funded the initial study from Code M; Paul Wetzell and Paul Herr (Code M), Sonny Morea, Jim Hester, Ben Shackelford, and Charlie Martin (MSFC) have provided valuable input all along. Hal Hikita spent long hours over several months painstakingly entering thousands of data points on the PC, besides extracting the first meaningful trends in the voluminous data. Connie Spencer skillfully produced a report from a set of disjointed notes.

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WELCOME

Dean Smerdon (member of the National Academy of Engineering) of the College of Engineering and Mines welcomed the group of experts to Tucson and the University of Arizona. He revealed the very recent news of the approval of a new building for the Department of Aerospace and Mechanical Engineering. This 24.8-million-dollar building will be the single largest construction undertaking on Campus. This should give an indication of the strong support that the College, the University, and the State have for the aerospace disciplines. He wished the meeting success and later met with some of the participants. A transcription of the meeting is given in Appendix B and a list of attendees in Appendix C.

INTRODUCTION TO SPACE RESEARCH AT THE UNIVERSITY OF ARIZONA

Professor Triffet (Director, UA/NASA Space Engineering Research Center) also welcomed the group and emphasized the importance of maintaining our competitive edge over other nations. He described the Space Engineering Research Center. This is the only center devoted to the utilization of Extraterrestrial Resources for manufacturing propellants (and other useful materials) in space. It is well known that a large fraction of the liftoff mass in traditional space missions is propellants. He also indicated the leverage available if one could make propellants "out there." Strong dependence on automation and autonomous controls with expert systems will be necessary for an economical, safe, and successful production of propellants extraterrestrially.

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INTRODUCTION TO THE PROBLEM

Kumar Ramohalli

General Background

The United States of America entered space with Explorer I, whose success was ensured through highly reliable solid propellant rockets in the second, third, and fourth stages; the use of clusters of identical motors (eleven, three, and one) is a characteristic typical of solid motors, namely the ease of "mass production" after development. Solid propellant rockets have been used extensively in space missions ranging from large boosters to orbit-raising upper stages. The smaller motors find exclusive use in various earth-based applications. The advantages of the solids include simplicity, readiness, volumetric efficiency, and storability (the advantages in specific comparison with liquid propellant rockets are detailed elsewhere in this report). So long as we continue to use them, and consider them for current and future missions, it is very important to maintain competence in solid propellants. Without such "in-house" capability, costly and wasteful panic solutions become necessary as problems are discovered in the use of newer propellants. Some non-technical solutions have saved the day, but these are temporary solutions at best. These aspects are listed in Fig. 1. Several recent advances in micro-technologies seem to indicate that we may profitably use these developments to economically evolve improvements. Our objectives are outlined in Fig. 2.

- SO LONG AS WE CONTINUE TO USE THEM
 - Important to maintain competence
 - Avoid costly panic solutions
 - Non-technical "solutions" may help in the short run, but do harm eventually
 - IMPORTANT RECENT PROGRESS IN RELATED FIELDS
 - Combustion
 - Rheology
 - Micro-Instrumentation/Diagnostics
 - Chaos Theory
- CAN BE APPLIED TO SOLID ROCKETS TO DERIVE
MAXIMUM ADVANTAGE AND AVOID WASTE

Fig. 1. Aspects of research on solid propellants.

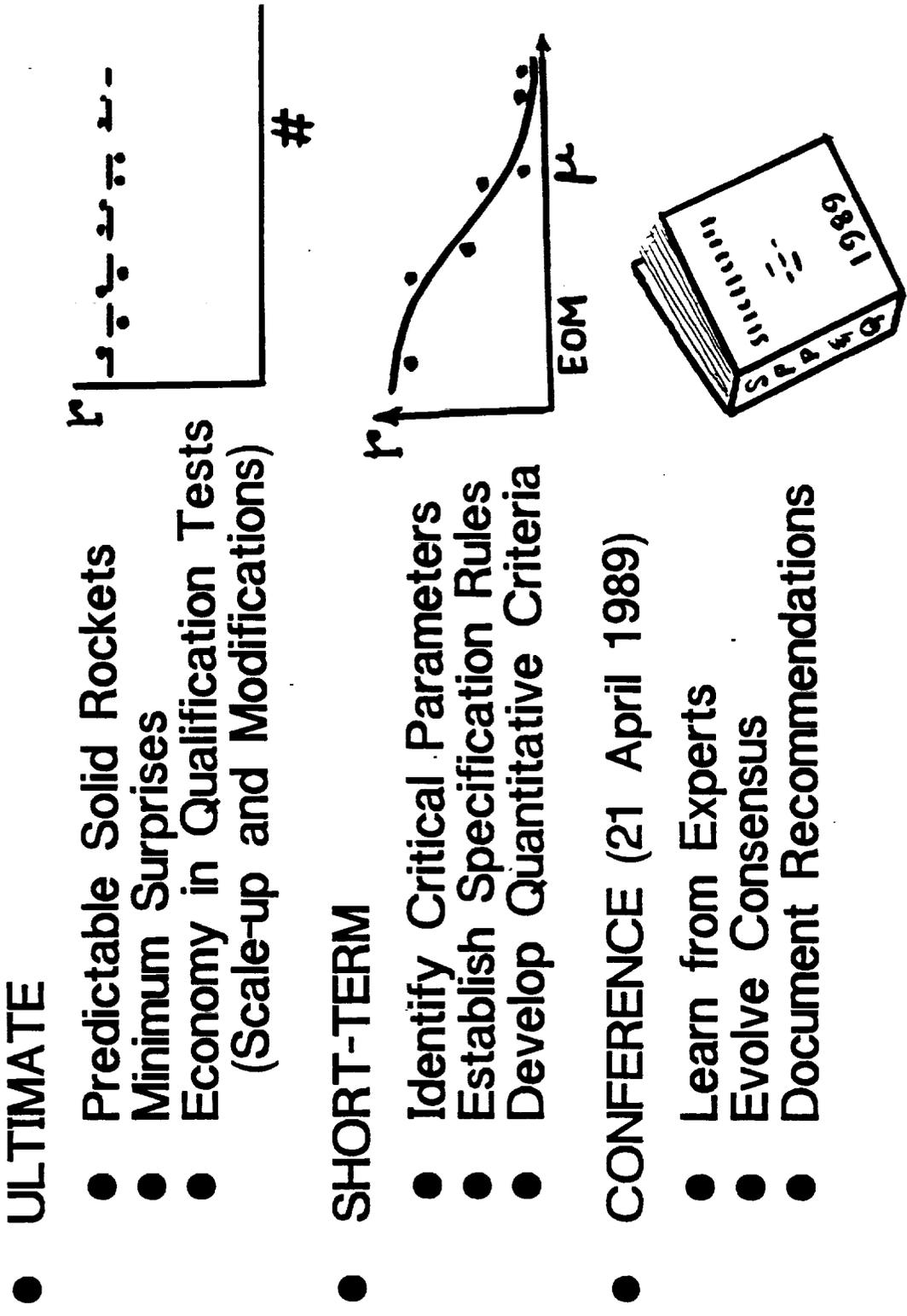


Fig. 2. Objectives of research in solid propellants.

It may be surprising to learn that we do not seem to have a good understanding of the fundamentals of solid propellants, especially after so many successful programs. The sheer bulk of data from almost five decades of (composite) solid propellant rocketry would lead one to suppose that very reliable rockets could be built based upon this data base. The fact that things are not that easy is best summarized by Ed Price, who notes the following:

An enormous amount of money has been spent during development programs on empirical approaches to meeting program needs for burning rate or mechanical properties. The totality of such efforts contributes very little to understanding or future ability for rational control because the myriad of relevant material, formulation, and test conditions have little commonality from one study to the next. . . .

. . . What they can't do by control of ingredient and processing specifications, they fine tune by testing liquid strands during batch processing and adding catalyst as needed. [His letter is reproduced in its entirety in Appendix D.]

With these clear revelations of the past and present status of solid propellants, one can obtain a better feel for the facts. The advantages of solid propellants have made them so desirable that a large number of these have been built and used without really understanding them well. Instead of a scientific "ground-up" approach, most solid propellant rockets have been built based upon past experience, educated guesses, and extensive corrective procedures during the design evolution. To ensure a sufficiently good understanding that results in verifiable quality and dependability, we will have to do better. The rewards will be substantial.

In the specific context of the Space Transportation System (STS), or the shuttle, we can realistically expect several important advances through a better understanding of solid propellants. These are outlined in Fig. 3. Basically, the payload increases because the liquid propellant margin can be reduced, the thrust vector control (TVC) system used to balance out imbalances in the two boosters can be a lot lighter, and several other systems can be made lighter. All these directly result in a lower cost per pound of material placed in orbit. The indirect cost reductions are far more substantial. These come from decreased developmental costs of the future motors.

Motivations

There are at least four important motivations for this scientific approach:

1. *Long-term economy through quality, reliability, and safety.* There has been a growing awareness in the rocketry community, and particularly at NASA, that a thorough scientific understanding is the only way to achieve long-term satisfactory performance and economy; this awareness was reflected in the formation of Code Q at NASA.

- P A Y L O A D I N C R E A S E S B E C A U S E O F
 - Decreases in the liquid margin
 - Decreases in the TVC system weight needed for the two SRB mismatches
 - Decreases in several other controls/instruments
- C O S T D E C R E A S E S B E C A U S E
 - ASRM and RSRM can be better designed
 - HTPB can be used instead of PBAN
 - Clean propellant can be quickly developed
 - Insulation (non-asbestos) can be tailored
 - Alternative propellants can be quickly implemented
- F U T U R E N A S A D I R E C T I O N S
 - Can be easily followed

Fig. 3. Advances derived from a better understanding of solid rockets.

2. *New and Revised Designs.* Many advanced designs (e.g., ASRM) and revised designs (e.g., RSRM) are planned or are being executed.¹ Specific examples include (1) the attempts to replace PBAN with HTPB in the STS SRBs and (2) the alternative propellant being considered for pollution reduction through AN instead of AP. Such **new designs** can be economically handled only through a better understanding of the fundamentals. Safety, reliability, and quality cannot be ensured if the general feeling is one of "Don't touch it! We just got it to work with great difficulty. Don't alter anything."
3. *Advanced Process Control.* For safety reasons and also to introduce modern computer-controlled processing, it is very important to understand the fundamental relations among the process variables. It is simply not practical to introduce advanced process control techniques if human monitoring and qualitative judgments (based on experience) are constantly required. This specific aspect of autonomous controls has become very important lately. With the recent NASA (and the USA) thrusts toward space exploration and a permanent presence in space, it is easily recognized that extraterrestrial propellant production is a major enabling technology. This *in situ* propellant production must be demonstrated robotically.

Some of the communication time lags between earth and other planets and asteroids mandate an autonomous processing plant. Such autonomous propellant production at remote sites can only be accomplished through a thorough understanding of the process variables, contingency margins, and "beyond-the-envelope" knowledge. This general area of autonomous propellant production using local resources provides a strong motivation for a better understanding of the fundamentals.

4. *High-Technology Devices.* This decade has seen a rapid advance in several high technologies. Microfiberoptics, IR/UV real-time imaging, free radical chemical techniques, *in situ* non-obtrusive sensors, microchips, and microcircuitry provide only a few examples of a wide variety of innovations. Many aspects of solid propellant monitoring and control that were beyond the technologies of the 1970s can be almost routinely handled through state-of-the-art technological advances. These recent high-tech devices and the definite promise of imminent advances provide an important motivation for revisiting many unsolved issues in solid propellant rockets.

Technical Background

The technology of solid propellants and high explosives has developed into a maturing *art* rather than a precise *science*. The variables and factors associated with typical composite propellant processing are so many in number that they may elude traditional, deterministic analyses. Quality control standards have been set based on *known* factors that influence performance, but the unknowns continue to cause surprises. It is not uncommon for propellants with "identical" ingredients processed in "identical" batches to reveal perceptible, and frequently unacceptable, variations in burn rates and mechanical properties (e.g., the tensile modulus). Two typical examples are shown in Figs. 4 and 5. Figure 4 shows a normalized burn rate, while the propellant in Fig. 5 indicates actual burn rates. It is thought that, in both of these cases, the propellants were processed in very similar, if not identical, manners. It is easy to recognize two aspects of this problem. One is the obvious indication that the propellant may not meet the expected performance; the other is the more important, genuine doubt about the performance of future batches. Of course, a major factor that precludes conventional quality assurance analyses and reliability predictions is the fact that usually, especially in larger motors, the number of batches will be too small for a reasonable statistical analysis. Many of these anomalies in recent experiments have been discussed.²⁻⁶ It is clear that, for all the attention the problem has received, attempts at analyses are rare.

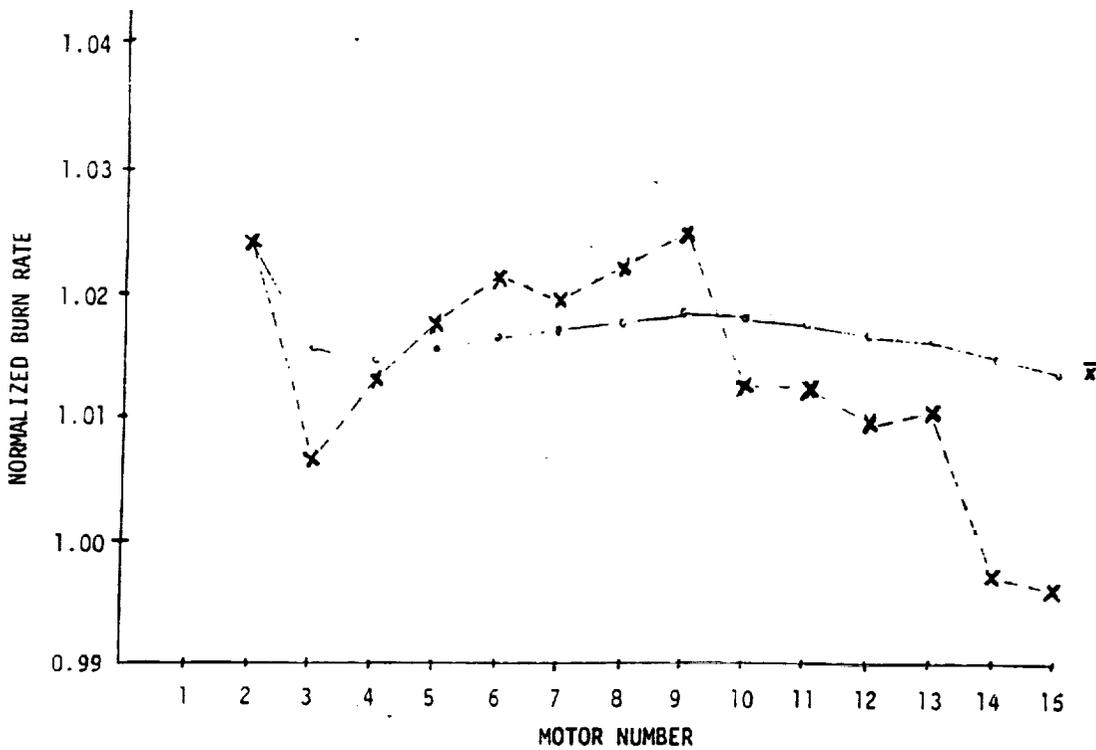


Fig. 4 Normalized burn rate of a solid propellant from batch to batch.

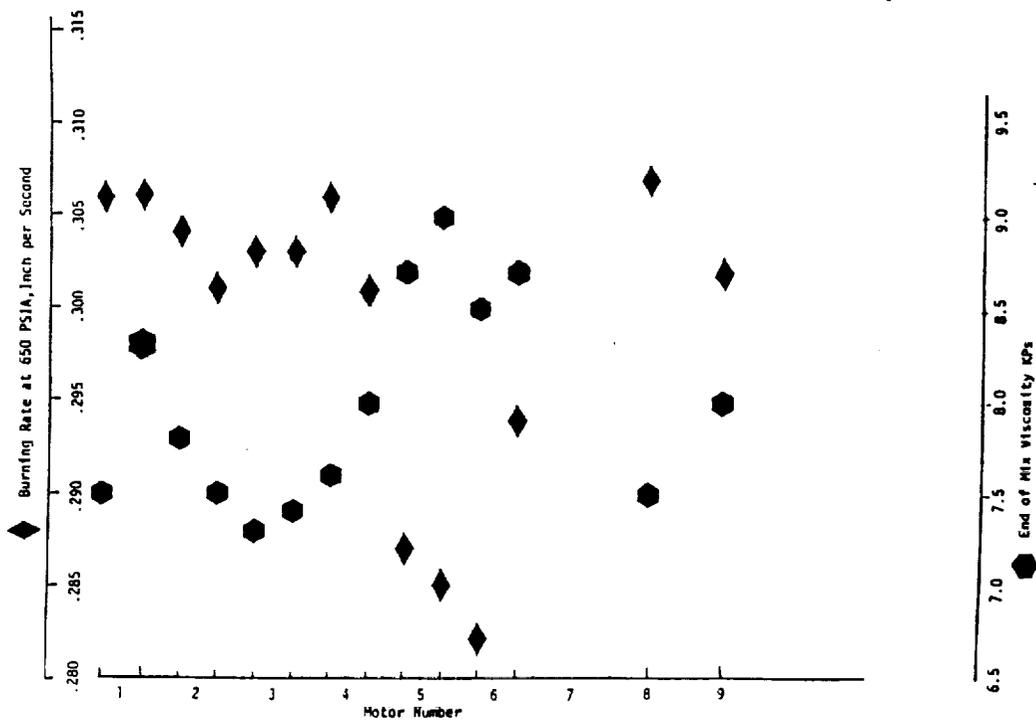


Fig. 5. Actual burn rates of a solid propellant.

ORIGINAL PAGE IS
OF POOR QUALITY

Quality control in solid propellant rockets has not been thoroughly understood, mainly because of the very large number of parameters involved in the manufacture of solid propellants. The parameters (Fig. 6) involve the ingredients (at least 10 different ingredients are used, typically; see Table 1) and the processing (at least 30 steps have to be followed, typically; see Fig. 7). The end-use parameters of interest include the steady-state (really, "time-dependent") burn rate, susceptibility to instability or oscillatory combustion, ease of ignition, uniformity of burn rate, completion of combustion (i.e., product distribution), mechanical properties, aging characteristics, environmental effects, and a host of related issues.

The fact that no two batches of solid propellants are identical in performance has been well recognized for many years; it has been thought adequate to maintain quality control standards within, for example, JANNAF recommendations to meet specific needs. Occasional "malfunctions" have not provided sufficiently strong stimuli for a detailed scientific analysis of the problem. A significant shortcoming (12,000-foot altitude loss) in the fourth launch of the STS in 1982 appears to have been the first problem to cause a pink, if not red, flag to be raised⁷ (Fig. 8). Subsequent revision of the SRB burn rate downward (Fig. 9) appeared to have solved the problem, at least temporarily.⁸ This incident resulted in a thorough examination of the entire burn rate prediction procedures in large SRBs.⁹ The general conclusion appears to have been that more work is needed for a better understanding of the mechanics of propellant manufacture, but it is simply not practical to process, cast, cure, and test-fire hundreds of rockets, each containing literally millions of pounds of propellants. Also, as the batch size increases, the potential for non-uniformities in ingredient distribution and processing increases. Better techniques are needed not only to ensure economy and quality control, but also to raise our confidence in the entire manufacturing technique. We simply cannot wait for the "next" firing to provide one more anomalous data point.

The understandable reticence of concerned manufacturers to openly discuss their experiences with malfunctions has not helped to alleviate the problem [however, a good start has been made by one company (see Fig. 10)]. The session organized by Bob Geisler at the AIAA Propulsion Meeting in 1982 appears to be the first to openly describe the experiences.²⁻⁶ No specific recommendations were made, however, to guide future efforts. Two papers^{10,11} attempted to isolate one specific subprocess (final mixing time) for a detailed analysis in a carefully controlled experiment where all other parameters were held strictly constant. Use of the same lot numbers for the ingredients minimized ingredient variations. The first theory attempted to relate the progressive grinding of the coarse AP to burn rate and initial tensile modulus. The experimental results were consistent with theory.

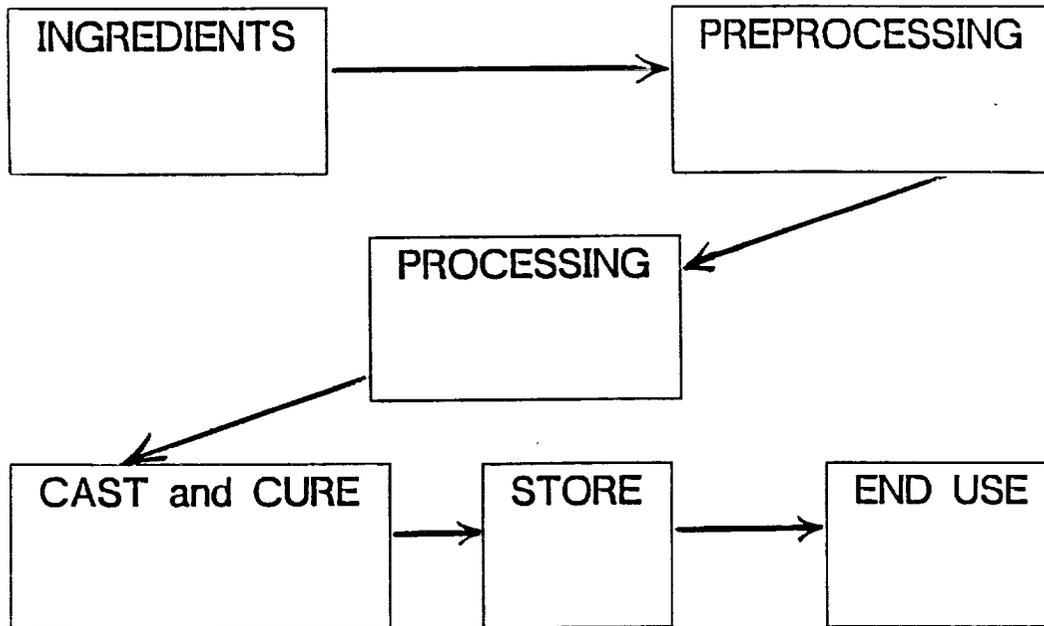
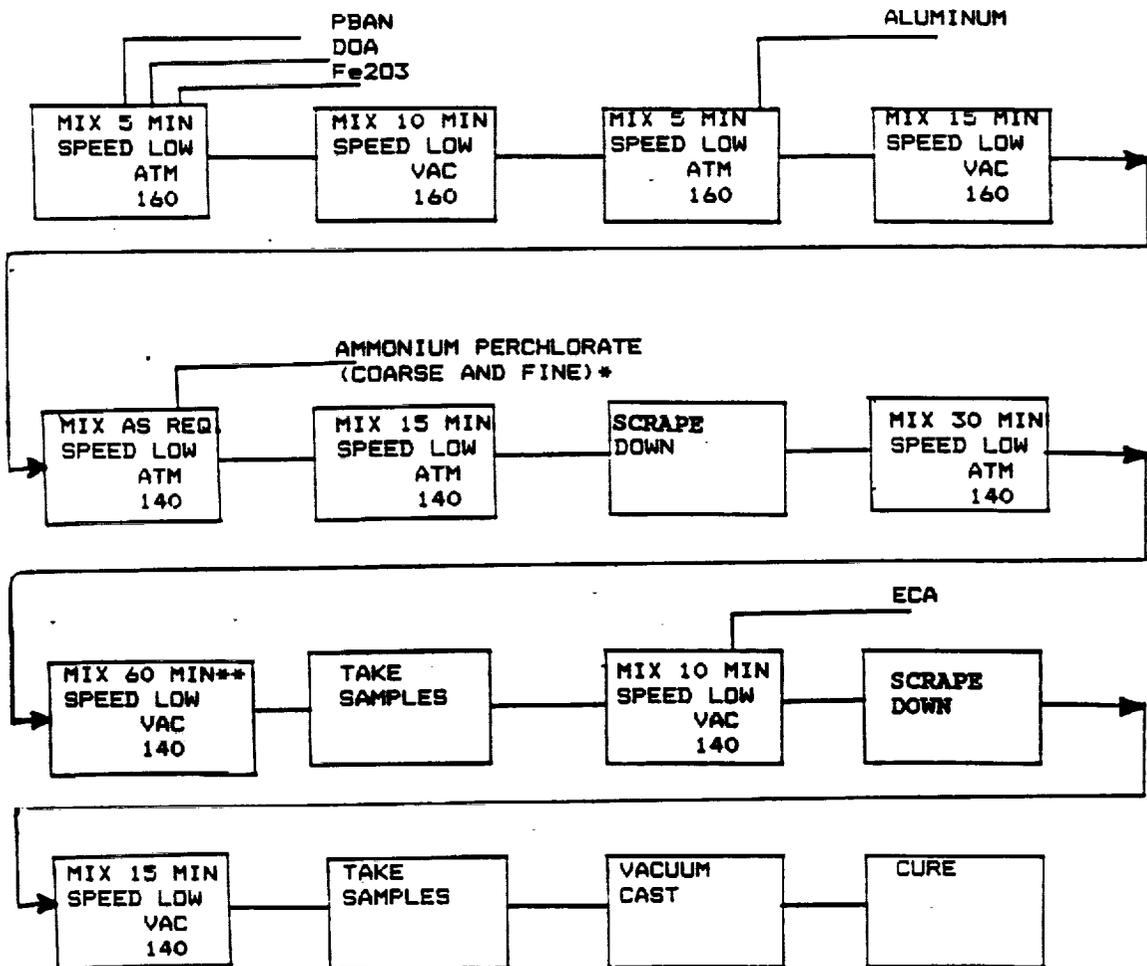


Fig. 6. Parameters involved in the manufacture of solid propellants.

Table 1. Ingredients for a typical propellant (EB-248).

| Ingredient | Lot No. | Percentage | Weight (g) |
|---------------------|---------|----------------|------------|
| Butarez HT | 4760 | 4.1452 | 658.050 |
| R45M | | 7.6395 | 1212.771 |
| Alrosperser | | 0.2180 | 34.6075 |
| Iso Stearyl Alcohol | | 0.5473 | 86.8839 |
| A0-2246 | | 0.1400 | 22.2250 |
| IPDI | | 1.3100 | 207.963 |
| MT-4 | | 0.200 | 31.7500 |
| A1 1230 | | 18.00 | 2857.50 |
| AP, unground 5272 | | 47.60 | 7556.50 |
| AP, grind 8 | | 20.40 | 3238.50 |
| TOTAL | | 100.200 | |

^aNote that the actual numbers seemingly exceed 100% by weight.



*Alternate: Coarse-fine-coarse-...-coarse
**STORED RUNS: Store material at 140 . Mix for 30 min. before adding ECA.

Fig. 7. Mixing procedure for 150 gallons. (The numbers 140 and 160 are temperature in °F.)

Depressed Launch Profile Causes Concern Initially

Kennedy Space Center—Space shuttle's fourth launch, on June 27, caused concern among flight controllers when less-than-planned solid rocket booster performance created a depressed trajectory, lifting the vehicle lower and slower than desired during first-stage flight.

Columbia flew 8,000 ft. below its planned trajectory line, costing a theoretical 2,000 lb. in payload, Johnson Space Center engineers said.

The performance will be an issue for future flights. Engineers are investigating how booster performance is predicted prior to liftoff.

The depressed trajectory did not falter to the point where it seriously affected flight safety. Flight controllers were concerned that it would become a serious problem, but about 30 sec. into the lower trajectory the shuttle began correcting back toward the desired flight path.

Flight controllers said that if they had not seen a similar but smaller solid rocket booster digression on Mission 3, the Flight 4 solid rocket performance would have been even more of a real-time concern. The performance resulted in delayed abort mode calls to the crew and the separation of the solid rocket boosters at a lower altitude and at a slower velocity.

To compensate for the lower performance, the Rocketdyne main engines burned for 2-3 sec. longer than planned, expending about 2,000 lb. worth of the 12,000 lb. of payload performance margin carried by the vehicle.

Maximum Trajectory

Even with the depressed flight path, astronauts Navy Capt. Thomas K. Mattingly and Henry W. Hartsfield piloted the Columbia through its first maximum performance ascent trajectory, verifying the basic flight profile that will be employed most often in the shuttle program.

Mission 4 was the first to fly due east out of Kennedy Space Center, Fla., into a 28.5 deg. orbital incline. It is at this angle that the shuttle can benefit most from the Earth's rotation when boosting payloads into equatorial orbit. About 95% of shuttle missions flown from Kennedy will follow this profile.

Columbia's liftoff weight target of 4,484,585 lb. was about 5,000 lb. heavier than Mission 3. The high-performance trajectory was selected for this flight to assist vehicle propulsion with the heavier mass. The Defense Dept. payload weighed about 8,000 lb.

Immediately after liftoff from Launch Pad 39A, Columbia rolled 90 deg. to the right to establish a 090-deg. due east

heading over the Atlantic. This was a departure from earlier missions when a 113-deg. or greater liftoff roll maneuver was used to direct the orbiter northeast into a higher 38-40.3 deg. orbital inclinations.

Columbia's ascent profile was structured using both solid and main engine performance data acquired on the first three missions as opposed to the earlier procedure of using analytical engine performance data. Flight directors expected this to provide a more accurate trajectory compared with predicted values.

A desire to increase the dynamic pressure envelope of the vehicle while at the same time providing a softer ride in the Mach 0.8-1.2 maximum dynamic pressure region, where additional data are needed, also dictated changes between this and previous launches.

Engineers achieved a higher dynamic pressure than during the last flight at a point later in the ascent in order to reduce the loads in the more critical Mach 0.8-1.2 region. The maximum dynamic pressure (Max-Q) for Mission 4 was targeted at 691 psf. compared with 648 psf. on the last flight and a maximum operational dynamic pressure limit of 760 psf.

Almost immediately after liftoff at 11 a. m., the vehicle began exhibiting characteristics indicating lower-than-desired solid motor performance. Main engine throttle down to 65% to reduce loads at Max-Q occurred 2-3 sec. late, and throttle up was also delayed. During first-stage flight the vehicle flies with open-loop guidance, where attitude is a function of velocity. The targeted throttle down from the 100% point was at 13.5 sec.

Vehicle angle of attack at Mach 1 was programed flatter than on Mission 3 to provide a more optimum performance for the heavier ascent mass.

Mission 4 ascent flight test objectives above Mach 1 allowed for a higher dynamic pressure in this regime. This was a change that allowed a higher performance/relative flight path angle in this phase of the flight compared with the first three missions.

