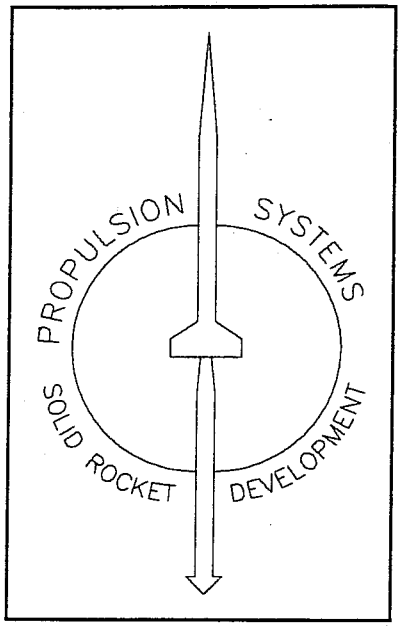
HOW TO FORMULATE AND PROCESS COMPOSITE PROPELLANTS



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Published by:

Propulsion Systems
P. O. Box 130077
Edmond, OK 73013

Introduction

This book is the culmination of more than 15 years of research and development with composite propellants. The methods described in this book and used in the SOPROC motor development program are based on the way most amateurs, not professionals, work to develop composite propellants. No thousand dollar test equipment is required to develop composite formulas for your own use when you use the testing methods described in this book in conjunction with the SOPROC motor development program.

HOW TO READ THIS BOOK: This book is designed to be read thoroughly from front to back. The first two chapters are an introduction. *Don't worry if the concepts in chapter two seem strange or that you don't understand them all at first.* Understanding even the simplified formulas used in this book can take time. The rest of the chapters are laid out in the sequence you will go through to process, cast, cure and work with a propellant.

The second book in this series, *How to Build High-Performance Composite Rocket Motors*, covers the construction of nozzles and the actual motor hardware. This second book is designed to be used in the construction of standard size rocket motors like those being sold commercially for use in the high-power hobby market at a fraction of the cost. In addition to these two books we recommend the use of the computer program SOPROC to design motors. This program is available from PSI and will greatly simplify your work!

Read and reread this book if necessary. Work safely, diligently and in the scientific tradition and you too will join "the club" of those of us who have successfully produced working composite rocket motors. By the time you understand the concepts in this book you can legitimately call yourself a "Rocket Scientist."

Feel free to write to PSI at the address on the cover with any questions you may have or to tell us results of your experiments. While we cannot promise a personal reply, selected letters and questions may be published in future booklets, in the catalog or in articles in the RRS or Tripoli journals.

The cost of this book represents the cost of the research and development effort that was undertaken to write it, not the publishing cost or number of pages.

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LEGAL DISCLAIMER

READ THIS NOTICE NOW !!!

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The development of rocket motors and the processing and handling of propellant can be extremely hazardous and dangerous under the best of professional working conditions. It is even more dangerous for persons unfamiliar with the handling of low explosives. Not all of the dangers and hazards can be foreseen. **READ THE SAFETY APPENDIX FOR GUIDELINES ON CONDUCTING YOUR WORK.** No person under the age of 21 years should attempt to use the information in this booklet without competent adult supervision. ***ANY PERSON WHO ATTEMPTS TO USE THE FORMULAS, PROCEDURES AND TECHNIQUES IN THIS BOOK ASSUMES ALL THE RISK OF THIS POTENTIALLY DANGEROUS ACTIVITY.***

The amateur worker may be governed by laws enforced by the DOT, ATF, and local and state Fire Marshals. ***It is your responsibility to make sure you are in compliance with any state or federal laws before you begin working!***

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1

SELECTION OF PROPELLANT

Development of Solid Propellants

Solid propellants have a very long history. Although the date is uncertain, the Chinese are believed to have made the first rockets, used in fireworks, from black powder. Black powder consists of potassium nitrate, charcoal, and sulfur in various proportions. It is rammed into the casing to form a grain. Obviously, this method places limitations on the size of the grain that can be formed and black powder is also of very low-performance and more hazardous to work with than modern propellants.

The next development in solids was not until the time of World War I. Researchers began using double-base propellants based on nitrocellulose and nitroglycerin. The grain was formed by allowing the nitroglycerin to dissolve the nitrocellulose and formed a solid matrix upon heating. Although some double-based propellants are in use today, particularly in their modified form with other oxidizers and fuels incorporated into the grain, they normally require working with liquid nitroglycerin and are very hazardous.

During World War II, Frank Parsons, an explosives chemist working with experimenters at the California Institute of Technology, came up with a propellant called GALCIT. The propellant incorporated crystalline potassium perchlorate in a mixture of molten asphalt and oil. The mixture was formed into a grain by pouring while hot and allowing to cool. Although there were numerous difficulties associated with this propellant, such as poor physical properties, unreliable ballistic performance, short shelf-life of the finished motor, and many explosions of the motors, the first crude composite propellant was born.

The basics for modern composite propellants were set. The propellants would consist of a solid oxidizer, usually a perchlorate, bound in a substance which was initially a liquid but, through some process, cured to a solid mass. An **oxidizer** is necessary in a rocket propellant because it must function in a chamber to produce gasses for propulsion. Even if the rocket remained in the atmosphere, oxygen would be necessary in the combustion chamber for the combustion reaction. The simplest method is to incorporate an oxygen releasing substance in the solid propellant.

The search began for better fuels which also acted as binders for the solid propellant. In 1946 at the Jet Propulsion laboratory, chemists incorporated perchlorate oxidizers into Thiokol LP-3 polysulfide polymer and cured it to a rubbery solid with p-quinone dioxime[1]. This was a major advance in propellant chemistry. With the use of chemically crosslinked binders, the propellant mix was a very thick semi-liquid mass which could be cast into any shape by "pouring" directly into the motor and then curing to form a rubbery solid. The propellant did not deliver a very high specific impulse, or power output, however.

Modern Composite Propellant Binders

Development efforts concentrated on finding a fuel/binder which would impart good physical properties to the cured grain while having a high fuel value. Numerous *polymers*, plastics and synthetic rubbers, were investigated. One of the earliest plastics used was unsaturated *polyester* cured in combination with *styrene*. In 1950, a state of the art propellant, developed by Aerojet General Corporation, consisted of a mixture of ammonium perchlorate, polyester, and styrene which was cured by cumene hydroperoxide[2]. Controlling the physical properties of the cured propellant was still a problem; however, as was the heat generated during the curing reaction.

Polyethers have been used in propellants and their availability in large quantities is good. They are low in viscosity which means that the uncured propellant is easy to mix and cast. The polyethers also cure at a good rate, but are not as high in fuel value as some other modern binders.

The search for high fuel values led chemists to work with several variations of *polybutadienes*. PBAA is a copolymer of butadiene with acrylic acid and was the first to be used. These propellants showed poor reproducability due to the method used in preparing the pre-polymer. A pre-polymer is a partially polymerized plastic which allows the final cure reaction to proceed more smoothly. The physical characteristics of the cured propellant were improved by using a terpolymer based on *butadiene, acrylonitrile, and acrylic acid*, which is referred to as PBAN. The mechanical properties of this propellant are very good and more tons of propellant have been made from this formulation than any other. Chemists also developed pre-polymers with functional groups, the reacting portion of the pre-polymer, in the terminal, or end, positions to improve physical properties. *Carboxyl-terminated polybutadienes*, or CTPB, have excellent physical properties and are used in motors where the mechanical properties of the propellant grain are of prime importance. While the potlife

(the amount of working time before the mixture solidifies) of the polybutadiene based propellants is excellent, conversely the cure times can be very long. They are all cured with epoxides or aziridines and some of these compounds are capable of dissolving or decomposing ammonium perchlorate. They generally have to be cured in ovens at elevated temperatures[1].

A completely different type of propellant is the PVC plastisol variety developed by Atlantic Research Corporation. No chemical reaction takes place during the formation of the solid propellant grain. A plasticizer, usually dibutyl sebacate or dioctyl adipate, is mixed with plastisol grade *poly(vinyl chloride)* and the mixture, with an oxidizer and several minor ingredients, is heated to form the solid plastisol. Potlife is excellent with these propellants, essentially being any amount of time desired since no chemical reaction takes place, only a physical solution process when heat is applied during the curing process. The solvation time to form the grain can also be quite short, consisting of the time it takes to raise the grain to the curing temperature. Unfortunately these propellants do require a very high solids loading, which is the percentage of solid ingredients in the uncured propellant mixture[3]. High solids loading can make a propellant mixture stiff and difficult to "pour" into a grain.

A very versatile group of binders are the *polyurethanes* which are based on cure reactions with *diisocyanates*. Although great care must be taken to exclude water from the chemicals forming the propellant (water reacts with the diisocynate), this class of binder has the advantage of relatively short cure times so that a number of propellant grains can be made in relatively little time using a minimum number of molds. With some polyurethane formulations room temperature cure is possible, eliminating the need for expensive ovens. The advantage of short cure time can also be a disadvantage; however, in that potlife can be very short.

Most recently chemists have developed a binder specifically for use in propellant manufacture. HTPB, or *hydroxyl-terminated polybutadienes* were developed by Aerojet General Corporation in the early 1960's. They were formulated to deliver high specific impulse, or fuel value, as well as good mechanical properties of the finished propellant grain. A variation of the polyurethanes mentioned previously, they can be cured with diisocyanates at room temperature or slightly higher and, with careful selection of the curing agent, provide adequate potlife at moderate working temperatures[1].

Polymers for Amateur Use

Several of the polymers discussed above have found their way into use by amateurs. The author has experimented with the polyester type, but found the potlife with available curatives to be far too short. Many people are using the PBAN pre-polymers, but these require careful selection of the curing agent. The HTPB system has many advantages for general amateur use. With the right diisocyanate, potlife is adequate and the grain can be cured at room temperature or slightly higher temperatures with heat lamps, usually in 48 hours or less. *Plasticizers* can be used which result in a "pourable" propellant mixture so that complicated injection casting is not necessary. The HTPB polymers also have the property of minimizing the combustion instability problems associated with developing a new propellant. For these reasons, propellants based on HTPB will be discussed in this book.

Oxidizers for Use in Composite Propellants

In addition to the polymers, which comprise the fuel for the rocket propellant, an oxidizer is necessary for the mixture to function as a propellant. An oxidizer, in this context, is a substance which releases oxygen in a chemical reaction.

Several crystalline oxidizers have been used in modern composite propellant formulations. Among them are *potassium perchlorate, lithium perchlorate, ammonium nitrate,* and *ammonium perchlorate.* Several high-energy oxidizers have been investigated including nitronium perchlorate and the hydrazine perchlorates. A decade of research went into the use of nitronium perchlorate in propellants because of the promise of very high specific impulses; however, no usable propellant has ever been formulated using this oxidizer. Some of these compounds are primary high-explosives and their use should be attempted only by persons having a degree in chemistry and experience in handling high-explosives.

Ammonium nitrate, AN, has been used in many propellant formulations. Advantages of AN include low cost, relatively clean exhaust, and for some applications, its low flame temperature is desirable. Its disadvantages include hygroscopicity (the tendency of some substances to absorb water from a humid atmosphere) and change of crystalline state with temperature. For many applications AN's relatively low rate of burn is also a disadvantage. The change of crystalline state means that the crystals occupy a different amount of space in the grain at different temperatures causing grain expansion and contraction problems. In the late 1980's,

phase stabilized ammonium nitrate was introduced overcoming this one problem. The hygroscopic nature of AN usually requires that the crystals be handled in a controlled atmosphere away from moisture and humidity. Additives are necessary to insure proper burning of the propellant.

The metallic **perchlorates of lithium and potassium** have been thoroughly investigated for use in propellants. Lithium perchlorate is extremely hygroscopic, and although it delivers relatively high-performance, its use in amateur propellants is limited because of handling difficulties and its high cost and low availability. **Potassium perchlorate, KP**, is not hygroscopic and does not form hydrates. Although it is relatively low in performance, it is cheap and readily available. Both of these perchlorates are not particularly hazardous to handle although all perchlorates present a fire hazard when in contact with organic matter. They may irritate the skin and mucous membranes requiring the use of gloves, face masks, and a respirator, especially when working with small particle sizes. Potassium perchlorate also has the advantage of forming solid particles (smoke) in the exhaust which makes the tracking of amateur rockets easier. The major disadvantage of KP is that the rate of burn of its compositions is extremely pressure sensitive. Its linear rate of burn is also too high for most core burning motor geometries but, it can be used successfully in end-burning applications (see the chapter on grain geometry).

Ammonium perchlorate, AP, is the most commonly used oxidizer for high-performance solid propellants. It is stable, non-hygroscopic, reasonably high-performing, and easy to handle. AP forms no hydrates, but should be stored in sealed containers. Most AP composite propellants have a moderate rate of burn compared to other oxidizers and its burn rate can be modified by varying the particle size and/or using burn rate catalysts to make it suitable for most any application. One disadvantage of AP is that it forms no smoke in fully oxidized formulations except under conditions of high humidity. However, this problem can be overcome by adding to the propellant formulation aluminum or magnesium metal in the form of a fine powder. Adding finely divided metal to this propellant also insures against serious problems with combustion instability or "chuffing". AP can be a strong irritant to the skin and mucous membranes, but this problem can be overcome by wearing gloves and, if necessary when working with small particle sizes, a dust mask. With reasonable care in storage and handling, it makes an excellent oxidizer for amateur use[4].

