very likely! In such cases, a helpful chemical to add is known as 'fumed silica'. This is simply silicon dioxide (sand!) that has been manufactured in such a way as to produce particles of an incredibly small size: less than 10 microns diameter (0.00038 inch) to even less than 1 micron diameter (0.000038 inch)! These particles have the property of forming large (on a molecular level) web-like structures that are all linked together by surface charges shared amongst themselves. These large structures impede the flow of the liquids in which they are dispersed and act to increase the apparent viscosity of the liquids. But, only very small amounts, again, are needed: usually less than 1-2 % by weight of the liquid, and often much less. Levels of 1-2% can cause a watery-thin liquid to become a spreadable paste (!), and lower levels can produce viscosities that are high enough to prevent particulate materials from settling out, while not interfering much with the abilities of the thickened liquid to flow. The possible use in low-oxidizer-level propellants is obvious. Believe it or not, a very convenient source of this material is in almost any good hobby shop! The material is sold as a thickener for epoxies used to form fillets around wing roots, model rocket fins, etc. Although it is rather expensive by weight, a little goes a VERY long way! If you use a little too much, though, you can probably still continue by using vibration to help move things along (pun intended!).

It should also be mentioned that there are organic materials available that do pretty much the same thing. These materials are usually of the organic group called polymethacrylates. Typically, they are used in the paint industry to thicken paints without introducing undesirable side effects that fumed silica can produce. Research would have to be done into the source and cost of these materials as well as the specific type of thickener to use for a specific type of resin system (consult, for example, industry literature such as The Journal of Coatings Technology, etc.), but they do have the distinct advantage of being organic and, therefore—unlike fumed silica—are BURNABLE!!

POLYURETHANE POLYMERS:

Theory:

Raw Materials:

Polvols (POLYmeric alcohOLS): Long organic molecules that have ends made up of alcohol groups (hydroxyl groups). These molecules can have various numbers of these groups contained within themselves: one, two, three or more. It is even possible to have fractional numbers of these groups, for example, 2.3-2.5, etc. This is because none of the manufacturing processes is perfect and some molecules have various numbers of these groups on them. The overall average for reaction purposes, however, is expressed as a whole number plus a fraction. The end result is that, on the average, each molecule can react with more than one molecule of isocyanate curing agent, a fact which makes crosslinking possible. Cross-linking is the bridging of the curing agent with more than two resin molecules so as to form a three-dimensional network instead of just long chains. Molecules that form long chains, only, can form compounds that seem to be just like rubber but, when heated sufficiently, will eventually melt. This is not desirable in a propellant, especially if the melting point is anywhere near the temperature at which the propellant is expected to operate. A three-dimensional network, however, forms a nonmelting type of rubber (or plastic). When heated to extreme temperatures, it merely chars without melting. This is a more desirable trait for a propellant as it means that the propellant will retain its shape when heated.

Isocyanates: Isocyanates are named for the type of end group on the molecule. This end group has the formula: —N=C=O. The nitrogen atom has a single bond connected to some organic compound, R. It is, in turn, connected to a carbon atom by a double bond.

which, in turn, is connected to an oxygen atom by a double bond. Isocyanates are the curing agents used in polyurethanes. As will be shown below, they react with the hydroxyl end groups on the binder resin to form the polyurethane rubber that holds everything together.

The two most commonly used isocyanates are di-isocyanates and polyisocyanates. The di-isocyanates have just two end groups, but the polyisocyanates, very much like the polyois described above, can have more than two end groups or even fractional numbers of end groups. If a di-isocyanate is used as a curing agent, then, if the binder does not have more than two hydroxyl end groups per molecule, a third compound, known as a cross-linker, must be added if a three-dimensional molecular network is desired. This is usually a diol (i.e., a compound having only two hydroxyl end groups) but can be a polyol. It usually has a length which is much shorter than the molecule length of the binder resin. It helps to provide cross-linking but, because of its short length, also provides some stiffness to the resulting rubber chain. This helps to make the rubber a bit stronger than it would otherwise be if it were composed only of longer, more flexible chains.

If a polyisocyanate is used, however, then it is capable of providing the cross-linking all by itself, even if the resin used is only a diol. Of course, if a polyisocyanate is used with a polyol, wherein BOTH compounds have multiple end groups on each of their molecules, then the result is a VERY reactive mixture that probably will not even need any kind of curing catalyst to cause the rubber to cure in a short time. This will be the situation with the binder and curing agent used in this course. A by-product of this fact is that the reactivity will be so high that only a small amount of time will be available for processing the propellant once the curing agent has been added. The propellant will be starting to gel (i.e., forming the initial three-dimensional network) in an hour or less, giving a realistic working time of only about one-half hour! As a result, it will be important to work through the various procedures as quickly as possible, consistent with safety.

BASIC REACTIONS OF RAW MATERIALS:

The first reaction, indicated above, is the desirable one. An isocyanate end group reacts with a hydroxyl end group to form a urethane group. The second reaction, however, is very UNDESIRABLE! In this reaction, an isocyanate reacts with moisture that may be present in some of the liquid ingredients, or on some of the solid ingredients, to form an organic compound known as a urea plus forming carbon dioxide, which immediately becomes a gas. This gas then causes foaming of the propellant and, possibly, an uncontrollable increase in burning area due to

the bubbles. The result can be a propellant which is not as dense as it should be, producing a lighter propellant weight than desired, as well as a possible explosion.

Also, the organic urea compound formed contains one, or more, amine end groups (---NH2). If the compound formed is at all soluble in the binder, then these amine end groups will react strongly with the isocyanate end groups that are present to form their own polymer. This can be a problem because the amine end groups have the ability to accelerate the gelling of the binder system, which can result in premature gellation before processing can be completed! So, this is another reason that all ingredients should be kept as dry as possible.

PARAMETERS OF INTEREST

Thrust: $F = C_f * A_t * p_c;$ $F = A_b * r_b * d_p * I_{SD};$ Lb.

Propellant density: dp Lb/ Cubic Inch

Burning area: Ab, Inch²

Combustion Pressure: pc. psia

Combustion Temperature: T_c, degrees Rankine (degrees Fahrenheit +460)

<u>Coefficient of Force</u>: Cf (dimensionless)

Specific Impulse (Classical): Isp, 1b-sec/lb = seconds

Characteristic Velocity: c*, ft/sec

Burning rate vs. pressure (absolute): rb, inches/second

Burning rate pressure exponent: : n (dimensionless):

calculated from the burning rate vs.

pressure data

Specific Impulse vs. pressure (absolute): displayed on a log-log graph as an

approximately straight line

Area Ratio vs. pressure (absolute): also displayed on a log-log graph as

an approximately straight line

Acceleration: a. ft/sec²

<u>Velocity</u>: v, ft/sec

Distance: h or s, feet

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IGNITION

by

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INTRODUCTION:

One of the most important areas of rocketry is the subject of ignition of propellants. Depending upon whether solid, liquid, or hybrid propellants are to be ignited determines the actual design of the ignition system. Without proper ignition, proper and safe operation of a rocket engine is not possible. While this may seem to be overstating the obvious, nevertheless, ignition systems are typically not engineered by experimental rocketeers to the same extent as the rest of the rocket system. Perhaps it is because so little is known about ignition system design that this is so. One tends to put off this task until it can no longer be ignored. Then, likely as not, very vague and opinionated procedures are followed. The end result of this "technique" of design is, occasionally, faulty ignition of engines, below-par performance (such as hangfires, wherein an engine may, or may not, eventually ignite properly), burn-throughs, or outright explosions. The safety hazard aspect of the unpredictability of this situation is obvious.

This "seat-of-the-pants" procedure, while occasionally being VERY EXCITING (!), should not be allowed to take place. So, this section of the course is designed to present some well-known (in the rocket industry) information that will help produce an ignition system that is as close as possible to that which is needed.

IGNITION:

THEORY: To begin, realize that the purpose of ignition systems is to supply enough heat energy for a long enough period of time to ensure that the propellant in question (which WANTS TO BURN, ANYWAY!) starts to burn in a smoothly-controlled manner, that is, burning starts and causes an increase in both combustion temperature and pressure until the desired operating temperature and pressure are reached that will sustain proper combustion of the propellant. Providing too little energy and/or too little ignition duration can cause the propellant to either not ignite at all or to partially ignite at a pressure much lower than desired. If this low-level burning process is able to continue for long enough, it may cause enough heating of the engine casing to finally allow the propellant to get hot enough to burn or explode. Even if proper combustion, without explosion, eventually occurs, quite a bit of the propellant may have already been consumed which will cause the rocket to have lower performance than its design goal.

On the other hand, if too much energy and/or duration is provided, the propellant can "over-react", so to speak. The propellant may start burning at a rate which is much higher than the design value. Since the nozzle throat diameter is fixed and designed for a particular operating pressure, too much pressure upon ignition can lead to failure and explosion. Normally, solid propellants have a kind of built-in tendency to keep the

pressure at the design value. But, if extreme excesses of ignition energy occur, even this tendency cannot keep explosions from happening. Also, extremely high ignition energies can cause such a large increase in initial combustion pressure that the propellant itself may become torn or cracked as a result of its own strength being greatly exceeded. Then, the additional surface area that is exposed starts to burn. Since the burning area is now larger than the design value, excessive pressure increase is again produced, possibly leading to an explosion.

Some ignition compounds burn very rapidly, even to the extent that the ignition process becomes an explosion of its own. If a propellant is highly loaded with oxidizer, such that individual crystals of oxidizer and/or other particulate ingredients can physically touch each other (this can happen in propellants that have as low as 80% oxidizer loading), then it is possible, in some cases, for the propellant to sustain a shock wave within itself. If this happens, the propellant is liable to explode due to the shock wave passing through it. This is another reason that the ignition process should be as controlled as possible. Also, it is a good reason to design the igniter housing itself such that it can dissipate any shock wave that might accidentally be produced so that the shock wave does not directly strike the propellant.