At about 1 min. into the ascent, mission control center plots began showing a marked digression from the nominal trajectory line. This started controllers discussing the vehicle's energy state on the ascent flight director's communications loop.

Booster Separation

The flatter programed trajectory for Mission 4 had called for a solid booster separation altitude 5,750 ft. lower than on Mission 3. Actual solid motor performance on the flight, however, resulted in depletion of propellant and booster separation about 2,000 ft. short of that goal at a velocity of 4,293 fps. compared with the 4,336 fps. relative velocity target.

The overall Mission 4 solid booster separation parameters were for a lower altitude and a higher velocity separation in a flatter climb trajectory to provide more performance toward the 55-naut.-mi. main engine cutoff target.

The less-than-expected solid motor performance, however, resulted in a lower and slower situation than desired at this point, affecting abort and other vehicle energy milestones.

This became especially noticeable 2 min. 40 sec. into the flight, when Columbia was scheduled to be capable of achieving a Dakar, Senegal, emergency landing with one engine failed. The milestone passed with no notification of this capability from spacecraft communicator astronaut David Griggs in Houston.

The two-engine Dakar capability expected at 2 min. 40 sec. was not actually attained until about 3 min. 10 sec. Subsequent energy oriented milestones important for abort mode determination were delayed about 15 sec.

Throughout the remainder of powered flight on the main engines, the closed loop guidance phase that adjusts trajectory for the most optimum profile to achieve main engine cutoff targets took out the solid motor performance deficiency. Main engine cutoff was about 2-3 sec. later than planned, but was achieved at the 25,677 fps. velocity predicted.

The 55 mi. engine cutoff point was planned 3 mi. lower than on the last flight and also programed to occur at a higher vehicle flight path angle.

The ignition of the two Aerojet 6,000-lb.-thrust orbital maneuvering system (OMS) engines for the first OMS burn at 10 min. 32 sec. into the flight was observed through the Bermuda tracking station. The 1 min. 37.7 sec. burn provided a 154 fps. velocity change and an initial vehicle orbit of 130 x 33.3 naut. mi.

The second OMS burn was performed 37 min. 40 min. into the flight with the 175 fps. velocity change resulting in a 130 x 130 naut. mi. orbit completing the ascent.

Engineers believe more emphasis will be placed on how the thrust from specific solids can be characterized prior to each flight. □

Fig. 8. Article regarding deficiency in solid propellant performance.⁷

Performance of Solids Revised Downward

Kennedy Space Center—Space shuttle solid rocket motor performance is being reduced as a result of the depressed trajectory flown on Mission 4 and new test data showing the motors have a slightly slower burn rate than earlier believed. The change had been a significant prelaunch issue for shuttle Mission 5.

Performance revision considerations were necessary because it meant that software commanding Columbia's Rocketdyne space shuttle main engines had to be reprogrammed to provide a throttle schedule different from that originally planned.

The Thiokol motors that make up the United Space Boosters, Inc., solid rocket boosters still fall within specification limits even with their performance revised downward.

Initial shuttle Mission 5 liftoff planning was for throttle commands to reduce the main engines to 68% thrust to reduce accelerations when approaching Max-Q, the point at about Mach 1 when the vehicle encounters maximum aerodynamic pressure.

After finding the solid motors have a slower propellant burn rate than earlier believed, software commanding the main engines was changed so the oxygen/hydrogen powerplants would throttle no lower than 85% during launch to make up for the performance loss from the solids. This had to be done as a pre-liftoff measure.

During its fourth launch last June, the shuttle experienced what has been dubbed "the great depression," when lower than expected performance resulted in a depressed trajectory that missed solid booster separation altitude targets and affected the dynamic pressure experienced (AW&ST July 5, p. 20).

The boosters performed within specification on Mission 4, but the vehicle's programmed trajectory during that launch was based

on projections of higher performance. This resulted in a situation where modified vehicle steering and a slightly longer main engine burn time was needed to compensate. The situation was well within vehicle margins but resulted in delaying the availability of critical abort modes.

After that experience, the National Aeronautics and Space Administration and Thiokol started to determine how the motors could be characterized better before launch so the Mission 4 situation would not be repeated.

The earlier preflight characterization method was to use both ground test and flight motor data. These data then were combined with firing tests by several 5 × 12-in. tubes containing propellant from batches being used in the motors flying the upcoming mission. Between 48 and 52 small tube firing tests are done to characterize each motor before flight. The resulting data are used to help structure the trajectory from a performance standpoint.

The motor performance had been showing a slightly decreasing trend between flights one and three, but on Mission 4 the trend was more apparent.

On the last flight the propellant burn rate predicted was 0.366 in./sec. The actual burn rate was 0.359 in./sec., a difference that affected the trajectory significantly.

The NASA/Thiokol effort resulted in a decision to use only flight data in conjunction with the tube firing tests. This changed the scaling factor and has provided new expected burn rates that have been revised downward affecting Mission 5 prelaunch software preparation. Mission 5's original motor burn rate target was 0.368 in./sec. That has now been revised down to 0.365 in./sec.

Fig. 9. Article regarding revision of performance standards for solid propellants.⁸

| Problems Which Have Failed to Make-up on Castings | Description of the Specific Problem(s) | Reasons for Failure | Control Measures | Proposals | Remarks |
|---|---|--|--|-----------|------------------|
| Slag pourer required to stop the propellant in the 600 gallon mixer. | In the analysis of the 800 First Stage propellant, TP-8187, the mixer kept backing out on the upper pourer limit. During run of these castings, the drive belts were assembly bonded to half. | To correct this problem, causes were pulled on the propellant in a close relative manner. In this manner, the pourer requirements could be controlled. | Subsonic. One theory is that the gas produced by the reaction of formal with the AP adhering to the AP particles was additional pathway to form bubbles which caused to splint. | TP-8187 | SE |
| Horizontal properties of the propellant failed to match those of the substrate standardization mixer. | During the analysis of the 800 propellant, the stream in the 600 gallon mixer was lower than the 600 gallon substrate mixer. | The 600 gallon mixer was processed with a lower pourer rate to match the 600 gallon mixer during run. However, at this low pourer rate, variations were so great as a time was just applied to the proportion of the high pourer rate. | The reaction of the 800V with the AP produced 8V which adheres to the fine AP particles and caused to form bubbles without being AP. | TP-8175 | SEAS |
| Horizontal properties of the propellant failed to match those of the substrate mixer processed in the horizontal mixer. | When the lighter propellant, TP-8117, was scaled up to the 70 gallon mixer an analysis the horizontal properties were greatly reduced even though there was no volume change in the process. | The Argon mixer used to have the propellant dry and stabilized on a constant level. This action was based on the effects noted in alpha processing with and without Argon sweep. | Subsonic. This propellant contains 8E-712 which is not supposed to produce 8V. However, the propellant acts the same as those which do produce 8V. | TP-8117 | 0-4, SE |
| The burn rate of the propellant scaled up to the five gallon mixer was lower than predicted. | When five gallon mixer of 70V propellant were processed, no bad trouble occurred. When 800 gallons were fired, the burn rate was very low. | No new burn analysis requirements on the burn analysis of AP is required and is not needed. This action was based on mixer processed in which the AP was screened and weighed in a dehumidified bag. | Subsonic. The theory is that the mixer used the air caused the AP to agglomerate as it was screened. | 70V | 0-4 |
| The viscosity curve of the propellant failed to match up. | When 600 gallon mixer of TP-81035 propellant were processed, the low shear rate viscosity was about three times that of the five gallon standardization mixer. This caused the propellant to splash or harden that original curves were seen. | It was hypothesized that the mixer used the air caused the AP to agglomerate as it was screened. | It was believed that slight changes in the particle size distribution of the 800 had caused the AP to agglomerate as it was screened to shift up and cause the problem. | TP-81035 | 0-4 |
| The propellant coating properties failed to scale up. | When the TP-81028 propellant was scaled up to the 600 gallon mixer and kept into a 17 lb. container, the rate of the mixer failed to fill to spread the fine coating mixture wide. | The propellant AP particle size distribution was changed to reduce the high apparent viscosity at the low shear rates. In addition, the propellant level in the same formal was increased to provide a better seal. | It appears that the small changes in fine ground AP used to adjust the burn rate of the propellant has a drastic effect on the viscosity curve. | TP-81028 | 0-4 alone |
| The coating rate of the propellant could not be properly predicted. | When coating TP-81028 propellant into the 17 lb. container from a 600 gallon mix, the coating rate was about twice that predicted. | Coating differential pressure was adjusted to provide a reduced coating flow in the 600 gallon coating system. | Subsonic. It was felt that slip at the wall or turbulent interpenetration of the viscosity curve caused the error. | TP-81028 | 0-4 alone |
| Core react/pot life of propellant. | The use of TP-81076 propellant processed in the 600 gallon mixer caused up shift in the core level during the casting operation. | The pot life curves were run on three different temperatures to determine the effect of temperature on the pot life. Following this, the end-of-run temperature was reduced by 10°F. In addition, the pot life of the propellant was monitored every half hour to catch any way up in the viscosity signaling the start of cure. | The coating effect caused on composite 600 gallon mixer didn't take place because the burning time was used. Thus, the pot life being run as 120°F didn't represent the true cure curve of the propellant which was actually 120°F in the die head. | TP-81076 | 0-4 alone |
| The burn rate of the propellant in the 600 gallon mixer. | The burn rate measured in the 600 gallon mixer by liquid stream and chemical meters were about 0.020 in./sec less than substrate mixer using the same formulation and materials. | The verification 800 will be repeated and the material removed from the bin for substrate mixer will be obtained using a thief sample. | Subsonic. The scaling of the line used to the 600 gallon mixer to obtain raw materials to process the five gallon mixer in the standard mixer. The 800 mixer AP is used to scale during grinding with the coarse material going to the side of the bin. It is assumed that the samples for the substrate mixer were from the top of this zone. | TP-81076 | SE |
| The cure time noted on the early substrate mixer failed to scale up. | The first mixer of TP-81076 using 70V cured in 4-4 days at 120°F. The time to reach level curves on the 600 gallon verification mix was 12 days. | Investigation is proceeding. To date, additional 600 gallon mixer have not been processed. | Subsonic. 70V is known to require an acid environment to chemically change into the granular which is the actual catalyst in the polymer reaction. | TP-81076 | SE |
| Soft spots and soft streaks were observed in the larger mixer. | When the TP-81175 propellant was scaled up to the five gallon mixer, soft spots and soft streaks were noted in the mechanical properties samples. This soft, spongy propellant was also found in the 600 gallon mixer. | The level standards were increased all the way to the bottom prior to making addition in the five gallon mixer. Then later, the mixer blades were replaced with those which provide half the original timespan blade to level. In the 600 gallon mix level casting system, the propellant from the walls was not sent through the system they just stay before the wall propellant gets into the casting zone. | The high viscosity of the propellant which formed a film on the bowl wall could not be cleaned away from the bowl viscosity was lowered quickly by the addition of acetone. | TP-81175 | SEAS |
| A film of cured-like propellant formed on the surface. | The use of TP-81175 was preheated and sealed with Argon purge. When the mix was final mixer, the pressure was released. When the lid was removed in the casting area, a "curved" film was noted on the propellant surface. | The 80V used was allowed plenty of time to cure prior to the part being used around propellant. | The base and lid used to purge the die level prior to storage and shipping to the casting area had been sealed with 80V and the curing was involved in the cure of the 80V material with the propellant ingredients to cause the problem. | TP-81175 | SEAS |
| The propellant cured but had soft, lumpy areas. | The propellant cured into the first stage mixer former was soft and lumpy. There were also "spots" of soft propellant in the die former which raised ring area. | The 80V parts were vacuum dried at high temperature for extended periods of time to allow the alcohol time to diffuse out of the 80V part before it was placed in the casting tank. | The soft propellant areas were caused by the alcohol formed by the cure of the 80V in the die former which was not removed with the acetone in the propellant surrounding the cure. | 70V | 0-4 |
| Horizontal properties failed to make-up. | The mechanical properties of the 600 gallon mixer were not as good as the substrate mixer and densification of the propellant outside was noted. | The time which the preheated propellant was stored was increased to a minimum of 24 hours. | The processed time curves are prepared. First, it allows the polymer to wet the outside and second, it allows the chemical constituents to proceed to completion. | TP-81086 | Standard Results |
| Burn rate of the propellant failed to make-up. | The burn rate of the Mustang First Stage propellant varied over a considerable range as a function of mix time prior to addition of the primer. | The mix time of the oxidizer blending step of the mix cycle was tightly controlled to maintain the same burn rate throughout the mixing process. | The blending of the dry oxidizer in the horizontal mixer was usually grinding the AP by attrition. | TP-81011 | Mustang I |
| Burn rate of the propellant. | The burn rate of the Mustang First Stage propellant, TP-81011, increased as a function of time the polymer was moved to the tank zone. | See tanks were fabricated from stainless steel to eliminate the corrosion of the acid canal. | The loss from the acid canal tanks in which the 80 polymer was stored was entering as a burn rate anomaly. | TP-81011 | Mustang I |

Fig. 10. Discussion of anomalies presented by Thiokol [report to Jet Propulsion Lab].

More important than the arithmetical accuracy of the results was the first recognition that this complex problem may be amenable to scientific analysis after all. The point to note here is that the importance of such work was recognized long before 1986. The letter from Professor Summerfield (Appendix E) documents this.

A major step toward a scientific delineation of the quality assurance in solid rockets was taken at MSFC via the report "Solid Propulsion Integrity Program Technical Plan" (Preliminary Rept. No. 2-1635-7-14). Clear recognition was made of the fact that

in process management of particle size distribution, surface area and concentration of critical ingredients such as iron oxide, aluminum oxide and ammonium perchlorate should be developed, or improved. Measurement of in process viscosity is important and needs improvement. Process controls need to be evaluated for the capability of providing control of the important parameters within the necessary limits as they become known.¹²

A briefing to industry by Richard Brown^{12,13} also has important details and future plans to minimize surprises.

A program was established at JPL by Code M and MSFC to study these problems. As part of that larger program, one low-level effort in 1984-85 indicated the importance of actual temperatures as contrasted with global mixer jacket temperatures, for example. Especially in a large mix, it was shown that the actual propellant temperature could not only differ from the jacket temperature, but differ at different locations within the mix itself (Fig. 11). A simple Arrhenius rate cure analysis indicated that increases of only one to two degrees Fahrenheit in the mix temperature could result in a decrease of two to three percent in the burn rate of the cured propellant. This simple quantitative estimate was made in an unpublished interoffice memorandum at JPL in 1984. It is likely that one or two degrees difference in the mean temperature could be indicative of five or more degrees difference in local temperatures in the slurry, which could lead to significantly different curing rates, especially if these differences occur after the addition of the curing agent (see Fig. 12). A very careful entry was attempted of literally thousands of data points (mostly from JPL data sources obtained in a nozzle evaluation rocket program) in an unfunded study at the University of Arizona. This data base was generated on a PC by Hal Hikita.

At this point, it would be useful to recall two important aspects of solid propellant predictability. First, the number of parameters is so large that a traditional scientific formulation and analysis may be very difficult, even with the availability of large computers. Second, the key processes that finally result in the cured propellant (and its combustion) must be well understood in order to even look for meaningful trends. What this means is that unconventional approaches may be necessary to obtain a good feel for the variabilities and variations. In other words, we may have to make educated guesses about the probable influences before subjecting the data to a more careful scrutiny.

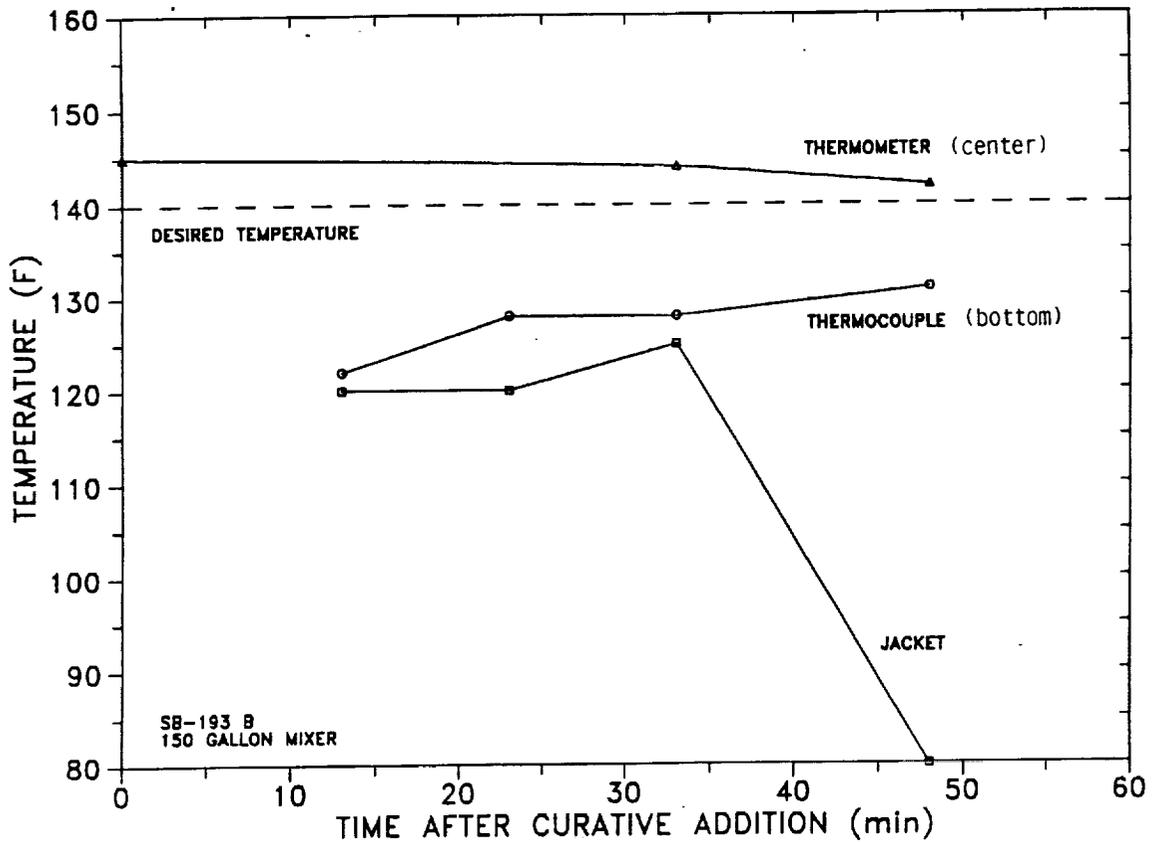
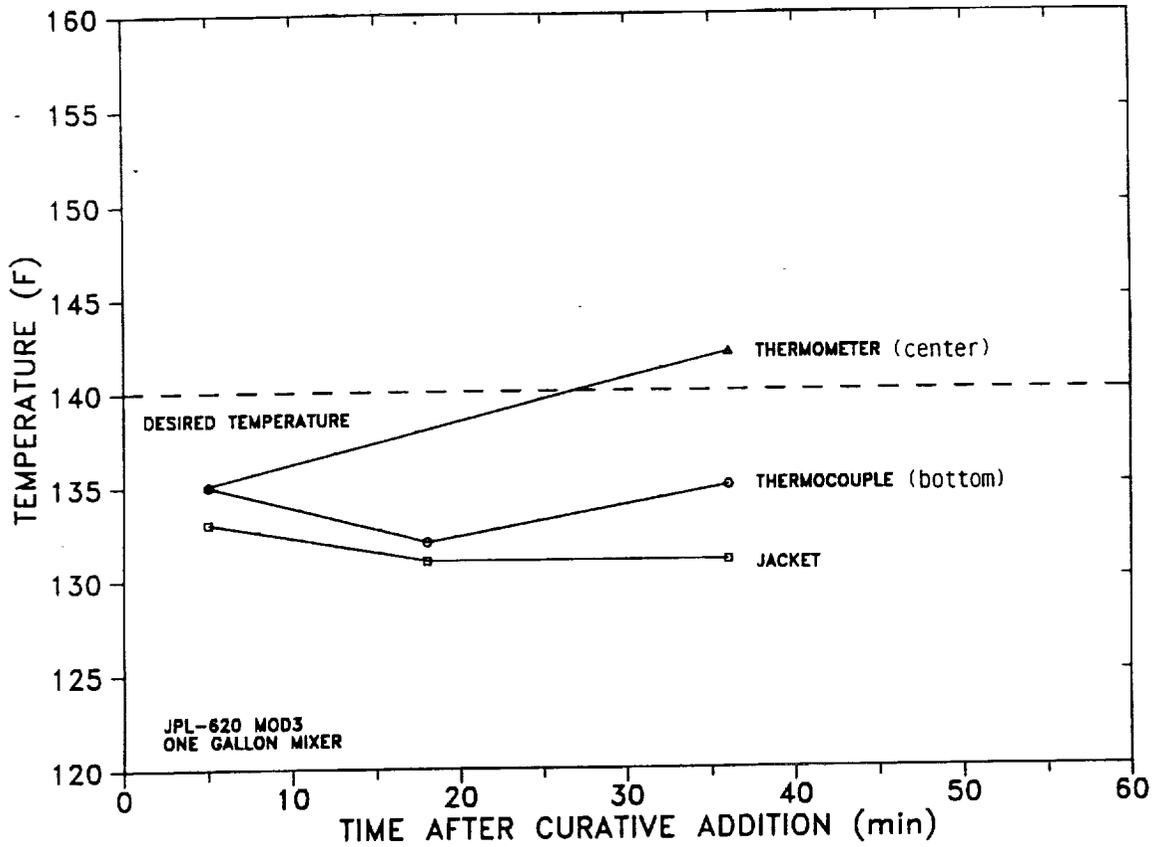


Fig. 11. Actual propellant temperatures in different locations in the mix.

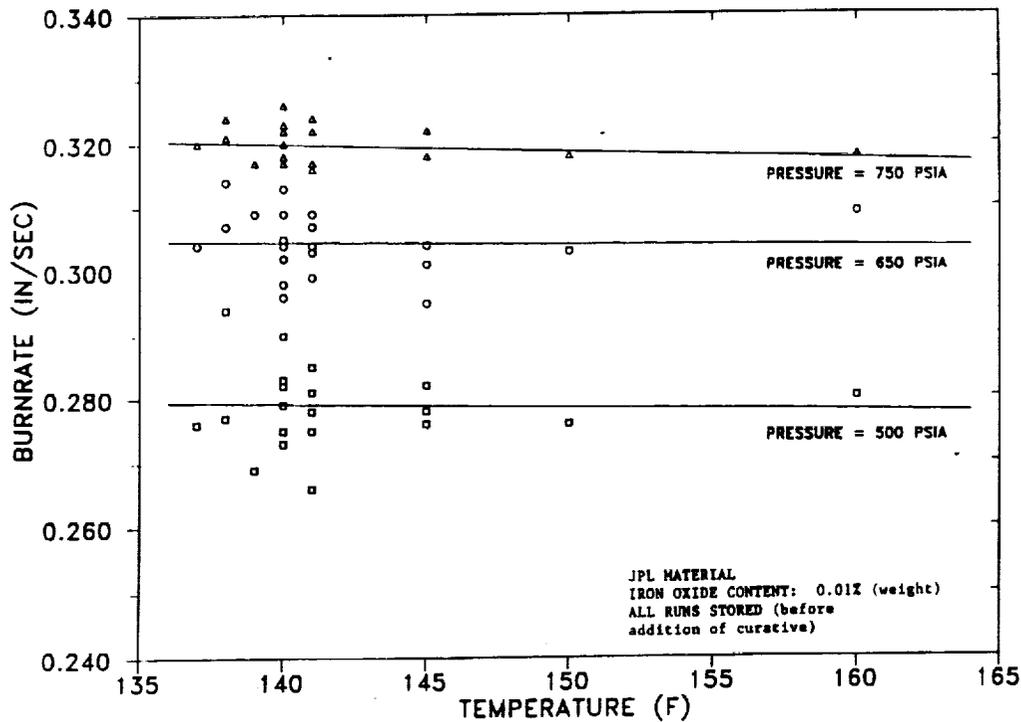


Fig. 12. Burn rate dependence upon maximum last mix stage temperature.

The author feels that it may be instructive to digress here and present two non-technical examples from Sir Arthur Conan Doyle. In the first example, investigators are attempting to reconstruct the events in the night that led to some unfortunate mishaps. Sherlock Holmes guesses that a candle light may have been used in the night, looks for a half-spent candle, and indeed finds it. If he had not looked for it, the candle would not have been found because of all the mud and slush. In another example, he is faced with extracting all the information he can from a small note written hurriedly on the back of a breakfast receipt at a hotel. While Lestrade is preoccupied with the contents of the note, Sherlock Holmes is more fascinated by the very expensive breakfast; this leads him to the hotel where the note was written. That is, what was merely "noise" to Lestrade was indeed the "signal" to Holmes. In a field as complicated as solid propellants, unorthodox and unconventional approaches are necessary to help introduce economical solutions. It is emphasized that such unorthodox approaches should be used only to *narrow down the field of our search* and should not be used as substitutes for scientific and mathematical solutions. End of digression!

Correlations were attempted based on scientific criteria; in the absence of guiding scientific analyses, attempts at obtaining correlations among these extensive sets of data would have been both meaningless and futile. Two of the most important correlations were seen between the end-of-mix viscosity and the burn rate (they are anti-correlated), Fig. 13, and between the shore A hardness and the burn rate (Fig. 14). The significance of these

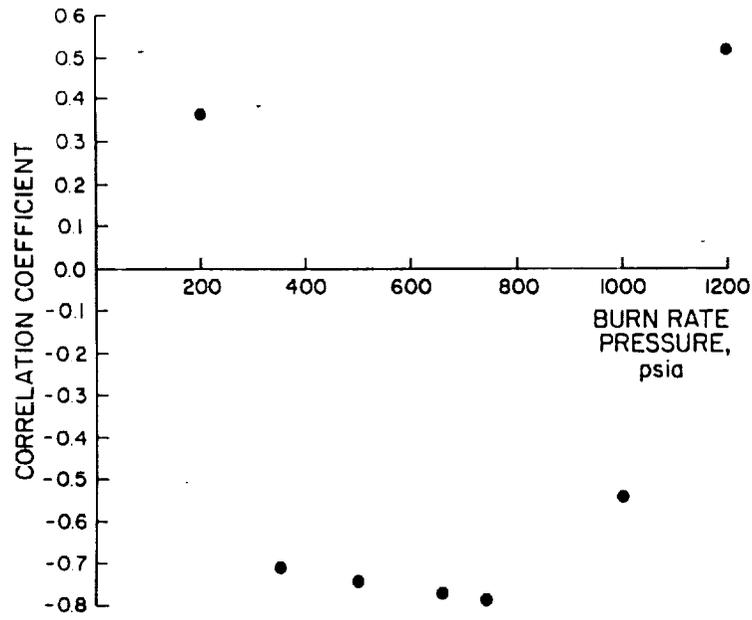


Fig. 13. Burn rate-end of mix viscosity correlation coefficient versus pressure.

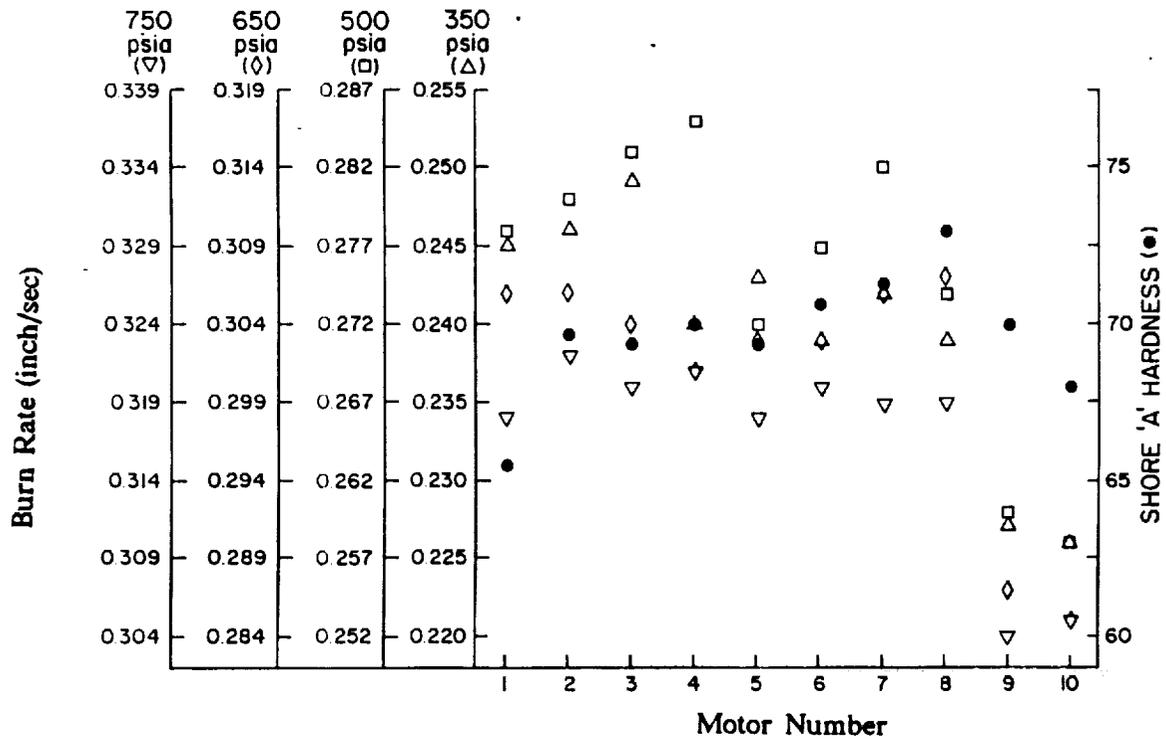


Fig. 14. Shore A hardness/burn rate data.

was described elsewhere.¹⁴ The mention of viscosity as a parameter does not mean that the determination of viscosity is simple, or easy. Measurement (and interpretation) of viscosity of a high solids slurry is by no means well understood. We find that in-situ measurements (where possible), batch-interrupt measurements, and others give different values. The rate of shear is very important. Recent results have also shown that the orifice diameter and edge shape can influence the measured values. In a senior design project, two students built a viscometer that gave continuous real-time viscosity in a mixer that used high-viscosity fluids, simulating propellants. The apparatus was somewhat larger than what could be conveniently included in a practical propellant mixer, but has provided a first step in a highly desirable approach. The main point to note is that the important parameter, namely slurry viscosity, does not appear to be measurable in an unambiguous way at the present time.