The use of ammonium and potassium perchlorates as oxidizers will be discussed in this book.

Appendix D

Notebook Forms

The forms on the following pages may be copied and filled out to keep as part of your records for potlife tests and processing batches of propellant. When performing the potlife test described in the testing chapter, it is important to keep a record of how long the mixture must be cured and at what temperature. Space for this is provided on the potlife test form. A typical batch for this test is one hundred grams so simply use the percentages in the formula as if they were grams when mixing a test batch. A similar form is included in book two for static testing motors.

2

COMPOSITE PROPELLANT BASICS

Introduction

If you have not read the introduction to this book, go back and read it before you go further. The concepts in this chapter, while simplified, may take a while to understand. It is not essential that you understand everything in this chapter to begin working. Your understanding of this material may become more thorough as you work.

Propellant Requirements

Rocket motors impart velocity to the vehicle by producing high-pressure hot gasses which are expanded and accelerated through a nozzle. To produce the gasses at high-pressure, the motor must be self-contained. The propellant must contain a fuel for energy and also supply an oxidizer for the combustion process.

Solid propellants have the added requirement that the finished propellant is a solid, referred to as the grain, of adequate mechanical strength to withstand the accelerations of rocket operation. Modern processing techniques also require the raw propellant mixture to be fluid enough to facilitate the mixing and casting of the grain. These requirements are met by using polymers, plastics and synthetic rubbers, as a binder to hold the solid ingredients, oxidizers and additives, in a solid matrix.

The first concern in **designing a composite propellant formulation** is usually the mechanical properties of the finished grain and, especially for amateur use, the fluidity of the raw propellant mix. Within these constraints the propellant chemist tries to create a formulation which gives the highest specific impulse. The required physical properties of the grain are imparted by using an adequately high percentage of binder ingredients, usually 12% or more of the total composition. The added amateur requirement of a pourable mixture usually means increasing the binder content to 20 to 25% of the total composition. Within this constraint it is still possible to achieve a theoretical specific impulse in the range of 210 to 240 lb-sec./lb. when using AP in the formulation. Actual delivered impulse will be somewhat lower because of combustion and motor inefficiencies.

When formulating a new propellant it is convenient to relate the isocyanate as a percentage of the total HTPB resin and crosslinker since the isocyanate chemically reacts with these ingredients. The plasticizer does not react with the other ingredients and is usually expressed as a percentage of the total binder content. The formulas in this book are expressed in percentages totalling 100% for ease of use but, can be reduced to the above form for workers formulating completely new propellants.

Basic Performance Relationships

Calculations of motor and propellant parameters are most conveniently carried out on a personal computer. All the calculations listed here can be performed by the SOPROC motor design software available from PSI; however, some understanding of the basic relationships is desirable.

Specific impulse, I_{sp} or I_s , is a measure of the energy which can be derived from a given unit mass of propellant. It is expressed by:

$$I_s = \frac{c}{g_0} = \frac{c^* C_F}{g_0} = \frac{I_t}{w}$$

where I_t is the total impulse of the rocket motor, **w** is the total mass of usable propellant, and g_o is the acceleration of gravity or 32.2 feet per second per second. The other symbols are explained below.

Effective exhaust velocity, c, is the average velocity at which the combustion products are ejected from the motor. It is expressed by:

$$c = c^* C_F = I_s g_0$$

Characteristic exhaust velocity, c*, is a theoretical value for a given propellant which may be determined from an analysis of the propellant energetics.

Thrust coefficient, C_F, is also a theoretically determined quantity which relates combustion pressures and nozzle expansion conditions to the propellant energetics.

Thrust, F, is usually expressed in pounds or newtons, and may be related to other parameters by:

$$F = C_F p_c A_t = \frac{m I_s}{g_0}$$

where $\mathbf{p_c}$ is the combustion chamber operating pressure, $\mathbf{A_t}$ is the area of the nozzle throat, and **m** is the mass flow rate of the burning propellant.

A very important parameter in solid propellants is the relationship of the area of the burning propellant, $\mathbf{A_b}$, to the area of the nozzle throat. This is called the **Kn ratio** or simply the K ratio. It is expressed as:

$$K_n = \frac{A_b}{A_t}$$

Kn is calculated from both theoretical values and the experimentally determined quantities of the density of the propellant and its rate of burn at specific combustion pressures.

The **nozzle expansion ratio, ϵ**, can be determined from the theoretical properties of a specific propellant. It is expressed by:

$$\epsilon = \frac{A_e}{A_t}$$

where $\mathbf{A_e}$ is the area of the nozzle exit and $\mathbf{A_t}$ is the area of the nozzle throat. Both quantities may be expressed in square inches.

Propellant Burn Rate

The burn rate of the propellant is one of the most important parameters in designing the motor and, unlike the thermodynamic properties of a new propellant, must be determined experimentally. The **factors which influence burn rate** of a particular formulation after oxidizer selection are: **(1) oxidizer particle size, (2) oxidizer percentage, (3) heat of combustion** of the binder and metal additive if any, **(4) particle size of the metal ingredient**, and **(5) additives** which modify the burn rate. As mentioned previously, the smaller the particle size of the oxidizer, the faster the burn rate.

Increasing the percentage of oxidizer increases the burn rate. A higher heat of combustion will increase the burn rate as will a smaller particle size of the metal ingredient. As mentioned in the first chapter, the oxidizer selection can also strongly influence burn rate; however, this data is meant to apply to a formula after the oxidizer has already been selected.

Ballistic additives may be added to the propellant formulation to control the rate at which the propellant burns. An increase in burn rate may be obtained by adding small percentages of ferric oxide, chromic oxide, copper oxide, or magnesium carbonate or oxide. Very small percentages of copper chromite can also increase the rate of burn substantially. There are also indications that copper chromite can reduce the pressure sensitivity of the burn rate[3].

A reduction in burn rate may be accomplished by the addition of calcium carbonate or oxamide to the propellant formulation[3]. Replacing part of the AP in the formulation with ammonium chloride has also been reported to reduce the rate of burn[7].

Testing for burn rate is usually done in strand burners and small-scale ballistic evaluation motors. The strand burners are useful for comparing propellant formulations and batch-to-batch consistency. Only a small motor firing can simulate the actual conditions of combustion closely enough to produce data useful for designing motors. Full-scale motors are occasionally instrumented and tested also.

Burning rate in the finished motor is also influenced by: **(1) combustion chamber pressure, (2) initial temperature of the propellant, (3) temperature of the combustion gases, (4) velocity of the gas flow** parallel to the surface of the burning propellant (called erosive burning), and **(5) in flight acceleration and spin factors**[6].

Rate of Burn and Pressure

Analytical models have yet to predict the rate of burn of a new propellant formulation. When plotted on log-log paper the rate of burn of most propellants using 70 to 80% AP in their formulation is essentially flat in the range of 200 to 1,500 psig. KP formulations with 60 to 80% oxidizer exhibit linear burn rates from 200 to 2,000 psig[3]. The pressures in amateur motors are typically between 300 and 1,000 psig. The burn rate versus pressure relation may be expressed mathematically as:

$$r_b = ap_c^n$$

where r_b is the burn rate usually expressed in inches per second, p_c is the chamber pressure expressed in psig, **a** is an empirical constant which depends on ambient grain temperature called the temperature coefficient of burn rate, and **n** is the burn rate pressure exponent. SOPROC will perform this calculation for you. The pressure exponent is essentially independent of grain temperature and shows the effect of chamber pressure on the rate of burn. Low values of n are highly desirable. Some propellants exhibit a phenomenon known as plateau burning meaning that they have a nearly constant rate of burn over a limited pressure range[6].

Rate of Burn and Temperature

The rates of chemical reactions are influenced by the temperature at which they are carried out. The variation in propellant performance with temperature is termed the temperature sensitivity of the propellant. Motors fired at high temperatures will have a shorter burn time and higher thrust and combustion chamber pressure than motors fired at lower temperatures. In practical terms this means that if motors are designed and tested at near freezing temperatures then taken to the desert for launch they may well CATO!

Erosive Burning

In some motors, the velocity of the combustion gasses over the burning surface of the propellant grain can significantly increase the rate of propellant burning. This primarily occurs in core burning geometries with a ratio of area of port to area of throat of 4:1 or less although some erosive burning may occur at ratios as high as 6:1 (see the chapter on grain geometry for an explanation of core and end-burning grain geometries). Erosive burning appears to function by increasing the heat transferred to the propellant surface.

Erosive burning increases the mass flow rate of the propellant and so increases the chamber pressure in the early part of the burning. Erosive burning is most severe at the nozzle end of the motor and can be controlled by making allowances in nozzle and grain design[6]. Motors designed without consideration to erosive effects may CATO. The SOPROC motor design software designed to go with this book can warn

against most types of commonly encountered erosive burning effects and help you to solve them.

Effect of Acceleration on Burn Rate

High g forces in a moving rocket can seriously affect the burn rate of composite propellants. Acceleration forces induced by spinning the rocket to a high rate of speed or in very high thrust-to-weight ratio rockets can significantly increase the rate of burn. Since these effects are primarily seen at accelerations not encountered in high-powered rocketry, they are not discussed further here. See reference 6 for a treatment of this phenomenon.

Introduction to Composite Propellant Processing

The processing of composite solid propellants involves the preparation of the ingredients, mixing, casting the liquid mixture, and curing to form a solid grain. The grain formed must be free from air bubbles and voids and of a consistent density. This requires vacuum processing at either the mixing or casting stage or sometimes as a separate degassing step. The author has known many experimenters, including himself, who have tried to cast grains without using vacuum, but no reliable, repeatable results have ever been obtained in this manner.

Vacuum processing requires the use of a good vacuum pump and these can be expensive. The author obtained the pump used in the development of formulas for this book for free from a heating and air conditioning firm which was discarding an old pump. An investment of approximately $35 in parts, some new pump oil, and a small amount of labor produced a very good working vacuum pump. When looking for a used pump, find one that was originally capable of pulling a vacuum of 29 to 30 inches of mercury or 1 inch of absolute pressure (less than 26mm). The better the original specs were on the pump, the more likely it is that it can be restored for use in propellant processing. A vacuum gage should be used to test the pump after it is repaired. New vacuum pumps with the above specifications start at about $500 dollars and, unless a lathe is purchased for machining nozzles, this will represent the largest single outlay for the equipment necessary to make composite motors. Resourcefulness in obtaining a vacuum pump can mean a major savings in start up costs for producing composite rocket motors.

3

EQUIPMENT FOR COMPOSITE PROPELLANT PROCESSING

Introduction

This chapter describes the various equipment needed to process propellant and make finished grains by the techniques set out in this book. The equipment used to make nozzles and finished motors is described in book two in this series.

The Workshop

You will obviously need a place to work. This may be simply a garage or work area but you will need space for one or two **workbenches** and tools. Allow room for storage cabinets to keep chemicals and motor hardware supplies in. Try to allow room for expansion if possible. Making your own rocket motors and successfully firing them can be addictive!

LEGAL CONSIDERATIONS: At the time of this publication, Tripoli was working with the ATF (Bureau of Alcohol, Tobacco, and Firearms) to allow Tripoli members to legally possess and store up to 50 pounds of raw propellant and finished motors. Contact Tripoli for the latest information. Whether or not you need clearance from the ATF to process propellant and make your own motors, you may still need permission from local and state fire authorities to work. Check with these agencies before setting up your workshop. Most Tripoli prefects are organizing bunkers to meet ATF guidelines for storing finished motors.

Safety Equipment

Safety equipment for handling propellants and chemicals need not be expensive. At the least you should have **rubber gloves,** a **face shield** or other eye protection, and a protective **apron** when working with the degasser and handling chemicals. While no accidents have occurred with the degasser described below, there is always the chance that an apparatus under vacuum could implode. Although implosion is the opposite of explosion, the results can be just as dangerous!

A **dust mask or respirator** should also be worn when working with small particle size solids, such as metal powders

and fine oxidizer grades. Work conscientiously and with an eye to safety at all times.

A **metal storage cabinet** is a worth while investment in which to keep oxidizers and finished grains and motors. Also, if you will be machining grains, it is very important to remove the fall off or shavings from the work area at frequent intervals. While the actual grain may be difficult to ignite, *propellant shavings are highly flammable and may start an intense fire from a stray spark. Always keep a* ***fire extinguisher*** *of adequate capacity near when machining cured propellant grains.* Keep all propellant shavings in a **sealable plastic jar** and dispose of in accordance with the procedure in the safety appendix.

Propellant Processing Equipment

Obtain a good **laboratory balance** accurate down to 0.1 grams for working with the size of batch mixed in the standard degasser. Ideally, a balance with a tare poise weight (a device that allows for the weight of the container in which you are weighing chemicals) should be obtained and an accessory weight set may be desired for large batches of propellant. These are expensive and you can certainly get by with a less expensive model but, they will make your work much easier.