In addition to shock wave dispersion, it is also desirable to have the flame and gasses produced by the igniter dispersed over the propellant surface. If not dispersed to at least some extent, then the flame may only strike a small portion of the propellant surface, causing it to burn away much faster than it should. This could alter the geometry of the propellant grain to such a degree as to compromise the performance of the engine. It can also lead to an excessive ignition delay if only a small portion of the surface ignites, since the flames of the burning propellant would then have to spread slowly over the rest of the propellant surface. Ideally, the entire surface of the propellant would ignite at the same time. Although when under pressure the spread of flames can be thousands of inches per second, at atmospheric pressure (the pressure at the start of ignition), this speed can be as low as only 2-5 inches per second. If the propellant grain is several inches (or feet!) long, then extremely long ignition delays can occur.

When discussing heat and heat flux, one can use two kinds of scientific dimensions, that is, metric or English. Typically, heat flux for ignition compounds seems to be measured in metric terms, that is, calories, rather than in English terms, such as BTUs. This is the convention that will be used in this course, as well. Experiments by many researchers over many years and using various kinds of equipment have shown that a reasonable value for the heat flux needed to just barely ignite a typical solid propellant, of almost any chemical composition, is about 30 calories per square centimeter of exposed propellant area per second of duration of ignition. So, if one has a propellant burning area of 10 sq. inches and wishes to have a duration of 0.05 seconds, then it is necessary to have at least:

$$30 \text{ cal/cm}^2$$
-sec x 0.05 sec x ($10 \text{ in}^2 \text{ x } 2.54^2 \text{ cm}^2/\text{in}^2$) = 96.774 cal.

However, this would only be enough energy to barely (translate that as <u>unreliably!</u>) ignite the propellant. Our goal is to have a definite, reliable ignition. The standard procedure is to use approximately four times as much energy as is required for minimum ignition, that is, use about $4 \times 30 = 120 \text{ cal/cm}^2$ -sec. So, for 10 sq. in. of area, we need $4 \times 96.774 \text{ cal} = 387.096 \text{ cal.}$, or, about 387 calories. (Note that at least one group has indicated that using less than, QR more than, 4X the minimum ignition energy can result in excessive ignition delays and possible ignition problems.)

An important factor in the quest for good ignition is the amount of gasses that are produced by the igniter. Some compounds produce little, or no, gasses while others produce nothing BUT gasses. How does one determine the level of gas production that is desirable and necessary? And how does one determine the proportion of gasses produced to any solids or liquids produced? The basic idea is that the igniter should produce enough gas, in addition to the heat flux, to raise the engine pressure to about 30-40% of the desired operating pressure. If a closure is used at the nozzle exit (known as a "burst disc"), then this closure should be designed to blow out at 90-100% of the desired operating pressure. If a closure is not used, then it would probably be better to have the igniter produce a pressure of about 75-85% of the desired operating pressure. Also, it helps to have hot particles and/or hot liquids produced.

The reason for the need for hot gasses is as stated above. But, also, the hot gasses are going to be coming out of the igniter housing, and traveling down the grain, at rather high velocities. This means that the gasses will be "scrubbing" along the surfaces of the propellant. This is known as being "turbulent". When gasses move quickly, and especially when they move in a strongly random fashion (turbulence), they are able to transfer very high quantities of heat from themselves directly to the surfaces with which they come into contact. This process is known in thermodynamics as "forced gaseous convection". This greatly enhances the temperature rise of the surface of the propellant and contributes to a quick ignition process.

The presence of hot particles and/or hot liquids (like molten aluminum oxide, etc.) provides an additional way to transfer large quantities of heat to the surface of the propellant. As the particles or liquids strike the surfaces (a process known as "conduction"), large amounts of heat are transferred. Also, as the liquids hit the surface, there is a certain amount of "forced liquid convection", as well. As with the gasses, this allows huge amounts of heat to be transferred in a short period of time. The other advantage of having hot particles and liquids in the igniter exhaust is that a significant amount of heat transfer to the propellant is due to the radiation of heat from this super-hot cloud of particles. This is an important source of heat for ignition and should not be done without, if at all possible.

COMPOUNDS AND COMPOUNDING:

ALCLO:

The name of this mixture is derived from the names of its ingredients, Aluminum powder and potassium perChLOrate. It is an extremely hot (about 3000 - 7000 F) ignition composition that spews out solids, liquids and gases. The gas is mostly potassium chloride (KCl) and some aluminum oxide (Al₂O₃). Roughly 30% by weight is gaseous KCl and about 70% by weight is solid and liquid Al₂O₃.

BKN:

The name of this composition is also derived from the names of its ingredients: Boron and potassium (K) Nitrate. It is a milder ignition compound than ALCLO and produces about 35% gaseous and 65% solid products of combustion. It is a safer mixture to use than ALCLO (which is impact sensitive as well as being sensitive to static electricity) but boron

is a very expensive element and so it is unlikely that experimental rocketeers will want to use it very often.

Smokeless Powder:

Smokeless powder is essentially nitrocellulose. For all practical purposes, it produces 100% gasses and almost no significant amounts of solids (hence, the name "smokeless"). Although it is effective in raising the ignition pressure, it produces almost no hot particles. So, an extremely important source of heat transfer is missing if this composition is used. Also, since it is so energetic, one must be very careful not to accidentally use too much.

Black Powder (BP):

This composition derives its name from its color. It is made up of a mixture of charcoal, sulfur and potassium nitrate. To work properly, it must be made up of finely-ground particles of each of the ingredients and they must be intimately mixed together. It should be kept dry at all times, as it can be erratic in how it burns if it is too moist. BP is manufactured in several different particle sizes, from very fine to a coarse size which is suitably slow-burning for use in home-made cannons. By mixing together different particle sizes, one can get the gasses, hot particles (essentially carbon particles), hot liquids (more than half a dozen solid and molten chemical compounds), heat transfer, and ignition duration desired. Adding a powdered metal to the mixture can increase its effectiveness, too, up to a point. If too much is added, however, it can rob oxygen from some of the other ingredients and contribute to a lowered level of gas production. In this course, BP, laced with titanium powder to give additional quantities of hot particles at a much higher temperature than BP would normally produce, will be used as the igniter composition.

SAMPLE CALCULATIONS:

When using BP, the following formula can be used to estimate the amount of BP needed to raise the pressure to a given value in a given volume⁽¹⁾:

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p = 2000 x ( W/v)^{0.86}, where p = pressure, psig; W = weight of BP used, grams; v = volume to be pressurized, cubic inches

So: W = v x (p/2000)^{1.16279}

Let: v = 10 cubic inches, p = 500 psig x ( (0.75 + 0.85 ) / 2 ) = 500 x 0.80 = 400 psig. (We are assuming that no nozzle closure is being used.)

Then: WBP = 10 x (400 / 2000)^{1.16279} = 1.539 grams or approx. 1.54
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(If a nozzle closure were being used, then only about 0.589 gram is needed since the pressure to which the internal volume would be raised would be only 150 - 200 psig instead of 400 psig.)

To this, then, would be added the titanium powder in whatever amount desired, but not so much as to interfere with the combustion of the BP. The combustion of the titanium to form titanium dioxide particles would raise the temperature significantly. Thus, it may be that slightly less BP would be needed for gas production, since the extra heating of the gas would also raise its pressure. This would probably require some experimentation, unless thermochemical calculations could be done.

When using ALCLO or BKN, calculations are done differently. It is necessary to know the heat flux output of these compounds⁽²⁾. ALCLO has an output of 2300-2500 cal/gram and BKN has an output of 1450-1550 cal/gram.

Let's do calculations for ALCLO. Assume 85 square inches of exposed propellant surface area, a desired heat flux of 120 cal/ sq. cm-sec, and an ignition duration of 0.05 second:

85 sq. in. x 6.4516 sq. cm/ sq. in. = 548.386 sq. cm. or about 548 sq. cm.

Then: $120 \times 548 \times 0.05 = 3,288$ calories of heat needed.

The average heat flux available from ALCLO is 2,400 cal/gram.

So: 3,288 calories / (2,400 cal/gram) = 1.37 grams of ALCLO needed.

While this shows about how much ALCLO is needed, it says nothing about the size (or size range of sizes) of the ingredients used to make up the igniter composition. If extremely fine powder is used, then it is likely that the duration will be less than the desired 0.05 second. On the other hand, if the powder were to be pressed into a pellet, the duration might be 0.05 sec, or even more. Typically, durations of no more than 0.100 second are needed to ensure adequate ignition of the propellant. If we assume that ALCLO burns at about 1 inch/sec, then, if it burns from both sides of a particle, that particle would need to be about (2) x $(0.05 \sec x \ 1 \ inch/sec) = 0.1 \ inch thick$. This is about the thickness of an ordinary aspirin-type tablet and, in fact, this is a common shape for igniter pellets, even up to several inches in diameter! Since a typical ALCLO pellet of this approximate size weighs about 0.38 gram, then one would need:

1.37 grams /(0.38 gram/ALCLO pellet) = 3.605 pellets or about 4 pellets.

In reality, the four pellets would probably have a small amount of fine ALCLO powder as well as a small amount of ALCLO granules added. These additional particle sizes, with their much larger surface areas and greater sensitivity to ignition, would help ensure the ignition of the bigger pellets.