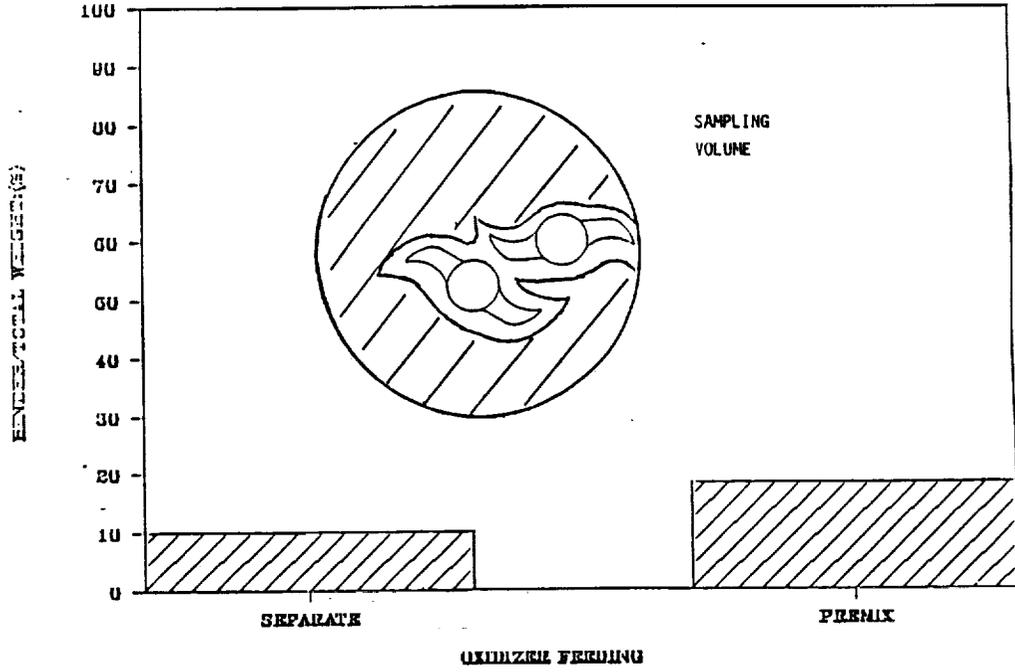
More recently, five other plots were discovered to be significant in information content.¹⁵ In Figure 15, we see the non-uniformity of the oxidized particles in the slurry. The composition near the blade is not the same as the bulk values. The basic message is that important pieces of information are available on the manufacturing of propellants. More are needed.

Long-Range and Short-Term Objectives

Development of a fundamental and scientific understanding of the complex processes involved in solid propellant manufacture and end use (combustion in a rocket motor) will need a commitment and should involve a well-coordinated nationwide effort among NASA, DoD, industry, and the universities. Meaningful results that will prove their use in quality assurance and predictability can be realistically expected in ten years after the initiation of such an effort. The results will quantitatively relate the performance of a rocket motor (the thrust time curve, for example) to the ingredients and processing variables; the program will also evolve unambiguous *a priori* rules for effecting desired changes in propellant systems. For example, one of the main results will be to evolve a table indicating the effect of propellant (slurry) mix temperature and the end-use burn rate. Another example is the prediction of the burn rate as a function of pressure as the curve is influenced by the variance of the fine particle size distribution from the mean. Yet another example may be the precise prediction of the burn rate when the shape of the coarse particles is specified as a deviation from spheres.

In a field that is as important and current as solid rockets, it would be appropriate to demand more immediate results. Recent work¹⁴ has clearly indicated the definite promise of such results. For example, it was shown that the final mixing time has a measurable

BINDER CONCENTRATION



WET AREA

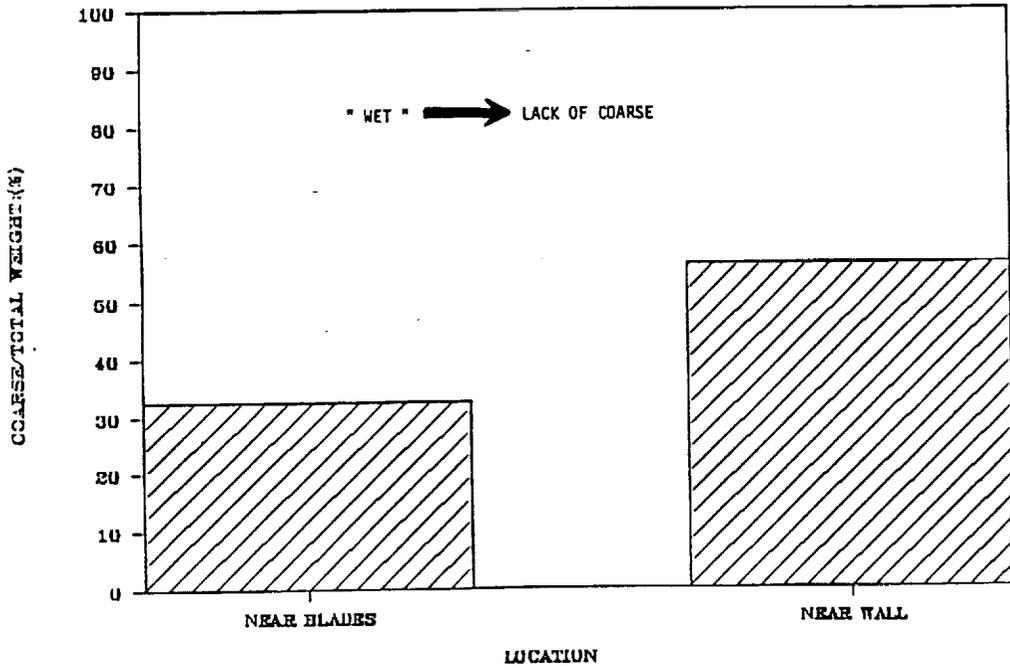


Fig. 15. Non-uniformity of oxidized particles in slurry.

effect upon the burn rate and the Young's modulus of the cured propellant. It was also shown that the end-of-mix (EoM) viscosity is a definite indicator of the burn rate variation of the cured propellant. Such quantitative observations are significant. For example, the processing could carefully monitor the slurry viscosity continuously, and when the viscosity deviates beyond a specified bound, corrective actions would be initiated. This would avoid the costly waste of the production of a full-scale motor of substandard, or unacceptable, quality. To some extent, such observations are indeed in use at the present time. The author admired the judgment of Joe Hance (who, incidentally, directed the processing and production of the T-17 propellant that was successfully used in Explorer I), who would make a decision to stop the processing of propellants based merely on observation of the "quality" of the slurry; the explanation would usually be something like, "the LP-3 had probably deteriorated during storage." This admiration invariably turned quickly into frustration upon realizing that solid propellant quality assurance was not scientifically prescribed, but depended instead on the feel of experience. In the short-term, a program, such as the one discussed in this report, would evolve quantitative, if semi-empirical, rules that will be useful in processing. The qualitative feel of experience will be made scientifically respectable and technologically acceptable through independent verifications. The point is that the benefits of a fundamental program will be felt immediately. These short-term objectives will be to provide clear, dependable guidelines for economical processing and a list of measurable parameters that give a tell-tale signal of the health of the propellant.

The Legacy of Black Art

Solid propellants have also suffered from their legacy of black art. Many of their manufacturing techniques cannot be traced to scientific evolution. The detailed batch sheets and SOPs (Standard Operating Procedures) are usually the result of experience. It would be most useful to revisit some of these.

Solids Versus Liquids

There appears to be a growing feeling among many concerned¹⁶ that eliminating solid rockets altogether, in favor of liquids, would completely "solve" all problems. The absence of a Challenger-class (liquid rocket) catastrophic failure¹⁷ belies the extreme vulnerability of liquid rocket motors. It would be wise to recall that there have been a number of near misses with liquid rockets in recent years. The major problems are systematically outlined by Feynman:¹⁸

- Turbine blade cracks in high-pressure fuel turbopumps (HPFTP). [May have been solved.]

- Turbine blade cracks in high-pressure oxygen turbopumps (HPOTP).
- Augmented spark igniter (ASI) line rupture.
- Purge check valve failure.
- ASI chamber erosion.
- HPFTP turbine sheet metal cracking.
- HPFTP coolant linear failure.
- Main combustion chamber outlet elbow failure.
- Main combustion chamber inlet elbow weld offset.
- HPOTP subsynchronous whirl.
- Flight acceleration safety cutoff system. [Partial failure in a redundant system.]
- Bearing spalling. [Partially solved.]
- A vibration at 4,000 Hertz making some engines inoperable, etc.

There have also been major catastrophic failures, involving key components, in static tests. The dramatic explosion of Ariane Spot 1's third stage provides a flight example in recent times (November 1986). Another serious problem with liquid propellant rockets is beginning to be recognized lately. This is the potential for orbital debris creation. While the exact cause is not yet known, many believe that a debris hit caused the Ariane third-stage explosion in 1986 (Fig. 16): **"Officials believe the most likely cause of the explosion was the detonation of residual oxygen/hydrogen propellants in the vehicle".**¹⁹ This "Ariane Spot 1 rocket body represents the single greatest source of debris now in orbit about the earth."²⁰ The pressure-fed systems used in liquid rockets are a source of catastrophic explosions upon impact. Many other problems include leaks, toxicity hazards in the vacuum of space, and extreme low temperatures in the vicinity of cryogenic tanks; many serious problems in several operational spacecraft have indeed been traced to these sources. Mechanically, liquid rockets are far more complex than solid rockets—a fact that has frequently forced long delays in launches due to last-minute repairs. At the fundamental level, the combustion processes of the liquids (providing thrust) are no better understood than those of solids. It would be prudent to keep all options alive at this time, and for the foreseeable future, unless a major advance is made in liquid rocket reliability and safety. After all, it is its intrinsic simplicity that has made the solid rocket so attractive for centuries. This simplicity allows for a great flexibility in the size of the motor at little cost. A well-proved solid propellant can be loaded into motors of any size. In extreme cases, a piece of propellant from a larger motor can be cut, loaded into, and used in a smaller motor. Such flexibility is totally absent in liquids, which still need the full system of components in the smaller motor.

We cannot give up the proven merits of the solid motor simply because some problems remain unsolved; in fact, the merits provide a strong motivation for scientifically solving these few remaining problems.

Used Ariane Stage Explodes, Creating Space Debris Hazard

Washington—A European Ariane booster third stage launched nine months ago exploded in space Nov. 13, creating potentially hazardous orbiting debris and prompting a U. S. request that Ariane-space investigate the incident to prevent a recurrence.

The Ariane 1 stage had been used Feb. 22 to launch the French Spot 1 Earth resources satellite (AWAST Mar. 3, p. 21).

The explosion could result in changes to avoid such incidents and limit the buildup of debris orbiting Earth, according to Frederic d'Allest, president of Ariane-space.

The explosion of the spent stage is not believed to be linked to problems experienced in the oxygen/hydrogen system during powered flight. The Ariane third stage has failed three times, most recently on May 30 (AWAST June 9, p. 21).

Before the explosion, the White House, State Dept. and National Aeronautics and Space Administration had begun an effort to alert international space agencies to the debris issue.

The Ariane stage was orbiting in about a 490-mi. Sun-synchronous polar orbit inclined 98.7 deg. when it exploded. The force of the explosion threw debris into orbits as low as 270 mi. and as high as 840 mi.

The incident occurred at 7:39 p. m. GMT Nov. 13 just after the Ariane stage passed the equator on a northbound path over the central Atlantic between South America and Africa.

Ground Tracking

U. S. Air Force Space Command and Navy Space Surveillance System radars are tracking about 200 pieces of debris one-half inch in diameter or larger. This suggests the presence of several hundred or thousands of smaller particles impossible to track with ground-based radars. Even a small particle orbiting at high velocity could cripple or destroy a spacecraft—manned or unmanned—were a collision to occur.

Officials believe the most likely cause of the explosion was the detonation of residual oxygen/hydrogen propellants in the vehicle. Space Command conducted computer analyses to determine whether the breakup was caused by collision with other space debris. Radar data, however, show no other trackable debris in the area.

Space Command analysts believe that other Ariane third stages launched into geosynchronous orbit may have exploded after long exposure in space. Evidence comes from tracking apparent debris from these vehicles, although such fragments are extremely hard to track since they orbit above the equator, where the U. S. has minimal radar capability. The Spot 1 stage was flying in an orbit where tracking is far easier.

Although the odds of collision with a useful satellite are small, many spacecraft have orbits that pass through the area in which the Ariane debris has dispersed.

There also is significant debris in this area from seven U. S. Delta second stages that exploded years ago after prolonged exposure to the space environment. The Delta incidents created a continuing space debris problem and subsequent Delta stages have been modified to prevent potentially explosive conditions from building. □

Some Simple Approaches

Composite solid propellant predictability and quality assurance can only come through adequate control of the ingredients and processing. As was evident throughout this meeting, and other information sources, we are beginning to identify some of the more important parameters that one must control and for which specifications must be established. After such specifications are proposed, they must still go through a series of independent verifications, different scales of mixers, different sizes of motors, and different firing conditions before they can be well received, accepted, and followed. In the meantime, some of the more straightforward procedures that the author has followed are described here.

1. Simple Physical and Chemical Examination of the Oxidizer.--Very simple SEM/EDAX examinations of the AP, as received, can be quite revealing. Shown in Figs. 17 and 18 are AP crystals from two sources; Fig. 17 shows AP from a source in the USA, while Fig. 18 shows AP from a Japanese source (Nahun Kaleet). The differences are dramatic. Not only are the Japanese AP much more spherical, but their sizes are far more uniform; the particle size seems to approach a unimodal distribution. Prilling produced the near spherical AP in Fig. 18. The precise quantitative influences of this difference in shape on the processing, cast, cure, and combustion are not clear. It would seem obvious that there will be substantial differences. While this example is intentionally chosen here to make a point, the utility of simple SEM examination of as-received AP should be obvious, even when the shape differences are not this dramatic. [It is most interesting that nearly three months after these shape influences were discussed at this meeting, a paper discussing very similar concerns and data was presented at the AIAA/ASME/SAE/ASEE 25th Joint Propulsion Conference, Monterey California, July 10-12, 1989.²¹]

2. Simple SEM Examinations of the Cast (Cut) Propellant.--Scanning electron microscopy has been extensively used in the diagnostics of quenched samples from combustion experiments; the pioneering work at NOTS/NWC is most familiar to those in the field of composite propellants. However, the use of SEM for simpler examination of cured propellants is not that prevalent. In one of the programs on low-smoke, high-burn-rate AP propellants, some candidate propellants exhibited unacceptably poor reproducibility (Fig. 19). Pressed for time, we attempted a simple SEM examination of the cured propellants. In Fig. 20, the propellant looks fairly good in terms of mixing, voids, and the coarse/fine distribution; this was indeed the propellant that burned reproducibly. In Fig. 21, we see a very different pattern. The propellant does not appear to have mixed well, voids are present, and the coarse/fine distribution does not appear to be uniform. This was indeed a

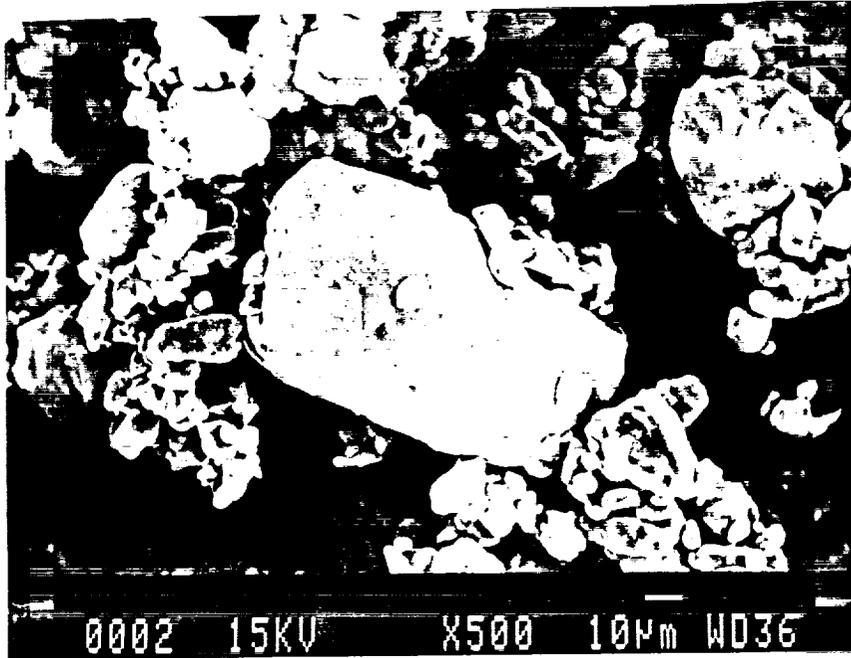


Fig. 17. AP crystals obtained from a source in the United States.

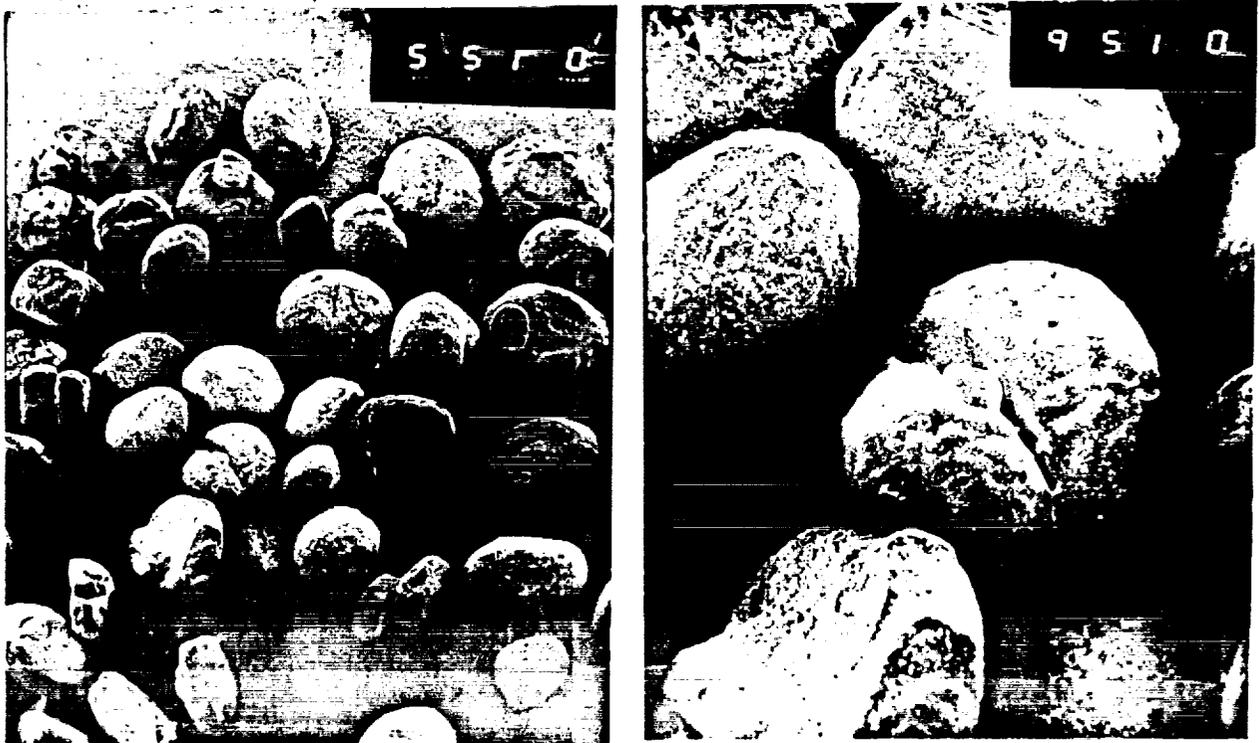


Fig. 18. AP crystals obtained from a source in Japan.

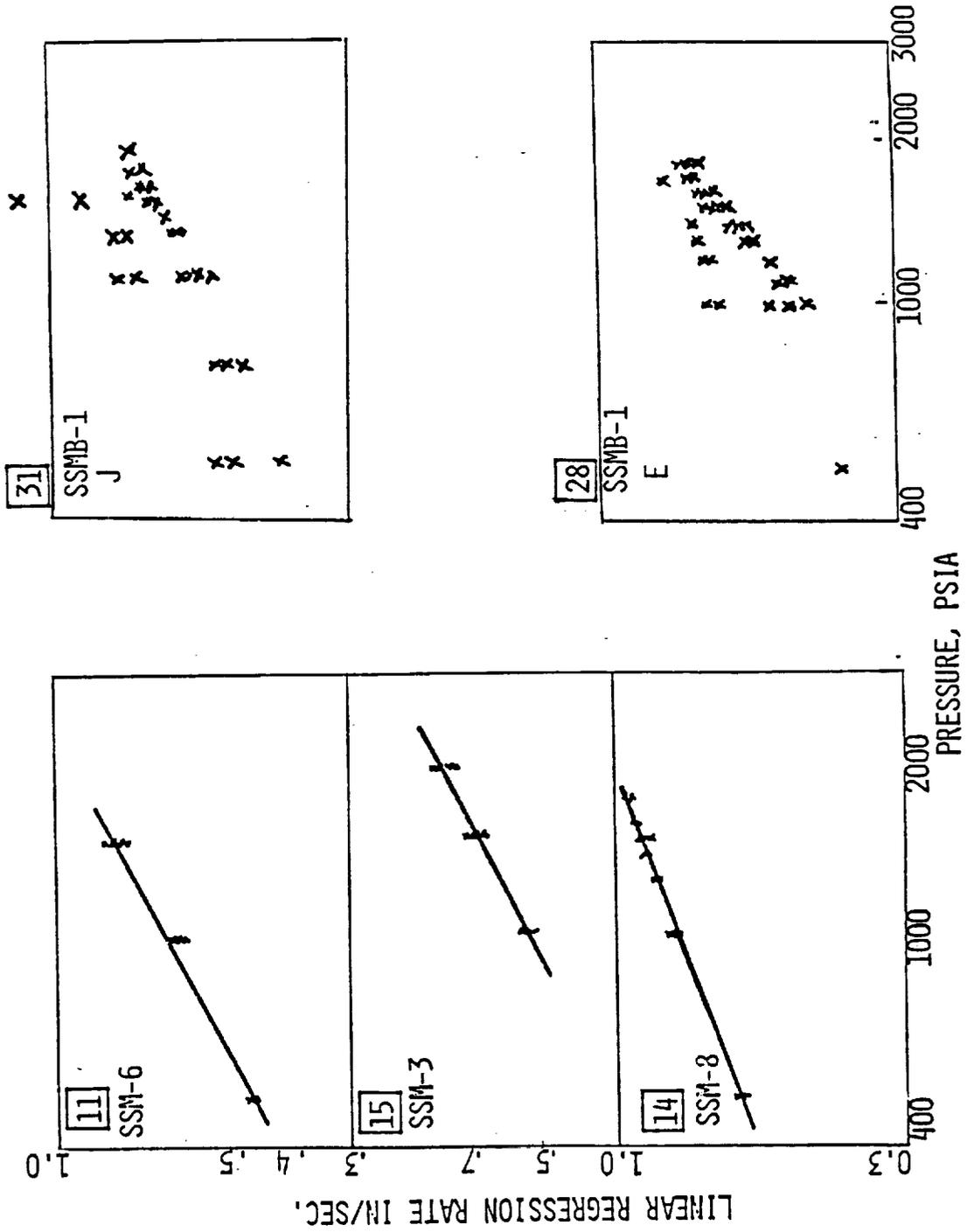


Fig. 19. Time-independent combustion in the Crawford bomb.

propellant that burned in a non-reproducible manner. While these SEM examinations do not solve the problem, they can economically reveal the problem source.

3. Complete Examination of the Particle Size Distribution.--Many ingredients in composite propellants are particles. Examples include coarse AP, fine AP, and aluminum. These particle sizes were designated by the commercially convenient 50% weight average point. This is wholly inadequate for our purposes. Different distributions can have identical 50% weight average points. Shown in Fig. 22 are two such distributions. Their influence on combustion was acutely felt in one program. A solid rocket motor was developed with the first grind and was stable within the pressure range of interest in a double BATES motor. Having exhausted our supply of fine AP, we borrowed some AP of the "same size" from a nearby laboratory to complete the motor tests. The new batch of motors went unstable in firing tests. As is evident in Fig. 22, the second AP had a narrower distribution, with the 100% weight average point at 20 microns, as contrasted with 40 microns for the first AP. A simple computation of natural propellant frequency (mean burn rate divided by the 100% weight average point of fine AP) shows that the frequencies for the two propellants are substantially different. In the first case, the frequency was not close to any of the natural acoustic frequencies of the rocket motor cavity; in the second case, it was. This example from 1973 may seem a little archaic. Today, more complete particle size analyses are indeed routine. Nevertheless, this experience is typical of many other ingredient characterizations that are inadequate to ensure quality and reproducibility in solid rockets.

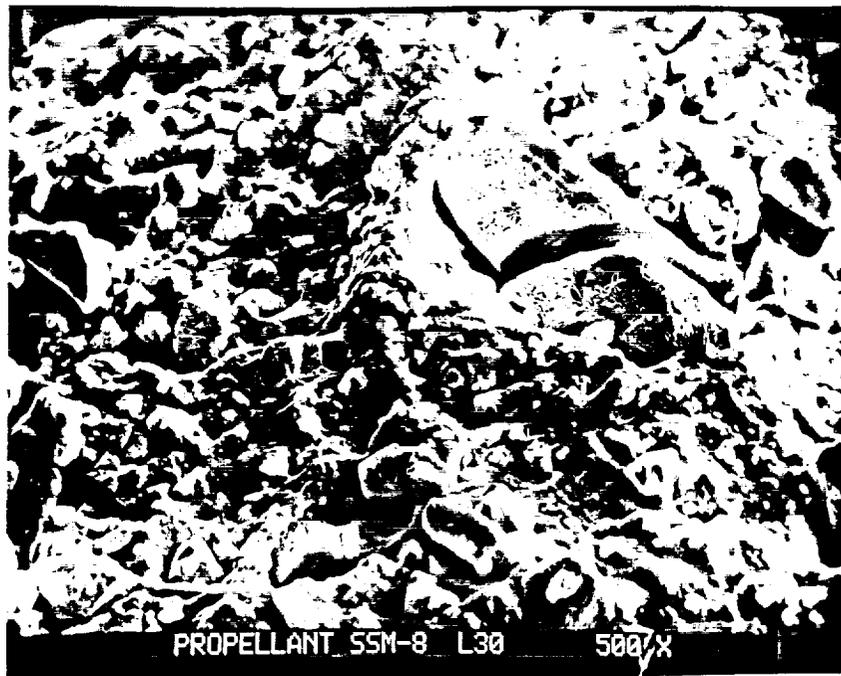


Fig. 20. A propellant whose burn could be reproduced.

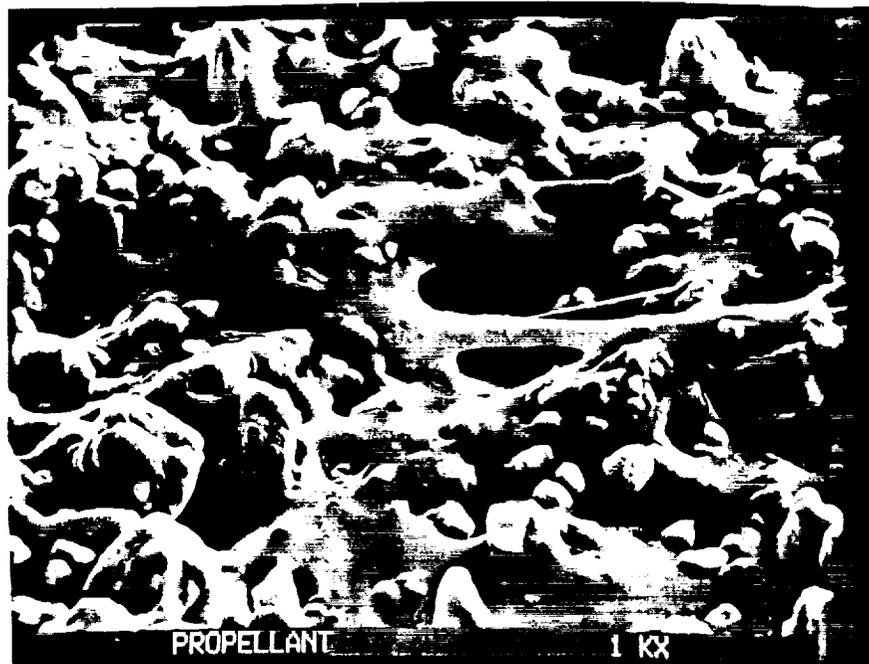


Fig. 21. A propellant that burned in a non-reproducible manner.