Use only **waxed paper cups** to weigh individual liquid chemicals in, never plastic. Some of these chemicals will attack plastic cups. Oxidizers and other solid chemicals can be weighed in **plastic cups**. Also, a good supply of **paper towels** will be needed for processing propellant.

A **vacuum pump** is required for degassing the raw propellant mixture and should be capable of pulling the degasser to as low a pressure as possible (about 29 to 30 inches of mercury). Good laboratory vacuum pumps for this purpose start at about $500. However, like the author, you may be able to find a **used pump** from an air conditioning firm or university laboratory for very little cash outlay. The author obtained a pump in this way that was being discarded and invested $35 dollars in parts to obtain a very good working vacuum pump. Change the **pump oil** frequently to maintain the full vacuum capability of the pump. There are vacuum pumps used in the plastics industry (for vacuum bagging) which can be purchased new for as little as $110. It is possible they may pull a low enough vacuum to provide adequate degassing but we have not tested these pumps. The vacuum used in the degassing of formulas for this book was

approximately 10mm absolute pressure or more than 29 inches of vacuum.

You will also need several **vacuum accessories**. See the vacuum accessories photograph in the photographs appendix. The flask is an Erlenmeyer type designed for use in vacuum filtration. It functions to form a trap to prevent any propellant from being sucked into the vacuum line. Unless your pump has a vacuum gauge on it, you will need to mount one in line on the vacuum accessories board. Use a standard pipe "T" for this connection. The valve pictured is very important. While a standard straight valve will work, the three way type pictured will make your work a lot easier. *Your set up does not have to be this elaborate* but, at the very least, you need a trap to keep propellant foam from being sucked into the vacuum pump and a valve of some kind. You will need several feet of thick walled **vacuum hose** to make the connections. The standard degasser is designed to take 1/4 inch I.D. vacuum tubing. Ideally, the vacuum board should be mounted to the wall next to the work bench where you will process propellant, NOT on the bench itself because of vibration from the **vibrator** used to settle the propellant.

The **degasser** itself is also shown in figure 7 in the drawing appendix. It is the simplest and least expensive way to vacuum process composite propellant mixtures. The top of the degasser is the removable part and consist of two circles of 1/4 inch thick polycarbonate sheet bolted together. The smaller inner circle is cut to just fit inside the tubing of the degasser body. The bolts are sealed on the top of the degasser with silicone rubber sealant. The polycarbonate circles are cut from sheet material with a wood circle cutter available at most hardware stores. Note the brass tubing in the center to accept the vacuum line. The tube making the body of the degasser is a 12 inch length of 4 1/2 inch outside diameter clear cast acrylic tubing, 3/16 of an inch thick. The base circle of polycarbonate is epoxied to the bottom of the tubing. The seal shown is cut from thin rubber sheet with an exacto knife using the tubing as a guide.

The size shown is considered standard but, larger degassers can certainly be made to handle more than the 700 gram capacity of the standard one. When designing a larger model keep in mind the important thing in degassing the propellant is to expose as much raw propellant mixture to the vacuum as possible in a short time. The surface area of the inside of the tube used will give a good estimate of the capacity of the one you are planning by comparison to the standard size. Thicker tubing and polycarbonate sheet may be needed in larger size degassers to withstand the vacuum.

WARNING: When working with any apparatus under vacuum there is always the danger of implosion. *Wear safety equipment to protect yourself!*

Grain Production Tools

If you will be machining the cured grain (refer to the chapter on grain construction) you will need several tools. A **miter box and saw** are essential for cutting the cured gain into individual BATES grains. This can be as simple as the plastic miter boxes sold at hardware stores for a few dollars. It is important to be accurate with the tool if you will be using a **drill press or drill stand** to cut the center grain perforation. A lathe is the most accurate way to cut the center core on BATES grains; however, the drill stands sold for use with hand drills do have some advantages. They usually come with an attachment for holding dowels or other cylindrical pieces in alignment with the drill bit. This is exactly what is needed to cut the center core of the grains.

The Notebook

This piece of "equipment" is important enough to deserve its own section in this chapter. Get a ringed binder and ruled paper to keep notes on every batch of propellant you produce. Record all the process variables discussed in the chapter on processing and the date you process the batch. Suitable forms can be found in appendix D. Record your observations and techniques in as much detail as possible. Small changes in formulas and techniques can have a major impact on the way the finished rocket motor performs.

You should also record each new motor design you try. Static test results such as burn time or time of motor CATO into burn should be recorded if you are to make progress. An analysis of the time into the burn that a motor fails, either from chuffing or explosion, can be compared with SOPROC thrust time data and yield the answer for the next successful motor. Forms for recording static tests are included in book two.

SOLID PROPELLANT GRAIN DESIGN AND CONSTRUCTION

Introduction

There are two basic types of grain design: (1) **end burning grains** in which the propellant burns from one end of the grain to the other much like a cigarette burns, and (2) **port burning grains** which have a central cavity where the propellant is ignited and burns from the central core to the outside of the cylindrical grain. Basically, a propellant grain will burn on all surfaces where the propellant is exposed and not inhibited. Although cylindrical grains are not the only type, they are the simplest for amateur construction. Cylindrical grains will be discussed here.

There are two basic methods of loading the propellant into the rocket motor. **Cartridge loaded grains** are made by casting or extruding the propellant into the shape of the desired grain. They are cured, machined, and then loaded into the motor. **Case bonded grains** are made by casting the propellant directly into the motor casing. A liner is used to promote bonding of the cured propellant to the casing wall. Because of their ease of production and versatility, cartridge loaded grains will be discussed in this book.

Common Terms

The configuration of the grain refers to the shape or geometry of the initial burning surface and determines to a large extent the shape of the thrust-time curve.

Burn time and thrust are described by relation to neutral burning. A **neutral burning** grain is one that produces a steady state of thrust and pressure over the entire burn time of the motor. **Progressive** grains show an increase in pressure and thrust over the burn time and **regressive** grains start with high thrust and pressure and reduce these parameters as burn time progresses. The neutral grain is desirable for maximizing the energy contained in the propellant since specific impulse degrades as combustion pressure is lowered from maximum values. The regressive grain is desirable when high thrust is required to boost a fin-stabilized rocket quickly to stabilizing velocities. Progressive grains show

poorer performance than the other two types when applied to fin-stabilized vehicles operating within the atmosphere.

Effective burning time, t_b, is the interval between the time that 10% of maximum pressure is attained and web burnout. **Action time, t_a ,** is the time between the initial and final points of 10% of maximum thrust or pressure on the thrust (or pressure) time curve.

Web thickness, b, is the minimum thickness of the grain taken from the initial burning surface to the point where the grain burns out. **Web fraction, b_f,** is the ratio of the web thickness to radius of the grain or:

$$b_f = \frac{b}{radius} = \frac{2b}{diameter}$$

Volummetric loading fraction, V_f, is an important design consideration with high values being desired in high-performance motors. It is the ratio of **propellant volume, V_b, to the chamber volume, V_c,** which is available for propellant, insulation and liners. It is expressed by:

$$V_f = \frac{V_b}{V_c} = \frac{I_t}{I_s \rho g_0 V_c}$$

where ρ is the propellant density. The other symbols were discussed in chapter 2. To a large extent, the selection of the grain geometry determines this critical performance parameter [6].

Common Grain Geometries

End burning grains are very desirable from the view point of volummetric loading fraction. They do, however, introduce the need for sometimes thick and heavy insulation to protect the motor walls from the hot combustion gasses. The **star burning grain** provides maximum protection for the walls of the motor and minimal insulation is needed if the grain is designed properly.

The internal burning tube must be segmented, also called a **Bates grain**, to provide a nearly neutral burn time curve for most applications. By machining the ends of a Bates grain to

a cone shape, it is possible to maintain combustion pressure within 4% of neutral burning.

C-slot grains provide a high initial grain surface for maximum acceleration of fin-stabilized rockets. However, both the C-slot and the **moon-burner geometries** expose the motor case to the combustion gases for most of the burn time and may require added insulation or heavy motor walls.

Grain Geometry for Amateur Use

End-burning and Bates grain geometries are shown in figures 1 through 4 in the drawings appendix. As mentioned, the end-burning grains may require the use of heavy insulation and metal wire technology is needed to attain high thrust-to-weight ratios. Star grains require the machining of expensive mandrels on a milling machine. The C-slots and moon-burners may require heavier than normal cases or insulation.

The simplest grains for amateurs to produce are the Bates and end-burning types. Bates grains keep exposure of the motor case to the combustion flame to a minimum and, because of their ease of construction and versatility, will be discussed first.

The most neutral burning Bates grains are obtained by having a length to diameter ratio of 1.64 to 1. The core should have a minimum diameter of one-third the grain diameter for most motors. Increasing the core diameter and/or lengthening the grain may be necessary in motors that show erosive burning.

Construction of Bates Grains

There are two common methods for producing Bates grains: (1) **cast the propellant in a cylinder and then machine the grain**, and (2) **cast the individual grain segments using a mandrel.** Both methods have advantages and disadvantages. Machining the grain is the only practical method for small grain sizes while casting individual segments allows larger motors to be made from a given mixer or degasser size, saves propellant since none is lost through machining, and is the safest method.

WARNING: *Machining cured propellant is dangerous and great care must be taken to clean up the shavings. Never cut the grain with power tools, such as radial arm saws. The motors and moving parts may become clogged with propellant, a very dangerous situation!*

To use either method, a mold is constructed from the same tubing used in making the motor casing or the liner for the grain in a reloadable motor is used. The mold is used to shape a piece of inhibited liner made from 60 lb. Kraft paper of appropriate size and coated twice with inhibitor (see the formulas chapter).

To make the liner for either method:

(1) Coat one side of a sheet of waxed paper with mold release compound and tape the coated side down to a flat smooth surface (Formica works well for this purpose). The sheet should be stretched smooth and free of wrinkles.

(2) Cut a sheet of Kraft 60 lb. wrapping paper slightly smaller than the sheet of waxed paper. Place the paper, curl side up, on newspaper and brush on the liner/inhibitor formula listed in the formulas chapter.

(3) Press the coated side of the Kraft paper down on top of the waxed paper and smooth out any bubbles or voids with a hand roller. Suitable rollers of the type used for pressing Formica counter top material are available at most hardware stores. Allow the material to cure overnight.

(4) Brush on a second coat on top of the Kraft paper and allow it to cure for an additional 24 hours or until it is only slightly tacky to the touch.

(5) Pull up the coated liner sheet (the waxed paper will now be stuck to the Kraft paper) and remove as much mold release as possible from the waxed paper side with a paper towel damp with rubbing alcohol. When dry, wrap the liner sheet around a dowel slightly smaller than the I.D. of the motor the grain will be used in. Use waxed paper again, applied to the coated side of the liner paper, to keep the liner from sticking to itself while it finishes curing. The original waxed paper laid down under the Kraft paper should stick to the back of the liner and this facilitates loading the grain into the motor. Wrapping the liner around the dowel while it finishes curing puts a curl on it which helps it to fit snugly in the mold tubing.

(6) Allow the liner to finish curing for at least another 48 hours.

NOTE: Brushes used for coating the liner may be cleaned by working the brush in mineral oil, wiping the mineral oil out on a paper towel, and then working the brush in acetone. The roller can usually be cleaned by wiping with a dry paper towel.

The **base of the mold** is made from a block of wood approximately one inch thick (see figure 5 in the drawing appendix). Cut the wood into a square somewhat larger than the O.D. of the motor casing you will use. Find the center of the square and cut a circle at least one-eighth inch deep and the same diameter as the inside of the motor tubing into the wood with an adjustable circle cutter. The motor tubing will be used to form the grain and should fit tightly into this circular cut. The inner circle of the base will come into contact with the raw propellant and should be coated with a thin layer of common canning wax to prevent adhesion of the cured grain to the mold.

It is important to adjust the circle cutter accurately. Use a piece of 1 inch scrap wood and start with a diameter of cut slightly larger than the inside diameter of the motor casing. Be certain the cutting bit is turned properly with the point of the cutter on the inside of the cut. Keep adjusting the tool to slightly smaller diameter cuts until the motor casing just fits into the cut. SAVE THIS PIECE OF WOOD. Use it to adjust the circle cutter the next time you need to make this size of circle. Simply lower the bit of the tool into the center hole and adjust the tool arm until the cutter fits snugly in the circular cut.

To Cast Stick Grains:

(1) Cut the liner sheet into a rectangle as long as the motor tubing used as a mold and slightly wider than the inside circumference of the tube.

(2) Curl the liner long wise and insert into the tube. Allow the liner to uncurl to match the inside of the tubing.

(3) Mount the liner and tubing on a large dowel slightly smaller than the I.D. of the tubing. Hold the dowel in a vice. Slowly pull the tube off while taping the seam of the liner together with a single piece of masking tape. The liner tube, now finished, should fit **snugly** into the motor tubing used as a mold.

(4) Reinsert the liner into the mold tube and position on the mold base (this will move the liner up in the tube to allow insertion of the mold base). Twist the tubing, with the liner inside, off the base and apply a thin coat of silicone rubber sealant to the area between the bottom of the tube and the liner. Also, apply this sealant to the tube and the liner at the top of the mold (the liner should now stick up above the tubing by 1/8 of an inch). The purpose of this sealant is to prevent raw propellant from getting between the liner and the mold tubing and making it impossible to remove the cured grain

from the mold. This seal will be broken with an Exacto knife after curing the propellant.