A NOTE REGARDING SAFETY: Pyrotechnic ignition compounds may be purchased from fireworks suppliers or made up by an individual who is doing his own experimentation. Only very small amounts of any (but, especially, NEW) pyrotechnic compound should be made at any one time, and gentle mixing techniques MUST be used. Adequate safety equipment and apparel must be used to avoid being burned if the mixture should accidentally ignite during processing, and a fireproof barrier must be between the mixture being made and the mixer. If larger quantities of igniter compounds are needed, it is better to make up several, even many, small batches and only combine them into one large batch when all processing has been completed. In this way, if there is a fire or

explosion, either of which is more likely to happen during processing than during combining into a larger quantity, the risk of personal injury or death is minimized.

If pelletizing of a pyrotechnic compound is done, be aware that it usually must be pressed at high pressure to make it hold together, from a few hundred to as much as 10,000 psig, and, sometimes, even more. There is also friction and impact involved in the process. When doing this, provide enough protection in the form of barriers, safety clothing, goggles, etc., so that no part of the body is exposed to the area of pellet formation in the event that the pellet explodes during manufacture.

Also, be sure to keep detailed records of any experiments. By reviewing these records, it will be possible to plan in such a way as to avoid previous mistakes, and to get as much helpful information from future experiments as possible.

Finally, always keep in mind that there is no substitute for experimentation. Anything that ignites and will sustain combustion in a closed space, like the inside of a rocket engine, may be useful as an igniter composition. Whether a new compound is discovered by experimentation or procured from a fireworks manufacturer, it is extremely important to think out the procedure to be used ahead of time, plan for any possible problems that might arise, and be prepared with adequate safety procedures, equipment, clothing and physical barriers in case of a fire or explosion.

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Beginning Composite Mixing, Casting and Curing

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This text was designed to be used with the "hands on" training provided during the RRS solid propellant course. It will provide a complete procedure for manufacturing a Bates type propellant grain. The article on mixing, written by John Rahkonen (and included at the end of this book), perfectly compliments the following procedural text and is highly recommended reading prior to the beginning of the course field activities. Since HTPB, and any other polymers you might choose for propellant work are mostly used in industry, there is a tremendous amount of information on these materials available simply by contacting the manufacturer or related businesses. In fact, many types of expensive polymer samples can be had just for the asking.

The following procedures, though applicable in general, are specific to the mixing, curing and firing at the Reaction Research Society's Mojave Test Area. These specific procedures require two days for propellant preparation. Mixing, casting and curing are completed in one day; Coring, prepping and testing will finish on the second day.

The following items are universally needed during any composite mixing evolution:

- Source of electricity For mixer and curing oven.
- A lab notebook, pencil Three ring binder for project documentation.
- A very easy to read stop watch that you don't mind getting propellant on.
- Mechanical mixer Kitchen Aid Type tare weight for mixing bowl.
 Triple Beam Balance accuracy to 1/10 of a gram
- Weight set large enough for maximum propellant weight. Used for propellant ingredient weigh out.
- Digital Scale accuracy to 1/100 of a gram
 - Capacity at least 70 grams. Used for igniter formulating.
- Heavy duty aluminum foil weighing out different material, general purpose.
 Small pieces of plywood or any stiff support transporting uncured propellant.
- Wax paper setting curing propellant ends on, general purpose.
- Small, chemically compatible plastic bottles weighing liquid ingredients.
- Ziploc gallon bags pre-weighed chemicals and finished grains.
- Several permanent marking pens.
- A case of good quality paper towels.
 Box of surgical gloves.
- Vapor mask.
- Mixing spoons and tongue depressors.
- Compatible solvents for polymer used Acetone for HTPB
- Water
 - Large bucket of water and fire fighting source for secondary fire control.

Water for washing hands and eye wash - Borax soap

MIXING - No matter what means of mixing you decide upon, you must use a clear check off list. Several dry runs should be done making inert grains. Sugar can be substituted for

the oxidizer. These practice runs allow for procedural improvements and refining technique. They also provide inert grains for discussion and study.

MANUAL MIXING - (Hand mixing) - 1s typically undesirable from the stand points of health, fire exposure and uniformity of propellant blending. Inhalation of uncured isocyanates is undesirable. Hands (gloved or not) covered with mixed propellant provides a opportunity for serious burns. Additionally, hand mixing can not provide the reproducibility of mechanical mixing.

Planning out how much propellant you will need to mix is calculated by finding the area of an individual Bates cartridge. Then multiply the number of grains in each motor by the number of motors. We will be mixing enough for 30 grains.

(See Cartridge Drawing) - D1 = 2.055 inches, D2 = 1.000 inches, L = 4.000 inches

Area of D1 = $(2.055/2)^2$ x = 3.317 square inches (Since we are creating the central core after casting the volume of the entire cartridge needs to be calculated.)

Surface Area = 2.532, Volume = Surface Area x Length, 3.317 x 4 = 13.268 cubic in.

DENSITY - The mass of a substance in grams divided by its volume in cubic centimeters will give its density. The density of our propellant, determined through experimentation with live and inert grains, is .054 pounds per cubic inch.

Therefore: $.054 \times 13.268 = 0.7165$ lb. $\times 453.6$ grams (a pound) = 325 grams per cartridge.

Always mix a little more to allow for what sticks to the mixing bowls, utensils, and gloves.

The math says 325 grams. However, to be sure we have enough, we will say each grain needs 358 grams of propellant. Then, 358 times the number of cartridges (30) gives us 10,740 grams of total propellant.

Mixing Equipment

MTA MIXING CHECK OFF LIST

 Review with mixing and loading crew, procedures (Summary).
 Check safety equipment - assign responsibilities
 Verify cellular capability in case of emergency.
 Check generator - gas/oil (top off before commencing test operations)
Check mixer and curing oven in actual running operation
 Set up scales and polymer prep table
 Lay out propellant formulation chemicals
 Level and tare balances
 Propellant team briefing

A good scale is essential. You can find them in the bargain box or at school auctions. A common scale is the Ohaus triple beam or platform balance. A extra set of weights is nice to have. If the extra weights are not available, you can weigh out pieces of metal and mark them with their weights.

A mechanical mixer is one of the most important tools needed for composite propellant formulation (along with an accurate scale). A mixer is also typically the most expensive item needed by an individual experimenting with polymers. However, one of the many assets that the RRS has to offer its membership, is the use of either a Kitchen Aid 5 quart and/or a planetary motion 20 quart mixer for a nominal donation to the Society's Research Fund. The Kitchen Aid 5 quart mixer is a must for any serious composite work. A lot of grief can be spared by having the ability to mix and perfect your pre-oxidizer inert grains at home. You can always sell the mixer to other members if and when you no longer need it.

If you decide to purchase the Kitchen Aid, a good source for a new mixer is Price Club. They charge around \$250.00 for a K5 mixer. Do not purchase the K45 model. For the small difference in price, it is not worth purchasing the smaller K45. The K5 bowl attachment and motor size lends itself to composite mixing. If you purchase second hand through the newspaper or garage sale, always test the unit before you purchase it. Typically, you can judge the condition of the unit from what the outside looks like. Always plug it in and listen for scrapping sounds. Such sounds, from either the planetary gear head or from the beater hitting the bowl, suggest you keep looking. If you pick up a used Kitchen Aid, there are numerous individuals that can repair them. Factory repairs can be expensive.

An advantage of having a K5 mixer is that one of the members of the society, Larry Teebken has designed a Plexiglas top that fits the K5 for vacuum formulation of propellant. As a general rule, any propellant that can be poured needs to be vacuum degassed. The beginning composite class propellant will not be vacuum degassed.

Our method of mixing involves using a planetary style mixer, such as a Kitchen Aid. We do our polymer premix in a 5 quart and our final mix (with oxidizer) in a 20 quart. Our experiments are done with very short mixing times. Typically, composite polymer mixing times are measured in hours. We are mixing in 5 minute intervals (4 five minute mixes). The 5 quart is more efficient because of size so we do the polymer premix in it. Since 77% of the propellant is oxidizer, the 20 quart is needed for the final mix.

CASTING TECHNIOUE FOR CLASS PROPELLANT

The casting technique utilized for this propellant is done without vacuum, external heating or mandrel pulling. It is the simplest method and produces quite satisfactory results. It seems to produce a uniform porosity throughout the propellant grain. Experiments to date show a uniform cured propellant density and burn rate.

Each step of the grain filling procedure is stepped through prior to actual processing with propellant. This rehearsal helps to maximize your efficiency after the propellant is mixed and the catalyst is actually starting the curing process.

Since the class rocket motor utilizes a PVC propellant cartridge, preparation of the cartridges is done first. The PVC liners need to be cleaned. Acetone is used to "clean" the surface so that the propellant will properly bond to the PVC.

Wax paper is cut to place under the grains during filling and transporting to the curing ovens. This is one of the little, easy to over look steps that causes unnecessary waiting while the propellant is curing in the mixing pot.

Pieces of wax paper are placed under each PVC propellant liner before filling with propellant. The mixed propellant is the consistency of bread dough. The wax paper

provides a means of closing off one end of the liner, and also allows transfer of the packed grain to the curing oven without it sticking to the tray.

Rubber medical gloves are wom during all propellant handling operations. A handful of propellant is placed inside the PVC cartridge. Simply pushing the propellant into the upended PVC liners using the center and index fingers fills the cartridge. The cartridge is finished off by patting the surface of the propellant with the flat surface of an open hand (rubber gloved) until filled to about 1/16 of an inch above the PVC cartridge.