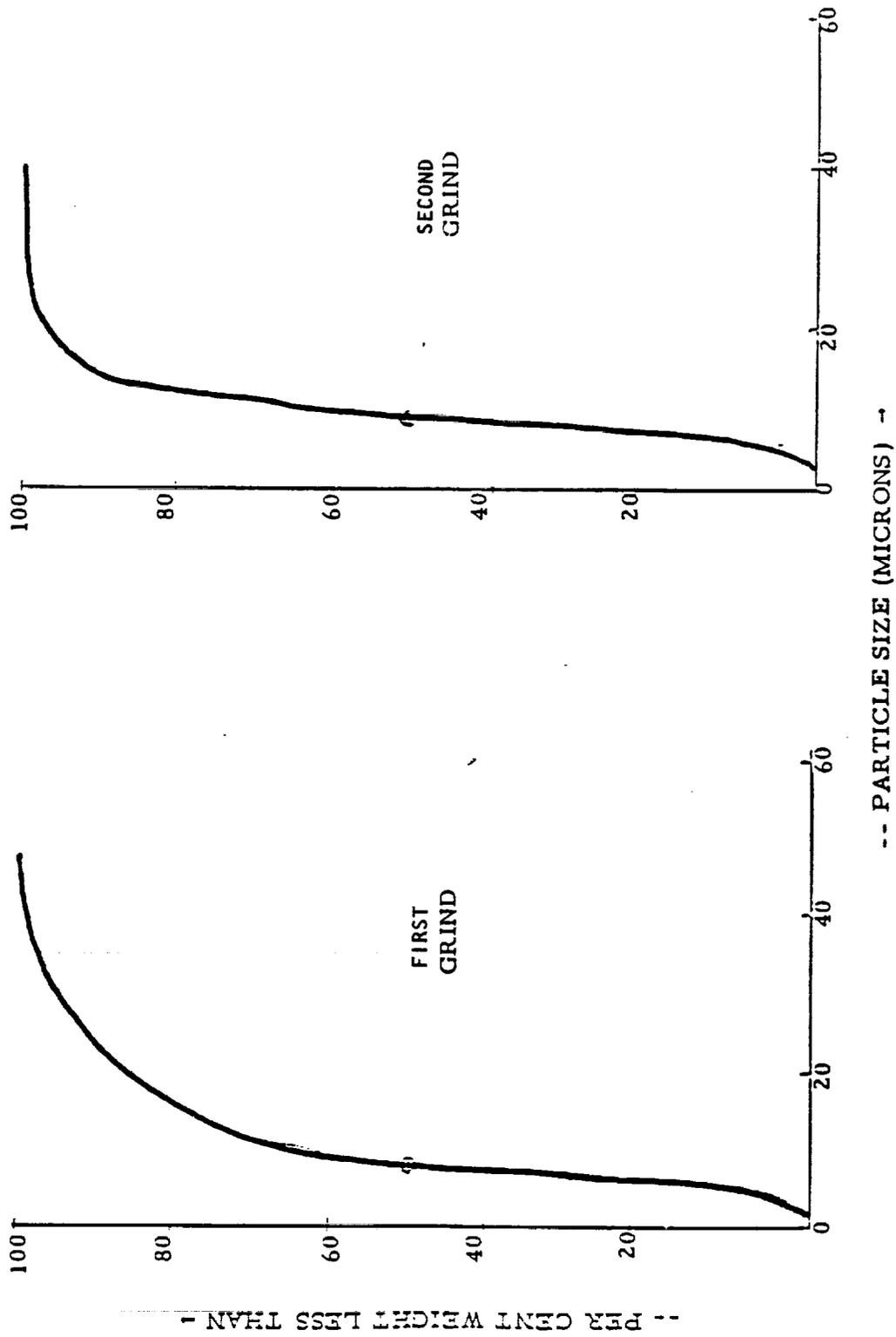


Fig. 22. Differences in the "fine" AP as revealed by micromerograph analyses.

THE UNIVERSITY OF ARIZONA PROGRAM IN SOLID PROPELLANTS

P30

Introduction - Kumar Ramohalli

The University of Arizona program is aimed at introducing scientific rigor to the predictability and quality assurance of composite solid propellants. As already noted, the main program in this area is conducted for NASA Marshall Space Flight Center. The statement of work is available in ref. 14. Two separate approaches are followed concurrently. One is attempting to use many of the modern analytical techniques to experimentally study carefully controlled propellant batches to discern trends in mixing, casting, and cure. The other is examining a vast bank of data, mostly obtained at JPL as part of a NASA MSFC study, that has fairly detailed information on the ingredients, processing, and rocket firing results, including mechanical property values of JANNAF standard dumb-bells (dog-bones). The experimental and analytical work is described briefly by Daniel Perez in this report. The principal findings have been that pre- (dry) blending of the coarse and fine AP can significantly improve the uniformity of mixing, the Fourier transformed infrared spectra of the uncured and cured polymer have valuable data on the state of the fuel, there are considerable non-uniformities in the propellant slurry composition near the solid surfaces (blades, wall) compared to the bulk slurry, and in situ measurement of slurry viscosity continuously during mixing can give a good indication of the state of the slurry.

In the related study of the voluminous data bank, several observations are important. First, this is perhaps the single most carefully controlled set of solid composite propellant data. Close scrutiny revealed that many of the "identical" batches had variations in the iron oxide particle size, concentration, source, batch size, motor size, etc. Thus, we found only a small fraction of the initial data bank to have been really "identical" within the available information records; there could be variations that were not noted. Even in this small fraction (approximately 31 data points), variations are apparent. The fundamental advance made at the University of Arizona has been the careful logging of all available data with a color-coded entry into a popular spread-sheet program for easy manipulations and the generation of graphs to show trends more readily. This work is currently continuing.

Arthur Mazer, a student in the Department of Applied Mathematics, has approached the problem of mixing in a mathematical way. His work is described later in this chapter in a highly abridged manner. The question of mixing of various ingredients has to be properly understood. In some mathematical formulations and approach, the mixture becomes homogeneous in the limit of infinite time of mixing. This is simply not the case in composite propellants. Even in the limit of infinite time, the propellant continues to be heterogeneous but more uniform than at the start. Thus, the concept of the smallest scale

for uniformity has to be established. Is this the size of the coarse particles? Is this several multiples of the coarse particle diameter? Is there a fundamental spatial scale that is truly representative of the homogeneity of the end product? Art Mazer and Professor Vincent will answer these questions. For now, it is most interesting that we may have to evolve the concept of **heterogeneous homogeneity** in order to mathematically characterize the mixing in composite propellants.

The importance of quantitatively accurate color displays cannot be overemphasized. Here, Mike Hicks (with Professor Nikraves) is programming the governing equations on the computer specifically to identify **dead zones** that could lead to improper mixing. His work is also briefly described here.

As part of the studies being conducted at the University of Arizona's NASA Center, extraterrestrial propellant production is examined by Paul Schallhorn. Although this is not part of the MSFC program, it is felt worthwhile to include his work here to indicate the important steps being taken to automate composite propellant processing and to minimize questionable human judgmental factors.

Some of these studies are less than six months old, but already indicate the promise of a better understanding of composite propellants.

An Interim Progress Report on Mixing - Dan Perez

Introduction

No research is readily available on high solid concentration mixing. Yet problems evident in the processing of such materials are well known. Data on the variations in mechanical properties and burn-rate performance have been given in the literature.^{9,11} Under "identical" procedures and with material from the same lots, propellants have been manufactured with distinct and discernible differences in performance.

A recent review of the process has opened the door to speculation on the culprits in the problem. Many causes of the variations have been listed, covering the areas of mixing, casting, and curing. Even the accepted testing techniques of the finished product (i.e., strand burn rate and uniaxial tensile tests) have been criticized. Any and/or all of these variables could be the culprit(s); the volume can be, and is, overwhelming.

Furthermore, the percentage of solid particles within composite solid propellants is extraordinarily high. With the addition of metals, concentrations within 80 to 88 percent by weight of solid material have been manufactured. The understanding of such material processing has an added complication due to the use of multidispersed particle-size systems.

It is quite evident that an endeavor to analyze the entire process at this time would be fruitless. Therefore, work will be pursued on one stage of the process in order to assist in the establishment of the proper route for research in the others.

Work within this research has been, and will continue to be, on the first stage of the process mixing. Since this first step defines the state of the propellant, it must be well understood. Any imperfections which arise within this stage will have to be dealt with in the following stages. In addition, the complexity of the mixing stage, as compared to casting or curing, allows future work to be minimized. Findings in the mixing stage may have the potential of being applicable to the less chaotic behavior of the other stages.

In the following sections, three areas will be briefly stated. These cover those areas of the investigation which are most crucial in the efforts to resolve this problem. The first will describe the JPL data base established to guide and substantiate any findings. The second will state the rheological understanding of the material presently available. Last is the series of testing techniques developed to define the state of the mixture.

Data Base

The data under review were acquired by JPL and consist of a series of 60 batch runs of ammonium perchlorate/PBAN propellant. Each batch constitutes 150 gallons of material. The mixing was done with a Baker Perkins Model 16-PVM vertical planetary mixer with a thermal jacket surrounding the mixing bowl. This is a two-blade dual planetary mixer.

The batch runs were vacuum cast into 48-inch-diameter cartridge molds. Small samples were taken from these molds for analysis. Samples for tensile, density, and burn rate tests were allowed to cure in separate molds. The detailed data sheets on which this information was supplied are included in Appendix F.

The ingredients were received from two sources, and the lots were examined for adherence to specifications. The ingredients and their respective weight percentage within the propellant are given in Table 2.

Table 2. Listing of propellant ingredients.

| Ingredients | Wt. % |
|------------------------------------|-------|
| Ammonium perchlorate (200 microns) | 48.99 |
| Ammonium perchlorate (10 microns) | 21 |
| Aluminum (granular) | 16 |
| Ferric oxide ^a | 0.01 |
| PBAN | 11.49 |
| DOA | 0.7 |
| ECA | 1.81 |

^aFirst 36 runs: 0.01; next 18: 0.04; next 6: 0.27 (AP coarse subsidized for %Wt. balance).

The standard operating procedure is shown in Fig. 7. Actual run schedules were recorded. Automated monitor readings were also noted and hand measurements for propellant temperature and viscosity taken.

An additional 11 runs on identical ingredient lots were completed for 1-gallon batches. These were used as a comparison for the end-product properties of the large batches.

Figures 23 through 26 show samples of several findings which have proven important. Each indicates strong correlations with respect to certain parameters.

Rheology

Unlike mixing, work on high solid concentration rheology is available. From these works, two regions are quite apparent in the rheology. The first lies in the shear rates below 1.0 sec⁻¹, where the material is extremely well behaved in the sense of flow mechanics, and is pseudoplastic in nature. The second is not so hospitable, with sudden viscosity jumps and shear thinning and thickening behavior. The maximum shear rate behavior recorded was as high as 1000 sec⁻¹, so the entire spectrum of shear rates experienced in the mixer was certain to be covered.

A list of the most dominate parameters is mentioned below with respect to each particular region. Note that these are the predominant factors which govern the flow mechanics and, that for high shear rates, two more parameters must be considered in addition to those for low shear rates. In decreasing order of importance, the parameters are:

Low Shear Rates

1. Volumetric solid concentration
2. Particle size distribution
3. Particle shape

High Shear Rates

4. Shear thinning
5. Wall effects

The most interesting and important work is on the rheology of bi-dispersed particle size distributions identical to those within solid composite propellants. Figures 27 and 28 show the behavior of this material based on the theoretical model of synthetic flow.²² This model has proven successful in mapping the viscosity characteristics of the material and plays a large role in future work.

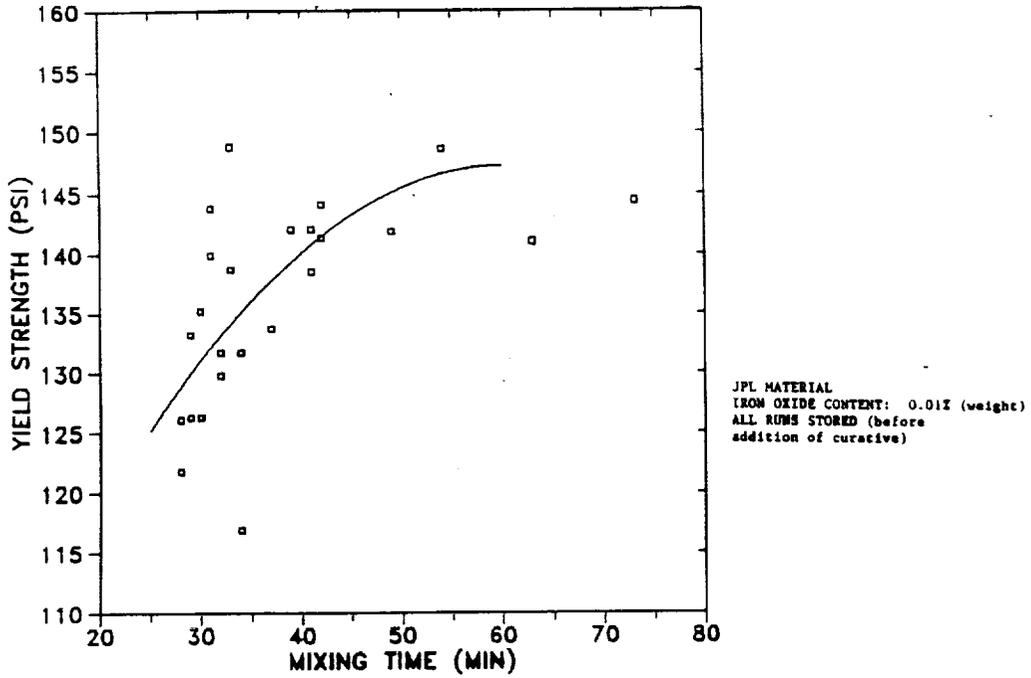


Fig. 23. Yield strength dependence on mixing time.

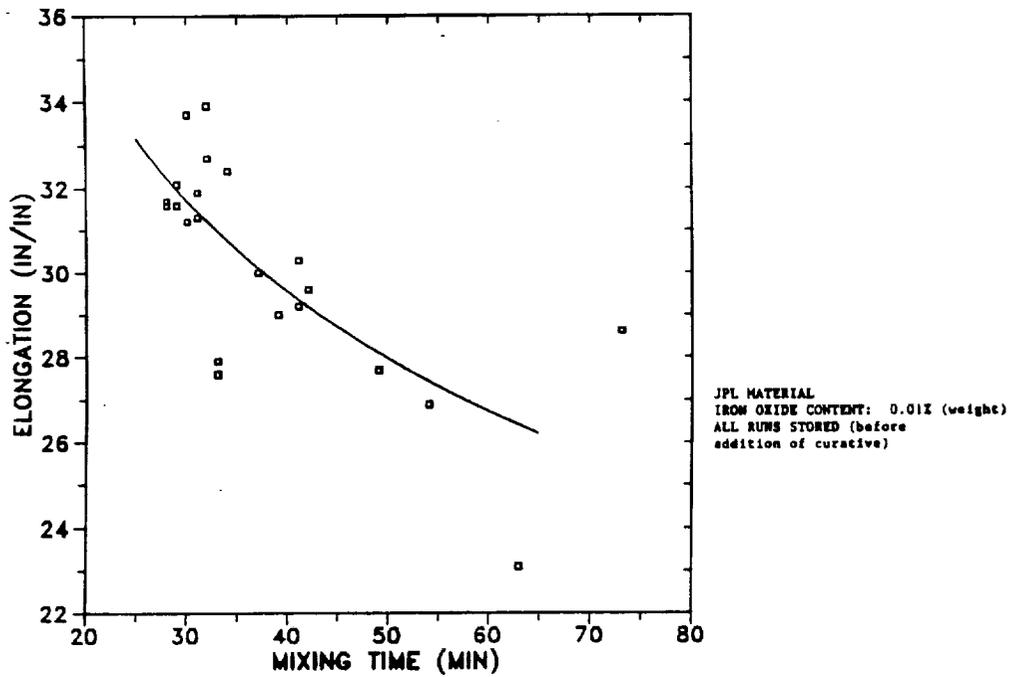


Fig. 24. Elongation dependence on mixing time.

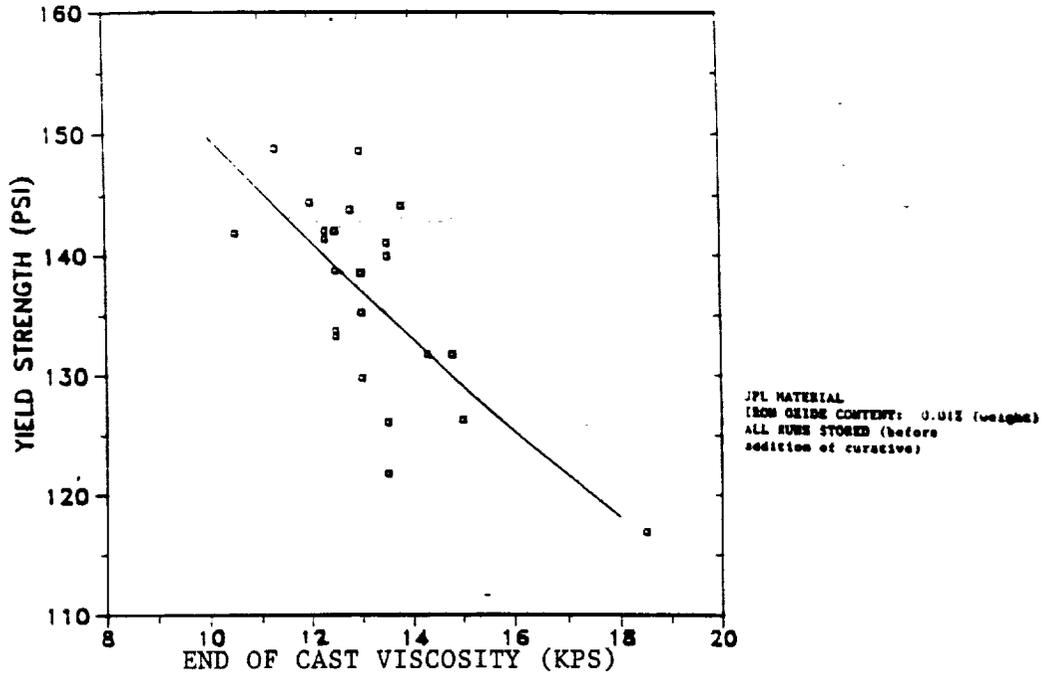


Fig. 25. Yield strength correlation with cast propellant viscosity.

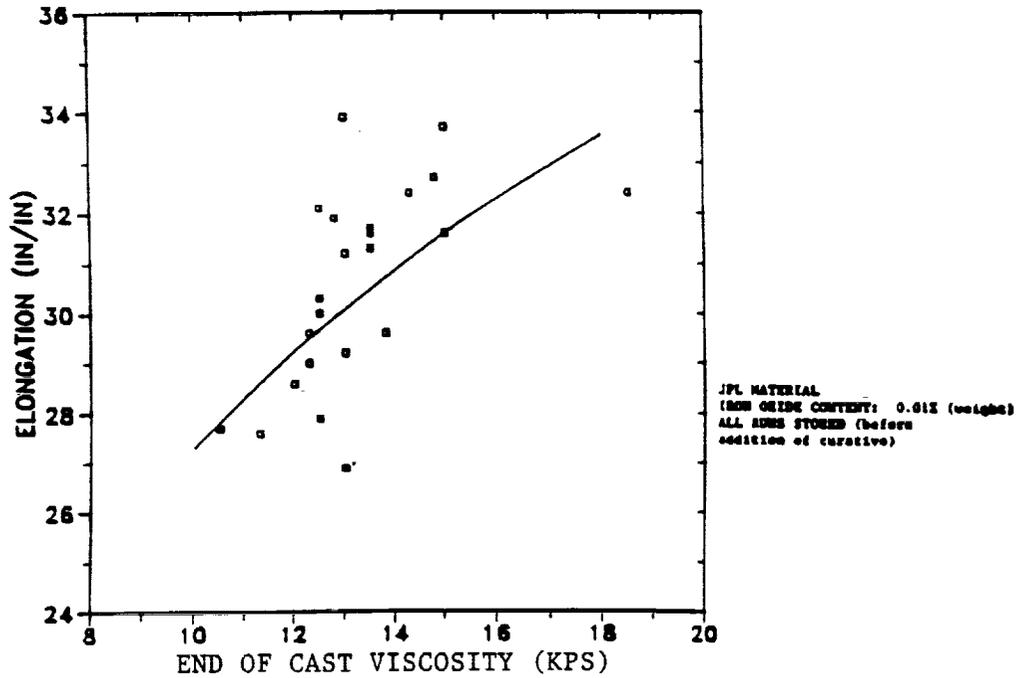


Fig. 26. Elongation correlation with cast propellant viscosity.

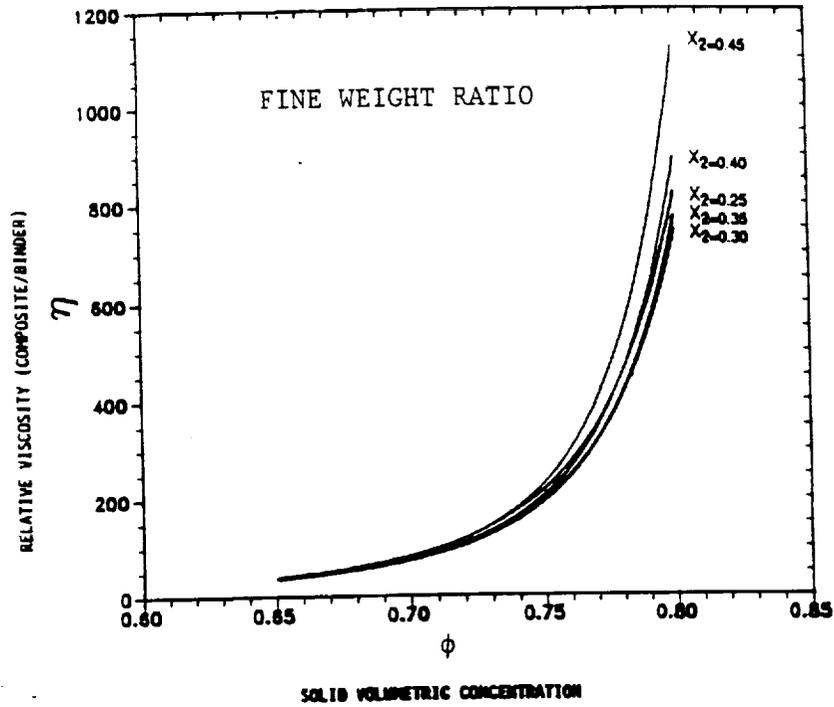


Fig. 27. Relative viscosity with solid volumetric concentration.

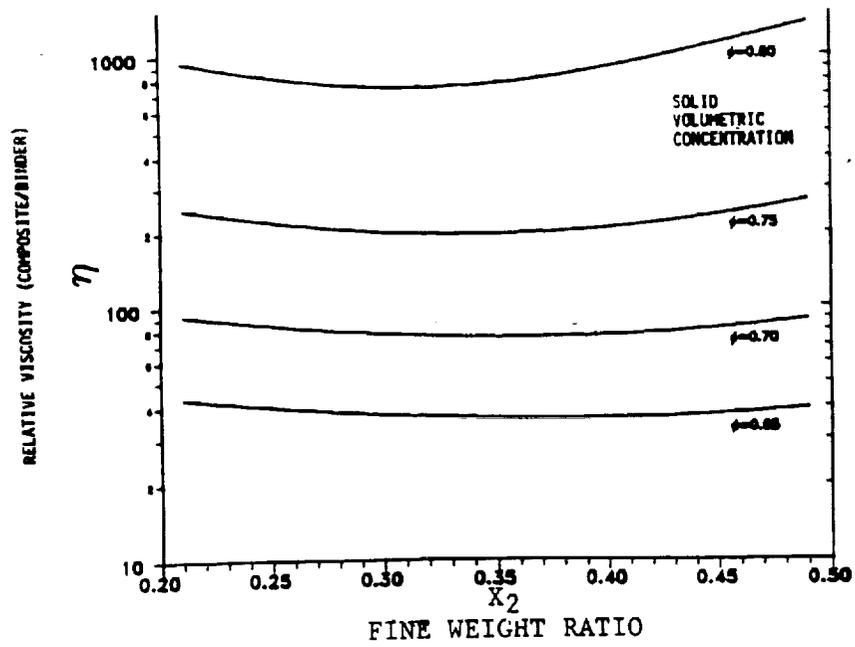


Fig. 28. Relative viscosity with fine weight ratio.

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Testing Techniques

Let it be assumed that, in the manufacture of a solid composite propellant, all the starting materials are properly inspected and conform to the specifications. Furthermore, curing is to be done as per the specifications without any variations. Under these conditions, it is therefore evident that to ensure reliable performance of a propellant from batch to batch, the final mix before curing needs to be well defined. In other words, the final mix has to be properly and fully characterized with reference to

- a. critical solid ingredients by way of particle size, particle size distribution, concentration, etc., and
- b. the binder matrix in terms of its cure stage (molecular weight buildup and cross-linking) in addition to the concentration of various ingredients.

If every propellant mix batch, whether small or large, is brought to conform to this definition before curing, batch-to-batch variation and scale-up problems can be further understood. To achieve this end, very fast and quick techniques have to be established for evaluation of the propellant. These tests have been developed. The analysis includes drawing samples of the mix and extracting, with a suitable solvent, the solid particles from the binder. With this complete, the constituents of the mixture can be inspected as follows:

- a. Insoluble portion--consisting of solid inorganic ingredients such as AP or AL--can be analyzed for particle size and distribution (i.e., with microtrac, microscope, and coulter counter).
- b. Soluble portion--consisting of primarily the binder--can be analyzed for ingredient concentration and polymer growth (i.e., with FTIR spectroscopy and GPC analyzer).

Figures 29 and 30 show the inferred spectrum of the soluble portion of the propellant. Figure 9 has been processed through the extraction technique and therefore in a solution of solvent.

A Mathematical Formulation of Mixing - A. Mazer and T. Vincent

In this section, we present a brief introduction to the design and analysis of a bladeless mixer. The motivation for designing a bladeless mixer is to overcome the shortcomings associated with the blades used in the mixing of solid fuel propellants.

The dynamics of mixing systems has interested mathematicians since Poincaré introduced a geometric viewpoint to the study of differential equations. More recently, the search for chaos has spawned many examples of mixing dynamics. The common feature in all mixing systems is the presence of a positive Lyapunov exponent which indicates that the dynamics stretches trajectories as they pass through certain regions of the domain.

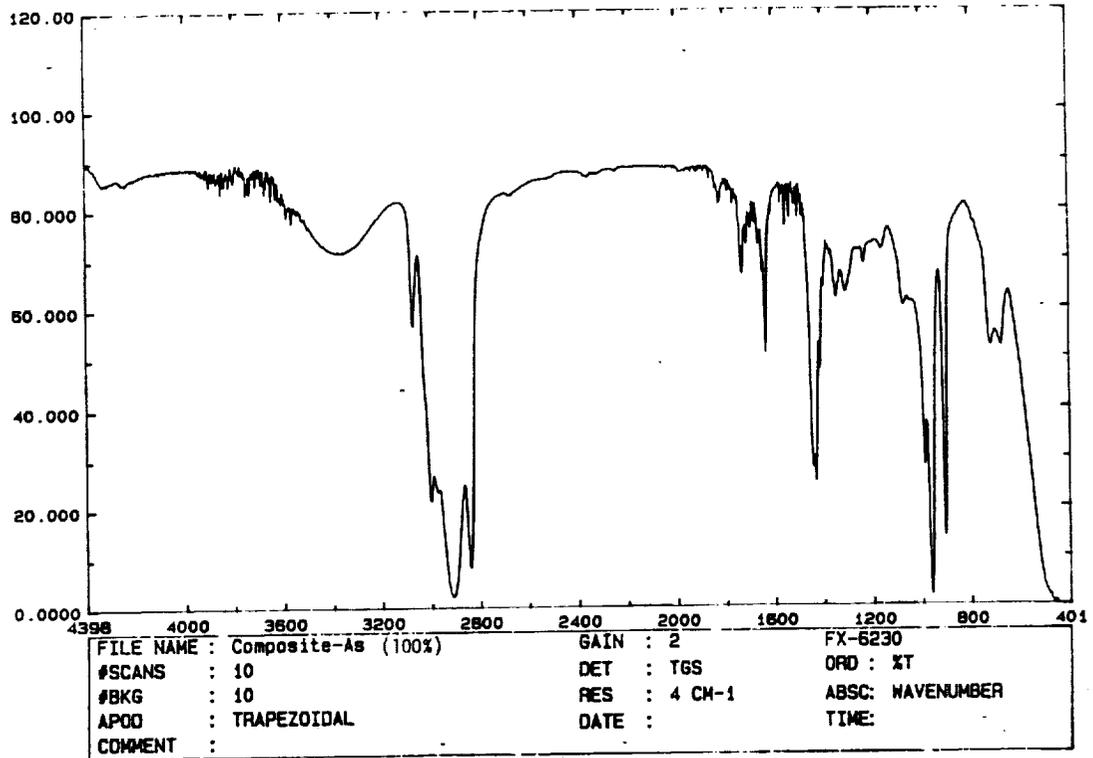


Fig. 29. FTIR spectrum for composite binder.

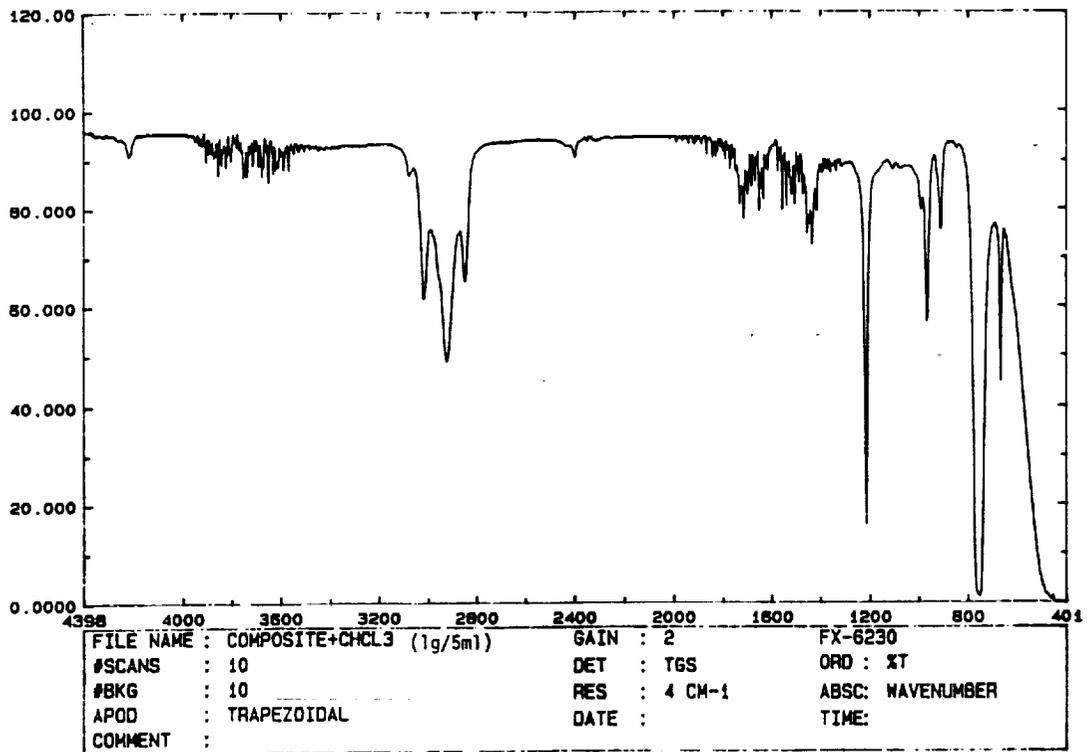


Fig. 30. FTIR spectrum for extracted composite binder.

As an example of a mixing system, consider the "Baker's Transformation." The Baker's transformation is a mapping of the unit square back to the unit square which is performed in two steps (Fig. 31) In step one, the domain is stretched by a factor of two. In step two, regions 2 and 4 are stacked on top of regions 1 and 3.

