

# Silicone II: A New Fuel and Binder for Fireworks

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## ABSTRACT

*This paper presents some initial work on the use of silicones including GE Silicone II (GE280 and GE5000) as a fuel and binder in fireworks. Some of the pros and cons of silicones are covered, as well as a partial list of safety concerns. A brief review of silicone chemistry is included as a background for the choice of suitable silicone materials. This is followed by some pyrotechnic chemistry, including observations, methods, and ideas for how to use silicone compositions in fireworks. Silicones show great promise for certain applications and may prove to be safer and less costly than some of the materials they could replace.*

**Keywords:** silicone, fuel, binder, rocketry, potassium perchlorate, ammonium perchlorate

## Advantages and Disadvantages of Silicones

Except for the military illumination flare mentioned by Stanbridge,<sup>[1]</sup> there seems to be no published material on the use of silicone in fireworks. Yet, like Parlon®, silicone rubber can serve as both a fuel and a binder.

Perhaps the most striking advantage of silicones, sometimes known as Room Temperature Vulcanizing rubbers (RTVs), is that they are almost unreactive at normal ambient temperatures. This makes working with them relatively safe. Most silicones are totally stable at 300 °F and some as high as 500 °F. In fact, it is somewhat surprising that they burn at all. Indeed, some formulations are difficult to ignite, while others take fire readily. Most are fairly energetic fuels in pyrotechnic compositions.

Unlike Parlon®, a chlorinated rubber, silicone rubbers generally lack the color enhancing

halogens—chlorine or fluorine. All tested varieties also appear to be free of sodium, which would interfere with color purity. Silicones burn colorlessly and somewhat luminously, like metals.

The chemical properties of silicone seem to reduce the sensitivity of compositions during and after manufacture. The water-resistant nature of the silicone serves to encapsulate the grains of the composition, not only protecting them from moisture, but also slowing such reactions as ion exchange between salts.

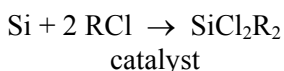
The physical properties of better silicone pyrotechnic mixes are also favorable. Silicones require no solvents to facilitate mixing. Ten to 15% silicone compositions tend to have a dry, crumbly texture and can be pressed into objects that hold their shape during and after cure. Compositions with 15 to 25% silicone usually have a workable, putty-like, plastic texture, and can probably be pumped. These properties would lend themselves well to low-cost, high-volume automated productions. After curing, the compositions can be bent and struck without losing physical integrity. The lubricity of the uncured material allows devices, such as rockets, to be removed easily from forming tools.

On the negative side, many compositions—but not all—generate large amounts of smoke. Silicone also has a no-stick property like Teflon®, so it is difficult to find materials that will adhere to it. This makes the priming of cured stars extremely difficult. The water resistance and chemical stability also assure that any unconsumed items such as unlit stars will remain as reactive pyrotechnic compositions indefinitely.

## Chemistry of Silicone Manufacture

The manufacture of silicones is a multi-stage process, in which several foreign materials may be introduced into the finished product. Some are beneficial for pyrotechnic purposes; others are not. There are many processes. According to Rochow<sup>[2]</sup> the following is typical.

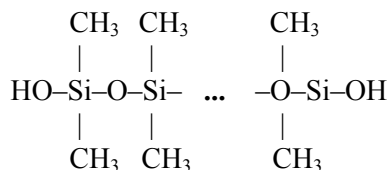
Elemental silicon (Si) is reacted with chlorinated hydrocarbons (RCl) in the presence of a catalyst to form a substance that can be polymerized. This reaction forms compounds with a varying degree of chlorine saturation. The resulting mixture is distilled to obtain the desired product in purified form.



The "R" in the equation represents a hydrocarbon radical such as  $-\text{CH}_3$ . The type or types of radicals chosen for R ultimately determine chemical and physical properties of the silicone because the R groups become the side chains of the final polymer. If the methyl group is chosen, for example, the result is dimethyldichlorosilane. Copper is typically the catalyst used in this reaction.

The dimethyldichlorosilane, or other starting material, is then hydrolyzed by adding water, forming short chains and rings, and evolving hydrochloric acid.