(5) Before the sealant sets up, press the tube and liner into the base and attach with duct tape so that it does not come lose during casting and vibrating. See photograph 3 in appendix F for an illustration of this. Do not use masking tape, it will not hold!

(6) After the silicone sealant has cured for at least two hours, process the propellant and cast it into the mold as described in the processing chapter.

To Cast Individual BATES Grains:

Use the same mold set up as above but, insert a dowel into the center of the base (see figure 6 in the drawings appendix). The dowel should be the same size as the core of the grain you want to make and at least as long as the grain or longer. Of course, the dowel will come into contact with the raw propellant so it too must be coated with canning wax. The motor tubing should be the same length as the grain you want to cast plus the depth of the cut in the mold base. Make the liner sheet into a tube and seal it with silicone sealant as described above.

To make a mold for high-temperature curing (above room temperature), the core mandrel should be made from Teflon rod stock or, in large diameters, thick walled Teflon tubing. A base may be cut from Teflon sheet stock with the adjustable circle cutter to fit on top of the wooden base. The Teflon parts are coated with mold release compound for added insurance against sticking. We have **not** found this step to be necessary when casting sticks by the above method, only when making single grains where the outer surface of the grain must be smooth when released from the mold.

After Curing:

(1) The mold casing is twisted off the base and the rubber seal at both ends is slit with an Exacto knife. The grain is ejected from the casing with a large dowel. *Care must be taken not to deform the grain during this process.* The grains cast as individual segments are now ready for step 4 below.

(2) Grains which must be machined are cut to length with a hand-powered miter box saw. Once again, *DO NOT USE POWER SAWS TO CUT THE GRAIN!!!*

(3) The cut grains are now mounted in a drill press or drill stand where the central perforation is drilled out. The grain should be held in place with a vertical dowel holder or similar device to insure that the core is parallel with the outer sides of the grain cylinder. Most drill stands, for hand held drills, come with an attachment for this purpose. The core must be closely centered in the cylinder forming the grain. Use a guide to center the core cut in the grain cylinder. Guides for this purpose may be cut from thin birch plywood with an adjustable circle cutter. The most accurate way to drill the center core is to use a machine lathe but, this is really not necessary.

(4) IMPORTANT: Remove the masking tape which held the liner together and build up the indented side of the liner tube with masking tape. Half inch masking tape works well for this purpose. Finish with 3/4 inch masking tape across the seam. The grain should now fit snugly in the motor casing with no gaps. This step keeps the flame from traveling up the void that would have been left between the grain and the motor wall.

It should be emphasized that machining grains is dangerous. The author strongly urges persons starting out to use an inert propellant simulant formula while gaining experience. *Clean up and dispose of shavings and fall off at regular intervals and especially after you are through working. Read the safety appendix before starting to work.*

Producing End-Burners

End-burners are also produced by the stick method described above. Be certain to cast the stick as long or longer than the size of the finished grain. The grain is finished after being cut to length. To make an **augmented end-burner** for higher lift off thrust, the X-form is cut into the end of the grain where ignition will occur. Use a hand saw of appropriate width across the teeth to get the width of cut you need. A guide for cutting the X-form can be made from scrap tubing of the type used to make the mold. *Do not cut the X-form too deep or over pressurization of the motor may occur.*

For both types, follow step 4 above using masking tape to fill the seam where the liner sheet was held together. If you do not your motors may CATO!!!

Erosive Burning and Grain Design

Erosive burning is, to an extent, determined by the grain geometry and the relation of the port area (core area) to the throat area of the nozzle. Erosive burning may be minimized by keeping the port area at least 4 times the throat area; however, this is not always feasible. Erosive burning is believed to be caused by the heat transfer effect of the moving gasses over the surface of the unburned propellant in core burners. Erosive burning is not an issue in end-burners.

With Bates grains, there are two primary ways to minimize the effects of erosive burning: (1) the core diameter can be increased; however, this cuts down on the volummetric loading factor of the grain and gives poorer performance, (2) the length of the grain may be increased, to obtain a lower initial burning surface, while decreasing the number of grains, if necessary, to hold a target total impulse value. This method may be effective up to the first incremental increase in grain length where SOPROC shows the peak thrust to occur at motor burn out.

A third alternative is to increase the area of the nozzle throat to accommodate the increased rate of burn. This works with grain geometries other than Bates. The SOPROC motor development software designed to accompany this book can help you minimize the effects of erosive burning in most motors; however, the mathematical analysis of erosive burning has not progressed to the point of being an exact science. *Motors which are flagged by SOPROC as being candidates for erosive burning should always be static tested and treated as though they may CATO upon ignition or at some time in their burn.*

5

COMPOSITE PROPELLANT FORMULAS

Formulating HTPB Propellants

An introduction to this subject was given in chapter 2, *Composite Propellant Basics*. You may want to review this information before proceeding. The formulas given in this chapter are meant to serve as guides. You may use them as they are or follow the information listed under each formula to tailor your own formula for your specific needs.

Oxidizers

The oxidizer for a composite propellant usually consists of a crystalline inorganic perchlorate. Recall that an oxidizer is necessary in the propellant to supply free oxygen for the combustion process. The perchlorates release oxygen when heated, especially in the presence of a fuel such as the binder or a powdered metal.

Ammonium perchlorate (AP) is the most widely used oxidizer in composite propellants. While the oxygen content of AP is not as high as some other perchlorates, it is desirable because of its ability to produce large volummes of hot gas. AP is supplied in ordinance grades of 400, 200, 90, and 55 microns. While the larger particle sizes may be handled with relative safety, smaller particle sizes (55 microns or below) should not be used by amateurs because of their ease of flammability and explosive nature. AP has an oxygen content of 54.5% by weight, a molecular weight of 117.5, and a density of 1.95 g/cm^3 [4].

Potassium perchlorate (KP) does not offer the high performance of AP but, is desirable because of its availability, exhaust properties, and the fact that it is safer to use in machined grains. There appears to be some confusion in the literature about the burn rate of KP propellants. KP is sometimes listed as having a low burn rate but, as Rumbel has shown in his work on PVC plastisol propellants, at standard conditions of burning (1000 psig) KP propellants typically burn faster than propellants containing AP. Actually, the burn rate sensitivity of KP to pressure (i.e., the pressure exponent, n) is greater than that of AP meaning that at low pressures KP formulations burn relatively

slowly and at high pressures they burn much more rapidly [3]. KP has an oxygen content of 46.1 weight percent, a molecular weight of 138.6, and a density of 2.53 g/cm^3 [4].

Both of the perchlorates should be obtained in a low moisture content form. Storage under humid conditions can introduce moisture into the powder and they should be stored in sealed containers.

All the perchlorate oxidizers produce hydrochloric acid in their exhaust and care must be taken to avoid human or mechanical contact with this corrosive substance.

The **particle size of the oxidizer** has a prime influence on the burn rate of the finished propellant. The range of particle sizes of the solid components of the propellant mix also influences the viscosity of the raw propellant. An ordinary flour sifter should be used to eliminate clumps in the 90 micron AP and finely powdered KP before use. Clean the sifter with distilled water and dry after use to prevent clogging of the screen which could change the particle size of the oxidizer you are using.

The oxidizers should be handled with care. Contact of perchlorates with organic matter must be avoided and spills should be cleaned up immediately. Safety glasses, an apron, and gloves should be worn when working with the perchlorates. Fireproof clothing is desirable; however, lacking this, clothing worn during the handling of the perchlorates should be washed immediately to prevent a fire hazard.

The perchlorates should be stored in sealed containers away from heat and humidity.

Metal Fuels

The addition of powdered metals to the propellant formulation can theoretically increase the specific impulse by increasing the heat of combustion. The more heat that is generated in combustion the more the working gases are expanded and the greater the thrust obtained from a unit mass of propellant. Burning metals in small motors is not highly efficient; however, as the metal does not have time to burn completely before being ejected through the nozzle. The addition of metal does prevent severe problems with combustion instability and, for amateurs, has the advantage of generating smoke in formulations using AP as the oxidizer.

The most commonly used metals are magnesium and aluminum. **Aluminum** appears to be more desirable since it has a higher

energy content than magnesium. Aluminum is also less expensive than magnesium [2]. The addition of aluminum to a propellant formulation can increase the rate of burn. Burn rate is further affected by the particle size of the metal with higher burn rates seen at lower particle sizes. Smaller particle sizes will also increase the combustion efficiency of the metal. Since addition of a metal to a propellant formulation with a fixed binder requirement means decreasing the amount of AP, it should be understood that the metal burns by reacting with the steam generated by the propellant combustion process, not with free oxygen from the oxidizer. There is usually no reason to add metal to a KP formulation since KP already has a relatively high heat of combustion and forms solid particles in the exhaust to provide smoke for tracking.

Powdered metals should be handled with care. Avoid the formation of airborne dust, since any airborne dust can be an explosion hazard. Like other ingredients, metal powders should be kept dry in sealed containers.

WARNING: *When using a motor to mix the propellant, care must be taken to insure that no dust is formed which could be ignited by a spark. Follow the instructions in the mixing chapter to prevent an airborne dust hazard.*

Binder Ingredients

The prepolymer starts out as a liquid which gives a reasonably fluid mixture of the ingredients. It is then cured to the solid state by the addition of a curative which promotes final polymerization. HTPB based propellants consist of the crystalline oxidizer, a powdered metal fuel, if used, other minor solid ingredients, such as cure catalysts and ballistic modifiers, and the binder ingredients consisting of a prepolymer, plasticizer, crosslinking agent, and one of the diisocyanates as a curative. A bonding agent is sometimes added to impart the desired mechanical properties to the finished grain.

A **prepolymer** is a relatively low molecular weight substance capable of undergoing further polymerization [5]. Prepolymers are used in producing solid propellant grains instead of the original monomer to overcome the problem of exotherm during curing. Exotherm would result in severe problems of grain expansion during cure, making case bonded grain technology very difficult to use. A minimum change in the volume of the propellant is desired from mixing to the formation of the solid grain. Common HTPB prepolymers are R45

and R45M which contains an antioxidant to prevent aging problems.

Plasticizers are useful in reducing the viscosity, or increasing the fluidity, of the raw propellant mixture and also impart desirable low-temperature physical properties to the finished grain. In working with the uncured propellant mixture, a relatively low viscosity is desired in order to simplify mixing and casting operations. Prepolymers tend to be rather viscous and a very low viscosity plasticizer is desirable to help reduce the viscosity of the raw propellant. Common plasticizers for composite propellants include *di-2-ethylhexyl adipate*, commonly called dioctyl adipate or DOA, *dioctyl sebacate*, and *isodecyl pelargonate* [1]. An additional requirement is that the plasticizer must not boil off during vacuum processing of the propellant mix. The plasticizer does not chemically react with the other ingredients during the curing process but, imparts its desirable physical properties to the finished grain by virtue of its physical state.

The **crosslinking agent** also imparts desirable physical properties to the finished grain. By reacting with the isocyanate, the crosslinker adds chemical bonds to the side of the polymer chain increasing its resistance to tear or shear. Common crosslinkers for polyurethane prepolymers include low molecular weight alcohols, polyethers, and in particular, glycerol mono and triricinoleate[1]. Glycerol triricinoleate is commonly known as *castor oil* and works quite well with HTPB compositions.

The **diisocyanate** chemically reacts with the prepolymer and crosslinker to effect cure to the final solid state. Common isocyanates for polyurethanes are 2,4-tolylene diisocyanate, TDI, and hexamethylene diisocyanate, or HDI [1]. These compounds are not very suitable for amateur use; however, since both of these agents are excessively toxic and HDI is a lachrymator as well. Better choices for HTPB are the PAPI isocyanates produced by Dow Chemical Company, DDI-1410, and IPDI or isophorone diisocyanate. Of the PAPI isocyanates, *PAPI-2901 or diphenylmethane diisocyanate* is especially useful for fast cure times and produces a grain which is easily machined. This isocyanate is sold by Fire Fox under the name *Isonate 145-L*. IPDI and DDI-1410 are useful as slower acting curatives. These isocyanates may be mixed in varying ratios with PAPI-2901 to obtain moderate cure times. All of the diisocyanates are toxic and should be handled with extreme care.

It should be noted that the isocyanates react with water forming carbon dioxide, a very undesirable gas, which may cause voids in the finished grain and possible motor failure.

Great care must be taken to insure that the propellant ingredients are dry and to protect them from moisture and humid conditions in storage and handling. All isocyanates are unstable and should be stored at controlled room temperature.

Bonding agents are sometimes used to insure that the solid AP particles are in close contact with the polymer. We have not found this physical bonding to be very important in small motors, however. The bonding agent also introduces a phenomenon known as "dry mix," meaning that the raw propellant mixture behaves like a powder or very thick dough until the isocyanate is added. Even after the addition of the isocyanate these mixtures may be very viscous. This complicates the mixing and casting operations and for this reason bonding agents are conveniently left out of compositions for small motors.