CURING

As conveyed in the propellant section of this course, curing can be done at all temperatures. Our propellant was formulated for cost, ease of preparation and speed of curing considerations. Speed of curing was the most important target during the formulation stage since all oxidized propellant work is done at the MTA over a weekend. To speed up the curing process, a thermostatically controlled, insulated oven has been designed and built. Many different types of ovens could be built, but whatever design is selected, it should have most or all of the following characteristics;

- Easy to move Lightweight (Portability)
- Rugged
- Distributed air circulation
- Simple circuits, simple shielded heat source (heavy duty light bulbs)
- Well insulated
- Stand Alone No shared parts

A curing oven designed for use at the MTA, located in the upper desert environment, requires heavy insulation and rugged construction. The RRS's curing oven was built by George Garboden and is constructed of a 35 gallon drum fitted inside a fifty gallon drum. Solid styrofoam fills the walls between the two drums. A electronic thermostat is mounted to the oven lid and is connected to two 100 watt heavy duty light bulbs and a 5 inch duct fan. Plywood internal shelves are provided and are suspended by three lengths of all-thread. The duct fan and a built in air conduit provides even temperature throughout the entire vessel. The propellant you will process will be cured at 115 degrees F. This oven design is inexpensive to build, works well, and will provide a lifetime of hassle free use.

Curing ovens can also be made simply from Styrofoam ice chests. However, the use of this oven is another item the Society can provide. If you need to build your own oven, make the shelves adjustable so that you have flexibility in curing different grain lengths.

DOCUMENTATION

Written records documenting every aspect of any propellant mixing operations always prove to be invaluable later on. Information recorded should include the timing of each procedure, any deviations from established procedure, notes on possible equipment or process improvements to be implemented the next time around. During your inert mixes, it is very useful to get into the habit of taking copious notes. You can never over document your experiments.

STARTING THE MIXING OPERATION

The information located on the next page will be used to prepare the chemicals required for the propellant mixing operations required as part of this course;

Propellant Formulation List MTA - Field Work

Formulation Weights:

INGREDIENT	PERCENTAGE	WEIGHT
AP 200 Micron	61.68	6626.58 grams
AP 400 Micron	15.42	1653.96 grams
Aluminum	6.70	719.60 grams
HTPB	10.00	1074.00 gram
DOA	4.40	472.60 gram
HX878	.70	75.18 gram
PAPI	1.10	118.14 gram
Total	100.00	10,740.00 gram

Anti-foam - SAG = 10 drops (1 drop per thousand grams of total propellant weight)

10,740 grams / 453.59 grams (one pound) = 23.67 lbs of propellant.

Weighing Procedure: (Tare weighing vessel on scale) Always have a second person verify incremental measuring amounts.

The following ingredients are measured (in order) into the tare'd 5K mixer bowl. BE SURE AND FOLLOW THE ACTUAL MIXING PROCEDURE ON NEXT PAGE.

GRAMS	INGREDIENT	TOTAL WEIGHT
1074.00 grams	HTPB	1074.00 grams
472.60 grams	DOA	1546.60 grams
75.18 grams	HX878	1621.78 grams
719.60 grams	Aluminum	2341.38 grams
118.14 grams	PAPI	2459.52 grams
24 10 drops	59.52 grams SAG (Silicon Oil)	
	1074.00 grams 472.60 grams 75.18 grams 719.60 grams 118.14 grams Total Po	1074.00 grams HTPB 472.60 grams DOA 75.18 grams HX878 719.60 grams Aluminum 118.14 grams PAPI Total Polymer Premix Weight 2459.52 grams

iner + 748 Srams 329.2 STAR 52.4 501.2 300 82. 2 gran 7 diops of SAG 5767.0 AP 200 micror

Mixing Procedure

Starting Time:			
PREMIX:			
Combine and mix for five minutes all polymer ingredients w/o AP for 5 minutes.			
TRANSFER:			
Transfer premix to 20 quart mixing bowl.			
AP ADDITION:			
Add AP all at once while mixer is unplugged. All of our propellant oxidizer mixing is done remotely. This is accomplished simply by use of extension cords. Unplugging and plugging in the mixer from at least 50 feet away. The mixer is plugged in remotely and mixed for 5 minutes.			
FIRST SCRAPE DOWN:			
Stop the mixer remotely and scrape down the propellant. After this swift scrape down, the oxidized propellant is mixed another 5 minutes for a total of 15 minutes (including Premix mixing time).			
SECOND SCRAPE DOWN:			
Another stop, scrape down cycle and mixed another 5 minutes. Total of 20 minutes. (Mixing is now complete)			
THIRD SCRAPE DOWN (PRIOR TO FILLING):			
Stop, scrape down the sides of the mixing bowl and proceed to fill the PVC liners.			
Note: Since the weight of the uncured propellant will slump out the bottom of the cartridge, transportation of the uncured propellant to the curing oven is done by sliding a thin flat sheet of aluminum or any thin stiff material under the wax papered end of the PVC liners, either before filling them or sliding them under after filling.			
Time in Oven			
Curing Time at			
115 ⁰ F for 5 hours Time Out Oven			

PREPPING OF THE PROPELLANT GRAINS - SANDING

The next step in grain preparation is the sanding of the grain ends. This provides a means of trimming the grains to final length and creates a fresh, oxidizer rich propellant surface. This step is accomplished with a special disk sander built with an explosion proof (sealed so that none of the sanding dust can enter the motor housing) electric motor. An alternate method of grain trimming (knife trimming) may be used with some students during this phase of the operations. This operation creates the greatest concern for accidental ignition of the propellant grain. It is accomplished remotely and, if there is no a wind, a fan is positioned to disperse the propellant dust away from the individual doing the sanding. There are several methods being investigated currently to replace the sanding operation.

PREPPING OF THE PROPELLANT GRAIN - CORING / DRILLING

Our Bates grain design calls for a 1 inch core in the propellant cartridges. This can be done several ways, either by using a mandrel during casting or by drilling after the grain is cured. Our method of creating the central core is to drill it out. We believe that an oxidizer rich surface is thereby produced and is desirable from an ignition standpoint.

When drilling each grain, you must pay attention to proper alignment of the core in the center of the grain. The drilling fixture you will used is designed to hold the grain and drill concentric with one another. The accuracy of this device is more than adequate for our purposes here. As a general note, alignment of this central port becomes increasingly important for motors with more propellant segments. The drilling itself must be done slowly. This ensures no heat buildup due to friction and it prevents tear out of the propellant as the drill emerges on the opposite side of the grain.

SAFETY NOTE

The number one source of fires during propellant handling operations is from work area cleanliness. No propellant tailings, dust, or cuttings should ever be allowed to accumulate. You must be diligent in this routine or you will not be experimenting very long. Drilling out the core produces a lot of cuttings, and sanding produces a fair amount of propellant dust. At least after every grain, depending on the size of the grain, all cuttings, dust, and shavings must be removed from the immediate work area and disposed of safely.

One of the points that speaks in favor of using a mandrel for creating the center core is the elimination of the drilling process and the need to dispose of center core cuttings. (There are other concerns, however, in the actual process of pulling the mandrel.) The only safe and permanent way to dispose of cuttings, is to remotely burn them off under controlled conditions.

One of the important aspects of this course is for you to see a propellant burn off in person and understand that once a propellant fire starts, it is impossible to put it out. It is also important to experience a propellant burn off so that you have a first hand experience with the ferocity of burning propellant. In the event of a propellant fire, the best you can hope to do is attempt the suppression of the secondary fire created in nearby flammable materials. Propellant processing should never be done near any appreciable source of combustible secondary materials. This is the single greatest reason for doing all of you oxidized propellant work at the MTA where the remoteness of the facility ensures that there is no hazard to surrounding personnel or property not directly involved. If at any time you have any questions or are at all unsure of what to do next, STOP immediately and get help from one of the instructors.

STATIC TESTING

by
Thomas Mueller
Pyrotechnic Operator, Second Class
Reaction Research Society, Inc.

INTRODUCTION

One of the main things that differentiates experimental rocketry from model rocketry, sport rocketry, high power rocketry or even basement bombing, is that in experimental rocketry the rocket motor is tested in a static firing to evaluate its operation and performance. Usually, data is collected and is used to characterize the performance of the motors, confirm the design parameters, and allow adjustments and improvements to be made.

STATIC TEST STAND

Static testing involves mounting the motor in a static test stand such that the motor is immobile. In the simplest static test, no data is collected, but the operation of the motor is evaluated by firing it in a restrained condition before committing it to use in a flight vehicle. Firing a new motor design for the first time in a flight vehicle is not prudent. After expending many dollars and hours of labor in designing and building a flight vehicle, it would not be a good time to find that your motor design is prone to blown cases.

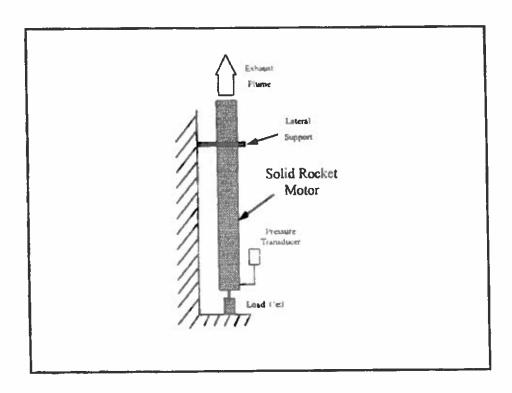


Figure 1. Static Test Stand

Probably the simplest way to conduct a static test, and one often used on new solid propellant motor designs, is to bury the motor in the ground with the nozzle pointed straight up. In this way, the motor is supported in a test stand that requires little more investment than a shovel and some labor. As an added bonus, if the motor should burst, most of the fragments are contained by the dirt.