Figure 32 illustrates that the Baker's transformation creates a mixing system. The unit square is partitioned into 16 cells and the orbit of the shaded region is tracked through four iterations of the transformation. After only four iterations, the shaded region, which was initially contained within one cell, is distributed uniformly in all 16 cells.

Figure 33 is a photograph of a bladeless mixer inspired by the Baker's transformation. The mixing medium is corn syrup, which is circulated in a clockwise direction. The narrowing of the tank, along with viscous effects and the manner of reinserting the fluid into the tank, induce the necessary stretching to create a positive Lyapunov exponent.

After running several experiments using different designs, we found that the most critical design criteria is the avoidance of stagnant regions where the fluid does not circulate through the pump. Such regions are known as invariant subsets. Figure 33 represents a design which produces no invariant subsets.

A portion of the corn syrup has been stained and visually monitored. (Our mixer is made of Plexiglas.) Stretching is observed and it is apparent that the stained region becomes mixed throughout the tank.

The analysis of the mixing process is an application of the branch of mathematics known as ergodic theory and can be carried out on other mixing systems.

We have two mathematical models of the mixer, a discretized and a continuous model. The first step in realizing the discretized model is to partition the domain of the mixer into n cells of equal size and label each cell uniquely with an integer between one and n . Define the number m_{ij} as the proportion of the mixing medium that is transported from cell i to cell j after mixing for a standard unit of time denoted by τ . (We take τ to be the average time it takes a fluid element to circulate clockwise through the mixer.)

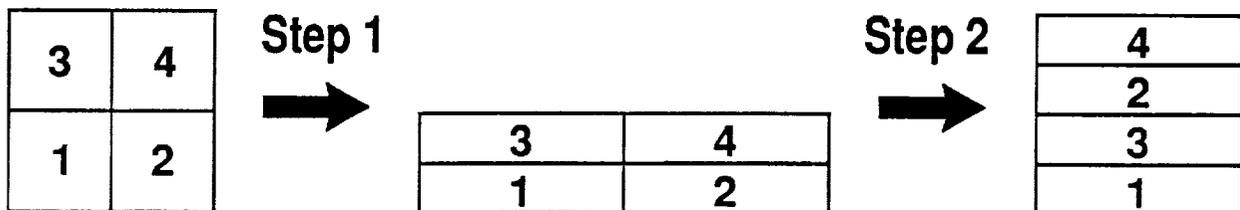


Fig. 31. The Baker's transformation.

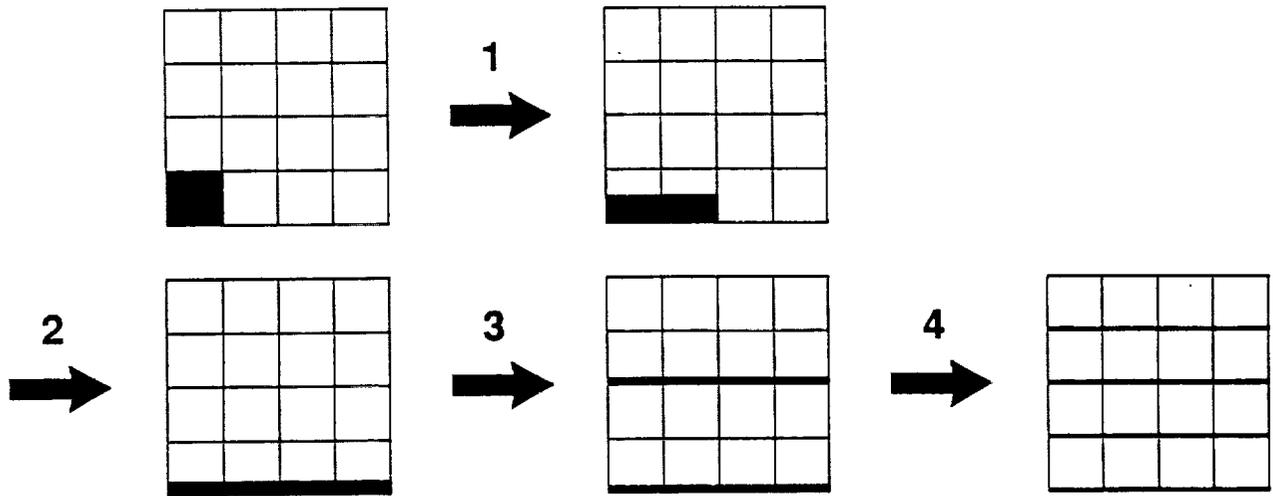


Fig. 32. Mixing system created by the Baker's transformation.

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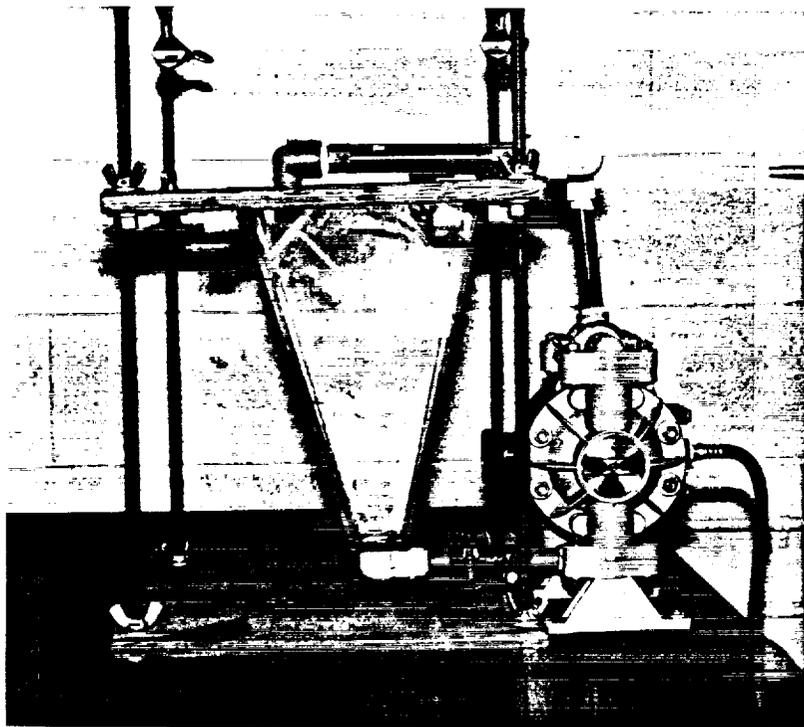


Fig. 33. The mixer.

The set of numbers, m_{ij} , forms a matrix called the transition matrix and is denoted by M . We say that the medium is being mixed if

$$\lim_{k \rightarrow \infty} M^k = [1/n] ,$$

where $[1/n]$ represents the $n \times n$ matrix in which every entry of the matrix is $1/n$. Physically, this condition states that the limiting proportion of mixing medium transferred from cell i to cell j is $1/n$ for any two cells.

A necessary and sufficient condition for mixing is that the magnitudes of all the eigenvalues for the matrix, $P = M - [1/n]$, are less than one. From the transition matrix, one can also address the following questions about the mixing properties of our system:

- i. What is the rate of mixing?
- ii. If we assign an initial density to the medium, what will the density distribution be at different times?

The continuous model is obtained by letting the cell size of the partition approach zero. Then, the transition matrix becomes a linear operator on the square integrable functions over the domain. We call this operator the transition operator. It provides more detailed information about the mixing process than the transition matrix does. To describe the features of this model would require a lot of background that cannot be provided here. Henceforth, we will only consider the discrete model.

We can determine the transition matrix for our mixer experimentally using the concept of ergodicity. If f is any function over the domain of the mixer such that $\int_{\Omega} |f(x)| d^3x < \infty$, then the mixer produces an ergodic system provided that

$$\lim_{k \rightarrow \infty} (1/k) \sum_{j=0}^{k-1} f[\phi_j(z)] = \int_{\Omega} f(x) d^3x ,$$

where Ω is the domain of the mixer. Also, z is considered to be any element of the domain and $\phi^j(z)$ is the position of this element after τ_j units of time. Physically, the system is ergodic provided that the time average coincides with the space average. We have determined that the mixer induces an ergodic system.

The experiment to determine the transition matrix is performed as follows: Place a particle in the mixer and take a measurement of its position at every τ^{th} time interval for many iterations. Let the number λ_i be the total number of times that the particle is located in cell i . Also, let the number γ_{ij} be the total number of times that the particle is transferred from cell i to cell j in succeeding measurements. Using the definition for the value m_{ij} and the definition of ergodicity, one can show that

$$m_{ij} \cong \gamma_{ij} / \lambda_i .$$

Dr. Summerfield, Dr. Hermance, and Dr. Dowler were concerned that the above model treats the medium within the mixer as if it were a fluid and that it does not account for distributions of solid particles such as one finds in fuel propellants. However, the ratio of particle size to domain size is so small that the mixing properties of the actual propellant should be similar to those of a fluid and so the model should remain reasonable. This conjecture could be verified by experimental observation.

Quantitative Computer Representation of the Governing Equations - Mike Hicks

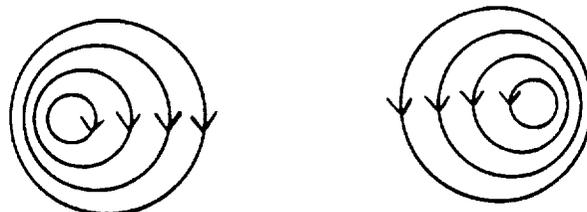
It is possible to consider the mixing process in an abstract sense as the operation of a function, F, which maps a domain back upon itself. Computational fluid dynamics are notoriously complex and time consuming, and this approach allows us to simplify the problem to a great degree and yet still be able to investigate certain fundamental and important aspects of mixing on the whole.

A two-dimensional mixing model has been developed using this approach. The equations were designed by Arthur Mazer (see his section of this report) to satisfy continuity and incompressibility conditions of two-dimensional flow in a unit circle. The differential equations of motion were solved numerically using the Rung-Kutta algorithm to obtain the mapping function. The results of this mapping were then displayed graphically on an IRIS workstation. The computational work was performed by this author.

The equations which were to be integrated are as follows:

$$\begin{aligned} \dot{X} = & [1 + \sin(t)] \left\{ \frac{2Y[4X - 2 + [(2 - X)^2 - 3(1 - X^2 - Y^2)]^{1/2}]}{3[(2 - X)^2 - 3(1 - X^2 - Y^2)]^{1/2}} + 2Y \right\} \\ & + [1 - \sin(t)] \left\{ \frac{2Y[4X + 2 - [(2 + X)^2 - 3(1 - X^2 - Y^2)]^{1/2}]}{3[(2 + X)^2 - 3(1 - X^2 - Y^2)]^{1/2}} + 2Y \right\} \\ \\ \dot{Y} = & [1 + \sin(t)] \left\{ \frac{2}{9} [2X - 2 - [(2 - X)^2 - 3(1 - X^2 - Y^2)]^{1/2}] \right. \\ & \left. \times \left[2 + \frac{4X - 2}{[(2 - X)^2 - 3(1 - X^2 - Y^2)]^{1/2}} \right] \right\} \\ & + [1 - \sin(t)] \left\{ \frac{2}{9} [2X - 2 + [(2 + X)^2 - 3(1 - X^2 - Y^2)]^{1/2}] \right. \\ & \left. \times \left[4 - \frac{4X - 2}{[(2 + X)^2 - 3(1 - X^2 - Y^2)]^{1/2}} \right] \right\} \end{aligned}$$

When we consider the streamlines generated by this velocity function, we see that the flow consists of two superimposed vortices rotating in opposite directions:



The two components of the flow are modulated by sinusoidal forcing functions which are 180 degrees out of phase. It is the periodic forcing functions that give rise to the chaotic behavior of the flow. By examining the graphical output generated (Fig. 34), we see that our model demonstrates mixing behavior very well. The photograph shows the results of 30 successive applications of the mapping function upon 4 sets of points initially very tightly spaced. Each mapping is overlaid in this output to demonstrate the mixing. There is one dead zone located in the lower right corner of the domain. This agrees with set theory, which states that there must be at least one invariant set, or in terms of mixing processes at least one dead zone, in any two-dimensional mapping of a domain upon itself.

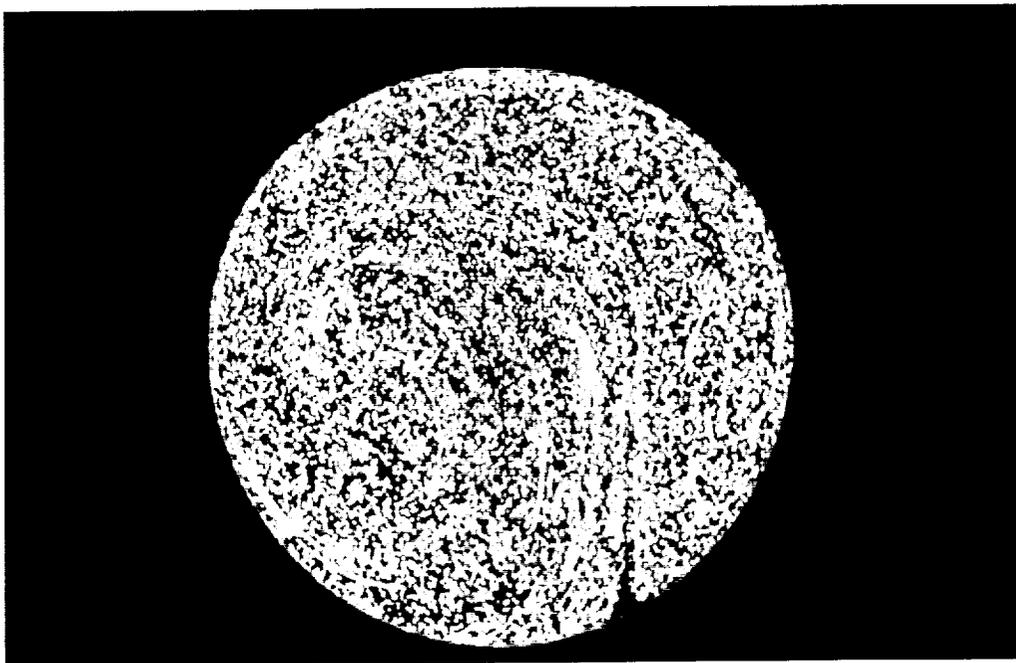


Fig. 34. Two-dimensional model after 30 mixing cycles.

Preliminary Work on Automation of Batch Processing - Paul Schallhorn

Abstract

For space-based propellant production, automation of the process is needed. Currently, all phases of terrestrial production have some form of human interaction. A mixer has been acquired to help perform the tasks of automation. We have designed, built, and installed a heating system to be used with the mixer. Tests performed on the heating system verify design criteria. An IBM PS/2 personal computer has been acquired for future automation work. It is hoped that by the end of the next academic year, the mixing process itself will be automated. This is a concept demonstration task--proving that propellant production CAN be automated reliably.

Introduction

The research work deals with the autonomous production of propellants. Because 80% to 90% of a spacecraft's weight is propellant, it is advantageous to produce propellants in strategic locations en route to, and at, the desired mission destination. This will then reduce the weight of the spacecraft and the cost of each mission. Since one of the primary goals of the space program is safety, a totally automated propellant production system is desirable. This system would thereby remove the constant human intervention currently required in production of many propellants from hostile, high-risk extraterrestrial environments. This enables the exploration of space to be more than the search for, and production of, propellants. As a proof-of-concept demonstration, one specific case was chosen for this study--composite propellant production; the principle is more important than the application.

Background

Currently, composite solid propellant production is done with constant human intervention. Using a control room, man has total control over all aspects of the propellant production. This is fine on Earth, but it is too costly in space. Thus, the need for automated composite propellant production exists.

Approach

We are currently completing testing of a heating system, which was designed by the student (Paul Schallhorn), for the one-pint mixer that is to be used for this project. Because composite propellant production requires mixing the ingredients at two constant temperatures (160 and 140°F), a self-contained water-heating system is required for space-based operation. Such a system is shown in Fig. 35. This system provides the required temperatures and only needs an electric power source to drive the pump motor and heat the

water heaters. This is not unrealistic considering that electricity is also required for the mixer and controlling computer.

One approach, therefore, is to use a personal computer to control the introduction and mixing of the composite propellant ingredients to the mixer (making sure that temperature is constant on the walls of the bowl, detecting local "hot spots" within the mixture, and taking in-situ measurements of the viscosity of the mixture to check if it is within an acceptable range). Then, pump the mixture, via computer programs, into a cast which will be placed in an oven for curing and then stored for future use.

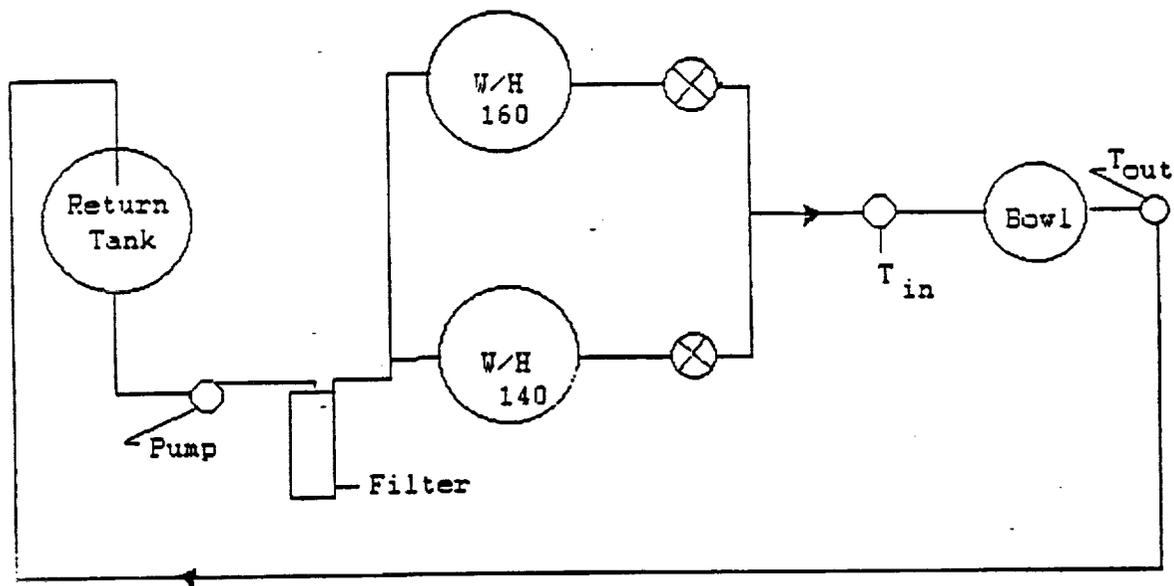


Fig. 35. The heating system.

Results to Date

The major results to date are as follows:

1. A used Baker-Perkins PX-2 mixer was acquired; this introduces a factor of 6 cost reduction (see Fig. 36 for the complete mixer setup). A heating system was required for its operation.
2. In September 1988, Schallhorn designed the heating system to be used for the mixer (see Fig. 35). It was determined that the minimum volumetric flow rate for the heating system for a 1-degree temperature drop across the mixer operating at steady state was 2.5 gallons per minute. Therefore, we selected a pump with a volumetric flow rate of 4.4 gallons per minute to ensure a negligible temperature drop across the mixer bowl. Since only two temperatures are needed, it was logical to have two separate reservoirs, each at

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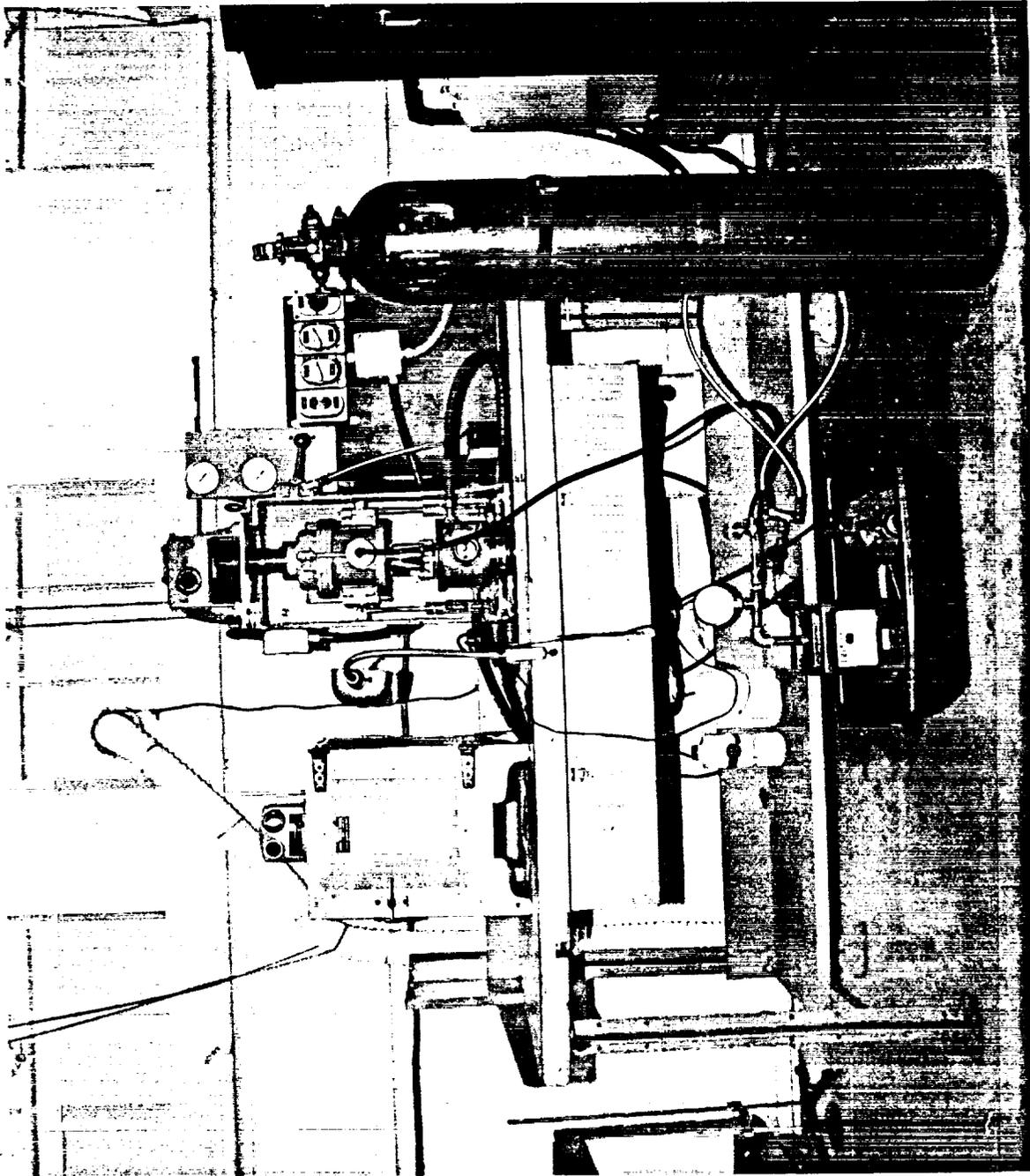


Fig. 36. University of Arizona's solid composite propellant production facility (including mixer, oven, vacuum pump, and heating system).

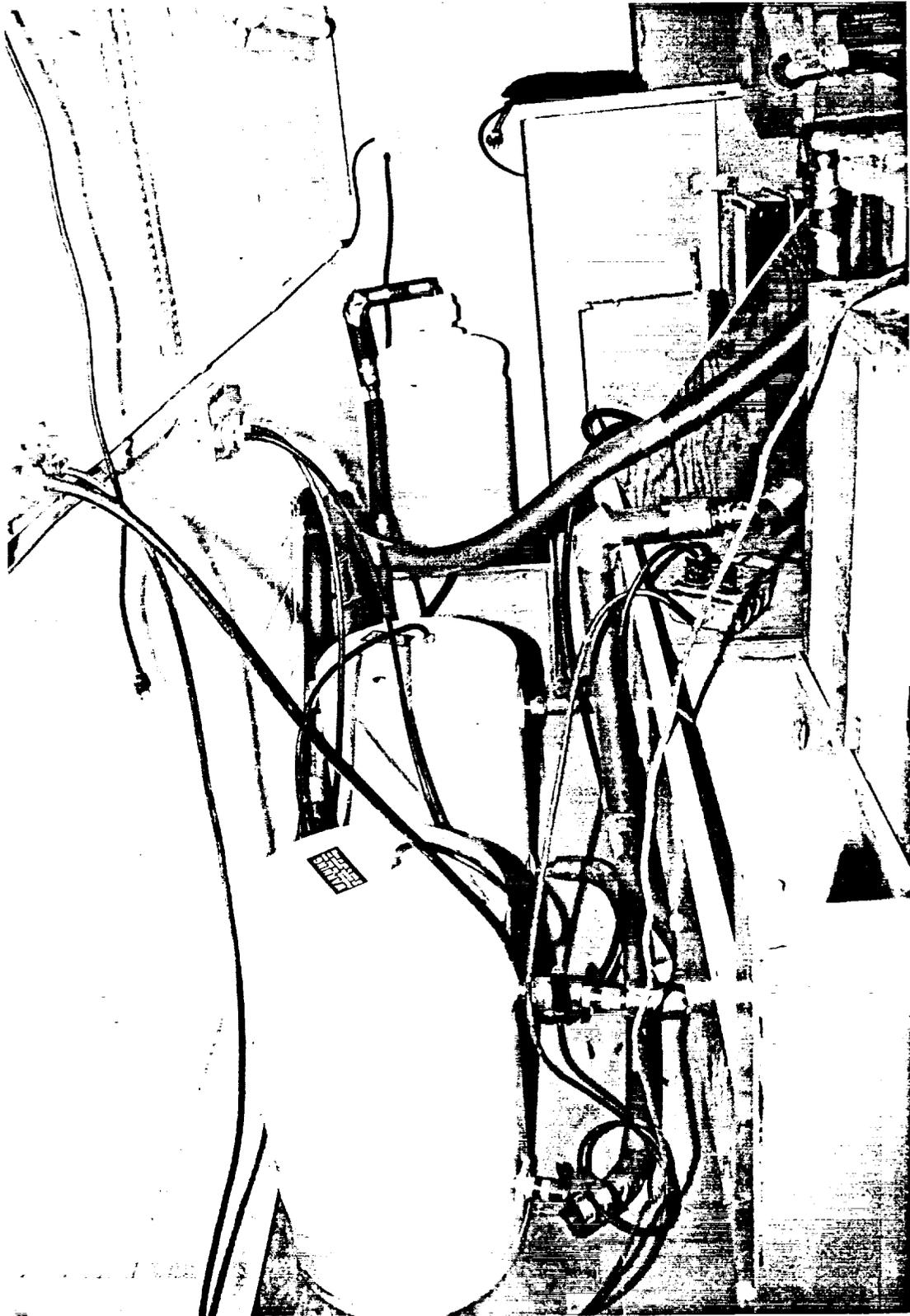
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one of the required temperatures. We chose to have both reservoirs be hot water heaters. Because we only had 120-volt a/c power available, we had to choose the most efficient heater size on the market. As we began to search for heaters for the project, it was discovered that the same heating element was commonly used in different-sized 120-volt water heaters. This made it clear that for maximum water heating, the smaller the water the heater, the more advantageous. That was the basis for the selection of two 10-gallon water heaters (see Fig. 37). The system uses distilled water to eliminate the possibility of scale buildup in the system. To further ensure the cleanliness of the water in the system, a filter is placed in the system immediately following the pump (see Fig. 38).

3. Acquisition of the components of the heating system was begun in October 1988. By the middle of November, all of the components were in and the heating system was assembled.
4. Initial verification of the temperature profile of the heating system was begun in December 1988. Verification of the heating system continued through March 1989, including verification of flow rate and the time required to heat the system from a cold start.
5. In August 1988, research was begun to determine which personal computer to purchase for this project. By the end of September, an IBM PS/2, Model 80 was selected, with an Intel 80386 microprocessor operating at 20 MHz, a 115-megabyte hard disk drive, and 2 megabytes of RAM. The computer was ordered at the end of September, along with the following peripherals: a 14-inch monitor, a 80387 math coprocessor, a modem, a 5.25-inch external diskette drive, additional memory, a mouse, and a Hewlett Packard Laserjet II printer. Due to shipping problems from IBM, the computer did not arrive until late in January, and the peripherals did not arrive until early February. By the middle of February, the computer system was operational. This computer system will be used on various other NASA Center projects, also.

Summary and Future Work

In summary, this task has shown that there is a need for automated production of propellants for space-based propellant production. We have also seen that there is no current system to produce composite propellants without human intervention. A mixer has been acquired to help perform this task. We have designed and built a heating system to be used in conjunction with the mixer to maintain constant mixing temperature. The heating



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Fig. 37. Side view of the heating system.

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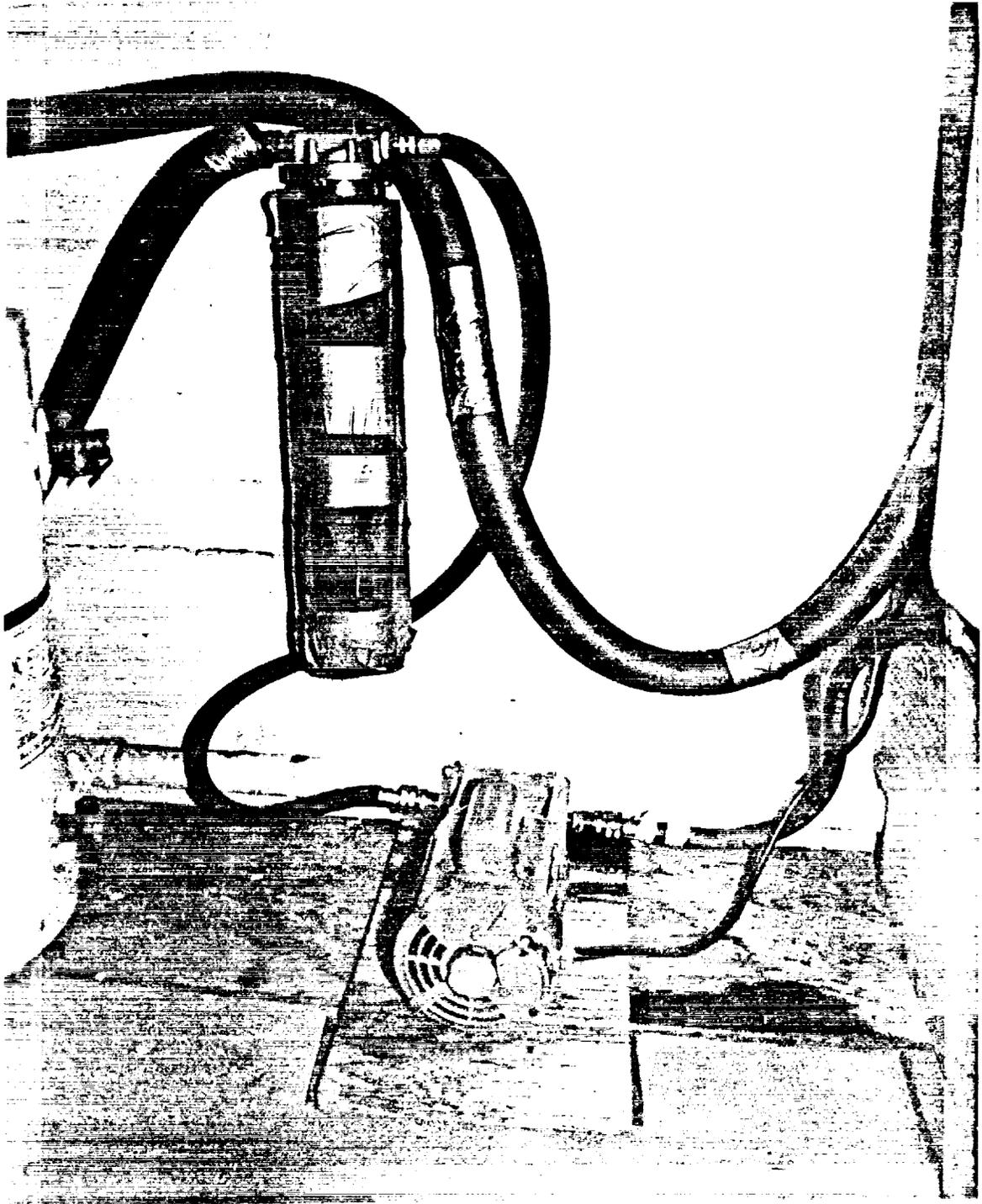


Fig. 38. Heating system pump and filter.