An **opacifier** is sometimes used in propellants that do not contain a "dark" ingredient. The opacifier is necessary to reduce the transmission of light and heat through the propellant grain which could cause irregular burning. Formulas containing sufficient amounts of powdered metals or copper salts do not require an opacifier since these ingredients make the propellant opaque to light. Carbon black or lampblack are commonly used as opacifiers.

An **antifoam agent** is particularly useful when vacuum processing with a degasser. PDS-8R111 is a silicone oil sold by Xerox Corporation for use in copiers and laser printers as fuser oil. It can be obtained in pint sizes directly from Xerox. The addition of as little as 0.10% PDS-8R111 to the formula significantly reduces foaming in the degasser. Without this antifoam agent, degassing is complicated since the vacuum must be pulled and then released and the foam settled. This process may need to be repeated several times to keep the foam from expanding into the vacuum line.

A slight depression in the ignition sensitivity of the propellant is seen at 0.10% levels of this antifoam agent. This problem can be overcome by adding 0.5 to 1.0% of a burn rate catalyst such as copper oxide. Formulas which do not include a burn rate catalyst and especially those containing a burn rate suppressant, such as oxamide, can be fired by using an ignitor boosted by a strip of high burn rate propellant.

Ballistic modifiers are sometimes added to the propellant to achieve altered burn rates. The oxides of iron, copper and magnesium and copper chromite are the most commonly used ingredients to increase the burn rate. Ferric oxide (oxide of iron) is the least effective (produces the smallest increase in burn rate) and copper chromite is very effective in small

amounts. For moderate burn rate enhancement, we suggest the use of copper oxide rather than magnesium oxide because of the tendency of the latter substance to form clumps which can lead to inconsistent burn rates. Depression of the burn rate is achieved by adding oxamide or calcium carbonate. Oxamide appears to be more desirable in this role since it is organic and can participate in the combustion reaction unlike calcium carbonate.

For slow acting isocyanates, such as DDI-1410 and IPDI, a **cure catalyst** is necessary to achieve cure at an acceptable rate. Triphenylbismuth, or TPB, is the most commonly used cure catalyst.

Using the Formulas in this Book

Your first batches should be inert propellant simulants using common table salt instead of an oxidizer. Go through the processing steps and all the way to cutting the grain with the simulants until you can produce a grain free from bubbles and voids. Only then should you consider casting live propellant.

All of the formulas in this book are listed as percentages totalling 100%. To find the quantities of the ingredients you need to mix, simply divide the percentage of each ingredient by 100 and multiply by the total number of grams of propellant you wish to cast. SOPROC will calculate your batch for you.

ABBREVIATIONS USED IN THE FORMULAS: The following codes are used in the formulas in this book:

AL	Aluminum powder, the grade is listed after the symbol. Example: AL; <400 mesh is Aluminum powder to pass a 400 mesh screen (this is available from Fire Fox).
AP	Ammonium Perchlorate, the grade is listed after the symbol. Example: AP; 200 mic is Ammonium Perchlorate 200 micron grade.
C-BLACK	Carbon Black which is a grade of carbon. You can substitute Lamp Black for this but, do not use charcoal dust!!!
C-OIL	Castor Oil, USP grade available at any pharmacy.

CUCRO	Copper Chromite burn rate catalyst.
CUO	Copper Oxide burn rate catalyst.
DDI-1410	A slower acting isocyanate curative.
DOA	Dioctyl Adipate plasticizer.
IPDI	A very slow acting isocyanate which gives a very long potlife. IPDI is useful when casting multiple batches in the same mold.
KP	Potassium Perchlorate. This is recommended for the experienced only because of KP's high sensitivity to pressure variations in the motor!
OXAMID	Oxamide burn rate suppressant (available from Prodyne).
PAPI-2901	A fast acting isocyanate also called MDI and Isonate 143L. Chemically it is Diphenylmethane 4,4-diisocyanate.
PDS-8R111	Poly-dimethyl Siloxane, 8R111 version from Xerox Corporation. The antifoam agent.
R45M	HTPB resin with antioxidant.
TPB	Triphenylbismuth, a cure catalyst used with DDI-1410 and IPDI to speed the cure process.

Liner Coating Formula

The liner coating formula presented here is simply the liquid fuel ingredients used in producing the propellants. It is necessary to coat the Kraft paper, used in making liners, with this formula to insure that the propellant mixture "bonds" to the liner during casting and cure. Failure to form this bond to the liner could cause unpredictable burning in the motor.

LINER COATING FORMULA:

R45M	52.0 %
DOA	30.0
C-Oil	7.2
PAPI-2901	10.8

Refer to the chapter on grain geometry for information on how to use this formula to coat grain liners. You will need approximately 0.135 grams per square inch of liner material per coat.

Tracking Formula

The best way to incorporate an integral time delay into your rocket motor is to use a manufactured time delay in the appropriate size. Time delays for this purpose are available from Aerotech for 54, 38, and 29mm motors. Commercially produced time delays are simply more reliable than those the average worker can produce; however, if you need to make a tracking grain only to produce smoke, the following formula can be used. It must be vacuum processed for best results. The formula burns relatively slowly and produces dense white smoke.

TRACKING SMOKE FORMULA:

AP, 200 micron	59.0 %
White Smoke, HH	18.6
R45M	12.1
DOA	5.7
C-Oil	1.3
PDS-8R111	0.1
PAPI-2901	1.1
DDI-1410	1.7
TPB	0.4

The white smoke is the Fire Fox high-heat white smoke mixture. Vacuum process and cast this mixture at 75 to 85°F in an appropriate size just as you would a propellant grain. Use an end burning grain. This formula can also be used as a slow burning propellant which is suitable for use with ceramic nozzles. The burn rate can be increased by using 90 micron AP and/or a burn rate catalyst.

Propellant Formulas

Once again, **MIX AN INERT PROPELLANT SIMULANT FIRST UNTIL YOU KNOW WHAT YOU ARE DOING!!!** The simulants are safer to work with and less expensive to practice on than live propellants. You must be able to produce a void-free grain before going on the live propellants. To mix a simulant, simply replace the oxidizer in the formula with common table salt.

Follow the suggestions given for tailoring the formulas to your personal requirements and working conditions. Before mixing any propellant you should read and thoroughly understand the steps in the next chapter on processing.

A FIRST FORMULA: *Procite A74ful*: a black smoke formula.

AP; 90 mic	74.0 %
CUO	1.0
R45M	13.3
DOA	7.5
C-Oil	1.6
PDS-8R111	0.1
PAPI-2901	2.5

Standard Isp with AP: 212.5 lb-sec/lb.

Solids Loading:	75%
Binder Percentage:	25%

This formula should be considered a winter formula. It must be processed below 72°F to have adequate pot life. The advantage is that it cures within 48 hours at room temperature without heat lamps. This formula can be used with either ceramic or graphite nozzles. The next formula is very similar and can be processed in the summer.

A HIGHER PROCESSING TEMPERATURE FORMULA: *Procite A73ful*: a black-smoke formula.

AP; 90 mic	73.6 %
CUO	1.0
R45M	13.3
DOA	6.9
C-OIL	1.6
PDS-8R111	0.1
PAPI-2901	1.2
DDI-1410	1.9
TPB	0.4

Standard Isp with AP: 212.1 lb-sec/lb.
with KP: 190.8 lb-sec/lb.

Solids Loading: 75 %
Binder Percentage: 25 %

This formula can be processed at 70 to 85° Fahrenheit. How to heat the mixture is explained in the processing chapter. Cure is complete in 24 to 36 hours at 130° F under heat lamps or in 2 to 3 weeks at room temperature. The burn rate may be slowed somewhat by using 0.1% carbon or lamp black in place of the copper oxide catalyst and increasing the oxidizer by 0.9 %. This altered mixture will not ignite as readily as the formula above. The burn rate may also be reduced by using 200 micron AP instead of the 90 micron listed in the formula.

KP may be substituted for the AP to obtain a fast burning formula suitable for use in end burning motors. Expect a burn rate near 1.0 inches per second when using KP as the oxidizer.

A FORMULA WITH ALUMINUM: *Procite AA6610ul*: a white smoke-white flame formula.

AP; 200 mic	66.6 %
Al; <400 mesh	10.0
CUO	1.0
R45M	12.1
DOA	5.7
C-Oil	1.3
PDS-8R111	0.1
PAPI-2901	1.1
DDI-1410	1.7
TPB	0.4

Standard Isp: 243.1 lb-sec/lb.

Solids Loading: 78%
Binder Percentage: 22%

This formula should use only AP as an oxidizer not KP. It is suitable for use with graphite nozzles only!!! The aluminum in the formula will attack ceramic nozzles severely. Process at 75 to 85° F to maintain adequate fluidity of the mixture. Cure is complete in 24 to 36 hours at 130° F under heat lamps or 2 to 3 weeks at room temperature.

VERY HIGH BURN RATE FORMULA: *Procite AA6510r2*: a white smoke-white flame formula.

AP; 90 micron	65.4 %
Al; <400 mesh	10.0
CUCRO	2.2
R45M	12.1
DOA	5.7
C-Oil	1.3
PDS-8R111	0.1
PAPI-2901	1.1
DDI-1410	1.7
TPB	0.4

Standard Isp: 243.4 lb-sec/lb.

Solids Loading:	78 %
Binder Percentage:	22 %

This is a very high burn rate propellant for use in end-burning and augmented end-burning grains only!!! The test for burn rate should be conducted with a standard end-burner and not an augmented type. Once again, formulas containing aluminum can be used with graphite nozzles only! This formula can also be used as an ignitor for other propellants. There is no need for vacuum processing when the formula is to be used as an ignitor only. Processing and curing of this formula is the same as the one above.

LOW BURN RATE FORMULA: *Procite AA6510o2*: a white smoke-white flame formula.

AP; 400 mic	49.2 %
AP; 200 mic	16.4
Al; <400 mesh	10.0
OXAMID	2.0
R45M	12.1
DOA	5.7
C-Oil	1.3
PDS, 8R111	0.1
PAPI-2901	1.1
DDI-1410	1.7
TPB	0.4

Standard Isp: 241.3 lb-sec/lb.

Solids Loading:	78 %
Binder Percentage:	22 %

This is a relatively low burn rate formula which can be used in BATES grain motors to approach the burn time performance of moon burners. This formula can be made to burn even more slowly by increasing the oxamide up to 6% and decreasing the AP proportionately. *Caution: long burn times may require the use of insulation on the nozzle.* Once again, formulas containing aluminum may be used with graphite nozzles only! Processing and curing of this formula is the same as the two directly above.

Since this formula contains only a burn rate suppressant, oxamide, ignition of the grain will be more difficult than the above formulas. Use a strip of the high-burn rate propellant above to boost a thermalite ignitor. Alternatively, nichrome wire can be embedded in or wrapped around a strip of the high-burn rate propellant to make a complete ignitor for this propellant.

ISOCYANATE SELECTION

The Isocyanate used depends on the processing temperature. Pure PAPI-2901 is only suitable for processing at 72 degrees or slightly below. IPDI and DDI-1410 can be used at much higher temperatures and even then require a cure catalyst. The addition of moderate amounts of PAPI-2901 to the other curatives gives a finished grain that is more easily machined than that obtained by using DDI-1410 or IPDI alone.

6

COMPOSITE PROPELLANT PROCESSING

Introduction

The basic procedure used by most amateurs for processing and casting composite propellant grains requires a propellant mixture with low enough solids loading (or possibly antiviscosity agents) to produce a pourable or nearly pourable mixture. It is in the vacuum step that workers differ in their approaches. Some mix under vacuum, some mix at atmospheric pressure and then degass in a chamber, and some degass only during the casting step. Each method has advantages and disadvantages; however, the primary advantage of the degassing method to be discussed in this text is cost. The degasser described in the chapter on equipment can be built for about $30 dollars in materials. Compare this with $300 to $2000 for a small vacuum mixer. The technique of vacuum casting requires that the propellant fall through an approximate 10 foot drop to obtain adequate degassing. A very large workshop is required for this method.

When using the degasser, the basic procedure is to weigh the chemicals on an accurate balance, mix them in the degasser, close the degasser and evacuate it with a vacuum pump while rotating and vibrating the degassing chamber. After degassing is complete, the propellant mixture is "pour" cast into a suitable mold and allowed to cure.

Processing Variables

In both the degassing method described here and the mix under vacuum method, propellant viscosity (fluidity) is extremely important. There are three main variables which interact to determine the viscosity of the raw propellant mixture. These are **(1) temperature of the mixture, (2) isocyanate selection/time**, and **(3) solids loading**. Each will be discussed in turn.

The **temperature** of the propellant can critically affect the processability of the propellant formulas at 78% solids loading. While the 75% percent solids loading mixtures may remain castable down to 68° F, formulas containing 78% or more solids are better processed at 75 to 85° F or even higher.