In the case of mature motor designs, a more sophisticated test stand is employed that allows the experimentalist to acquired test data from the motor. Unlike a liquid rocket, a solid rocket can be fired in any position without affecting the operation of the motor. This allows the convenience of firing the rocket vertically upwards, with the thrust reacted directly by a ground mounted thrust plate. This approach reduces the size and complexity of the test stand, since the stand doesn't have to resist the full thrust load. Figure 1 shows schematically how such a thrust stand is employed. The motor fires against a load cell that is mounted firmly on a thrust plate. The connection to the load cell is usually a gimbaled clevis joint, like an automotive tie rod end, that allows the axial load to be transmitted without any bending loads that could give an erroneous reading or damage the load cell. The top end of the motor is restrained in the lateral direction, but allowed to slide freely in the vertical direction. This is accomplished simply by supporting the end of the motor in a hole in a fixed plate that has a small clearance gap around the motor diameter. The plate should be fairly strong and rigid, since a significant side load can be generated by the motor during firing. For this same reason, the base of the test stand should be wide and fairly massive, or else have some sort of support such as guide wires to prevent a thrust side vector from tipping it over. The load cell must be mounted square to the axis of the motor, and should be mounted rigidly on a heavy plate to prevent any significant flexing, which will show up as "ringing" in the thrust data.

DATA ACQUISITION

WHAT IS DATA ACQUISITION?

Data acquisition is the process of measuring and recording certain parameters during the hot fire test, such as thrust and pressure. One of the most simple data acquisition techniques is to use a pressure gauge and scale to measure the pressure and thrust of a rocket motor, and to film or video tape the gauges during the firing in order to record the data. The data can then be manually recorded and plotted later by playing the tape back in slow motion and writing down the data points versus time into the burn. Time is resolved by counting frames or by a digital time stamp on the video recording, a feature of most video cameras.

A more sophisticated and useful type of data acquisition is an analog to digital converter that records data directly into a computer. This is the type of system that is employed by the RRS, and will be described in this section. Figure 2 shows a typical digital data acquisition setup for a solid rocket motor test.

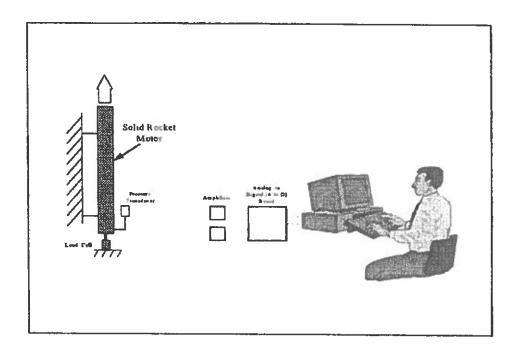


Figure 2. Typical Data Acquisition Setup.

DETERMINING WHAT TO MEASURE

The first task in setting up data acquisition for a static hot fire test is to determine what data to measure. For a solid rocket motor, two data channels are usually measured, thrust and chamber pressure. Thrust is important in determining the total impulse, specific impulse, and peak and average thrust values. Chamber pressure is used to determine propellant C*, burn rate constant and exponent, and peak and average Pc. Time is recorded by the computers internal clock. Additional data that should be recorded prior to the static test is the nozzle throat area, total weight of propellant in the motor, and ambient temperature.

GENERATING A SIGNAL - INSTRUMENTATION

The data acquisition system starts with an analog signal generated by instrumentation connected to the motor. The thrust measurement instrumentation is a load cell, which is usually based on a bending beam that strain gauges are bonded to. The thrust of the motor deflects the beam, which causes the strain gauges to generate a small voltage signal that is proportional to the load applied.

Pressure transducers are used to measure chamber pressure. A typical pressure transducer also uses strain gauges to generate the analog signal. In this case they are bonded onto a metal diaphragm that the pressure acts on and deflects. Other types of pressure transducers use silicon strain elements that generate a signal based on the strain applied to the silicon element.

GETTING THE SIGNAL TO THE COMPUTER

The analog signal generated by strain gauge or silicon element instrumentation is usually a very small signal, on the order of millivolts. This signal must be amplified to a higher value before it is converted to a digital value. Sometimes the amplifier is built into the transducer, so that the device puts out a high level signal, typically 5 volts full scale. In other cases the signal goes to a amplifier module, then to the computer.

At the computer, an A to D board in the computer expansion slot converts the analog signal to a digital value that the computer can store. A typical A to D board has 8 channels of data capability, and converts the analog signal to a 12 bit digital number. 12 bits equates to a resolution of $2^{(12)} = 4096$ steps. With a 0 to 5 volt input signal, for example, 0 volts would generate the binary number 0000 00000000 which is 0 in decimal, and 5 volts would produce 1111 11111111 which is equals to 4096 in decimal. The software converts these digital numbers to actual test data by applying a scale factor and offset to them to convert the data to useful units.

A typical A to D board can read 100,000 digital samples per second, which is divided over eight channels for a per channel maximum data rate of 12,500 samples per second. If such a high data rate is desired, a fast computer processor is required in order for the software to keep up with the data. In practice, such a high data rate is not usually needed, as more data is recorded than can be easily managed. Often, the response rate of the instruments are less than the sample rate, so no additional information is obtained by very high sample rates. A typical sample rate is 100 samples per second for a static test. This generates a file with reasonable size for plotting and data reduction.

ENSURING ACCURACY - CALIBRATING THE DATA SYSTEM

To get accurate data during a test, a method of calibrating the data acquisition system must be employed. Typically the instrumentation comes with calibration data that relates the signal out to the input force or pressure. Then, if the gain of the amplifiers are accurately known the analog signal to the A to D board can be calculated, allowing the digital number to be converted to the actual parameter being measured.

In practice, the RRS does an end to end calibration of each data channel prior to a static test. To calibrate a pressure transducer, the transducer is pressurized by a nitrogen or helium gas source which is measured with an accurate pressure gauge. A digital reading is taken by the data computer to verify that the digital number represents the gauge reading. The calibration is adjusted if needed by changing the scale factor or offset constant of the digital number.

In the case of thrust, calibration can be accomplished by placing a weight of accurately known mass on the load cell and recording it with the data computer to verify the calibration. This method is not practical if it is desired to calibrate to a thrust value in the hundreds of pounds. In this case, a force dynamometer is used to determine the force exerted on the load cell.

DATA REDUCTION AND ANALYSIS

Just prior to a static firing (typically at the count of 2) the data acquisition system is started. During the firing, the digital data is stored in the computer's RAM. After a static test is completed, the data from the test is saved in a file on the computer's disk drive. This data file is used to post process the data to determine the performance of the rocket motor.

Post processing involves plotting out the thrust and Pc versus time, as well as doing rata reduction algorithms to determine such things as total impulse (area under the thrust-time curve), specific impulse, C*, and average and peak values of thrust and chamber pressure. Figure 2 shows a typical thrust - time plot for a solid rocket motor test.

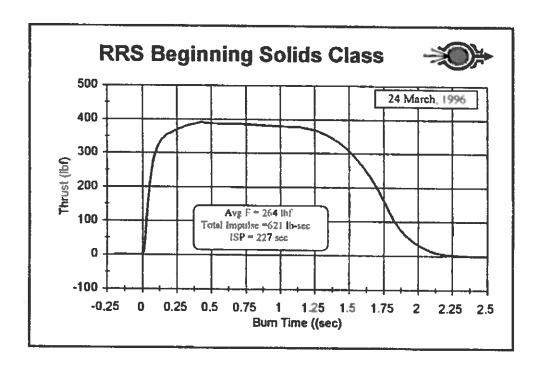


Figure 3. Thrust trace from solid rocket firing.

APPENDICES

The Art of Solid Propellant Mixing

By
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Before mixing solid propellants, there are several important precautions to observe which will increase your margin of safety.

- 1) Always mix in a location which is free of any combustible materials such as gas cans, paint, spray cans, paper, boxes, etc.
- 2) Always have fire extinguishers and open buckets of water in the immediate area in case of fire.
- 3) Always have safety equipment available such as face shield, respirator, rubber gloves, flame proof apron, etc.
- 4) Mix propellant only in a stainless steel mixer bowl or beaker because by grounding your hand or wrist to the outside of the mix bowl you can discharge static electricity safely from the propellant through your body so no sparking occurs at the propellent surface. The stainless steel mix beaker also distributes heat evenly, is easy to scrape down and clean up and takes a lot of punishment if dropped or banged up. Do not use glass or plastic mixing beakers.
- 5) Never smoke in a propellant mixing area.
- 6) Read all the Material Safety Data Sheets pertaining to the chemicals you'll be using.
- 7) Avoid dusting when weighing or adding the dry powders. If aluminum or AP dust occurs around your mixing area, use a small fan to blow it away.
- 8) Cured propellant can be cut with a sharp knife like a piece of cheese but never cut a hard propellant piece with a "harmmer chisel" type of operation. It can be ignited by impact.
- 9) Propellant cores can be drilled on a drill press or lathe but should only be done at the lowest possible speed to avoid temperature build up due to friction.
- 10) If an electric mixer is used to mix propellant, avoid using electric egg beater type of mixer. The high speed of this type mixer will whip a lot of air into the propellant. Also, the high viscosity of the propellant after all the oxidizer has been added can cause these mixers to overheat and burn out. The electric mixers to use are the "Kitchen Aid" type or Hobart mash potato mixers with a flat beater paddle. Use only at the lowest speed possible.

- 11) If an aluminum mandrel is used to mold a core in a bates grain of propellant, the mandrel should be coated first with a good mold release (Teflon or spray mold release) so the propellant does not stick to the mandrel when it is extracted. The method I use is to freeze the propellant with the mandrel in it after it is fully cured. Frozen propellant has a higher modulous of elasticity (is stiffer) and has a decreased tendency to stick to a mandrel when it is being pulled out of the propellant.
- 12) There is no substitute for common sense where safety is concerned. One of the most dangerous accidents I have witnessed in my 49 years of making solid propellant rockets was as follows:

A propellant hopper car riding on steel rails dropped a small amount of propellant onto the rails and was not noticed. When another hopper car ran over this small amount of propellant, the propellant became pinched between the steel rail and steel wheels of the car igniting it. The burning propellant dropped down into a casting pit containing thousands of pounds of propellant destroying the building and millions of dollars in equipment. Propellant should not be pinched in threads where two metal parts screw into each other.