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system has been, and is continuing to be, tested under operational conditions for design verification. We have acquired an IBM PS/2 personal computer for the computer portion of the automation.

For the 1989-90 academic year, the student plans to begin his Ph.D. research, which will consist of the actual automated propellant production. During the year, we will begin to automate the mixing process itself. It is hoped to have the computer control the addition of each ingredient from a "hopper" (yet to be built) to the mixer at required times and have the computer control the mixing of the ingredients for the required amount of time. We also plan on building and installing the in-situ viscosity measuring device for future integration into the automation system.

DISCUSSION AND COMBUSTION

Discussion

When discussing malfunctions, or less-than-desired performance, we would like to learn the way these are approached in similar programs. Fortunately, Code Q established a program to specifically explore failures, and to recommend improvements, in the closely related field of pyrotechnics. Larry Bement²³ has conducted a detailed study, for Norm Schulze, surveying the recent failures and substandard performance in NASA, DoD, and the Space Division. It is instructive to recall here the classification used to characterize the failures and anomalies. This is shown in Table 3. Extensive data accumulation is also systematically tabulated and catalogued. A typical example is shown in Table 4. Similar surveys of composite solid propellant rockets will be most valuable.

The rest of the discussion is best stated concisely in the form of the principal findings and recommendations, with one exception. It was felt by all that the end use, combustion, is poorly understood and that this must be rectified. Thus, the next section discusses combustion.

Combustion

An inescapable feature of solid propellants is that their end use will be through combustion. Thus, all of our efforts at understanding the ingredients, specifications, mixing schedules, processing, casting, and cure will be of little help in accurate predictions of performance unless the final combustion can be predicted accurately, too. Here again, many models are available and some have indeed proved useful in formulating good propellants with desired characteristics. Nevertheless, these combustion models are approximate at best, and none can claim to predict as simple a parameter as the time-independent burn rate purely from a specification of the ingredients. Some of these deficiencies, which may be adequately concealed in time-independent burning, are revealed when the propellant combustion becomes time-dependent, or unstable. For an adequate understanding of the solid propellant predictability and quality assurance, we must develop a better understanding of combustion.

Combustion of a composite propellant is inherently a heterogeneous, time-dependent process that involves key interactions among the condensed and vapor phases and physical and chemical processes, all within a time scale of milliseconds and within a spatial region of a few hundred microns. The conversion of the "room temperature" solid into vapors and gases that frequently exceed 5,000°F in temperature must be understood, at least to the extent of predicting the overall rates from the fundamental constituent rates. Hopefully, some of the constituent rates, such as the depolymerization rate of the binder, the

Table 3. Definitions for survey on pyrotechnic problems and failures.²³

1. Manufacturer's Poor Quality Control--All quality controls in place, but not followed.
2. Manufacturer's Bad Procedures--The quality control procedures or manufacturing methods are inadequate and in need of improvement.
3. Manufacturer's Decision--Unilateral decision by manufacturer affecting customer.
4. Bad Design--Technology exists, but not followed.
5. Lack of Understanding--Technology did not exist at the time of problem.

Table 4. Typical example of data collected--firing circuits.²³

| Date | Project | Problem | Impact | Source of Problem | Resolution |
|------|---------------------|--|---|-----------------------|---|
| 1983 | Aircraft Crash Test | Aircraft crash test program discovered stray voltage in facility firing circuits in flight checkout (poor grounding and corrosion of cables/connectors) | Delay of experiment and potential loss of life and loss of experiment | Bad test procedures | Rebuilt checkout/firing cables and consoles |
| 1982 | Shuttle | SRB decelerator parachute released prematurely in flight at frustum separation (pyrotechnic shock activation of water impact sensor) | Loss of spent SRBs | Lack of understanding | Redesigned, requalified |
| 1972 | Centaur | Shroud separation joint fired primary and secondary charges simultaneously in qualification and ruptured containment (connectors on firing circuits swapped) | Delay of system qualification, damaged test hardware, potential damage to spacecraft and flight vehicle | Bad design | Redesigned, different connectors |

decomposition rate of the oxidizer; and the melting rate of the metal, can be accurately determined through combinations of modern experiments and data analyses. The popular Arrhenius kinetics may be adequate to describe these, but the variations of the activation energy and pre-exponential factor as influenced by temperature, species, and pressure in the presence of intense radiation may need further study. The physical constants, such as the thermal conductivity coefficient, specific heat, and absorptivity, are usually averaged over all of the ingredients, and this procedure must also be examined. Many of the details of composite propellant combustion were reviewed and the more important theories presented in ref. 24, which describes time-independent (steady-state) burning. The time-dependent combustion aspects form the subject of a book (in press) in which the suppression techniques are scientifically described.²⁵ These books cover only those aspects of combustion that are known; more work is needed on the unknowns.

While the natural heterogeneity of composite propellants was adequately described, most combustion models used a "suitably averaged" homogeneous material when it came down to actual mathematical analyses. Clarke Hermance was the first to introduce heterogeneity explicitly in the analysis. The success of his model started a series of variations by other researchers. We now need another such step forward to improve the accuracy of our understanding and predictions. Many modern sensors, diagnostic tools, and microprocessors should all be constructively used in conjunction with powerful computational capabilities to evolve better combustion models. Such models should specifically address the importance of the following:

1. Condensed phase reactions, including those of the ingredients, between ingredients, and among the products of initial reactions (here, reactions include depolymerizations also).
2. Surface reactions, including the very definition of the "surface" itself.
3. Near-surface vapor phase reactions, including those within one fine (particle) diameter distance from the surface. What is the influence on heat transfer to the condensed phase from such close zones?
4. Main-flame reactions, including the proper definition of the flame, or the vigorous combustion zone.
5. Post-flame reactions, relaxation reactions, condensation reactions, and their importance to the overall burn rate.
6. Possible control of some of the "nature-prescribed" reactions through the powerful influence of free radicals and free radical donors.
7. Unambiguous verifications, independent checks, and repeatable tests; ability to predict small variations as influenced by ingredient or processing variations.

8. Realistic combustion experiments that reproduce the essence of solid composite propellant combustion without actually using solid propellants (the perforated porous plate burner provides one example).

PRINCIPAL FINDINGS

1. Unplanned variations in solid propellants have been quite prevalent.
2. With so many ingredients, each characterized by so many physical and chemical properties, quality control of the end product is subject to several uncertainties at the present time.
3. Parameters during processing (for example, the temperatures and mixing times) have varied around the desired values by magnitudes whose significance is not yet fully understood.
4. There does not appear to be a single case of a propellant that was scientifically studied, formulated, processed in various scales of mixers, cured, and tested in various sizes of rocket motors—all under conditions where nothing was changed in the formulation. We cannot fault the production specialists, because changes in the formulation of scaled-up batches are made on the basis of documented experience common to the industry.
5. The enormous "data base" in solid propellants is really unusable for a scientific study.
6. Most solid propellant rocket motors have been evolved based on empirical corrective procedures during development.
7. Bonded interfaces can be trouble spots.
8. The important **end use** invariably involves combustion; the current combustion models are too naive.
9. Even in academia, traditionally recognized for fundamental research away from the pressures of developmental programs, there are practically no universities in the nation capable of experimental pursuit of propellant formulation and rocket motor tests, even on small scales.
10. Extremely useful and revealing data may have been, and are continuing to be, lost when "unsatisfactory" propellant batches are simply discarded.
11. Unfortunately, many of the procedures followed in solid propellant formulation, processing, and production suffer from the legacy of black art; even the mixers we use are really borrowed from the bakers.
12. Eliminating solid propellant rockets in favor of liquid propellant rockets is hardly the solution, since there are even more serious problems with liquid rockets.

PRINCIPAL RECOMMENDATIONS

All of the deliberations and the consensus of the authorities (on solid propellants) present at this meeting are available in the transcript (Appendix A) and the body of this report. Here, the main recommendations are listed in the interest of concisely stating what is needed for increasing the quality and reliability of solid rocket motors. It is understood that long-term quality and reliability can only be ensured through better predictability which, in turn, can only be the result of a thorough understanding of the key parameters; it is important to note that thoroughly understanding the key parameters is distinctly different from an attempt to thoroughly understand all of the fundamental physical and chemical processes relevant to solid propellants.

Such an ambitious goal--to understand all of the fundamentals--would probably be instructive but would be prohibitively costly, besides detracting from intelligent and economical approaches that can identify and clarify the key parameters that directly affect the end-use performance. The recommendations of this working group are:

1. Establish at least one end-to-end facility where propellants can be formulated, processed, cast, cured, and tested in different size motors--all under strict control. [KR notes here that the only such facility in the U.S. still with an independent university is JPL; however, all of the experimental propellant processing capability has been moved from Pasadena to Edwards. In any case, support of this facility has been very meager in the last 15 years.]
2. Seek and establish a data bank from industry; this should include not only the mainstream successful programs, but also all of the seemingly secondary details that include failures, too.
3. Scrutinize the data bank for meaningful trends.
4. Since the end use will always involve vigorous combustion, establish a good combustion program in composite solid propellants. [KR notes that the establishment of a small number of highly focused, competitive, and selective grants in combustion will be much more productive than the establishment of a large program.]
5. Carefully study bonded interfaces. [KR notes that MSFC has recently started (with SAIC as the prime contractor) the SPIP Bondline program. More is needed to specifically study the propellant composition.]
6. Evolve the fundamental mathematical models for mixing and flow of heterogeneous mixtures, including chemical (curing) reactions.
7. Establish the bounds of physical and chemical variations of interest in practical composite propellants and exceed these bounds in the laboratory. These out-of-

bounds behaviors can be of immense value in understanding some of the unplanned variability in practice.

8. Utilize all of the latest high-technology developments in micro devices (sensors, processors, and chemical activators) to scientifically gather more information on composite propellants to help modeling.
9. Formulate one simple, model composite propellant and thoroughly study it at various independent facilities, including industry, universities, NASA, DoD, and other government laboratories. The results of such a study can be very valuable in understanding the bases of some of the baffling variations.
10. The last recommendation is very profound. All of the participants noted a general decline in the number of students and faculty actively working in solid propellants. To obtain and maintain a reasonable working knowledge of composites, it takes competence in several disciplines, dedication, and careful attention to details--all spread over at least twenty propellant families; and a careful, first-hand study is needed over the entire propellant program life, from uncured strand burn rates to full-scale motor firings. cursory supervision in a bystander role will simply not suffice; neither will any amount of theoretical work on model (ideal) systems. We must have more involvement by competent researchers, who should spend time actually working with the processing and end use (combustion).

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EDIT TO
END

APPENDIX A
MEETING PREPARATION

After initial telephone contacts that confirmed the interest of those contacted, a letter of invitation (see below) was sent to

Floyd Anderson, JPL
Julian Barry, JPL
Barry Butler, SAIC
Warren Dowler, JPL
David Flanigan, Morton-Thiokol
Robert Geisler, AFAL
Clarke Hermance, U of Vermont
Marshall Humphrey, JPL
Charles Martin, NASA/MSFC
Edward Price, Georgia Inst. of Tech.
Russel Read, NWC
Ben Shackelford, NASA/MSFC
Martin Summerfield, PCRL

In addition, several others from industry, government laboratories, and academia were contacted. These included Al Gent, Woody Waesche, Jim Hester, and others. For various reasons, mostly related to time constraints, they could not attend.

Text of Letter

March 28, 1989

Subject: Solid (Composite) Propellant Predictability Quality Assurance

First of all, I would like to thank you for your interest in this program. As I explained to you, there is a growing awareness that it is very important to be able to predict the performance (burn rate, susceptibility to instability, aging, mechanical properties, . . .) of solid rocket propellants. Ideally, one would like to feel certain of the quality and reliability of a propellant once the ingredients and processing are specified. In reality, a number of factors based on experience, educated guesses, and some analyses play key roles in quality assurance.

I have enclosed some material that may help you get a better feel for this problem.

If you need help with the non-technical details, please call Ms. Josie Tanner at (602) 322-2304. Of course, please feel free to call me also.

I look forward to seeing you here on Friday, the 21st of April.

Thank you,

Kumar Ramohalli, PI

APPENDIX B
TRANSCRIBED VERSION OF THE MEETING

SOLID (COMPOSITE) PROPELLANT PERFORMANCE PREDICTABILITY
and
QUALITY ASSURANCE

21 April, 1989
TUCSON, ARIZONA

WELCOME: 9:05 A.M.

DEAN SMERDON - Dean Smerdon, Engineering and Mines
Discussed the latest developments in the Engineering Dept, and also
in government (Mecham's decision to run for public office again).
The largest single building project on campus will be the
new Aerospace and Mechanical Engineering Building, 11 million from
the government was allocated for the projected 24.8 million
project. The benefits from this will be long range and positive.
Electrical and Computer Engineering is expanding its programs.
We are glad to have the NASA Center aboard, with Terry as Director.
The Flynn Scholarship program was developed to help students attend
college - at present there are 60 students involved with the
scholarship project - 50 of them at the U/A, and more than 25% are
Engineering majors.

Dr. TRIFFET - Director - UA/NASA Space Engineering Research Center
for Utilization of Local Planetary Resources.
The concept of the Center was to research and develop production
of propellants from extraterrestrial resources so we can refuel in
outer space. The key is to refuel there - practically everything
that has ever been thought of has been researched on this topic -
there are a lot of possibilities and we hope our research can be
responsible for the final answers. This is the only center
established for utilization of local planetary resources.

Dr. RAMOHALLI - P.I. and Professor
The purpose of this meeting is to evolve a general consensus on the
state-of-the-art in solid (composite) propellant predictability,
reliability and quality assurance; it is also the aim to generate
a few implementable, realistic and useful recommendations for use
by NASA. We must identify the KEY parameters and understand how
they influence quality of the end product.

BOB GEISLER: The key thing to be concerned with is there is only
one AP supplier - therefore you must requalify some.

Dr. SUMMERFIELD: About 17 years ago we tried to get the burn rates
- small 2% quantity - must search for that needle in a haystack -
nail down the size. We sent it out to different people and got back
all different numbers. It depends on the grinding, equipment,
processing, etc. They all reduce particles - all measured, by
micro-merograph.

Better to use equipment and get a number, but spherical particles -

is there a reason for discerning - everything we think of relates to the 2%.

Anomalies are problems - pressure - we need a better understanding. Are different size motors grinding particles to the same shape, etc.?

BOB - Many of our grains were made through the same size molds. Motor casting is dependent on viscosity.

SUMMERFIELD: - Strand burners and multiburners - is difficult to get better than 1-2%. JANNAF standard is 1.5%, usually have 3 kinds, if they fall beyond 1.5% take a look at new strands. Research done by us concerns the burning rate and flame height.

BOB: Identify today the mechanisms to get the numbers. We are at a stage now to do better than before.

SUMMERFIELD; How can you control the 2%? Is it a regulated 2% - a hump effect, reusable. There are systematic effects or random effects.

Look for many burn rates - wanting to localize it - see if the cast is laid down for burnable rate - systematic effects.

PRICE: Recognized reproducible - it's not understood - but we do understand the causes, particle segregation and flow casting.

HUSBAND: Cannot yet control it - so many different flows to consider. Need to requalify other old AP - investigating AP's.

SUMMERFIELD: Hercules work: proper particles orientation by strand cuts was verified in Miller's paper.

HUSBAND: Particle segregation is necessary to see changes in burn rates.

KUMAR: Are we chasing random variations, or more ordered variations? - random ones are complex. High density seems to find significant variations in burn rates - take into consideration the separation.

SUMMERFIELD: Random variations in motors are what? Have you analyzed the Strand burner notes? (i.e., details?)

KUMAR - It's never better than 4-8%. Different in labs than in factory process. Study with burn rates in same batch; CIT-2 was the best - very controlled burn rate.

BOB- Sometimes look at the histogram - it is wise to do that.

PRICE - We abandoned use of this type because it doesn't show the bumps.

BOB - Taking 50% particle size is not a good idea.

KUMAR - Unorthodox approach is necessary.
No substitute for conservation equations.

SUMMERFIELD - If you decide to - make certain the mixture measures out on a scale at .802 - transfer of material into mixing chamber - some of it hangs up - we're looking for only 1% - should reweigh it and see what happens. Could be, mixture ratio of liquid hangs to the sides - you have to sample it. Can't be sure it's not same as solid group.

BOB - Look at motors, solves some problems but can create others.

KUMAR - Looking for data on motors.

BOB - If you have it computerized it may not work out right.

SUMMERFIELD; BATES Motor is a better test of the burn rate. Gives radiation affects you don't get in the Strand - but Strand is affected by radiation too.

The difference between the small and large BATES Motor is how density gets "saluated" and whether its smokey.
BATES motor data are hard to find.

Memoirs have way of separating motors and strands - some propellants are better than others. What we use is ammonium perchlorate.

JPL is where we get the experience.

MARSHALL: Use cured strand - by variation.

KUMAR: Depends on propellants.

Ultimate Objective - Predictable Solid Rockets
Minimum surprises
Economy in qualification tests.

Short Term - Identify critical parameters.
Establish specification
Develop quantitative criteria.

Our Conference Today - Learn from Experts
Evolve consensus
Document Recommendations

DANIEL PEREZ: Problems - Data well/predictability
3% variations
two basic predictions to analyze

One company - goes on and on analyzing a series of batch runs - saw probable causes - motor burn rate - whether they noticed the temperature change or not was noted.

SUMMERFIELD: Assume they compared same ambient temperature -

relation between rocket motor and ambient temperature - duration of thrust divided by burn rate expected. How did they measure this? Kumar answered saying the condition had small variations. The burn rate separates at 7 - 8%.

PRICE - would like to see burn rate plotted for a month of the year.

KUMAR - Let's let Dan give his presentation.

DAN: It is a lengthy task of finding what it is - mixing process - lots of factors - immense problems - which one can we control? Many have spent as much as 20 years looking, but we want to know what will give the best answers right now.....Can be a problem with gases - segregation, which ones will contribute most? Have a volume of mixers - will get the differences from these.

DOWLER: Scaled propellant mixers are supposed to produce the same product as the batch size volume changes; however, they are not devoid from such considerations. Baker-Perkins specifications are not adequate to help Dan. Going from 1.5 pint batch size to several gallon batch size with the same or modified mix procedures will not produce same product uniformity and variability.

BOB: People are going to JH Day for mixers.

DAN: Burn rate -vs- pressure - 1 gal. Have to reanalyze these problems. We did a shot gun approach to see where it may be deviating. If it is different we have something to consider. Did two different batches - only volume was different. Data finding was correlating with particle/non particles. If volume changes it proved some things.

BOB: Could be mix viscosity.

KUMAR: We are working on it.

McKAY: In comparing burning rate, if made with identical batches - procedures were considered different depending on RPM's of blade.

SUMMERFIELD: Facing fact we have problem testing - difference could be between different things.

BOB: Jim Koury - if cast propellants out of box show difference on blades, it depends on where the strands were, etc.

PETERSEN: With 1 batch it looked like good data.

KUMAR: The main point - if specifying ingredients and mix and cure you should get the same result but there are so many variations.

PRICE: Looking for quality control or systematic system. This material and problem were not just propulsion and propellant itself

- looked at research - had different systems/ 25 by particle\ coarse and fine particles. Dr. Chung did it - found viscosity varied greatly - should be a correlation. There must be a ratio of how much fine particle you have to coarse particles. Must go to the lab and look at it - detect the changes in viscosity - equations are many on the subject as to how the concentrate will change. When looking at monitoring this device -see rotational viscometer. What type of testing do we have...

PETERSEN: Is Brookfield being used?

BOB: Have to pick right shear range/slip plane, mixer, so forth.

PIB used. No curing - thermo set.

DAN: Problems - particle shattering - nonuniformity - are they coarse or fine? Chemical uniformity.

PETERSEN: Big unpredictability

HERMANCE: Are results due to non-linear viscosity, shear rate, yield criteria.
If shear stress - radial is large size - larger than particle - we will have sheared particles.

BOB: Used lots for shear rates.
If part of the polymer family it makes a difference.

DAN: Particle looking for particle shattering - is its nature. Found its abrasion to be sole contributor. Got a few little specks.

BOB: British are making a propellant they could melt and use again; high (90%) solids - only coarse ammonium perchloride - let mixing process do it all. They receive the burning rate - they get shattering and alternately achieve rational distribution of particles. Using AP.

HERMANCE - Maybe it's the ammonium perchloride - not what they use -I think they beat it to death. Brings up the relationship between particles.

DAN: Further work - got separation better 300/589 and anything below it - used screen for the pictures of it. If had a 3% burn rate deviation - got a shift of 16% - or 10% - separation was 3%.

BOB: Never extracted HTPB, did you?

BOB: Found that HTPB below .10 can't extract - use 6 micron aluminum -went 3 months on harsh extractions - on SEM looked great - Just 6 micron up wouldn't break it.

KUMAR: Last summer Dan spent time talking to chemists exploring how to separate 8-10 limit.

Maybe we should try their solvents.

DAN: Uniformity factor - sulfate and HTPB - What happens if you mix fine and coarse together (dry blending) and then tried just coarse.

Premix with 3.1% - separate much higher (just fine over solvent).

BOB: Clusters only will be formed by anvil like action. Sometimes you can add solids to lubricate problems in mixer.

DAN: Looked at data - burn rate is motor size - one day versus storage process (batch 150 gal). Even for stored term it made a big difference in burner. Found chemical nonuniformity.

Did Experimental techniques - FTIR 5 Analyses - GPC polymer growth. Did some successful separation techniques of ammonium sulfate, ended up with a better overall picture.

Flow cavity 1 mil in thickness for quantitative result.

Need at least way to predict which way they are going. Found:

computational model - finite - difference program, base FLUENT.

monodispersion model

synthetic (bispersional model)

HUSBAND: Don't waste time - too far behind on theoretical.

Argonne is trying to do this with all their work force plus computers - interested in flows in nuclear reactors/gas phase.

HERMANCE: Not sure some can duplicate what fluid can do - national labs aren't the only ones who can come up with the answers.

HUSBAND: We are too far behind in that technology. Need some experience now to prove theories.

KUMAR - 2 arguments on that - one for and one against. I feel if you can do something in one specific area you can use it as a tool after we understand it.

Data gained in 1938 showed if velocity varies, flame height remains constant - mixing with 2 species - 2 cancel out...when this was all put together they tried to modify it and did not put it on the computer. They found they need observations of a model world.

HERMANCE - lets get some data in fluid analyses .

HUSBAND: Finite elements need parameters - now parameters are not significant.

HERMANCE: Computational help is an asset too.

DAN: We hope to model a flow; of a model disperse system - study things in mixing - shear rate in particles, make a mixing condition.

KUMAR: We were lucky to get Professor Vincent and student to do basic research of mixing - Mr. Mike Hicks also will show the basics of mixing.

ART- will give highlights from applied math department. Concentrate the means to get mixing analysis. Need process - mapping (stirring) revolution, needed to create mixture and how to get it uniformly distributed.

SUMMERFIELD; Have you any background on bakers/dough. Have you rejected automation against mass production as to automated production? Some are interested in continuous mixing - Batch mixing is only for the batch - must have safety measures.

ART: This system bakes it and stacks it/ cutting and stacking - replaces the folding like with cookie dough. Stretch it evenly throughout the shell - any set uniformly distributed. Envariance set - set the force of fluid through it - particles will start stretching out -

HUMPHREY: Close to fluid energy mill is another theorem - can tell by tracking what happens to one set of domain. Want to get a more mathematical definition of why its doing what.

HERMANCE: Wondering about conservation of mass -

ART: All seems equal.

Then Art gave his talk - was difficult to follow;;; If MIS is not equal to 0, you get mixing - can get how fast you're mixing from this process. Ergodicity - evaluate intergrals in domain by tracking only one point in the domain.

SUMMERFIELD: How do you get homogeneity - ultimate mixture is particle mixture - how to modify - how can you have uniform mixing?

HERMANCE: Added conditions which can go into any cell size have grind of mix - like molecules - smallest relevant in diameter.

SUMMERFIELD: You cannot incorporate this into variables. Inside cell - is an infinite number of cell functions - akin to balstran distribution.

HERMANCE: We can only make that assumption on microscopic scales - infinite number are still in homogeonus.

DOWLER: Making dough is a better analogy to propellant mixing -- particle dispersion is what we need to model -- it would be interesting to take a whole pot down to pencil tip size and see if all the mix volume have fractal dimensions.

KUMAR - Have to impose physical limits of size - if mapping is fine, etc.

SUMMERFIELD: Liked the funnel approach.

Mike Hicks gave his video display (integrating equations) from

Art's presentation on camera. A video of mixing at random - can see the actual dead zone. Mike is using Fortran - simple equations for solving - takes hours to go through one cycle.

KUMAR: Recent program at NASA on pyro techniques and pyro tech area. (showed slide)

Identified: (Larry Bement)

1. Manufacturers poor quality control.
2. Manufacturers bad procedure
3. Manufacturers decision
4. Bad design
5. Lack of understanding.

PRICE: They called some bad shots.

BOB: No single document could say it all.

Lots of resources are available - data - open literature - internal documents - restricted access banks, knowledge/engineering scattered - NASA DOD Industry, related fields - (chemical, industry and food industries), routine scientific meetings, AIAA, JANNAF.

Recommendations:

1. Verbal is best and economical approach. OBTA - best overall should specify quantity - ingredients, preprocess, process, cure.
2. Overall most economical ability
Quality reliability - safety, flexibility, adaptability, scale.

KUMAR:

We need some constructive things happening - NASA is now taking it seriously, JPL is working toward it.

SUMMERFIELD: Overall comment is: take this program seriously - and timely to get a facility and do your own work. You don't need to go to other people for their results - doing your own work gives you skills, and enhance to grad students. Safety inhibitant to the fact - but all through my years, (15 of research) never had an accident, persuaded sponsors so I could get my own data. You need own rocket motors/burners/hands-on-experience with burning rates, etc. Get problems taken care of or explore possibilities. Recommend experimental lab be established - get your own data - funds are available and the university has the facilities. May be more difficult to take particle size as a given - what if it is measured 3 times and you get 3 results - it becomes more apparent as you get more hands on experience.

KUMAR: What is most important?

SUMMERFIELD: Put the emphasis on understanding combustion process - different by large scale/small scale, etc. If velocity is right, these are questions that provide the basis for experiments.

BOB: If the Air Force gave a billion dollars to do anything you wish they would require a proposal to establish the reliability.

The Air Force looked at the question for the launch system - the cost of reliability was too costly - took all the shuttle experience, 90% success - as they had to start someplace. Solid rocket commission say they do 99.2 but we have always said our #1 place of failure in the solid rocket is interfaces.

Some of the patches are together. Classic failures are seldom due to poor propellant - only bonded interface is the problem. Despite what we know it's generally not the cause of failure.

Conclusion: Our design margin is smaller than they ever have been. You wouldn't believe the margin of safety years ago compared to now. As the design margin becomes larger there is more understanding.

Bonded interface failure is due to manufacturing.

The Air Force focused on O rings or pressure seals - The problems in the technical end are very serious. Quality of workmanship is a problem and the lack of rigorous service control.

Solid rockets are handmade objects of art. If you had to spend a dollar on solid rockets it should be on bonded interface.

When there is a problem on DOD - back out.

People in the Air Force are thinking about what to do before failure starts - fiberoptics, sensors, etc. What to do when you get a reading - take some action.

Final Item: Good old inspection and failure criteria to make x-ray. Still a fertile field. The way it's done to this day is a group of colonels get together and vote. That's the way the system works.

SUMMERFIELD: Can dry mix be implemented?

HUSBAND: Usually it's a turn around - liners are necessary for a good bond - need perfect thickness for paint.

BARRY: On an air force job they loaded sidewinder for GAP propellant - what happened, why is one good, or bad...why did it set up in a pot? In a mixing bowl the blades go around - they had a 5 gallon mixer...but imperfect mixing - there was no adhesion anymore.

R. MCKAY: The assumption is all is moving - can be important to do a Sherlock Homes, but could create more indepth way. Using other people's data is risky - can't depend on other people. In some of our work years ago we checked 1 pt. mixer and looked at it critically and noticed zones around the wall not as close set as others - we specified special slurry -(in 60's) and in-house talked about heat transfer and penalties. Turns out we have in-house 130/150 gallon if we can squeeze out the money. Some work was arrested because we can't make changes in the mixer. Perhaps used parameters in their designing - embarked on 2nd generation to incorporate certain variables we didn't have in the past. I suggest in lieu of possibility of doing all the work in-house - that there should be a request for critiquing other data - some things are just not obvious.

BARRY: 2 new viscometers have helped - work on the testing and burn

rate method (thanks to Kumar's work).

KUMAR: J. Barry - we would like to know in your experience did you find any correlation from looking at propellant that didn't look/feel right?