The **isocyanate selection and time** variable has a simple relation to viscosity. Immediately after the isocyanate is added to the mixture it begins to thicken. In the case of IPDI alone this can take up to 24 hours at 80° F before the mixture becomes unworkable. With MDI alone the two hour minimum working time is only possible at 72° F or less. It is best to run a test on several small samples to get the best isocyanate (and possibly cure catalyst) ratios for your working temperature. This has been done for you with the formulas in the previous chapter; however, the percentages expressed are for fresh isocyanates. If you use aged or somewhat decomposed curatives you will need to conduct your own tests. See the chapter on testing for details on how to conduct this test.

Solids loading, or the percentage of solid ingredients in the propellant formula, also significantly affects mixture viscosity. Seventy-five percent solids is the practical lower limit if reasonably high specific impulse is to be maintained and 78% solids loading is normally the practical upper limit. The particle size (micron size) of the oxidizer also affects viscosity but, to a lesser extent than the other variables mentioned here.

Propellant Processing Procedure: Using The Degasser

The following propellant processing procedure is to be used with the degasser. Familiarize yourself thoroughly with this procedure and the steps for casting the propellant before actually processing a batch of propellant. Propellant formulas should be processed in a batch no larger than 600 to 700 grams for the standard size degasser when using PDS-8R111 or about 500 grams without it. It is important that as much surface area of the raw propellant as possible be exposed to the vacuum to complete the degassing.

WARNING: *Protective gloves and eye protection must be worn throughout this procedure and until the equipment has been cleaned following the casting step. If you are working with solids in fine particle sizes, a dust mask should be worn as well.*

STEP 1: SET UP. The mold and liner into which you will cast the propellant should be ready (see the chapter on grain geometry). All equipment which comes into contact with the propellant should be thoroughly cleaned. A sufficient quantity of the oxidizer(s) should be sieved through a flour sifter and ready (sieving is only necessary with 90 micron AP or fine KP). A thin coat of Vaseline should be applied to the degasser rubber seal.

STEP 2: WEIGH INGREDIENTS. Weigh the oxidizer(s) and other solid ingredients in separate containers. Weigh the liquid ingredients in waxed paper cups, allowing for or taring off the weight of the cups.

STEP 3: MIXING. Mix the liquid ingredients to a homogeneous consistency in the degasser, ideally using a variable speed hand drill with a paint mixing attachment. Use a rubber spatula to get all the liquid ingredients into the degasser. DO NOT ADD THE ISOCYANATE YET!!! Add any solid ingredients, except the oxidizer, and mix again.

WARNING: *When mixing fine powders, such as 90 micron AP or magnesium or aluminum, always wet these ingredients by mixing them into the liquids with a dowel. Ventilate the area until there is no danger of airborne dust forming an explosion hazard. Dust suspended in the air could cause an explosion if set off by a spark from the mixer motor.*

Add approximately one-quarter of the oxidizer and mix again. Continue adding about one-quarter portions of the oxidizer and then mixing until all the oxidizer has been added. *Thorough mixing is essential for the propellant to function properly in the finished rocket motor.*

If you are processing above room temperature, heat the ingredients in the degasser by setting it in a pail of hot tap water until the proper temperature is reached. In a cold workshop, this temperature may be maintained by using heat lamps on the processing bench.

Finally, add the isocyanate(s) and mix again for at least 5 minutes.

STEP 4: INITIAL DEGASSING. Place the top plate of the degasser on the unit, making sure the seal "wets" the plastic. Connect and start the vacuum pump. **If you are NOT using PDS-8R111, watch to make sure the mixture does not foam too high and get pulled into the vacuum line.** If the foam gets within about two inches of the top plate, shut off the vacuum at the valve and settle the mixture by tamping and vibrating the degassing chamber. This may have to be repeated several times before the initial foaming subsides.

When the full vacuum has been reached and foaming has subsided, (this will occur very quickly when using PDS-8R111!!!) place the degasser on its side and begin rotating, exposing as much of the mixture to the vacuum as possible. Use your vibrator to help move a viscous mixture around the sides of the degasser. See photo #2 in the photographs appendix. Be careful that no propellant gets near the vacuum line at the top of the degasser. Continue rotating in this

manner, moving the mixture around the sides of the degasser with the aid of the vibrator, for at least 15 minutes (10 minutes is adequate when using PDS-8R111).

STEP 5: SETTLE THE PROPELLANT. Return the degasser to its upright position and settle the mixture to the bottom with the aid of the vibrator. When as much propellant has been settled as possible, slowly release the vacuum. ***NEVER TURN OFF THE VACUUM PUMP WHILE IT IS ATTACHED TO A VESSEL UNDER VACUUM.*** Instead, close the valve and pull off the hose going to the pump, then slowly open the valve to allow air into the degasser. Remove the top plate and scrape the mixture from the sides of the degasser with a rubber spatula, putting the scrapings in the bottom with the rest of the mix.

STEP 6: FINAL DEGASSING. Replace the top of the degasser, reconnect the vacuum hose, and turn on the pump. When full vacuum has been attained, vibrate the degasser in the upright position for at least two minutes. Slowly turn the degasser on one side while vibrating until the propellant mixture almost touches the top of the unit. ***DO NOT ROTATE THE DEGASSER AT THIS POINT.*** When the mixture is on one side of the unit, slowly release the vacuum as in step 5 above. Prop the degasser in position on its side so that the mixture does not flow. Remove the top of the degasser. You are now ready to begin casting the propellant.

Casting the Propellant Mixture

The mold, previously set up with a liner as described in the grain design chapter, should be ready to receive the propellant. The technique used here depends on the fluidity of the mixture.

If the mold is large enough and the propellant is fluid enough, the propellant may be literally poured into the mold which is held at a 45 degree angle and vibrated to settle the propellant. **It is vitally important to use care not to introduce voids or bubbles into the propellant mixture at this point.** *The action of pouring a fluid mixture into the mold is similar to pouring beer into a glass while maintaining minimum foam or head.* This procedure may need to be practiced until you can feel confident with your technique. This is another reason your first batch should be an inert propellant simulant. After casting, you can section the cured grain with a saw to look for bubbles and voids and adjust your technique accordingly.

If your working temperature is too low or the solids loading is too high to allow pouring the propellant mixture,

the technique is somewhat different. The propellant mixture may be carefully scooped out of the degasser with a spatula and inserted into the mold. ***CAUTION:*** *ALLOW ROOM FOR THE AIR IN THE MOLD TO ESCAPE PAST THE PROPELLANT MIX BEING "POURED" INTO THE MOLD!!! Great care must be taken not to introduce voids or bubbles into a mixture of this high viscosity!* Once again, practice with an inert propellant simulant until you are confident of your technique. It may be necessary to slowly move the mold to an upright position on the vibrator to get the propellant to settle to the bottom. Allow sufficient time for the vibrator to fully settle the propellant. **Successive scoops of mixture should be small in this case, only filling up to half the diameter of the mold at once!!!**

Once the casting operation is complete, set the molds aside to cure. The propellant will be cured to the point that it is ready to be cut into grains or inserted into the motor when it is firm and no longer tacky to the touch. Heat lamps may be necessary to speed the curing process when using slow acting isocyanates such as DDI-1410 and IPDI.

WARNING: ***POSSIBLE FIRE HAZARD!!!*** *When using heat lamps to cure the grain, make certain that the lamp cannot fall over and touch the propellant grain causing a fire. Use ceramic bases for 250 watt lamps and use a guard around the lamp. Use a base for holding the lamp that positively, absolutely will not tip over!!! Use a thermometer to monitor the process and do not allow the temperature to exceed 140° F. Remember the grain will be hotter at the end toward the heat lamps.*

7

TESTING

Introduction

There are many variables in solid propellant production that need to be tested before motors can be designed. The *density of the propellant* is one of the most inconsistent variables and must be tested to determine an average value for best results. The working time, or "*potlife*" of the raw propellant mixture must be monitored for various working conditions and kept close to ideal if minimum cure time is to be obtained. Also, in the development of new propellants, the all important *Kn ratio* must be established along with an experimentally determined *rate of burn*.

Propellant Density

Because of variables in the production of the finished propellant grain, propellant density tends to vary more than any other factor in the design of motors; however, it can be easily determined.

The **basic procedure** consist of cutting cured propellant into small pieces with an Exacto knife or similar tool, weighing the sample on an accurate balance, then dropping the sample into an graduated cylinder with water in it. The difference between the final and initial volume of water is the volume of the propellant. A simple formula can then be applied to determine the propellant density.

The total weight of the sample should be at least 30 grams. This is a piece of propellant approximately 1.25 inches in diameter (the size of a 38mm grain) and about one to one and a half inches long. Allow for at least this much extra propellant to be cast in your first few batches of a new propellant.

The formula for converting volume and weight in milliliters and grams to density in pounds per cubic inch as used by the SOPROC motor development program is:

$$d = 0.03613 \frac{W}{V}$$

where **d, is the propellant density in lbs/in^3**, **W** is the weight in grams of the sample and **V** is the volume of the sample in cubic centimeters or milliliters. ***SOPROC will perform this calculation for you.*** Simply enter zero when asked for the propellant density.

Potlife Determination

The formulas in this book are designed to be used with fresh isocyanates to provide at least two hours working time (potlife) at the temperatures suggested. The use of isocyante more than one year old will necessitate performing a potlife test. You may also want to provide yourself with more processing time for multiple casting operations to form large single grains. Professional motor manufactures make use of expensive viscosity test equipment to determine the potlife of the propellant formulas they are using; however, there is a simple method to adequately determine the potlife for your isocyanate mixture and working temperature using the equipment you will already need to process the propellant.

The **procedure** consist of mixing a small (about 100 grams) sample of propellant in a waxed paper cup. There is no need to vacuum process this mixture. After the chemicals are thoroughly mixed and have been brought to the working temperature, scrape the mixture to one side of the cup with a spatula and record the amount of time it takes on the vibrator to return the propellant to level in the bottom of the cup. Repeat this test every 15 to 30 minutes until the settling time exceeds 20 to 30 seconds.

This time is your personal working time or potlife. You must be able to complete all phases of mixing, degassing, and casting in this time. It is recommended that a minimum of two hours be allowed for potlife. If your time is significantly shorter than this, you need to adjust your isocyanate and\or cure catalyst. Cure the sample completely, after testing, to be sure it will set up. Recall that PAPI-2901 is a fast acting isocyante. Adjusting the ratio of DDI-1410 to PAPI-2901 or using IPDI instead of DDI-1410 (with a cure catalyst) should be the first solution tried. A very long potlife will require DDI-1410 or IPDI as the sole curing agent with TPB as the catalyst.

Record your time and temperature in a notebook. Remember that the potlife time you achieve will vary with the temperature at which you are processing. If you change the working temperature by more than a few degrees, repeat the

test. Again record the conditions in your notebook for reference the next time you work at this temperature.

Determination of Kn Ratio and Rate of Burn

Determination of the Kn ratio, or the ratio of the propellant burning area to the nozzle throat area, is very important in developing a new motor. The Kn ratio determines the combustion chamber pressure at which the motor will operate and is specific to the type of propellant being used. Professionals use expensive pressure sensing equipment, from which data is fed into an analog/digital converter and then into a computer, to analyze small motor firings. Fortunately, the amateur motor designer can gather adequate information by static firing a small motor and using either a movie camera or video recorder (possibly rented) to record the test data.

You will need to determine the burn time of the propellant at a particular nozzle throat diameter before using the SOPROC motor development program. The program will compute the actual Kn ratio and combustion chamber pressure when you enter your test data and nozzle throat size from a successful burn test.

To use the video recorder, or camcorder, you must have a VCR capable of single frame playback in high speed (SP) mode (this is usually done with a shuttle control). You will also want a model of VCR that displays the play time elapsed in seconds but, lacking this, simply record the second hand of a watch or clock in high speed mode and then count the number of frames per second. Use the shuttle control on the VCR to advance frame by frame.

To use a movie camera, again record the second hand of a large clock and determine the precise number of frames per second. Suitable movie cameras for this purpose may be purchased for around $30. You do not need a projector to analyze the test data, a small viewer which will allow you to advance single frames is actually better (and less expensive). Most large cities have movie camera and camcorder rentals available, check your yellow pages for listings.

To test a new propellant for use in Bates grain motors, cast enough propellant mixture to produce at least four or more 38mm or larger single grain Bates motors. The ideal length of the grain should be 2.125 inches in the 38mm size. The core diameter should be at least one third the diameter of the grain; however, the area of the port (core) must be at least six times larger than the area of the nozzle throat. This will insure that no erosive burning is encountered which

would interfere with the test. A core diameter of 3/8 to 1/2 inch is most often used in the 38mm size. This grain geometry allows an approximate 20% variance in the area of burn. Obviously, the burn time you determine will be an average over this range but, this is considered adequate for most purposes.

To test a new propellant for use in end-burners, simply use a standard (not augmented) end-burner for the test. The length of the grain needed is determined by the rate of burn of your propellant.

The test is conducted by placing the motor in the ground, nozzle end up, and firing. Record the count down and firing on the camcorder or movie camera to determine the burn time. Count the number of frames from full flame on after ignition until the motor burns out. For example, suppose you determined that your camera records at 30 frames per second and the firing time of the motor was 70 frames, divide the number of frames in the firing time by the frames per second to arrive at the burn time. In this example, 70 frames of burn divided by 30 frames per second yields a burn time of 2.33 seconds. The data collected from two successful burns at different nozzle throat sizes is all the information you need to be able to use the SOPROC motor design program.