The second great cause of propellant mishaps is with the fine shavings and dust produced from drilling cores out of propellant grains. This dust is extremely static sensitive and should be continuously removed with a wet and dry vacuum cleaner containing water.

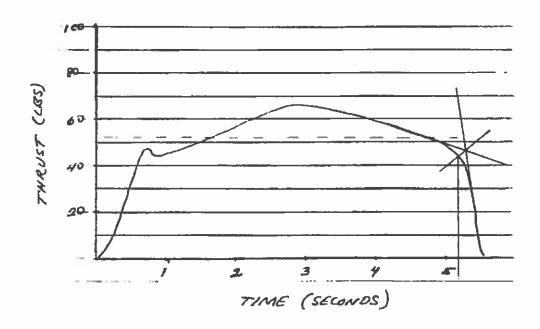
There are as many formulas for making a propellant as there are recipes for baking a loaf of bread. The bread baked at home is usually of a better quality than the mass produced bread at a large bakery since the prime prerequisite of a good loaf of bread is in the taste. We do not go by taste when we compare two propellants, so what are the characteristics we use to evaluate whether a propellant is so-so, good, or excellent?

A few parameters by which we compare propellants, which are common to all regardless of which ingredients are used in the formula are as follows:

VISCOSITY is a measure of the amount of resistance to mixing. A thick propellant which is difficult to mix has a high viscosity and a thin liquid propellant has a low viscosity. There is usually a trade-off between one extreme and the other. For example, as the solids loading of a propellant is increased (having a greater percent of dry ingredients) the propellant is dryer and more difficult to mix by hand. However, as the oxidizer and powdered metals percent is increased, the density and specific impulse are also increased as we approach the stoichiometric ratio. A low viscosity propellant is much easier to hand mix but the energy level and density are lower and it requires more reactive curative to obtain a full cure in a reasonable time period. It is also easier to degas a low viscosity propellant in a vacuum chamber since the air voids migrate to the surface easier.

SPECIFIC IMPULSE is a measure of the propellant energy in a rocket motor. During the testing of a solid propellant in a rocket motor, the hot burning gasses are expelled through the nozzle at ever increasing velocity as they approach the exit of the nozzle divergent cone. At the nozzle throat, the gas velocity is always at the speed of sound. The speed of sound is not a constant. The higher the temperature, the higher the speed of sound. Therefore, if you want a higher gas velocity at the throat, increase the flame temperature by either increasing the chamber pressure in the motor or increase the flame temperature of the propellant. If we increase the motor chamber pressure, we also must increase the tensile strength of the motor casing or make it thicker to withstand the higher pressure. This is a trade off between the inert weight of the motor and the specific impulse of the propellant. To understand the term specific impulse, let us static fire a rocket motor

on a thrust stand having a load cell calibrated in pounds of thrust. If we weigh our rocket motor before and after firing, an accurate propellant weight will be obtained. Our thrust/time curve will be:



In this illustration, the average thrust is about 52 lbs for approximately 5.25 seconds or 273 lb.-sec total impulse. If the amount of propellant consumed was 1.5 lbs., the equation for lsp becomes:

total impulse	273 lb-sec =	182 lb-sec
propellant consumed	1.5 lb	lb

If the pounds are canceled out on the top and bottom of the equation, the lsp of that formulation is listed as 182 sec. This is the delivered specific impulse of that particular propellant formulation, at that particular chamber pressure and nozzle expansion ratio and ambient atmospheric pressure. There are obviously many factors that effect the amount of energy obtainable from a propellant grain other than just the solids content of the formula itself. The theoretical specific impulse lsp (t) is the hypothetical maximum obtainable from a particular propellant formulation under the theoretical conditions of having no heat loss to the nozzle or case, complete combustion of the fuels, perfect nozzle expansion in a vacuum, etc., etc.

Of course these conditions are unobtainable in any practical application, so it is just used as a way to compare one propellant to another. Most rocket technicians and propellant chemists compare propellants by quoting the specific impulse at 1000 psi chamber pressure fired at sea level barometric pressure with an optimum nozzle expansion ratio. This "corrected" value of Isp is the only realistic one to use when comparing different propellant formulas.

PROCESSABILITY (pot life and cure time) If the propellant is mixed by hand, manually, it soon becomes quite tedious to blend all the ingredients properly unless the viscosity is relatively low (below 10 kilopoise). Therefore, if a propellant being mixed becomes too thick to mix properly by hand, even before the curing agents are added, there are only four practical solutions:

- 1) Reduce viscosity by decreasing the percent solids in the formula (or adding more liquids, polymer and/or plasticizer)
- 2) Increase the temperature of the propellant being mixed. (most high energy propellants are mixed at 135 to 150 degrees F.)
- 3) Use various oxidizer particle sizes such as 45 micron to 400 micron this gives a higher solids packing density by allowing the finer particles to fit between the larger ones. Also, if no oxidizer particles are finer than 45 micron, try adding 4 to 7% aluminum powder which is 20 micron or smaller and spherical.
- 4) Use an electric mixer (discussed previously), or a combination of any of the above listed techniques. If it takes 30 minutes to cast a batch of propellant, it becomes necessary to have at least a 45 minute pot life so that no propellant is wasted. There are few things more frustrating than to have a batch of freshly mixed propellant become too viscous to cast before there is time to get it all into motor paper tube liners. The length of pot life, (the time it takes for a slowly curing material to become too stiff to work with) is largely determined by the type of curative used, the type of cure catalyst (accelerator) used and the percent of each used in the formulation.

At the other extreme, a propellant can have a pot life of 10 or more hours which cures so slowly that after a month at room temperature it is still a sticky mass and it has not become completely cured even after several months of time. It becomes obvious that somewhere between these two extremes is the optimum pot life and cure time. It is also reasonable to conclude that the length of pot life is a good indication of the length of time it takes to fully cure a propellant. Many hours are spent in a propellant processing research and development laboratory to determine the pot life and cure time of a propellant formula. This is done by reading the viscosity of a propellant just after end of mix (EOM) with a Brookfield viscometer. The "T" bar of the viscometer is inserted into an 8 oz paper cup containing propellant and switch "on" the viscometer. As the "T" bar rotates slowly in the propellant at a rate of one revolution per minute, a dial attached to the "T" bar turns with it. The "T" bar as it slowly turns, is also sinking slowly into the propellant sample at a rate of 1/2 inches each revolution. Three readings are taken and recorded in a notebook at each of the three depths which are averaged out. This is noted as the end of mix reading (EOM). The temperature is also recorded using a Weston dial thermometer.

The propellant sample cup is then placed into a lab oven set at 135 degree F. Each hour, the sample is removed from the oven and another three readings are taken as before. These are all recorded in the notebook along with the temperature of the sample. After taking readings each hour for the next ten hours or so, a viscosity of 75 kilopoise is reached. Temperature is a critical factor in determining pot life and cure time. Comparing two samples of the same propellant, one at room temperature (75 deg F and one at 135), the propellant at 135 degrees will have a lower viscosity initially than the room temperature sample. However, the sample at 135 F will increase in viscosity at a higher rate and become more viscous than the other sample after about 4 or 5 hours.

The sample cup of propellant is not discarded after it has become too hard to take a viscosity reading. It is kept in the 135 F. oven for a day or two and then cut in half with a butcher knife. Using the freshly cut flat surface, a Shore "A" hardness gage is used to measure the hardness of the sample. This hand held gage, about the size of a pack of cigarettes, has a small round headed pin which is spring loaded. As this gage is pressed against a propellant sample, the softer the sample, the deeper the pin will penetrate into the sample. The dial on the gage will read a given value depending upon the depth of the pin in the sample material after a given number of seconds, between 15 or 20. This hardness test is repeated each day for the next 6 or 7 days or until there is no further increase in the hardness reading after two consecutive days. This is an indication that the propellant sample is completely cured.

This writer does not expect you to go out and purchase expensive equipment to test your home-made propellant. The above information is only given so you'll have an idea how the large solid propellant rocket corporations test various formulas to obtain the optimum material in their rocket motors. The making of good propellant is 10% chemistry and 90% trial and error with the continual testing of one formula or procedure versus another. In making and testing rockets, experience is far more important than having a college degree, provided that accurate records are kept which can be reviewed when needed.

The most important piece of equipment-you can have other than the "Kitchen Aid" type of mixer is an of oven to heat up" raw materials and mixing and casting apparatus. This oven can also be used to cure propellant grains and speed up the cure of epoxy used to bond nozzles and head caps. It is absolutely indispensable in colder climates or during winter time. This "oven" can be made very simplify purchasing a wooden packing crate in a surplus yard or building one from plywood. It should have a hinged door on the side and a hasp and padlock to secure it when it is used to store unused propellant grains. When the hinged door with handle is completed, cut and nail one inch thick Styrofoam insulation on the inside to decrease heat loss. This Styrofoam can be coated with a liquid fire retarding spray. Use a small electric heater with a fan to heat the inside. A hair dryer can be used for smaller ovens but they have the tendency to burn out coils during continuous use. The temperature can be controlled with sufficient accuracy through the heater thermostat or by using a variable rheostat (Variac). Drill a small hole in one side and insert a dial thermometer to check the inside temperature.