BARRY: We took a slurry test a month ago - it didn't turn out alright even though it looked ok.

MARK HUSBAND: If you look deeper there are explanations - only a certain amount of homogeneity. You have to know the facts and control them. Need to know you can't get the same mix from different size mixers. I compliment your enthusiasm. A lot of work is already out there in this field directly related to this (reference 26). JANNAF Rheology Panel - is active and meets every January - they looked at particle separation real logical - are finding now which parameters are important. There are new methods, new rheometers. Still are mixing problems - but major problems are after the mixing. Virtually in all cases what you get after the mix cycle is all you're going to get. Look at the advanced technologies - its a continuous process. Look at new technology up front - to go forward you need to do this. Your treatment of data needs better understanding. Motorburn rates and strand rates need more rigorous attention.

KUMAR: Of over 4000 data points, used on large and small motors, large and small batches, we were left with 30/31 data points. Some data was shown for history, making progress. Particles are different sizes. Recent data was very scrutinized. One reason I called Bob, is because I need to know what is going on. We found that mixing is the first thing we need to begin studying. JPL has big reports (we subtopic in order to understand if it is amenable to scientific analysis.)

KUMAR showed highlights of his March 3rd meeting. Stated there are smart materials available. What can we do with them? How do you initiate this - put 1 or 2% in these materials...can have control, can have FR donors - will control chemical reactions. Incorporating to special degree - then have active real end. Want to do low level, concept demonstrations - select one important system for study.

MARSHALL: We're taking an archaic method - instead of using something specialized - never really operated - also his ability had a lot of innovation to this phase - is a static mixture used in the chemical industry today. Used loss of weight feeders - only as accurate as the material you put into them. This was a back stage process. Static mixing is done by the way the material is moved through - no blades - like a flowing propellant going through

a pipe and split and reassembled. Ingredient food control.

PRICE: Big solid boosters for NASA not for much longer, be a long while.

Liquid engine run first to be checked before flight.

Solid rockets no chance to compete unless you can guarantee same identical product will be produced - is crucial.

Advice on solid rockets - When sitting out at Kennedy several weeks waiting to be sent out solids were just fine. Liquids wouldn't have been ready for awhile. Liquids are easily prepared for the flights though - and that's a big launch site cost.

Have to PROVE we can make solid propellants the SAME EVERYTIME or it will never work or be accepted.

Big solid boosters is NASA's big concern.

HUSBAND: Liquid's assumable reliability and reusability is why it is gaining ground. Reliability of liquid engine to whole solid load isn't the true picture. They don't see all the pumps, tubes, etc., that go along with the liquids.

PRICE: The impression is that they're looking on unnecessary improvements. But 1% improvement has a lot to do with NASA needs and the military view. A big variability, whole system to be on a burn rate...But, perception is all on control. For NASA applications they might appreciate 1%. Military has Titan and not built in latitude - don't know margin rate of what degrees. Needs to be addressed.

Specifiability of ingredients - chemical problems and specifications - chemical purity is very important. Need to specify the ingredients thoroughly. Spherical particles are specifiable - that's good. Use a unimodel, but then we can't make a propellant thats a model!

Scientific side and understandable data need to specify ingredients.

Alumminum from 2 different sources showed that they had difference in coating on oxide coating. Need to specify ingredients for construction and to change variable.

In 10 years new ingredients will probably be available, but we need to know the combustibile factors FIRST.

Try and convince people we don't know about combustion - no program in place - must take bigger interest in ingredient first, then combustibility, before manufacturing.

Theory for hydrocarbon binders still is far behind - need theory to explain why particles are as they are - can be done through computer computation. No one wants to support the combustion theory. Is 0 now.

Met a man who wants to use all computer capabilities with mixing flows, the scope is tremendous. But I believe we should take the principal system and find out what asumptions you can get from this. No practical stuff to appropriate is tractable.

No combustion work is going on - need Dick Miller ONR for his ideas.

NWC crowd are working doing things no one can imagine - there needs to be some bounds set....Also have guys who mix propellants and fire shots - they don't control variables or characterize materials well.. They get megabucks but don't keep data for others to utilize - no database for future work.

Kumar has problems with a poor database and NASA should be aware of this fact. When JPL makes a decision on what variables they are using, everyone should get together so it's all usable. We have pushed JANNAF for years with little success.

KUMAR: Saw NASA-Marshall Space Center - appears to be amenable and offer suggestions. Richard Brown has funding for it to a certain extent.

KUBLIN: We are attempting to increase propellant work - am happy to discuss propellant problems. Today's climate is better for this.

BOB: A big change from 1986 then.

DOWLER: Encourage mathematical analysis of mixing - it is important to know that what has been done so far in the mix can be based from knowledge of previous mixture. No matter what machine is used it won't improve motor variability unless you look at the casting operation; casting is also the way to understand mixing.

If a military mission was sent in anger but the motor didn't work then what happens with this data; we see few results of motor functional firings.

No one has analyzed casting process.

I also encourage you to build something experimental to show that the model works; then get model more complicated - see what happens with heterogeneous bimodal particle sizes; then add aluminum powder into propellant so the model can provide trimodal interactions.

We have flubbed into a mode where we put our faith in what manufacturers make - 2% control is good in one place but not in another. There is little interest in improving the burn rate measurement.

Look at the design of advanced solids motors, then, figure how you are going to meet the requirements. I don't know how far you can go with investigating only one propellant. It is critically important the University is backed up by work at another agency that can do propellant mixing in collaboration.

Need to pay attention to particle surface parameters.

Really, we have propellants with far too many ingredients. I do not see why we need to have so many ingredients. Had to add carbon black to transparent propellants to prevent "worm-holing;" then we

added aluminum powder as fuel; both prevent worm-holing so it doesn't seem necessary to retain both of them in formulation. It probably causes more problems because both ingredients have to be controlled. Need to go back and justify why each ingredient is needed.

Also need to link the motor insulation bond line surface to propellant and motor variability.

Government has no control over mix specs; they are really only manufacturer's specifications to prevent prior known problems from occurring. When AP, binder, curative and aluminum and other ingredients are changed a new factorial analyses is required for proper specifications. The propellant recipes just keep growing, and it is not necessary. We need to get some of those extra ingredients out of the future propellants.

To correlate burning rates with motors we need to confirm what we mean by "burn rate;" and confirm that we are measuring our definition. Propellants burn normal to surface, but often burn rate samples show burning at an angle due to the sample. Need to look at new techniques so as to develop burn rate instrumentation; will need specific data requirements. Need to get diagnostic burn rate measurements into motors; there are ways to make these measurements now. Maybe you can also back out better burn rate information from flight data. The price, compared to static firing, is not all that expensive.

Make variables you investigate wide enough so you can see the effects - make certain you know and verify. Widely acclaimed by Ed Price.

KUMAR: Artificially expand mix time - 1982 - looked at it, for 15 minutes to 10 hours - then no one gave any support.

HERMANC: If all propellants must be mixed - current testing is good so far...Need more combustion modeling working at 1/2 meter of propellant - why does it do that? (There are 14 ideas so far!) Further combustion modelling work needs done - we're still at physical modelling. Mathematics of mixing is a good idea though.

PRICE: Make sure mixing students look closely at the safety standards.
Need a thermal mixing model.

BARRY: Usually the outside factor creates problems with mixing accidents.

PRICE: You don't want a mathematical model devised that could be dangerous in life.

KUMAR: Mixing is very complex - we attempt to monitor it for local hot spots.

DOWLER: Look at the way the model is causing the mixing. Is only a scientist working with the mix. Someone with practical experience needs to take a what is being done experimentally.

PRICE: See if you can cut the mix in half.

DOWLER: Need ACCURATE burn rate. Casting is also important, and it is more difficult to model and control than the mixing. Every propellant company will want to use a different casting technique.

SUMMERFIELD: You must get your own funding and your own work; get your own students to do the work; I had many graduate students doing such propellant work, and never had an accident. Have small batches and rockets and get burn rates. Understanding the combustion processes for strand burners, small rockets and large rockets should all be done by you. I'll also mention this to your dean later today when we meet.

GEISLER: Bonded interfaces are culprits. Margin of safety is getting better and better. "O" rings are troublesome. We should use the DOE safety approach to nuclear safety. We had an \$800 million failure in Titan. Sensors are important.

HUSBAND: A lot has been done.

PRICE: Ingredients can cause bad troubles. Nature of Al₂O₃ on Al is not well understood. What is the role of its thickness? Specificifiability of ingredients is a problem at all levels. They should be meaningfully done.

GEISLER: "In spec. but out of family!"

PRICE: Will we change the ingredients? AN? Thermoplastic elastomers? We do not know enough about combustion to do what we should be doing. Learn more about ingredients early on. Combustion is still very naive. We have come a long way, but we still do not know much. Support of combustion is ZERO. At ONR one young man wants to do all kinetics data and do modeling and combustion. The scope is so horrendous that it is not useful. We must do some pretty crude stuff (first) for five to ten years to narrow it down in scope. Dick Miller and NWC should be interested.

RAMOHALLI: In a way, the situation is analogous to what we had in the 1950's. Practically all burn rate data were obtained experimentally in composite propellants. It appeared unthinkable that anyone could model, write equations for and obtain burn rates theoretically, applying conservation equations and boundary conditions. Solid propellant combustion was, of course, beyond all that. And yet, a start was made at Princeton. Many students, now all leaders in the field, did outstanding theses under Summerfield actually applying equations, refining results and comparing them with experimental data obtained carefully. Look where that approach has brought us in the 1980's. Similarly, the processing of propellants is considered beyond equations and a scientific

scrutiny today. Maybe, we can change that. Maybe, those complex processes are also amenable to a scientific analysis after all.

Adjourned at 4:00 p.m.

APPENDIX C
LIST OF ATTENDEES

SOLID (Composite) PROPELLANT PREDICTABILITY
and QUALITY ASSURANCE
APRIL 21, 1989

NAME J.A. BARRY
ADDRESS BOX 458 EDWARDS, CA 93523
TELEPHONE #: 805-275-7451 FAX #:

NAME Richard A McKay 512-102
ADDRESS Jet Propulsion Laboratory, 4800 Oak Grove Drive, Pasadena, CA 91109
TELEPHONE #: 818 397 9310 FAX #:

NAME Theodore V. Kublin - George C. Marshall Space
ADDRESS Flight Center Marshall Space Fl. Ctr, FL 32903
TELEPHONE #: (205) 544-8622 FAX #:

NAME E.W. PRICE, SCHOOL OF A.E.
ADDRESS GA. INST. OF TECH. ATLANTA GA. 30332
TELEPHONE #: 404 894 3063 FAX #:

NAME Capt Mark Husband
ADDRESS AL (AFSC) / RKPA, Edwards AFB, CA 93523-5000
TELEPHONE #: (805) 275-5534 FAX #: (805) 275-5144

NAME Robert L. Geister
ADDRESS Astronautics Laboratory, ATTN: VS, EAFB, CA 93521
TELEPHONE #: (805) 275-5420 FAX #: (805) 275-5144

NAME MARSHALL F. HUMPHREY
ADDRESS JET PRODUCTION LAB. 4800 OAK GROVE DRIVE PASADENA CA
TELEPHONE #: 818-354-3523 FAX #: CANT REMEMBER

NAME WARREN L. DOWLER
ADDRESS JPL 125/112 4800 Oak Grove Drive Pasadena
TELEPHONE #: 818/355-3169 FAX #: 7

NAME CLARKIE E. HERMANE
ADDRESS CEMIE DEPT, UNIV. OF VERMONT, BURLINGTON, VT 05405
TELEPHONE #: 802-656-3800 FAX #: 802-656-1924

NAME MARTIN SUMMERFIELD
ADDRESS PCRL, INC., 4275 U.S HWY ONE, MONMOUTH JCT., NJ 08852
TELEPHONE #: 609-452-9200 FAX #: 609-452-9205

NAME Dr Petersen
ADDRESS _____
TELEPHONE #: _____ FAX #: _____

NAME Dr Vincent
ADDRESS _____
TELEPHONE #: _____ FAX #: _____

NAME Daniel Perez AME # 16 / RM 301
ADDRESS _____
TELEPHONE #: _____ FAX #: _____

Carol research assist

APPENDIX D
LETTERS

**PRINCETON
COMBUSTION
RESEARCH
LABORATORIES, INC.**

4275 U.S. HIGHWAY ONE, MONMOUTH JUNCTION, N.J. 08852 TEL.: (609) 452-9200 FAX: (609) 452-9205

May 9, 1989

Memorandum to Prof. Kumar Ramohalli, University of Arizona:

This is a brief and hurried note to cover the main points that I suggested at the Conference on Solid Propellants held at the University on April 21, 1989. I apologize for the brevity of this note; I was not aware that my recommendations should be submitted in written form, and so this is being rushed at the last moment, before you are scheduled to depart on your long sabbatical leave.

I recall that I offered two principal suggestions. One was the observation that we may be searching for burning rate phenomena that are relatively small, perhaps as small as 2 to 5% of the "nominal" burning rate. Since that is so, it is necessary to be quite sure to eliminate all the usual disturbing effects that can introduce variations of this order of magnitude. For example, although two AP grinds may nominally be the same with the same reported particle size, it is quite possible for them to produce two burning rates that differ by the small amount suggested. More generally, it can be shown (look at the results reported in the Ph.D. thesis of J.A. Steinz, The Burning Mechanism of Ammonium Perchlorate Based Composite Solid Propellants, Princeton University, 1968, Appendix C, Experimental Procedures and Accuracy of Measurements, pp. 196-211) that many factors enter into the matter of accuracy and reproducibility. Most of them have been, and are still, overlooked or simply wiped away by practitioners in the field. Therefore, instead of taking burning rate data that other labs may furnish, one has to obtain specimens and test them oneself in one's own lab. Those other labs may not have given attention to the factors that may disturb the results. For example, manufacturers may be content to measure burning rates simply to assure quality control; that is, the rates may be in error by some small percentage, but if their mixes are always tested in the same way, deviations can be attributed to changes in the monomer or some other ingredient. That conclusion is probably true, but the burning rate data would not be acceptable for the more scientific investigations at the University.

The second main point that I made is that, if the University group under Prof. Ramohalli wants to pursue the factors that might cause systematic variations in burning rate, it will be necessary to set up a laboratory for this very purpose at the University. Two steps are involved in this matter: one, to set up a laboratory for processing and making the necessary "sticks" of solid propellant, and two, to set up a laboratory for measuring burning rates, with the "sticks" thus produced, with all the disturbing factors under control. Hazards are involved, of course. The laboratories for each of these two projects are

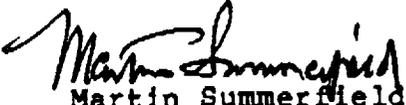
(Continued . . .)

Memorandum
Prof. Kumar Ramohalli
May 9, 1989
Page Two

not simple. It takes more than this short report to get into what is involved in setting up such laboratory facilities within a university. It can be done, and such facilities were in operation for nearly 20 years at Princeton University. Similar, but more modern, facilities exist at Penn State,* and plans for such facilities have been developed at Georgia Tech.

We discussed to a small extent the so-called "hump" phenomenon in burning rates deduced from firings of large rocket motors, but the matter was not resolved. It was reported that such "humps" have been eliminated, but this matter deserves some follow-up. There is probably a difference between burning rates measured with "sticks" of propellant in a strand-burner and rates deduced from firings of large rocket motors. The reason should be pursued, at least to "nail down" the small differences mentioned at the beginning of these notes. Something significant may be at the basis of it all.

This memo covers the main points on which I commented.


Martin Summerfield
Consultant

MS/jem

*While Penn State has an impressive array of solid propellant testing and research, manufacturing solid propellants is not one of them, yet (19 July 1989).

Warren L. Dowler
526 Camillo Street
Sierra Madre, California 91024-1402
818/355-9707
April 23, 1989

Professor Kumar Ramohalli
Department of Aeronautical and Mechanical Engineering
University of Arizona
Tucson, Arizona 85721

Dear  Kumar:

I want to thank you for the experience Thursday and Friday. I hope that it will be profitable to you. The social hour at your home was very nice. Your mounted photos also are good.

I would suggest that you send a draft copy of the report to each participant so that he can correct any errors made in the transcription of verbal comments. It would be a serious mistake to misquote someone. They also may wish to add other comments--unsaid or thought of after the meeting. However, you should give a drop dead time limit; like, if I don't hear within two weeks it will be assumed that you have no corrections or comments.

Separate subject. In thinking about the future of your Center for extra terrestrial materials, it seems you should now begin

Again, thanks for the opportunity to participate, and congratulations on the Professorship. Its been a long time.

Sincerely yours,



Warren L. Dowler

4800 Oak Grove Drive
Pasadena, California 91109
(818) 354-4321

April 27, 1989

Refer to: 353CP-89-144

Dr. Kumar Ramohalli
UA/NASA Space Engineering Center
4717 E. Ft. Lowell Road
Tucson, AZ 85712

Dear Kumar:

Thank you for having me at the recent meeting on propellant mixing and quality assurance. Enclosed you will find a flow sheet of what I remember of the Aerojet Continuous Mix Facility. It is crude but will indicate the principle features of the installation. The mixing screw was very important and many hours were spent trying to obtain the ideal design. Also the system was designed with fire breaks so that only a limited amount of propellant was exposed to a fire situation. Heating and cooling capabilities were also incorporated in the design. Many modern improvements could be incorporated into a new installation. However, the principle problem remains and that is the ability to produce more material than required. This of course is a scale problem, but it is also a problem of being able to halt production and maintain the quality upon start up of the system. A lot of interesting modifications come to mind for solving these problems.

Some solid propellants contain as many as 16 ingredients. This is entirely too many. Each material must be controlled as to purity, uniformity and various other properties. A reduction in materials would simplify the process.

I will list just a few of the many subjects or operations that should be examined to perfect the quality, uniformity and integrity of solid propellants:

Mixers or preferable compounders i.e. conical screws, ribbon blenders, static mixers or motionless mixers, modern sigma mixers, planetary mixers with modifications, ultrasonic, etc.

Reduce times in mixing

Temperature conditions for mixing

Vacuum vs. pressure and changes during processing

Continuous solids blending, testing and combining

Oxidizer particle size has always been a problem

Mixing times vs. necessity

Testing, microwave characteristics to determine BR, viscosity, solids distribution, density in situ

Ramohalli

-2-

353CP-89-144

Various rheological tests for quality assurance

Viscometer applications

and many more

Your centers' aims sound very interesting and presents excellent opportunity for developing the new space scientists that will be drastically needed in the future.

Again thank you and congratulations on your promotion.

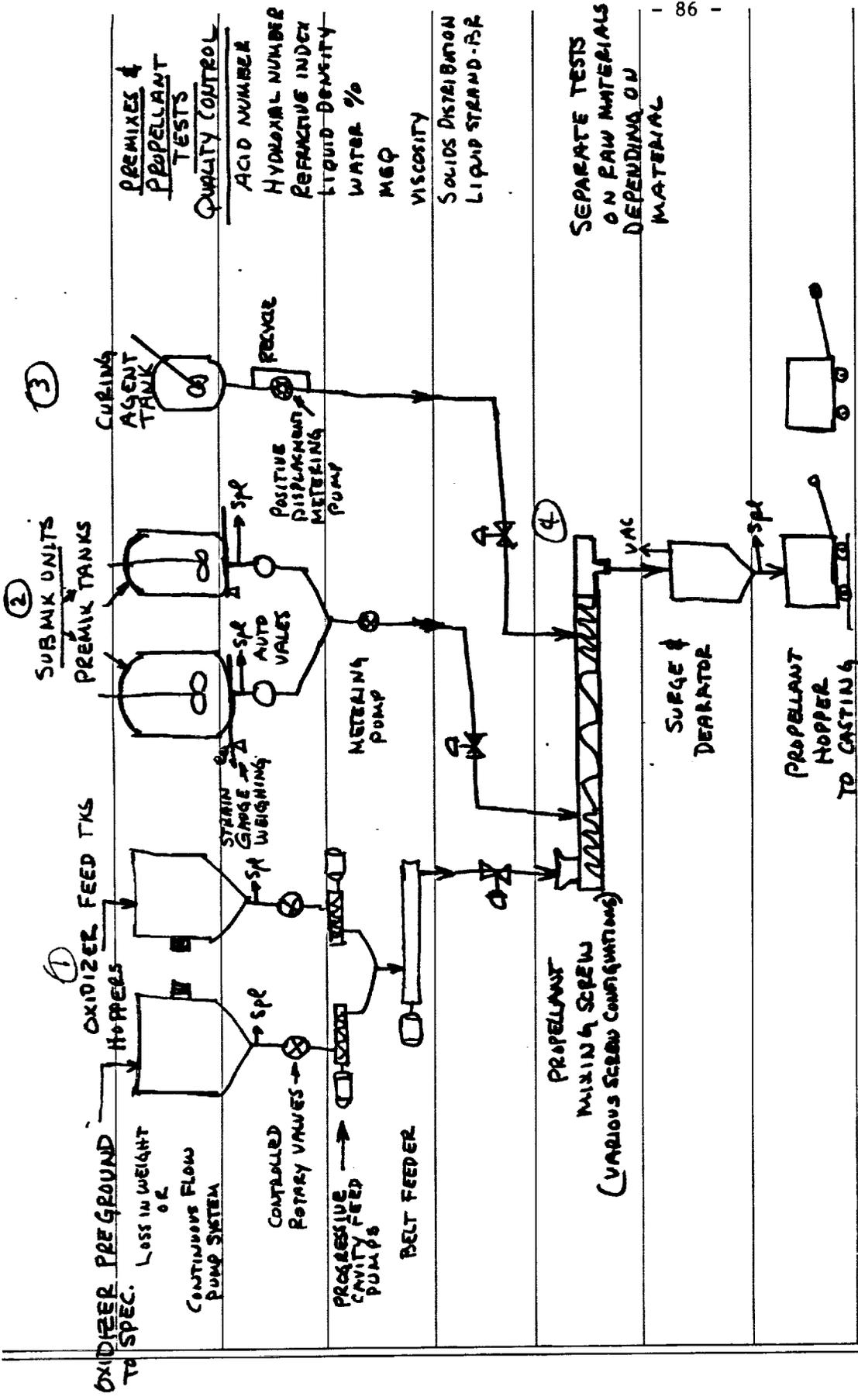
Your friend,



Marshall F. Humphrey
Member Technical Staff
Propulsion Systems Section

MFH:tdaw
Enclosure

cc: D. P. Maynard



SEPARATE TESTS ON RAW MATERIALS DEPENDING ON MATERIAL

NOTES ① OXIDIZER WAS PREGROUND AND PRE BLENDED - TWO FEED MECHANISMS TRIED, A WEIGH BELT CONVEYOR AND A LOSS OF WEIGHT SYSTEM [I BELIEVE A PROGRESSIVE CAVITY PUMP WOULD WORK]

② A SUBMIX WAS PREPARED CONTAINING PPG, MTDA, PBMA & LECITHIN

③ CURING AGENT WAS TDI (but it was DILUTED WITH?)

POLARIS
CONTINUOUS MIX FACILITY

④ MIXER WAS A VARIABLE SCREW

PREMIXES & PROPELLANT TESTS
QUALITY CONTROL
ACID NUMBER
HYDROXAL NUMBER
REFRACTIVE INDEX
LIQUID DENSITY
WATER %
MSQ
VISCOSITY

SOLIDS DISTRIBUTION
LIQUID STRAND-PSR

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332-0150

SCHOOL OF
AEROSPACE ENGINEERING

404-894-3000

DANIEL GUGGENHEIM SCHOOL
OF AERONAUTICS

2 May 1989

Prof. Kumar Ramohalli
Department of Mechanical
and Aerospace Engineering
University of Arizona
Tucson, AZ 85721

Dear Kumar,

Enclosed is a hasty write-up of the summary points I raised at your Workshop on April 21, 1989. Hope the write-up is useful. The comments that seem negative are not advanced as criticism, they are advanced as examples of arguments that you will have to address from time to time (and in most cases, already have).

Best wishes.

Sincerely,



Edward W. Price
Regents' Professor
School of Aerospace Engineering

EWP/ed
pltr.363

SUMMARY COMMENTS
AT RAMOHALLI WORKSHOP

April 21, 1989

1. A general point: the ongoing national promotional campaign to replace solid propellant systems by liquid propellant ones is going so one-sidedly in favor of liquid systems (for launch vehicles) that the goals of the University of Arizona propellant studies may become irrelevant unless the case for solid rocket motors is more vigorously promoted (sorry; that's a political issue!).

2. If the major propellant manufacturers were here, I believe they would say that they can make propellants now with burning rates that conform to all specifications, and that they could meet more stringent specifications if asked to. What they can't do by control of ingredient and processing specifications, they fine tune by testing liquid strands during batch processing and adding catalyst as needed. The case for better control by better initial control of ingredients and processing alone has to be argued in terms that acknowledge the present company capability.

3. The discussions here give the impression that the target control on burning rate ($\pm .5\%$?) is unreasonably stringent. This impression would be justified in the context of most military systems, which either

- a) have to deal with much larger variation in burning rate due to a wide service temperature range,
- b) have thrust termination systems that provide accommodation for variability in burning rate.

The need for fine-control on burning rate has to be assessed according to the application. The need for Shuttle and Titan boosters is the most obvious and most often posed.

4. If the burning rate is to be controlled through proper specification and control of ingredients and processing, the ingredients and processing will have to be specified in much greater detail than they are now. We are not even sure what properties and processes need to be specified and controlled (e.g., the burning rate and mechanical properties' dependence on AP particle shape, binder curative, or oxide coating on aluminum particles are unknown). Further, there is no work contemplated to bring these variables under rational control.

5. Whenever we begin to think our combination of art and science are getting things under control, somebody changes the rules of the game by introducing a new ingredient that doesn't conform (e.g., for smokeless propellants, low hazard propellants, low cost propellants, etc.). What predictive capability we have for formulation effects rests in part on familiarity with past results for the ingredients that are involved. Even the theory is tailored to match the observed combustion behavior. New

ingredients often render the old results misleading or irrelevant. The understanding of the combustion should be at a sufficiently fundamental level so that the relevant characteristics of new ingredients can be measured, and the effect on combustion can then be forecast.

6. The present theories for steady state burning are still very naive, and almost no research to improve the situation is in the works (funding is lower than at any time since the mid-1950s).

7. An enormous amount of money has been spent during development programs on empirical approaches to meeting program needs for burning rate or mechanical properties. The totality of such efforts contributes very little to understanding or future ability for rational control because the myriad of relevant material, formulation, and test conditions have little commonality from one study to the next. Some yield of understanding might result if some standard values of propellant and test variables were adopted "industry wide". Where feasible, standard values would be chosen. At least, each study might include a control test to establish comparability of results from different studies.

APPENDIX E
AN EARLY LETTER

June 24, 1983
PCRL-L-83-273

Dr. Kumar Ramohalli
Aerospace and Mechanical Engineering Dept.
Aero Building No. 16
University of Arizona
Tucson, AZ 85721

Dear Kumar:

Many thanks for sending me the program of the 1981

You might be interested in a recent development that provoked considerable reference to your paper on processing effects on combustion of composite propellants. I participated in a workshop conducted last week at the Naval Ordnance Station at Indian Head under JANNAF auspices, where the topic was the problems arising from the peculiarities of the combustion properties of composite propellants based on solid nitramine oxidizers (HMX and RDX) in non-energetic binders, when used as gun propellants (high pressure combustion). Although there were plenty of good reasons to be concerned with the complexity of the burning process and the ignition process, the question of whether a good part of the non-reproducibility could be traced to the variability of the propellant itself came up. I brought to the attention of the participants, most of them from the gun ballistics community and not from the rocket community, the paper that you had recently written on the subject. I did not have a copy with me so I summarized the highlights, and so there were a number of requests. Dr. Leon Strand was there, and he volunteered to get copies of the paper or the report from the JPL files and send several copies to the people at BRL and NOSIH who are concerned.

Sincerely, with best regards,



Martin Summerfield
President

MS/jmw

**APPENDIX F
DATA SHEETS**

PROPELLANT PROCESSING SCHEDULE

SNM No. 5 Cartridge No. 004R WEEK OF 31 Oct 1983 SHEET 1 OF 17
 BATCH NO. SB-191A MIXER 150 gl. Baker-Perkins JOB NO. 365-40530-0-3440
 PROPELLANT NO. PBAN-Mod 8 BATCH SIZE 1900 lbs. ENGINEER B. Morrison
 DATE MIXED 31 Oct 1983

| CHARGE NO. | CHARGE TYPE | MOTOR NO. | MANDREL CONFIGURATION | INSULATION AND LINER CONFIGURATION | CURE TEMPERATURE AND DURATION | PROCESS TECHNIQUES OR PROCEDURES | PURPOSE OR END USE |
|------------|----------------------------------|-----------|-----------------------|------------------------------------|-------------------------------|--|--------------------|
| 1 | Tensile Mold | | with density bar | | 140°F, 9 days | S.T. PD-FA-011-003 | |
| 2 | Burning Rate | | Block | | 140°F, 9 days | S.T. PD-FA-011-003 | |
| 3 | 48 in. Cartridge No. <u>004R</u> | | PL-BCT-1 Liner | | 140°F, 9 days | Processing Plan 48 in. dia. cartridge | |
| 4 | 1 gallon Sealrite | | | | 140°F, 9 days | | Ambient Storage |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

SPECIAL PROCESSING PROCEDURES MICROMEROGRAPH YES NO SIEVE ANALYSIS YES NO

1. Follow attached mix procedure.
2. Following the slurry pre-mix, obtain the propellant slurry wt. Remove a 1 lb. slurry sample for viscosity test as per TASK PLAN for MULTIPLE PROPELLANT BATCHES MIXING AND HANDLING. Record the slurry viscosity on the QCD form.
3. Return slurry sample to mixer pot. 4. Following the final mix cycle, measure EOM and EOC viscosities and record on QCD form. 5. Follow the logistics and handling instructions outlined in the TASK PLAN for MULTIPLE PROPELLANT BATCHES MIXING AND HANDLING.