Most ammonium perchlorate based propellants begin to chuff or experience **unstable combustion** below about 200 psig. The last test *before* this condition is encountered is the lowest possible Kn ratio you can use (the largest nozzle throat area). The last test before motor failure or explosion occurs is the highest possible Kn ratio (or smallest nozzle throat area) you can use. ***You do not need to test for these conditions.*** Simply tell SOPROC the maximum pressure under which you want your motor to operate and the program will do the rest. Maximum recommended combustion chamber pressures for the different types of construction described in book two are included in the SOPROC manual.

If a motor you are testing chuffs, decrease the nozzle throat area by at least 20%. DO NOT ADD CARBON TO THE PROPELLANT FORMULA OR CHANGE THE FORMULA IN ANY WAY!!! This has been proposed as a remedy for chuffing in other publications and shows á gross lack of understanding of the phenomenon of combustion instability!!! Chuffing occurs when the nozzle throat diameter is too large for a given motor/propellant combination.

If a motor you are testing CATOs, (blows off the nozzle or bulkhead or if the gasses eat through the seals) enlarge the nozzle throat area by at least 20% or more.

Continue in this way until you achieve stable burning. With experience you may be able to hit a working nozzle throat area on the first try!!!

To determine a 20% area change for the throat size you are working with, use the following two formulas:

$$A = \pi(\frac{d}{2})^2 \qquad\qquad d = 2\sqrt{\frac{A}{\pi}}$$

where A is the area of a circle and d is its diameter. The first formula is used to determine the area of the throat from a known diameter. The second formula will give the diameter from a known area of throat.

EXAMPLE: suppose your first test motor chuffs and you want to decrease the throat area by 20%. Lets say your nozzle throat diameter was the same as a #7 drill bit (0.201 inch diameter). Substitute this diameter in the first formula and you will find that your throat area was 0.03173 square inches. Multiply this area by 0.80 for a 20% decrease (a 20% decrease is the same as 80% of the old area) to give a new area of 0.02538 square inches. Substitute this area in the second equation to find the new diameter of 0.1798 inches. For the next nozzle use a #15 drill bit (diameter 0.180 inches). To enlarge the throat diameter (as in the case of motor CATO) multiply the old area by 1.20 and then substitute in the second formula to find the new diameter.

The exact Kn ratio you will use is also determined by the type of motor you are designing. Long high-performance motors which are subject to erosive burning may need to be designed around an average value midway between chuffing and test motor failure or even lower. SOPROC can help you find the right value to use. Be aware that very large motors in the M to O impulse range may require retesting in a larger Bates test motor than the 38mm size. This is because the *propellant burn rate is to some extent governed by motor size and heat transfer considerations.*

Appendix A

References and Reading List

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Appendix B

SAFETY

The following list of safety precautions should be adhered to at all times when working with composite propellants. **This list is not meant to be exhaustive.** Common sense should prevail at all times!!!

1. Always **wear the minimum safety equipment** described in the book. At the least, this consist of protective eye goggles or a face shield, gloves, and a lab apron or coat when working with raw propellant and chemicals. Remember that some chemicals are poisonous!

2. Always use a **dust mask and open ventilation** when working with fine powders of any kind. Fine powders can form explosive mixtures with air. When mixing propellants, always mix powdered ingredients into the liquid components of the mixture with a dowel and ventilate the area before resuming mixing with a power drill.

3. ALWAYS keep an ABC rated **fire extinguisher** on hand when mixing, casting, or cutting propellant. Take it with you when you static test your motors.

4. **POWDERED PROPELLANT SHAVINGS ARE EXTREMELY FLAMMABLE!!!** When machining grains, sweep up the fall off at regular intervals and store in a plastic bottle with a tight lid. Dispose of this fall off by igniting electrically in an open area away from grass or other flammables. IGNITE IN SMALL QUANTITIES ONLY!!!

5. **NEVER USE POWER SAWS TO CUT PROPELLANT GRAINS!!!** Always use a hand powered miter box saw for this operation. Getting propellant dust into or near motors may cause a flash fire with intense heat!

6. **Vacuum apparatus may be hazardous!** The use of a strong vacuum on various apparatus presents the risk of implosion. Always wear the goggles and gloves when working with vacuum devices, even if you are only testing for vacuum!

7. **Practice working with inert propellant simulants** before working with live propellants. Simulants are safe to work with and represent minimal fire hazard. Work the bugs out of your process with simulants first!

8. Always use extreme caution with loaded rocket motors. **Treat every new motor design as though it may explode.** Maintain the minimum safe distance suggested by Tripoli when static testing motors.

9. **Keep all oxidizers away from organic material and flammables.** Mixing oxidizers with fuel providing substances presents a fire hazard. Store them in sealed containers preferably in a locked metal cabinet.

10. **Read all chemical data safety sheets** that come with the chemicals you will be using. They contain safety data and poison information as well as the proper procedure for handling spills and fires.

11. **Exercise great care when using heat lamps** around propellants and chemicals. Use a ceramic base with 250 watt lamps and a guard around the lamp. Use a sturdy base to be sure the lamp cannot fall over and contact the propellant starting a fire. Do not leave heat lamps unattended.

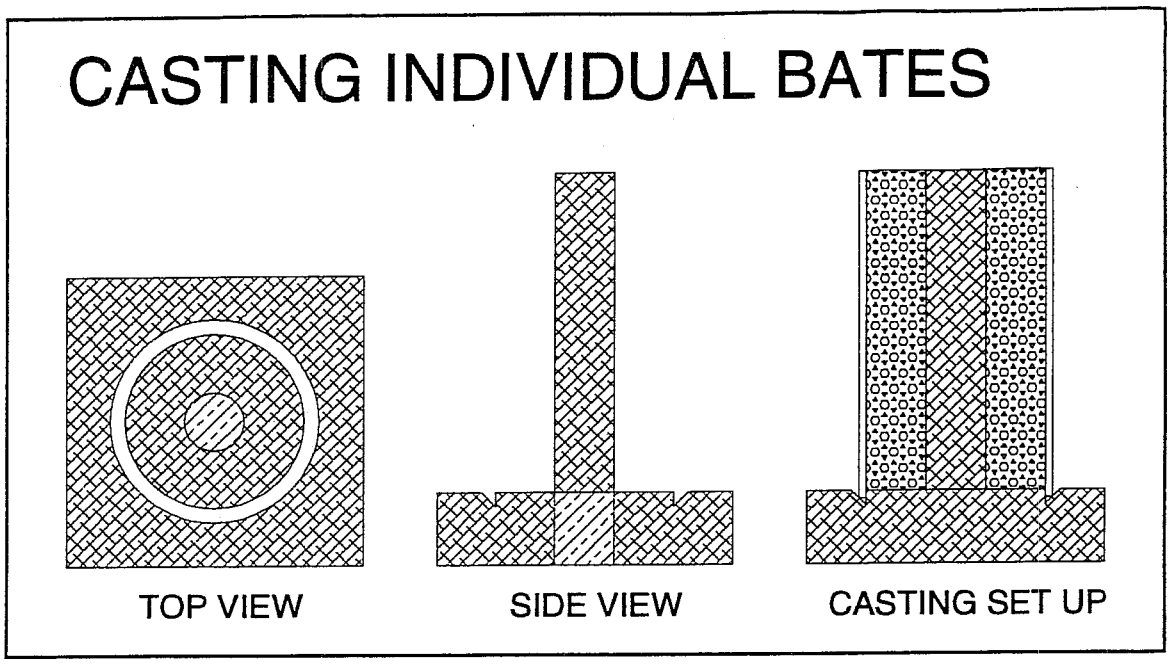

Figure 6 The mandrel base for casting individual BATES grains is essentially the same as the one used for casting a solid stick of propellant (figure 5). The addition of a dowel in the center of the base allows for the formation of a core in the grains. Recall that all wooden surfaces that come into contact with the propellant must be coated with canning wax!

The tubing is the same: the same tube stock used for the motor. The same coated Kraft 60 lb. paper is used inside the tube portion of the mold as a liner for the grain.

This method is more time consuming than cutting the grains from a single stick. It is also safer than machining live propellant grains. It becomes practical with motors 54mm in diameter and larger since smaller motors may not allow enough room for the propellant to be cast around the core mandrel. A single large motor may be made by this method with a relatively small degasser.

Once again, seal the ends of the liner tube to the mold tubing with silicone rubber sealant, as described in the text, before casting propellant into the mold.

When curing at elevated temperatures, the core mandrel should be made of Teflon rod or tubing. A base plate made from Teflon sheet stock is cut with the adjustable circle cutter to fit on top of the wooden base. Coat the Teflon parts with a small amount of mold release compound before casting. Too much mold release can cause ignition difficulties.

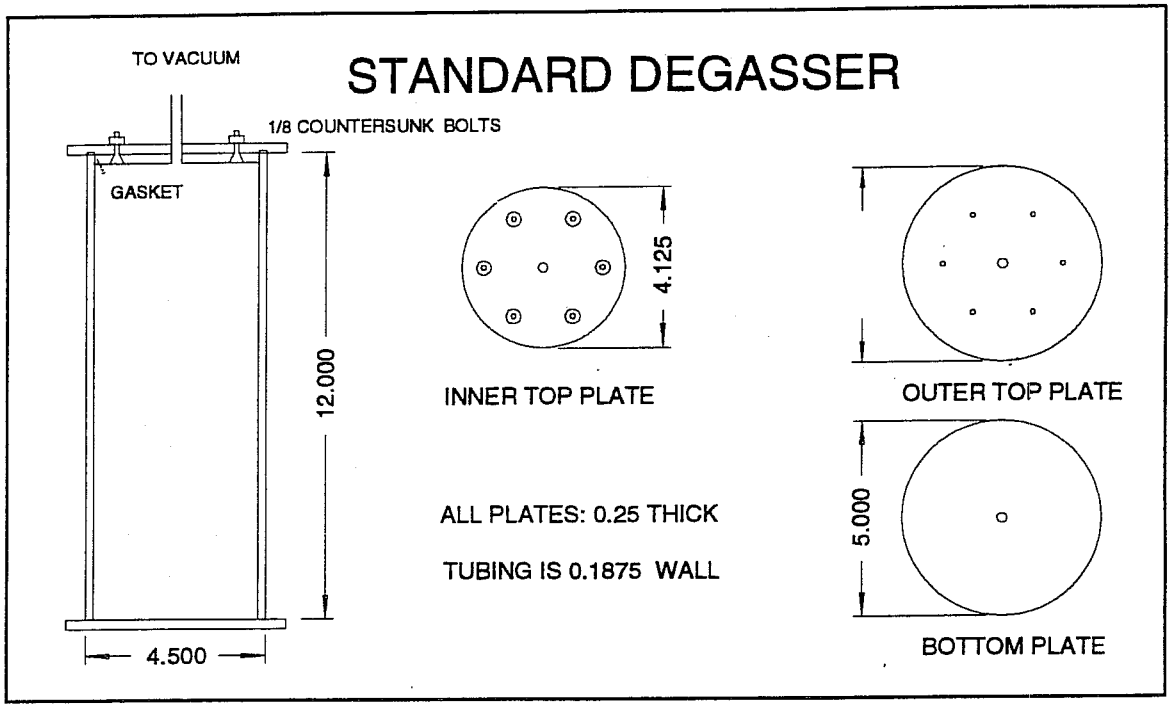

Figure 7 Refer to the equipment chapter for details on construction of the standard size degasser. Note the placement of the six countersunk bolts which hold the inner and outer top plates together. Brass or stainless steel is the best material for the bolts. Note the direction of countersinking the bolts. This ensures easy clean up of the degasser top. Epoxy will not hold the plates together but should be used to hold the bottom plate to the acrylic tubing with a generous fillet. Be sure to ruff the plate and tubing with sandpaper to ensure a good bond.

The holes in the center of the plates are created by the circle cutter used to cut the disks. The hole in the bottom plate is plugged with epoxy and the center holes in the top plates are enlarged to accept the 1/4 inch brass tubing to connect to the vacuum line.

Not shown is the silicone sealant around the outside of the bolts on the top plate. Simply use silicone rubber sealant from an automotive supply to seal the outside of the bolts and nuts against the vacuum.