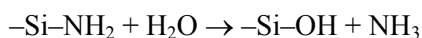
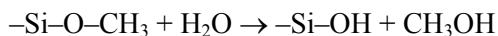
In the next step, the rings are broken and the chains further polymerized with the help of an additional catalyst. Strong acids like sulfuric and nitric, strong bases like sodium hydroxide, amides and other materials are used for this purpose. The result is called a silicone oil or grease. Its chains are terminated with hydroxyl groups. These terminal groups may be replaced to alter the curing chemistry. But for some purposes, this is the final product. A typical result is



## Silicone Rubber Curing Chemistry

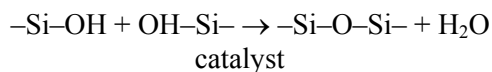
The curing of silicone rubber is similar to the building of Tinkertoys®. The chains described above are the sticks, except that they are quite long and flexible. The round Tinkertoys connectors correspond to molecules of cross-linking material added to the silicone. These molecules are typically composed of one silicon atom with three or four reactive groups attached, corresponding to Tinkertoys connectors with only three or four holes. During curing, the sticks become attached to the connectors until very few ends remain unattached. Failure to react most of the chains with cross-linkers could affect the chemical and physical properties of the product.

The cure reaction of GE Silicone II and other one-part silicones, as they are called, is initiated by atmospheric water vapor. The reaction releases various acids, bases, esters, alcohols or other chemicals as byproducts. The particular byproducts give a silicone its characteristic odor, for example:



The first equation generates methanol as a byproduct; the second, ammonia. These may be the source of the methanol-ammonia odor present in GE Silicone II (GE280). Note that both reactions leave hydroxyl groups attached to the exposed silicon atoms.

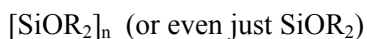
The hydroxyl groups then react, under the influence of a catalyst, to yield water. This catalyst is problematic to the pyrotechnician because it is often listed as a trade secret ingredient by the manufacturer, and derivatives of it may remain in the cured product.



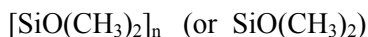
The cured product is typically free of the carbon-carbon bonds or reactive groups that cause instability in other binder-fuels. Thus silicones are unaffected by strong chemicals, excessive heat or UV light. However, possibly undesirable chemicals may still remain in the cured product, for example:

- catalysts, including copper
- HCl, H<sub>2</sub>SO<sub>4</sub>, NaOH, KOH, etc.
- unreacted organic groups from the cross-linkers
- cure reaction byproducts (ammonia and methanol for GE280, acetic acid for traditional RTV)
- any possible reaction products of the above

The cured product is mostly composed of long-chained, silicon-oxygen backbones, with side groups attached. The cross-links and terminations make up only a small portion of the molecule and can be ignored for stoichiometric purposes. This allows the silicone material to be represented in a simplified way in chemical equations:



For most silicone elastomers, the side groups are methyl radicals. Thus, this becomes



### Characteristics of Pyrotechnic Formulations

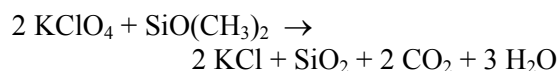
Tests were made with GE280 and GE5000. For oxidizers, only potassium and ammonium perchlorates were investigated in any detail. Potassium nitrate simply did not produce compositions that burned well. Other nitrates were not tried. Chlorates were tested for sensitivity but were otherwise avoided altogether because of the risk of forming highly sensitive or spontaneously combustible mixtures.

The chlorate mixes would snap when pounded on an anvil with a force that might be used to drive a large finishing nail. Nitrate mixes required a severe blow to cause a reaction but were nearly refractory in combustion. Apparently nitrate is not a strong enough oxidizer with silicone.

### Potassium Perchlorate

Potassium perchlorate created slow-burning compositions that might be suitable for microstars or lance; although smoke tended to be a problem. Excellent blues and good yellows were created with simple compositions. Some blues were tested as comets and in aerial shells, but they tended to wash out to a purplish blue and did not look quite as good as they did on the ground.