On a hot summer day, if the temperature outside is over 90 degrees F, the heater will not be needed since the oven will stay warm if it is painted black on the outside and it is placed in the sunlight. Have you noticed how hot it becomes in your car in summer with the windows rolled up? This oven should be placed outside, in the back yard, placed on a small wooden stand, a foot off the ground where it can sit in direct sunlight. Secure it with rope or cable to the ground so that wind will not blow it over. In winter time, when the sun does not help to heat it much, place the oven in a storage shed so the rain and snow does not warp or deteriorate the wood. It can also be coated with sheets of black rubber or black polyethylene Visqueen to protect the wood from rotting.

A minimum oven could be made from a cardboard box with two 100 watt lights to supply the heat. Never cure propellant in your home or basement where the isocyanate curative emits toxic fumes or children might knock it over. Before placing live solid propellant into your oven, test it for several days each hour to check its reliability in maintaining the desired temperature. The oven I built is 6 foot high, 5 foot wide and 4 foot deep. It has a 1000 watt electric heater with a built-in thermostat. When I heat up mix bowls and casting funnels, I leave the door completely shut but when I place propellant to be cured into the oven, I prop the door slightly open with a wooden block and two bungee cords to leave a half inch gap in the door opening. With the door propped slightly open, the air can

circulate more freely and will not pressurize and blow up if a propellant grain does ignite. The hot air from the heater is deflected so as not to blow directly against the curing propellant so no local "hot spots" can occur. This temperature conditioning box/oven has given me good service for many years and my initial cost was less than \$50 for it.

The next topic will cover the method I use to determine precisely the amount of curative to use in each propellant formulation depending upon the solids loading, curative type used and when I use a new lot number of the same type of curative. I first mix a 1000 gram batch of propellant which has all the ingredients thoroughly blended except the curing agent and cure catalyst. Next, I weigh out 50 grams of this propellant into each of 14 small plastic cups. Each cup is labled on the side and bottom with the letters of the alphabet A.B.C. etc. through the letter "N". Then I take a clean eye dropper and fill it full of the curative being tested. I carefully weigh out 30 to 50 drops of this curative into a small plastic cup which has been zeroed out on the balance. If the drops are counted and the 30 to 50 drops weigh 5 grams, it is simple to calculate the average weight of each drop. With the 14 propellant sample cups containing 50 grams of propellant each (labeled "A" thru "N"), I add 2 drops of curative to the sample cup A, 3 drops to B, 4 drops to C and so forth. The last cup, marked N will have 15 drops of curative added to it. Using a clean stainless steel spatula or a butter knife, I thoroughly mix the curative into each propellant sample, wiping the spatula clean with a paper towel after each batch is mixed. Then I record the data in a mixer log book so I know how much curative was added to each sample cup.

The next step is to take 14 clean plastic cups and label one of them A-1, another B-1, C-1 and so forth up to N-1. I split the propellant in cup A and put roughly half of it into the empty cup marked "A-I". This split of propellant between the two cups need not be accurate. A rough approximation of about 25 grams in each cup is sufficient. Then I do the same with cup B and B-1 and C and C-1 and so forth. Then, all the samples with the letter and number (A-1 thru N-1) are placed into a 135 degree F oven to be cured at that temperature while all the samples with just the letters alone are placed into an empty box and stored on a shelf. These samples will be cured at ambient (room) temperature.

After 18 to 24 hours have elapsed, check the samples in the oven by pushing a spatula into each propellant sample to see if any have cured up. The sample marked N-1 will be hard while the A-1 sample will still be soft. Test the samples at room temperature on the shelf in the same manner. Record in your notebook the time tested and the condition of the propellant in each cup. Repeat this testing procedure each day, at the same time or at regular intervals and record the condition of each propellant. At the end of six days, after most of the 135 degrees samples are fully cured and the room temperature samples are partly cured, the hard propellants can be removed from the plastic cups with a spatula and tested by bending it slightly with the fingers. The sample labeled N-1 will most likely be hard and brittle while the sample "A" will still be soft and hardly cured at all. Select the sample which is the strongest and has the best flex to it. It should be slightly stiff like a piece of rubber tire but should not break while bending it. If it is flexible like a rubber band, it is too soft.

Once you have zeroed in on the percent of curative to use in your formulation, all subsequent testing can be accomplished using only 5 or 6 samples instead of the 14 shown here. Each time your propellant formula is changed, with respect to amount of polymer, plasticizer or solids loading, you might run a small test like the above to insure that the proper curing agent ratio is being used. As you slice off a one inch slab of propellant, rub the cut surface with your thumb exerting some pressure to see if the oxidizer can be rubbed off the surface. Oxidizer can not be rubbed off the surface of a good propellant with the thumb.

I am assuming that all of you who read this have a limited amount of technical experience and a low budget. I am also assuming that you want to conduct your experiments in the safest possible manner without becoming a casualty or a statistic. As a child has to learn to look both ways before crossing a street, you will have to learn how to conduct your experiments scientifically and avoid all activities which might be unsafe. This instruction sheet is written for you, the beginner in the simplest of terms so you'll have no doubt about the ideas I'm trying to get across to you.

I began to build rockets as a high school junior in 1946, using the only propellant available at the time, the tried and tested black powder. From 1955 on, I graduated to using Micrograin (zinc/sulfur)propellant. In 1957, I made several batches of Galcit propellant using asphalt and KC104 oxidizer. In 1958 I began to make composite propellant using Polysulphide LP-2 binder with ammonium perchlorate oxidizer. All this time, I was reading every technical book on rockets that I could find. In April of 1961, I became employed as a chemist in the Research and Development Lab of a large solid propellant rocket company. In those days, the computer was not available so we did all our data reduction on static firing tests and instrument testing using slide rules and hand cranked Frieden calculators. It was the best way to learn what actually took place in developing a good propellant.

Over the past 40 years, perhaps 20 to 25 people have been killed in the solid propellant rocket industry, but most of these fatal accidents were caused by not being thoroughly familiar with the chemical ingredients being used or not complying with standard operating safety procedures. The safety record of solid propellants processing is very safe when compared to activities like fireworks manufacture or explosive manufacturing plants. A person should never become complacent when working with propellants and the work area should be kept clean and free of contamination and clutter at all times.

There are several mandatory pieces of equipment you will need even if you are hand mixing your propellant. The most important is the top loading balance to weigh the ingredients. This could be either a triple beam Ohaus or digital balance. Both should weigh in the metric system (grams and kilograms) since it is easy to convert to percent if the total mix weight is 500 grams, 1000 grams or 10 kilograms (10,000 GMs). The next piece of equipment to have is a stainless steel mixing beaker and a wooden mix paddle. This can be made from a paint mixing stick. A pen and laboratory notebook are vital to record all testing and results obtained. As a heading, it should have a date and the purpose of the test and what is going to be achieved. A good solid work bench is very important to place the balance and the mix beaker upon. Do not use a card table or a rickety work bench that is not flat. You will also need strong stainless steel spoons that will not bend, stainless steel spatulas or flat butter knives. You will also need a variety of small plastic cups, pint size plastic "sour cream" type containers with lids, eye droppers, paper towels and a dial thermometer.

A preheat oven which can also be used to cure propellant is optional depending upon the heating system in your garage or work shop. In the winter time it becomes far more important than in summer since without it I could not mix my 85% to 86% solids propellant very well and would not be able to deaerate or cast my propellant at all. There are many other pieces of equipment you will need later on but probably not as a beginner. Place the triple beam balance on the work bench and get zeroed out by moving all the sliding weights to zero. If it does not swing freely in the zero position, the knurled balance weight under the pan can be screwed in or out until it becomes balanced. Also, the balance used be perfectly level from left to right. A bubble level can be used to determine whether the table top is perfectly level and not higher on one end than the other. If the weighing is done

outdoors, the balance must not be swayed by even a slight breeze blowing past it. The surrounding air must be completely still to get accurate readings. If a digital balance is being used, simply press the bar in the front to zero out the balance.

Next, place the mixing beaker on the balance and zero in the weight of the empty beaker. The Prodyne stainless steel mixing beaker (sold as stock # 210) weighs approximately 459 grains without the cover lid so slide the balance weights to 459 until the beam swings freely and the reference lines on the beam line up indicating that it is zeroed out. This is called the "tare" weight and should be recorded in the lab book as such. If a digital balance is used, simply press the bar to zero out the balance. For those who have ever mixed a batch of propellant before, I would recommend a 500 gram propellant batch just to learn how it is done. With more experience, mix sizes can be increased to 1000 gram batches.

The first ingredient to be weighed is the liquid rubber binder, the R-45M. All the materials shown in this formulation are listed in the order in which they are weighed up and added into the mix beaker.

Material	<u>%</u>	<u>Grams</u>
R-45M HX-878 DOA A.P. 400 micron A.P. 200 micron TPB DDI-1410	15.5 0.5 1.5 29.8 50.0 0.2 	77.5 2.5 7.5 149.0 250.0 1.0 _12.5 500.0

This formula is known as an 80% solids propellant since the dry portion totals 80% of the formula. If aluminum powder is used, add 5% of it in place of 5% of the 200 micron A.P. which then becomes 45%, and the 200 micron A.P. becomes $500 \times .45 = 225$ grams. Adding aluminum powder to the mix formula will accomplish three things. It will increase the flame temperature of the burning propellant giving it a higher specific impulse. It will also increase the density and reduce the mixing viscosity since it has a higher density than the A.P. it replaces. The solids loading will remain the same at 80%.