Appendix F

Photographs

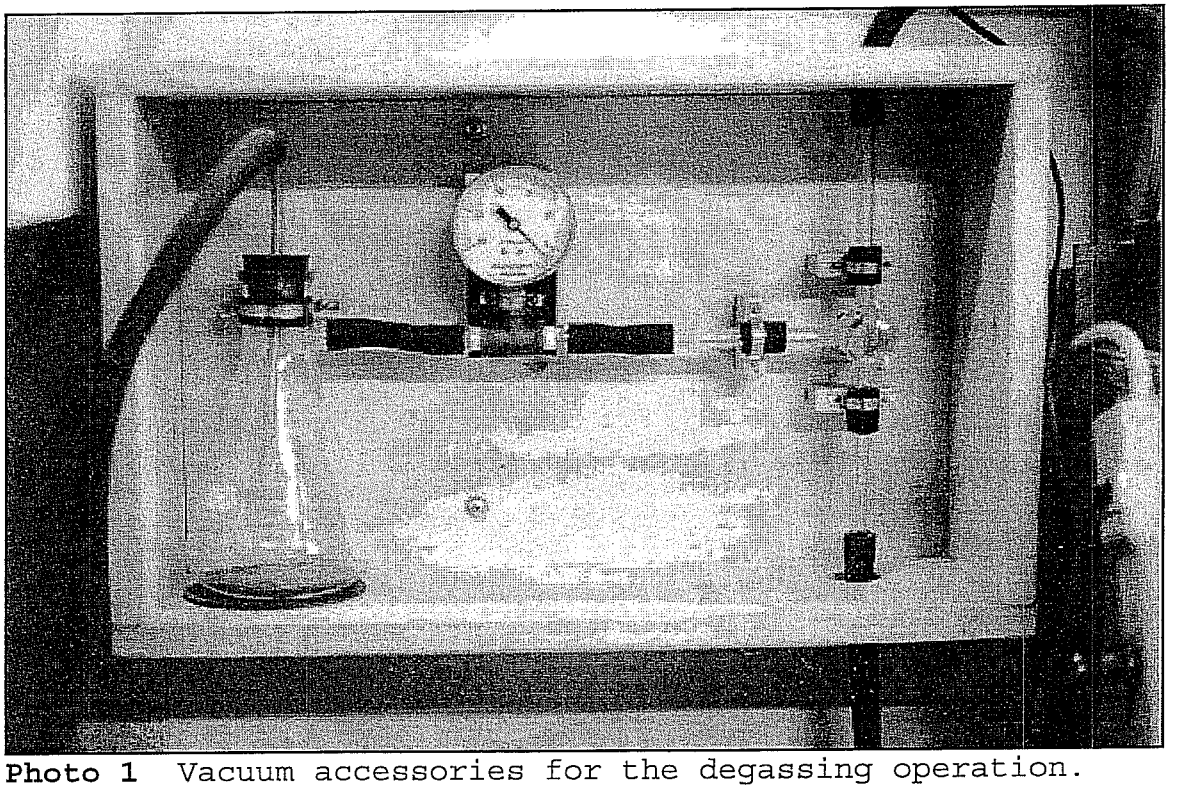

Photo 1 Vacuum accessories for the degassing operation.

The vacuum accessories described in the equipment chapter are shown in photograph 1. The vacuum flask on the left is designed for use in vacuum filtration but here serves as a trap to prevent propellant from being sucked into the vacuum pump. The vacuum line going off the top of the flask is connected to the degasser. The rubber vacuum hose connects to the top of the flask via a metal tube running through the rubber stopper in the top of the flask. This metal tube should go down almost to the bottom of the flask.

The side arm on the flask is connected with 1/4 inch vacuum hose to a plumbing "T" with the vacuum gage on top. The other side of the "T" connects to a 3-way glass valve used to control the application of vacuum to the system. The top connection is a vent to allow air into the system when releasing vacuum and the bottom connection goes to the vacuum pump.

Note that the accessories are mounted in a box to protect against accidents in the shop. The box is mounted to the wall above the work bench to isolate it from the vibrator used to settle the propellant. The box shown is only one possibility for housing the system.

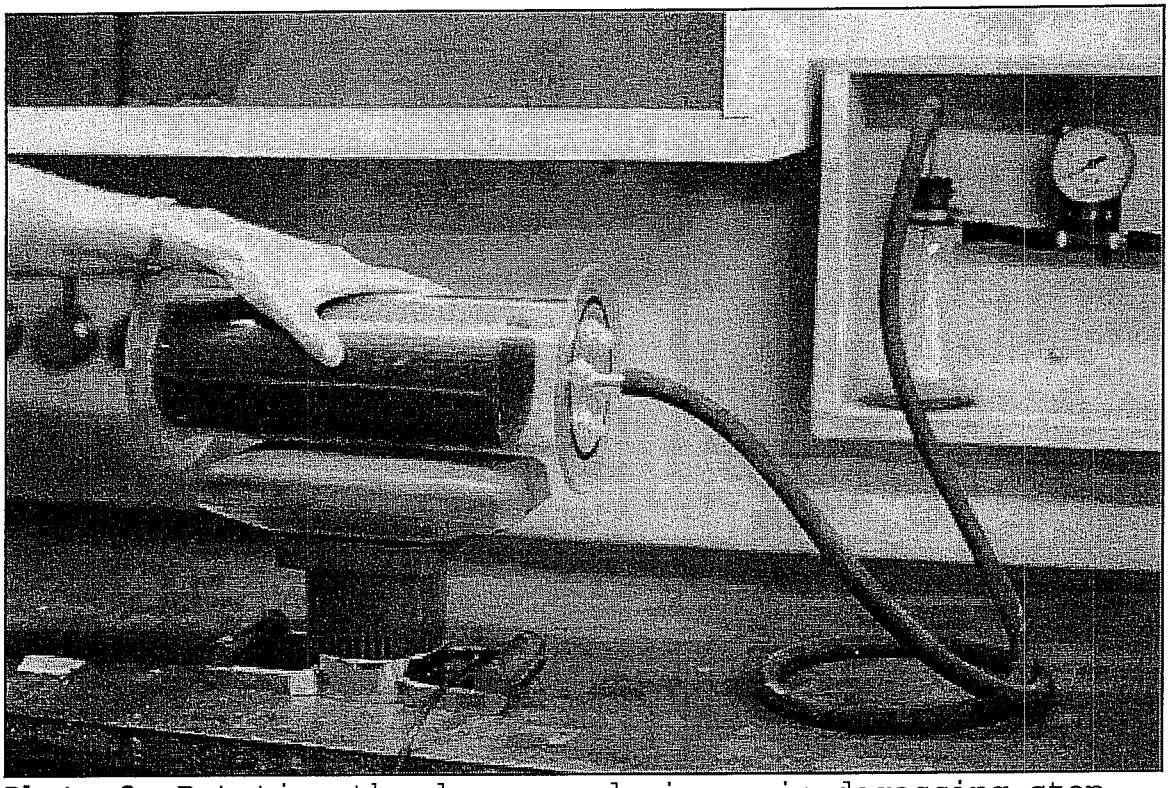

Photo 2 Rotating the degasser during main degassing step.

The rotation of the degasser on the vibrator is shown in photograph 2. Note the hose going to the vacuum accessories and the fact that the accessories box is mounted above the workbench. The degasser is rotated in this position for at least 10 minutes during the main degassing step described in the processing chapter. The idea is to expose as much propellant to the vacuum as possible in a short time by moving the propellant around the walls of the degasser.

Note that the black propellant mixture is not allowed to reach the top of the degasser where it could be sucked into the vacuum system. The next step will be to settle the propellant to the bottom of the degasser with the aid of the vibrator, release the vacuum and scrape the propellant off the walls of the degasser. Vacuum is reapplied and the propellant vibrated to one side of the degasser without rotation. The propellant is then ready for casting.

The procedure for casting the propellant is shown in photograph 3. After a small amount of propellant is placed in the top of the mold, the mold is held and vibrated in this position to allow the air inside the mold to escape. After the propellant is moving down the side of the mold the mold may be slowly held more and more upright on the vibrator. Vibration is continued in a nearly upright position until the propellant has settled in the bottom of the mold. A good overhead light is essential for monitoring this process. Only after the propellant has completely settled is the next portion added and the process is repeated until the mold is full. Be aware that some of the propellant will stick to the side of the mold. This is normal.

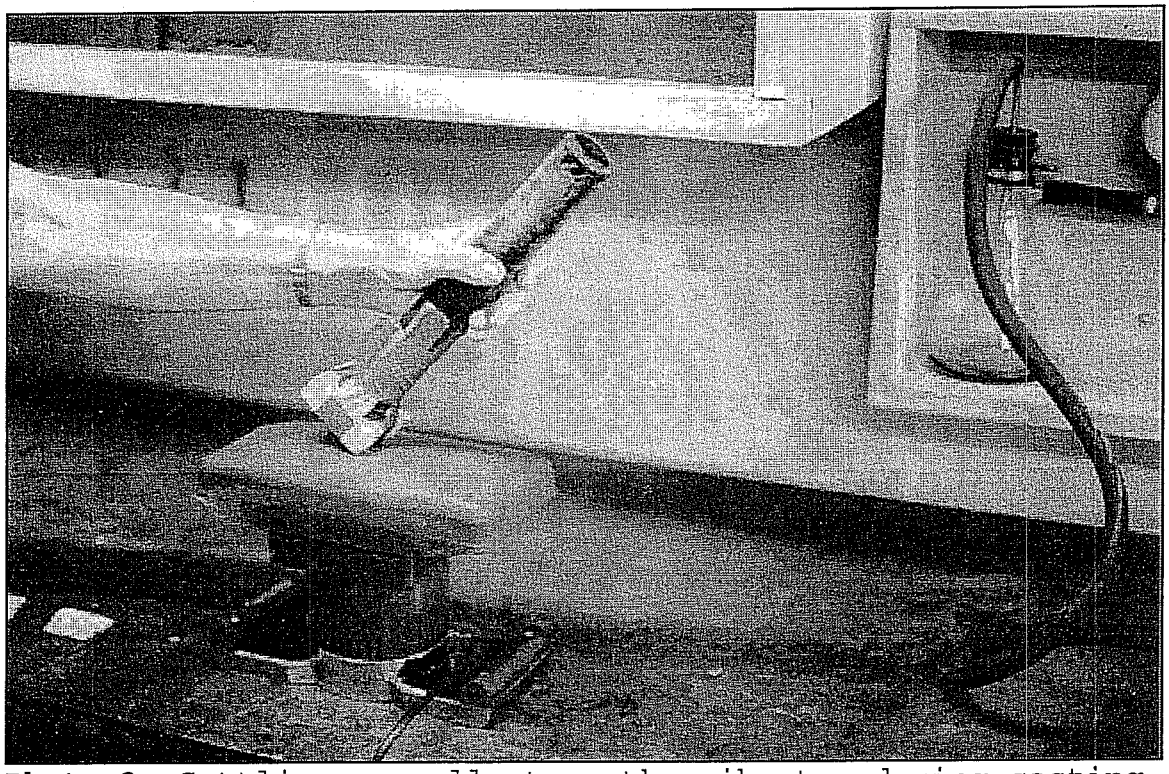

Photo 3 Settling propellant on the vibrator during casting.

Note the duct tape applied to the base of the mold and holding the base firmly to the tubing. This is essential to prevent the base from separating during the settling operation on the vibrator. Strips of duct tape form an X pattern over the base of the mold and up onto the tubing. Another piece is wrapped around these strips on the tubing section of the mold.

Appendix C

Suppliers

CHEMICALS

All of the chemicals used in the formulas in this book can be obtained from the following sources. The antifoam agent PDS-8R111 can be obtained directly from the Xerox Corporation's toll free order line listed below.

Prodyne, Inc.
P. O. Box 12806
Ogden, UT 84412-2806
(801) 392-7205

Firefox Enterprises, Inc.
P. O. Box 5366
Pocatello, ID 83202
(208) 237-1976

Xerox Corporation (PDS-8R111 only!)
(800) 822-2200
Ask for reorder # 8R111

LABORATORY APPARATUS AND SAFETY EQUIPMENT

Fire Fox, listed above, caries some of these items. One of the best sources of lab equipment, such as balances and vacuum flasks, can be found in magazines such as Popular Science. There is a classified ad section called *Science & Chemistry* in the back of the magazine. Also, you may be able to get these items locally. Look in the yellow pages under Chemicals and Laboratory Supplies.

VACUUM PUMPS

As explained in the book, the best source of vacuum pumps is the used equipment market. Heating and air conditioning companies use vacuum pumps and occasionally have them available in used condition. The Physics and Chemistry departments of colleges and universities and various research and development laboratories use them and sometimes have auctions where old equipment is sold. Make sure that the pump was originally capable of pulling a vacuum down to several millimeters of absolute pressure or greater than 29 inches of vacuum. Make sure you can still get parts for the model you

are considering and make your bid accordingly. If you buy a new pump with these specs, expect to pay around $500. For a used pump in good condition you'll pay around $250, and a used "fixer-upper" will run from $0 to $60. Prodyne occasionally has used and reconditioned pumps for sale as does Reynolds Scientific (listed in Popular Science magazine) but their prices are at the top end of the scale.

THE DEGASSER

The cast acrylic tubing and polycarbonate sheet needed to produce the degasser can be purchased from plastics suppliers. Look in the Yellow Pages under the *Plastics - Rods, Tubes and Sheets* classification. The polycarbonate sheet can be cut from scraps these suppliers usually have available.

ROCKET MOTOR HARDWARE

The various **fiberglass and phenolic tubing** needed to construct motors is best found through the Thomas Guide available at your local library. Look for suppliers and manufacturers of phenolic and fiberglass tubing. Specifications for ordering these tubes are given in book two. PSI is considering offering some of these tubes for sale if enough interest is shown.

Prodyne and Firefox, listed above in the chemicals section, also carry these items but the products they sell are not what we consider first quality products for rocket motor making. They are usually scraps of tubing used for other applications. Try them at your own risk.