NOTE: UNLESS OTHERWISE INDICATED CASTING SEQUENCE WILL BE IN ORDER OF CHARGE NUMBERS

SAFETY INSTRUCTIONS



2

PBAN - Mod. 8 PROPELLANT FORMULATION

BATCH NO: SB191-A
BATCH SIZE: 1900 lbs.
MIX DATE: 31 Oct 1983

CARTRIDGE NO: 004R
SNM NO: 5

| <u>INGREDIENTS</u> | <u>LOT NOS.</u> | <u>WT. %</u> | <u>WT. LBS.</u> | <u>WT. GRAMS</u> |
|--------------------------------|-----------------|---------------|-----------------|------------------|
| AP, 200 (70) | 5049 | 48.99 | 930.81 | |
| AP, Ground*(30) | 5049 | 21.00 | 399.00 | |
| A1, S-392 | 7676 | 16.00 | 304.00 | |
| Fe ₂ O ₃ | 18612599 | 0.010 | 0.19 | 86.18 |
| PBAN (1.0 eqs.) | 876 | 11.49 | 218.31 | |
| DOA (5% of Binder) | 48-664 | 0.70 | 13.30 | |
| DER-331 (1.3 eqs.) | WT. 8109252 | 1.81 | 34.39 | |
| | | <u>100.00</u> | <u>1900.00</u> | |

NOTE: Completed slurry mix should weigh 1865.61 lbs.

*Hammer Speed = 9600 RPM; Feed Rate = 80; Screen Size = 0.020"

SOLID PROPELLANT BATCH SHEET - INGREDIENTS

Batch Number SB-191 "A"

Formulation Number PBAN-Mod. 8

Total Weight 1900 LBS

Date 24 OCT 1983

Engineer J. GARRY

Weigh Room Temperature 76 °F

Relative humidity 29 %

| Ingredient | Lot#/Drum# | Treatment/by Date | Weight | | Balance | Initial |
|--------------------------------|---------------|--|----------|--------|---------|---------|
| | | | grams | pounds | | |
| AMMONIUM PERCHLORATE | 5049 | | Net | | 930.81 | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
| AMMONIUM PERCHLORATE (GROUND) | 5049 | H/S 9600 F/S 80 .020 SCREEN GR. # 222 | Net | | 399.00 | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
| ALUMINUM S-392 | 7676 DR. # 10 | | Net | | 101.00 | |
| | | | Dry Tare | | 13.76 | E-50 |
| | | | Gross | | 114.76 | % B |
| | | | Wet Tare | | | |
| ALUMINUM S-392 | 7676 DR. # 10 | | Net | | 101.00 | |
| | | | Dry Tare | | 13.98 | E-50 |
| | | | Gross | | 114.98 | % B |
| | | | Wet Tare | | | |
| ALUMINUM S-392 | 7676 DR. # 10 | | Net | | 102.00 | |
| | | | Dry Tare | | 14.30 | E-50 |
| | | | Gross | | 116.30 | % B |
| | | | Wet Tare | | | |
| Fe ₂ O ₃ | 13612599 | | Net | 86.18 | | |
| | | | Dry Tare | 25.77 | | E-2 |
| | | | Gross | 111.95 | | % B |
| | | | Wet Tare | | | |
| PBAN | 876 DR. # 11 | | Net | | 109.00 | |
| | | | Dry Tare | | 13.84 | E-50 |
| | | | Gross | | 122.84 | % B |
| | | | Wet Tare | | | |
| PBAN | 876 DR. # 11 | | Net | | 109.31 | |
| | | | Dry Tare | | 13.50 | E-50 |
| | | | Gross | | 122.81 | % B |
| | | | Wet Tare | | | |
| DOA | 48-664 | | Net | | 13.30 | |
| | | | Dry Tare | | 4.46 | E-50 |
| | | | Gross | | 17.76 | % B |
| | | | Wet Tare | | | |

= 304.0

= 218.31 LBS.

4

SOLID PROPELLANT BATCH SHEET - INGREDIENTS

Batch Number SB-191 "A"

Formulation Number PBAN-Mod. 8

Total Weight 1900 LBS.

Date 24 OCT 1983

Engineer J. BARRY

Weigh Room Temperature 76 °F

Relative humidity 29 %

| Ingredient | Lot#/Drum# | Treatment/by Date | Weight | | Balance | Initial |
|------------|------------------------|-------------------|----------|--------|---------|-------------|
| | | | grams | pounds | | |
| DER-331 | WT8109252 (NEW LOT) | | Net | | 34.39 | E-50 B B |
| | | | Dry Tare | | 7.62 | |
| | | | Gross | | 42.01 | |
| | | | Wet Tare | | | |
| | | | Net | | | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
| | | | Net | | | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
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| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
| | | | Net | | | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |
| | | | Net | | | |
| | | | Dry Tare | | | |
| | | | Gross | | | |
| | | | Wet Tare | | | |

4
JP

SOLID PROPELLANT OXIDIZER WEIGH-OUT

BATCH NO. SB-191A , BATCH SIZE: 1900 lbs DATE WEIGHED 10.28.83
 Cartridge No. 004R
 OXIDIZER TYPE: 200 μ AP Ground AP
 LOT NUMBERS: 5049 5049
 WEIGHT REQUIRED: 930.81 399.00 Net. Req. 1329.81
 INCREMENTS: 4 3
 WT./INCREMENT: _____

| Steps | Container plus Increments | INCREMENT WEIGHTS of OXIDIZER TYPES | | Scale Preload | Read |
|------------|---------------------------|-------------------------------------|-----------------|---------------|---------------|
| | | 200 μ | Ground | | |
| TARE Wt. | <u>180.30</u> | | | | |
| Inc. 1 | <u>330.81</u> | <u>330.81</u> | | | |
| Sum | <u>511.11</u> | | | <u>500</u> | <u>11.11</u> |
| Inc. 2 | <u>199.00</u> | | <u>199.00</u> | | |
| Sum | <u>710.11</u> | | | <u>500</u> | <u>210.11</u> |
| Inc. 3 | <u>200.00</u> | <u>200.00</u> | | | |
| Sum | <u>910.11</u> | | | <u>750</u> | <u>160.11</u> |
| Inc. 4 | <u>100.00</u> | | <u>100.00</u> | | |
| Sum | <u>1010.11</u> | | | <u>1000</u> | <u>10.11</u> |
| Inc. 5 | <u>200.00</u> | <u>200.00</u> | | | |
| Sum | <u>1210.11</u> | | | <u>1000</u> | <u>210.11</u> |
| Inc. 6 | <u>100.00</u> | | <u>100.00</u> | | |
| Sum | <u>1310.11</u> | | | <u>1250</u> | <u>60.11</u> |
| Inc. 7 | <u>200.00</u> | <u>200.00</u> | | | |
| Sum | <u>1510.11</u> | | | <u>1500</u> | <u>10.11</u> |
| Inc. 8 | | | | | |
| Sum | | | | | |
| Inc. 9 | | | | | |
| Sum | | | | | |
| Inc. 10 | | | | | |
| Total Wts. | <u>1510.11</u> | <u>930.81</u> | <u>+ 399.00</u> | | |
| Less Tare | <u>180.30</u> | | | | |

Oxid. Wt. _____ lbs. = 1329.81 lbs Net Wt.

Weights by: WBC
 Checked by: SAL

- 98 -
EDWARDS TEST STATION

SOLID PROPELLANT MIXING SCHEDULE

THIS SCHEDULE IS TO BE COMPLETED BY THE COGNIZANT ENGINEER PRIOR TO BEGINNING THE BATCH

BATCH: SB-191A COGNIZANT ENGINEER: B. Morrison
 CARTRIDGE NO: 004R DATE: 31 Oct 1983
 FORMULATION: PBAN-Mod. 8
 SNM NO.: 5

| <u>INGREDIENTS ADDITION</u> | <u>TIME (MIN.)</u> | <u>SPEED (RPM)</u> | <u>DESIRED PROP. TEMP. (°F)</u> |
|-----------------------------|------------------------|------------------------|-------------------------------------|
|-----------------------------|------------------------|------------------------|-------------------------------------|

ADD PBAN, DOA, & Fe₂O₃

| | | | | |
|--------|-----|-----------|------------|------------|
| MIX AT | ATM | <u>5</u> | <u>Low</u> | <u>160</u> |
| | VAC | <u>10</u> | <u>Low</u> | <u>160</u> |

ADD A1,

| | | | | |
|--------|-----|-----------|------------|------------|
| MIX AT | ATM | <u>5</u> | <u>Low</u> | <u>160</u> |
| | VAC | <u>15</u> | <u>Low</u> | <u>160</u> |

SCRAPE DOWN

ADD AP from hopper as per S.O.P. 2036

| | | | | |
|--------|-----|----------------|------------|--|
| MIX AT | ATM | <u>As req.</u> | <u>Low</u> | <u>160</u> |
| | ATM | <u>15</u> | <u>Low</u> | <u>160</u> - After all AP is in mixer. |

SCRAPE DOWN & BRUSH DOWN THOROUGHLY

| | | | | |
|----------------|-----|-----------|------------|------------|
| *MIX AT STAGED | VAC | <u>30</u> | <u>Low</u> | <u>160</u> |
| | VAC | <u>60</u> | <u>Low</u> | <u>140</u> |

Break Vacuum, lower pot, and weigh to obtain slurry weight. Take sample for slurry viscosity.

ADD DER-331

| | | | | |
|--------|-----|-----------|------------|------------|
| MIX AT | VAC | <u>10</u> | <u>Low</u> | <u>140</u> |
|--------|-----|-----------|------------|------------|

SCRAPE DOWN. Return excess viscosity sample to pot.

| | | | | |
|--------|-----|-----------|------------|------------|
| MIX AT | VAC | <u>15</u> | <u>Low</u> | <u>140</u> |
|--------|-----|-----------|------------|------------|

Take EOM viscosity sample. Vacuum cast all charges

*START MIXER PRIOR TO BEGINNING VACUUM PULL-DOWN. PULL VACUUM ACCORDING TO ATTACHED SCHEDULE.

SPECIAL INSTRUCTIONS: OBTAIN SLURRY, EOM & EOC BROOKFIELD VISCOSITY.

REMARKS: Layer the AP in the hopper as follows:
 Course-fine-course-finecourse.
 (i.e., begin and end with a coarse layer)

SB-191 - "A"

ATTACHMENT TO THE SOLID PROPELLANT MIXING SCHEDULE

VACUUM MIX CYCLE AFTER AP ADDITION

1. Make sure TV monitor positioned so that manometer in mix building can be observed.
2. Raise mix bowl into mixing position.
3. Start mixer at atmospheric pressure, low speed, and temperature control to maintain 140°F propellant temperature.
4. Continue operating mixer at low speed. Start vacuum pump and operate vacuum pump intermittently for the first 30 minutes of mixing to maintain the following pressures in mix bowl during vacuum transition mix cycle: Allow vacuum to stabilize at each setting before proceeding to next lower pressure.

| <u>Vacuum Pump Operation</u> | <u>Time (minutes)</u> | <u>Manometer Reading Leg (inches)</u> | <u>Mixer Speed (RPM)</u> | <u>Desired Prop. Temp. (°F)</u> |
|------------------------------|-----------------------|---------------------------------------|--------------------------|---------------------------------|
| Intermittent | 5 | 0901 11.0 ✓ | LOW | 140 |
| | 5 | 0906 9.0 ✓ | | |
| | 2 | 0911 8.0 ✓ | | |
| | 2 | 0913 7.0 ✓ | | |
| | 2 | 0915 6.0 ✓ | | |
| | 2 | 0917 5.0 ✓ | | |
| | 2 | 0919 4.0 ✓ | | |
| | 2 | 0921 3.0 ✓ | | |
| | 2 | 0923 2.0 ✓ | | |
| | 2 | 0925 1.5 ✓ | | |
| | 2 | 0927 1.0 ✓ | | |
| | 2 | 0929 0.5 ✓ | | |
| Full Time | 30 | 0.0 to 0.1 0931 ✓ | | |

ORIGINAL PAGE IS OF POOR QUALITY

10

QUALITY CONTROL DATA FORM
(QCD)

| | | | |
|-------------|--------------------|---------------|-------------|
| BATCH NO: | <u>SB-191A</u> | POT I.D.: | <u>JPL</u> |
| PROPELLANT: | <u>PBAN-Mod. 8</u> | CARTRIDGE NO: | <u>004R</u> |
| BATCH SIZE: | <u>1900 lbs.</u> | SNM NO.: | <u>5</u> |
| MIX DATE: | <u>31 Oct 1983</u> | | |

GROUND AP PARTICLE SIZE, μ

Grind Run No.: 222

MM Analysis: 10.5

F.S.S.: 8.7

PROPELLANT SLURRY WT., lbs.

Total Wt. (Pot + Lift Fix. + Prop. slurry): 4726

Tare Wt. (Pot + Lift Fixture):* 2856

Net Propellant Slurry Wt.: 1870

PROPELLANT VISCOSITY, KPS

| | <u>TIME</u> |
|-----------------------------------|--------------|
| Slurry: <u>13.5 @ 137°F</u> | <u>10:55</u> |
| End of Mix: <u>8.67 @ 147°F</u> | <u>11:56</u> |
| End of Cast: <u>12.75 @ 143°F</u> | <u>13:54</u> |
| Time: <u>1 hr. 58 min</u> | <u></u> |

*Weigh the pot after hot water has circulated through the jacket for at least 30 minutes.

PROPELLANT DATA FORM

BATCH NO: SB-191A

MIXER USED: 150 gal. B-P

FORMULATION: PBAN Mod. 8

END USE: To load cartridge 004R
for SNM No. 5

BATCH SIZE: 1900 lbs.

MIX DATE: 31 Oct. 1983

| <u>INGREDIENTS</u> | <u>LOT NOS.</u> | <u>WT. %</u> | <u>WT. LBS.</u> | <u>WT. GRAMS</u> |
|--------------------------------|-----------------|---------------|-----------------|------------------|
| AP, (70) 200 μ | 5049 | 48.99 | 930.81 | |
| AP, GRND* (30)~10 μ | 5049 | 21.00 | 399.00 | |
| AL, S-392 | 7676 | 16.00 | 304.00 | |
| Fe ₂ O ₃ | 1B612599 | 0.010 | 0.19 | 86.18 |
| PBAN (1.0 eqs) | 876 | 11.49 | 218.31 | |
| DOA (5% of bndr) | 48-664 | 0.70 | 13.30 | |
| DER-331 (1.3 eqs) | WT8109252 | 1.81 | 34.39 | |
| | | <u>100.00</u> | <u>1900.00</u> | |

MIX VISCOSITY:

Slurry 13.5 KPS at 137 °F *Grind No: 222

EOM 8.7 KPS at 147 °F Avg. Particle Size: 10.5 μ

EOC 12.8 KPS at 143 °F at 2 hrs.

PHYSICAL PROPERTIES AT 9 DAYS CURE

S_m, psi - 136.0

E_m, % - 28.6

S_b, psi - 130.7

E_b, % - 35.0

DENSITY, lbs/in³ - 0.0640

Shore "A" Hardness - 70

BURNING RATES AT 9 DAYS CURE

| <u>PSIA</u> | <u>IN/SEC</u> | |
|-------------|---------------|---------|
| 350 | 0.224 | |
| 500 | 0.258 | |
| 650 | 0.281 | 0.289** |
| 750 | 0.300 | |
| 1000 | 0.329 | |

COMMENTS: JPL Ingredients

* Hammer Speed = 9600 rpm; feed rate = 80 ; screen size = 0.020"
1 lb. = 453.5924 grams

**Retest Nine Months Later

12

SECTION



CRAWFORD BURNING RATE DATA RECORD

Page 1 of 2

T.O. R. Ray BATCH SB191A DATE 11-21-83 BY Len/Ron
SNM#5 11-22-83

| PRESSURE (Psig) | PSIA | LENGTH (In.) | TIME (Sec) | RATE (In/Sec) | REMARKS |
|-----------------|----------------|---------------|------------------|---------------|---|
| 986 | 1000 | 5" | 15.22 | .329 | Head # 1 G ₂ /C ₂ Temp. 81° |
| " | " | " | 15.27 | .327 | } "Avg. 329" |
| " | " | " | 15.11 | .331 | |
| | | | Av. | 0.329 | |
| 736 | 750 | 5" | 13.95 | — | 2 G ₂ /C ₂ 81° F |
| " | " | " | 15.43 | — | " " " |
| " | " | " | 16.66 | — | " " " |
| 1.36 | 650 | 5" | 17.70 | .282 | 1 " 81 |
| " | " | " | 17.86 | .280 | } Avg " " |
| " | " | " | 17.76 | .282 | |
| | | | Av. | 0.281 | C ₂ /G ₂ 77 |
| 486 | 500 | 5" | 24.84 | — | 2 " " |
| " | " | " | 19.35 | .258 | " Avg. 258 ← → |
| " | " | " | 19.49 | .257 | " " " next page |
| | | | Av. | .258 | |
| 336 | 350 | 5" | 22.27 | .225 | 1 } C ₂ only 77° |
| " | " | " | 22.20 | .225 | } Avg. 224 " |
| " | " | " | 22.43 | .223 | |
| | | | Av. | .224 | |
| 736 | 750 | 5" | 16.71 | .309 | 1. } C ₂ /G ₂ 79° F |
| " | " | " | 16.53 | .302 | " " " |
| " | " | " | 16.57 | .302 | " " " |
| | | | Av. | .301 | } Avg. 300 |
| | | | 1 " | | C ₂ /G ₂ 80 |
| 736 | 750 | 5 | 16.64 | .300 | 2 " " |
| " | " | " | 16.67 | .300 | " " " |
| " | " | " | 16.77 | .298 | " " " |

on by "istalo"

MICROMEROGRAPH DATA SHEET

| \sqrt{C} | dia. | % | A | Chart |
|------------|-------|------|------|-------|
| ∞ | | | | |
| 269 | 192.6 | | | |
| 180 | 128.9 | | | |
| 150 | 107.4 | | | |
| 120 | 85.9 | 100 | 86 | 0 |
| 90 | 64.5 | 98.8 | 85 | 1 |
| 75 | 53.7 | 97.7 | 84 | 2 |
| 66 | 47.3 | 96.5 | 83 | 3 |
| 60 | 43.0 | 95.9 | 82.5 | 3.5 |
| 54 | 38.7 | 94.8 | 81.5 | 4.5 |
| 48 | 34.4 | 93.6 | 80.5 | 5.5 |
| 42 | 30.1 | 91.3 | 78.5 | 7.5 |
| 36 | 25.8 | 87.2 | 75 | 11 |
| 30 | 21.5 | 82.6 | 71 | 15 |
| 27 | 19.3 | 79.7 | 68.5 | 17.5 |
| 24 | 17.2 | 75.6 | 65 | 21 |
| 21 | 15.0 | 69.2 | 59.5 | 26.5 |
| 20 | 14.3 | 66.3 | 57 | 29 |
| 19 | 13.6 | 64.0 | 55 | 31 |
| 18 | 12.9 | 61.0 | 52.5 | 33.5 |
| 17 | 12.2 | 57.6 | 49.5 | 36.5 |
| 16 | 11.5 | 54.7 | 47 | 39 |
| 15.1 | 10.8 | 51.7 | 44.5 | 41.5 |
| 14 | 10.0 | 47.7 | 41 | 45 |
| 12 | 8.6 | 41.3 | 35.5 | 50.5 |
| 10 | 7.2 | 33.7 | 29 | 57 |
| 8 | 5.7 | 26.7 | 23 | 63 |
| 6 | 4.3 | 17.4 | 15 | 71 |
| 5 | 3.6 | 13.4 | 11.5 | 74.5 |
| 4 | 2.9 | 9.3 | 8 | 78 |
| 3 | 2.1 | 8.1 | 7 | 79 |
| 2.5 | 1.8 | 4.7 | 4 | 82 |
| 2.0 | 1.4 | 0.6 | 1.5 | 85.5 |

SAMPLE: NH₄ClO₄ RUN# 218
 Density: $\rho = 1.95 \text{ g/cc}$ DEAGGLOMERATOR: 250
 $\sqrt{\rho}$: 1.396 PRESSURE: 200
 TECHNICIAN: HBC
 Sample Size 50 m
 MATERIAL USAGE: Date Analyzed 10/20/8

Prop. Batch # 58 190 →
 Other Avg of 6 Batches
Composite Cast into cartridge 002 for SMM-5

DESCRIPTION OF SAMPLE:
 Grind Date 10.14.83
 Grind No. 222
 Lot No. 5049 Drum No. _____
 Hammer Speed 9600 RPM
 Feed Rate 80 RPM
 Screen Size 0.020

AVERAGE PARTICLE SIZE:
 Total pen deflection (C_f): 86
 Average pen deflection ($C_f \div 2$): 43

| | |
|------------------|-------------|
| P.S. (DIA) | CHART |
| <u>10.8</u> | <u>41.5</u> |
| <u>Ave. P.S.</u> | <u>43</u> |
| <u>10.0</u> | <u>45</u> |

$(.8) (\frac{2}{3.5}) = .457$
 $10.0 + .46 = 10.46$

AVERAGE PARTICLE SIZE = 10.5 μ

DATA REDUCED BY: Plkay

STANDARD TENSILE TEST DATA SHEET

BATCH NO. 58191A
FORMULATION NO. JPL PBAN M00B
PROGRAM NO. OR S.O.P. 365-40530-0-3440
CURE TIME AND TEMPERATURE
DATE 11-18-83
OBSERVER WRIGHT/CLAY

Table with 3 columns: Test Parameters, Specimen 1-1 (x 5119), and Specimen 1-2 (x 5120). Rows include Gage Length, Load Cell, Charge, Instron Run No., Specimen Source, Run Temperature, and various mechanical properties like Load Scale, Crosshead Speed, Chart Speed, Width, Thickness, Transverse Area, Elongation Factor, Max. Load, Chart Extension, Load at Break, Shore 'A' Hardness, Maximum Tensile Strength, Elongation at Max. Load, Tensile Strength at Rupture, and Elongation at Rupture.

REMARKS:

ORIGINAL PAGE IS OF POOR QUALITY

DENSITY OF PROPELLANTS

LABORATORY WORK SHEET

TECHNICIAN JERRY CLAY DATE Nov 21, 1983
 COPY TO R. RAY BATCH NO. SB 191 A
 FORMULATION JPL PBAN MODB
 ENGINEER J BARRY
 PROGRAM 365-40530-0-3441

SOP 16

SAMPLE NO. 344

SAMPLE NO. 345

WHT IN AIR

WHT IN AIR

WHT SAMPLE + WIRE 14.07707

WHT SAMPLE + WIRE 16.32805

WHT WIRE (-) 0.05189

WHT WIRE (-) 0.05189

WHT SAMPLE (W₁) 14.02518^v

WHT SAMPLE (W₁) 16.27616

WHT IN ISO-OCTANE

WHT IN ISO-OCTANE

WHT SAMPLE + WIRE 8.61398

WHT SAMPLE + WIRE 10.01620

WHT WIRE (-) 0.05189

WHT WIRE (-) 0.05189

WHT SAMPLE (W₂) 8.56209

WHT SAMPLE (W₂) 9.96431

TEMPERATURE OF ISO-OCTANE 24.4 °C

TEMPERATURE OF ISO-OCTANE 24.3 °C

DENSITY OF ISO-OCTANE (d) .6886 ✓ GM/CC

DENSITY OF ISO-OCTANE (d) .6887 GM/CC

PROPELLANT DENSITY

PROPELLANT DENSITY

$$1 - \frac{w_2 \left(\frac{8.56209}{14.02518} \right)}{d \left(\frac{.6886}{.6886} \right)}$$

$$1 - \frac{w_2 \left(\frac{9.96431}{16.27616} \right)}{d \left(\frac{.6887}{.6887} \right)}$$

= 1.767816 GM/CC

= 1.775928 GM/CC

X 0.03613 = 0.063871 LBS/IN²

X 0.03613 = 0.064164 LBS/IN²

0.064017

APPENDIX G
SOME SIGNIFICANT OBSERVATIONS

SOME SIGNIFICANT OBSERVATIONS

The detailed discussions, deliberations, consensus, and recommendations are presented in the bulk of this report. It is worthwhile to extract some of the more important comments made (or quoted) during the day:

"Unplanned variability of solid propellant properties is a widespread problem in propellant production." [Ref. 12, 2.2, page 11]

"Space Shuttle's fourth launch on June 27 caused concern among flight controllers when less-than-planned solid rocket booster performance created a depressed trajectory, lifting the vehicle lower than desired during first-stage flight." [Ref. 7, opening sentence]

"On the last flight, the propellant burn rate predicted was 0.366 in/sec. The actual burn rate was 0.359 in/sec., a difference that affected the trajectory significantly." [Ref. 8]

"We are not even sure what properties and processes need to be specified and controlled." [Professor Edward Price, April 21, 1989]

"Whenever we begin to think our combination of art and science are getting things under control, somebody changes the rules of the game by introducing a new ingredient that does not conform." [Professor Edward Price, letter of May 2, 1989]

". . . one has to obtain specimens and test them oneself in one's own lab." [Professor Martin Summerfield, letter of May 9, 1989]

(. . . for historical reasons) "Some solid propellants contain as many as 16 ingredients. This is entirely too many. . . . A reduction in materials would simplify the process." [Marshall Humphrey, letter of April 27, 1989]

"Solid rockets are handmade objects of art." [Robert Geisler, April 21, 1989]

"The legacy of black art in propellant manufacture persists today, and does not lend data to analyses." [Ref. 14]

"All propellants must be mixed first, then cast, then cured. Therefore fundamental is mixing and current emphasis (of the MSFC grant at University of Arizona) is correct." [Professor Clarke Hermance, April 21, 1989]

"The present theories for steady state burning are still very naive, and almost no research to improve this situation is in the works (funding is lower than at any time since the mid-1950's)." [Professor Edward Price, letter of May 2, 1989]

"Recent advances in high-technology devices and micro (submicron) miniaturizations provide a strong motivation for revisiting many of the unsolved problems in solid rockets." [Kumar Ramohalli, April 21, 1989]

"For a successful technology, reality must take precedence over public relations, for nature can not be fooled." [Richard Feynman, Ref. 18, last sentence]

APPENDIX H
INDUSTRY RESPONSE TO A FIRST DRAFT OF REPORT

[Note: These comments have been incorporated in this report.]

Atlantic Research Corporation

5945 Wellington Road
Gainesville, Virginia 22065
(703) 642-6033

Virginia Propulsion Division

James D. Martin
Vice President

September 15, 1989

Professor Kumar Ramohalli, MS 65
HSCA, Harvard University
60 Garden Street
Cambridge, MA 02138

Dear Kumar:

Thank you very much for the opportunity to review the draft of the report covering your April 21 meeting on the predictability of composite solid propellant. As I indicated in my telephone call, I believe you have made an excellent start on an approach to the solution of a problem whose extent is, as yet undefined; however, the perception that a problem does exist in the production of reliable batches of solid propellant is a nagging one which must be addressed in order to instill confidence into potential users.

As I also indicated, there are some portions of the report which I feel leave misconceptions as to the current state of the solid-propellant industry. The most disconcerting one deals with the role of and measurement of viscosity. Although you quite properly state that this characteristic is probably the single most important determinant for the ballistic behavior of production lots of composite solid propellants, you leave the impression that not much has been done to evaluate the viscoelastic characteristics of highly-loaded composites during processing. Our conversation clarified your understanding that this impression was not correct, and I believe that you will utilize your knowledge to remove this impression in the final meeting document.

Another suggestion I wish to offer concerns your finding no. 4 on p. 52. The solid-propellant industry has found it necessary to adjust propellant formulations to meet the variables associated with the wide range of mixers encountered during the development of a propellant formulation and its application to large motors. These adjustments are documented within company archives and their underlying principles are understood, viz., shear rate and its dependency upon mixer size, blade clearance, etc. Such adjustments are not merely "black art"; your proposed program will add to the data base existing for the scaling of propellant formulation, so its value cannot be questioned. Also, the program will not be hampered by the strictures of production schedules. In short, finding no. 4 should be restated! As is, it ignores the data base generated by the industry.

Some expansion of your findings on p. 22 as to the morphology of AP crystals is desirable. The effects of such morphology on propellant reproducibility are not to be questioned; it would be helpful if you could comment upon the cost increment associated with the utilization of the (more) uniform AP and the potential for increased reproducibility which utilization of this oxidizer represents.

Atlantic Research Corporation

As I also indicated, the use of the term "burn rate" offends me! I appreciated your concurrence in this attempt to employ proper usage of the English language despite the prevailing attempt to employ slang.

Once again, thank you for the opportunity to review the draft of your report. Other personnel at Atlantic Research who are involved in the fabrication of production lots of propellant have reviewed the draft and found it quite valuable. I look forward to hearing of your further work in this critical arena.

Very truly yours,

ATLANTIC RESEARCH CORPORATION

R. H. Woodward Waesche

R. H. W. Waesche
Principal Scientist, Technology

RHWW:sbt



Science Applications International Corporation
An Employee-Owned Company

September 27, 1989

Dr. Kumar Ramohalli
HSCA Infrared and Optical Division
Harvard University
60 Garden Street
Cambridge, MA 02138

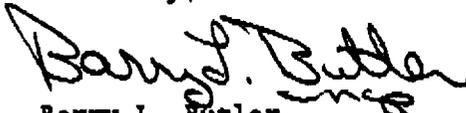
VIA FACSIMILE

Dear Kumar:

I appreciated the opportunity to review your report on (Composite) Solid Propellant Predictability and Quality Assurance. Since the propellant mechanical properties have a major affect on the propellant-to-insulator bondline properties, we in the NASA SPIP Bondline program are very interested in your initial data compilation on propellant mechanical properties and how they are affected by mixing and particle volume fraction. We are particularly interested in the measurable parameters monitored during mixing.

I have circulated your report to our team members and look forward to working with you to apply the results of your work to our program.

Sincerely,


Barry L. Butler
Program Manager

BLB/ncp

Kumar, (While you were out of the room) (prior to your leaving E Martin)

Dr. "McKay" (I think that's his name) came out very strongly in favor of Martin Summerfields recommendations that you do everything yourself - i.e. - have & use your own real propellant facility for at least small batches. Also rec'd. critiquing the work reports from other people, regarding large batches.

Ed Price

Big solids in future won't exist if reliability / reproducibility are not guaranteed. SRP's: don't need continuous maintenance (applies to skuttle) - must sell these to NASA, Forces

Impression given to group: 1% needed - others covered. Others need to cover needs

1%: Military - critical (-40 + 140°) hence large numbers in r are expected due to temp.

NASA - push 1% needed for space needs for testing duration, + starting.

Reproducibility / Definability / Reliability of components very important. Clarke

(2)

But must require more detailed specs on ingredients

Warren Dowling:

Proof of ingredient need. is desirable

Want - prop't that ignites & burns in a predictable fashion

All propellants must be used first

then cost

then cured

∴ fundamental is missing & current emphasis is correct

∴ However, more work on ^{combu} modeling, accounting for including more reality is necessary.