If your desire is to improve this propellant even further, you can grind a portion of the 200 micron A.P. in a mortar and pestle until it becomes finer and begins to look like powdered sugar. This finer A.P. replacing a portion of the 200 micron A.P. will increase the burning rate and make it easier to ignite. It will also make the propellant less sticky and not as grainy. A ball mill is the easiest way to grind the A.P. to a finer particle size but costs more than the mortar and pestle. Both of these items can be purchased in a ceramics shop since they are kiln fired in an oven. The R-45M HTPB polymer is weighed directly into the empty mixing beaker zeroed out on the balance. Pour some R-45 out of the gallon can into an 8 oz plastic cup or plastic lined paper "Dixie" cup. Set the weights on the beam balance to 77.5 grams plus the tare weight of 459 gm for a total of 536.5 grams. Slowly pour the R-45 into the mixing beaker until it balances out. If you "overshoot" and get too much in the beaker, some of it will have to be removed with the spatula until it balances at the 536.5 setting. If a digital balance is used, simply stop adding more when the 77.5 grams is shown on the lighted dial.

The next material to be added is the bonding agent (HX-878). Since so little of this is required, it is added a drop at a time by dipping the tip of a clean spatula into the can of

HX-878. Since this amount is 2.5 GMs-add this amount to the 536.5 already set on the balance so the new total of 539 grams should now be set on the balance. With the spatula tip coated with the HX-878, touch the inside of the mix beaker, near the level of the R-45, with the spatula until the required amount sticks to the beaker. It might require two or three applications with the spatula to get the proper amount. These are the only two materials which are weighed directly into the mixing beaker. All other materials are weighed separately and then added to the beaker. Remove the mixing beaker from the balance and with a clean spatula thoroughly blend the HX-878 into the polymer until the color is uniform. Set the weights on the balance back to zero and check to see that it is balanced out.

The next material to be added is the DOA plasticizer, 7.5 grams. Place a small clean plastic cup on the balance and pour approximately 10 grams of DOA into the cup. Place a clean eye dropper into the cup and squeeze the bulb until the tube is about 80% full of DOA. Next, lift the dropper out of the cup and squeeze the bulb again expelling the material into the cup. Now, pour all of the DOA in the cup back into the DOA can. Some will remain in the cup and eye dropper since the liquid will stick to the sides. Do not wipe this material out. Place the cup, with the wet eye dropper in it back on the balance and zero it out. The reading on the balance is known as the "wet tare" of the cup and eye dropper together. This "wet tare" weight should be added to the 7.5 grams of DOA required. For example, if the wet tare weight is 25 grams, adding this to the 7.5 GMs required equals 32.5 GMs total. Set the balance to this 32.5 gm level using the sliding weights and pour this amount back into the cup until it balances out. With the cup and eye dropper balanced on the scale, and the balance weights indicating 32.5 GMs., slide the weights back to the original wet tare value of 25 grams. Using the eye dropper begin transferring the DOA out of the cup into the mix beaker until the 7.5 GMs is transferred and the balance is once again swinging freely. Remember that the eye dropper is part of the wet tare weight and must be placed back into the cup each time the weight is being checked.

This method of weighing liquid ingredients into a mix is known as the "weigh back method". With a digital balance, it is very simple. I'm assuming that all of you who can afford the expense of an electronic digital balance are also quite proficient in its use. It is the only type of balance to use if you are making 5 or 6 propellant test mixes each day since it cuts the weighing time in half. The plastic wet tare DOA cup and eye dropper can be placed into a plastic zip lock sandwich bag and labeled "DOA" and used at a later time. The weigh back method of adding small quantities of liquid ingredients to a mix is the only method to use when adding curing agents since if you weighed the materials directly into the mix beaker it would ruin a mix to "overshoot" and get too much material into the mix.

It is extremely difficult to get excess quantities of small amounts of materials out of a mix and retain any accuracy. Needless to say, by using a digital balance, it becomes much easier since the digital lights lets one know continuously how much more ingredients are required and there is less chance of overshooting the correct amount of a liquid ingredient. The weighing of dry ingredients, such as the oxidizer and powdered metals is by comparison very simple since they do not stick to the weigh up cup like liquids do. If any of the powders do stick to a cup, simply tap the bottom of the cup with a spoon to free any material when dumping it into a mix. Always weigh the A.P. directly into a plastic cup and add it to the mix in spoonful increments, a little bit at a time, with a lot of mixing in between additions, to wet it down before the next increment is added. If you add a lot of powdered oxidizer to a mix all at once, it would create clouds of dust and cause the mix to become very dry. These two conditions are to be avoided at all costs. If powdered metals are used in a propellant formula, the aluminum, magnesium, iron oxide, etc. must be completely blended into the liquid binder and plasticizer until it becomes a gray or red colored "paste" before any oxidizer is added.

This "premix" of metals and liquids can be preheated in the oven along with the mix beaker and mixing stick to over 150 deg F since without the oxidizer it is practically inert. This will thin out the material so that the oxidizer can be stirred in easier. Add the oxidizer by alternating between the fine and coarse particle sizes; a spoonful of the 200 u AP and then a spoonful of the 400 u AP until they are all mixed and added in. If the mix looks too dry or thick to cast, before the curing agent is added, put the cover on the mix beaker and let it "pre-batch" over night in a 135 deg oven. It becomes "soggy" like wet corn flakes so the next day it mixes easier than it did the previous day. A propellant mix can be stirred with a vertical electric mixer, the "Kitchen Aid" type for several hours until it is completely and totally mixed.

There is no comparison between this propellant and the material mixed by hand for 30 or 40 minutes which burns slightly erratic. The hand mixed propellant, however, is adequate for the enjoyment of testing and flying model rockets. A few small air voids in the propellant are not catastrophic to the performance of a well designed rocket motor. The curing agent is added to the propellant mix last along with the triphenyl bismuth (TPB). Being a liquid, they reduce the viscosity considerably. The TPB is a white powder which should be weighed out separately and added to the propellant mix first and then the liquid DDI-1410 placed directly on top of it with an eyedropper using the weigh back method described for the DOA addition. Once these curatives are added to the propellant mix, the pot life clock starts to tick and there is only a limited amount of time to get the propellant into the motor tubes.

Using an electric mixer, I mix only 10 minutes more with only one scrape down of the propellant from the sides of the mix beaker before the end of mix (EOM). If no vacuum apparatus is available, I would scoop the propellant out of the mix beaker and pile it on a table top covered with polyethylene sheet. If it is too runny, set it out side to cool so it becomes stiffer like the consistency of pizza dough. Cut chunks of it with the butter knife and roll it into cylindrical cigar shapes and with both hands, insert it into the paper tubes. Seal the ends of the tubes with masking tape or aluminum foil and place them into the oven to cure. Cleaning out the mixing beaker of remaining propellant can be accomplished using a rag soaked in Stoddard solvent or a mixture of DOA and liquid dish soap. Wear latex or vinyl gloves to protect the hands. Thirty years ago, strong solvents such as trichlorethylene were used to clean out mixers, but the EPA banned their use for this purpose and now they have banned the use of inhibited trichloroethane (methyl chloroform) and acetone also. The environment is now safer so I guess the extra scrubbing of mixer bowls and blades by hand is a small price to pay for a cleaner environment.

If there are any questions on any of these proceedures or data given, write to me, John Rahkonen at Prodyne or call on (801) 392-7205 (but only during normal working hours please).

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ANALYTICAL DEVELOPMENT OF NEAR-NEUTRAL-BURNING BATES GRAINS

by

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Currently, near-neutral-burning BATES-type composite propellant grains are being used by members of the RRS and by the RRS itself in its solid-propellant training classes. The design of this type of grain lends itself to convenient manufacture and its geometry is simple enough to be easily analyzed. The following article is intended to give readers a more in-depth look at the actual design of these grains. This will allow independent design variations and will avoid slavish adherence to a single set of dimensions, thus providing a lot more flexibility for the designer, especially if larger or smaller grains are going to be made. As will be seen in the following analysis, there is a small, simple set of equations that describe BATES grains no matter what their size.

Although BATES grains are generally thought of as nearly-neutral burning, that is, the thrust and pressure tend to be nearly constant throughout the burning time, it is possible to design versions which are either progressive or regressive; it depends upon the relative dimensions of the grain and their exact ratio to each other. Furthermore, BATES grains can NEVER be exactly neutral due to the physical design. However, it is possible to come within just a few percent of neutrality by proper attention to the relative sizes of the dimensions. Neutral pressure is desirable because it means that it is not necessary to contain excess pressure. The engine can be designed to have a thinner, lighter wall. An additional advantage is that propellant testing can be done at nearly constant pressure, giving more reliable estimates of specific impulse, burning rate and area ratio.

Throughout this analysis, please refer to Fig. 1, attached, in order to keep track of the variables being used. The heavy "C"-shaped line in the diagram emphasizes the initial burning area and the heavy short line shown at the outer grain diameter is meant to emphasize the final burning area. The dashed "C"-shaped line in between the initial and final burning areas is meant to show the typical burning surface at some time during the burning phase. The dotted lines at each end of the grain that are at 45 degree angles are meant to show how the points of intersection between each burning end face and the burning core "move" until they intersect the final burning area. They are lines in a two-dimensional drawing but in three dimensions they are actually cones. Fortunately, it's not necessary to do a three-dimensional analysis, so our two-dimensional drawing will do just fine. Because of the fact that burning is taking place on each end face as well as inside the cylindrical core itself, as the core gets larger in diameter it simultaneously gets shorter in length. This is the reason why the grain can be designed so that the decrease in length almost compensates for the core diameter increase, giving nearly-neutral burning.

VARIABLES:

 A_i = Initial burning area A_f = Final burning area

Az = Burning area at some time "t" during burning

A₂• = Burning area at which the intermediate value is either a maximum or a minimum (whether it is a maximum or a minimum will be determined during this analysis.)

L_i = Initial grain length L_i = Final grain length

 r_1 = Initial port radius

 r_2 = Outside radius of propellant (r_2-r_1) = Weh thickness of propellant

z = Distance burned at some time "t" during burning

z* = Distance burned at which burning area is maximum (or minimum)