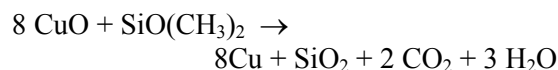
The simplified stoichiometric reaction of potassium perchlorate with silicone is



That represents a 78.9 to 21.1% weight ratio.

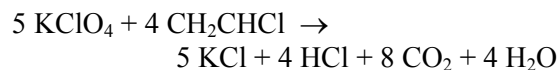
When burned, the silicon accepts a second oxygen-creating silicon dioxide (silica) mostly in a molten state, forming a slag. The potassium tends to hold onto its chlorine, so a chlorine donor is needed. During burning, it is plausible that methyl groups and/or hydrogen are initially left unreacted, leaving the slag as a gas. They can burn in the atmosphere without consuming oxygen from the oxidizer. This means that higher fuel loadings should be possible.

For blue compositions, copper(II) oxide serves as an optional oxidizer, sometimes being reduced, and sometimes not. The presence of elemental copper can easily be detected in the ash of a test, indicating that the copper(II) oxide is being reduced. The reaction when copper(II) oxide is fully reduced is



giving a 89.8 to 10.2% weight ratio.

Polyvinyl chloride (PVC) was used for the chlorine donor. The simplified stoichiometric reaction of potassium perchlorate with PVC is



giving a 73.5 to 26.5% weight ratio.

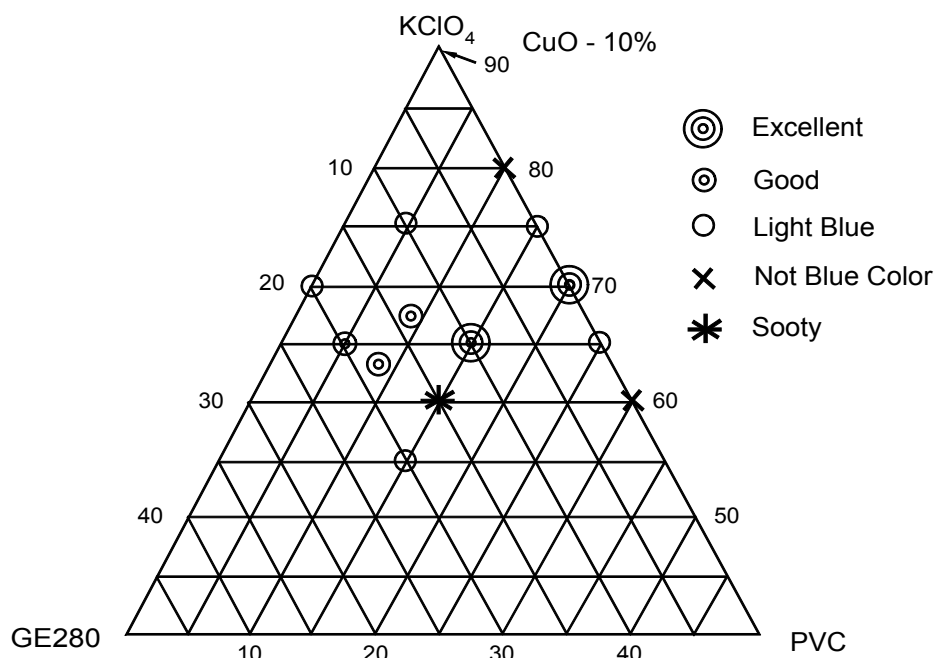


Figure 1. Good slow-burning blue mixtures can be produced using potassium perchlorate, PVC and up to 20 % silicone.

The triangle diagram in Figure 1 shows the range of blue color generation with 10% copper(II) oxide content. Five percent copper(II) oxide was also tested yielding a similar pattern but with inferior color quality. The nomenclature of Shimizu in Pyrotechnica VI<sup>[3]</sup> was used, and his formula B11 (see Table 2) was used as a standard to judge results. Shimizu's data correspond with the right hand edge of the triangle, where the silicone content is zero. His results are shown for reference.

Little data was taken on burn rates. Formula KB33 with 75% oxidizer (see Table 2), burned at 36 s/in, while compositions with lower percentages of oxidizer burned slower.

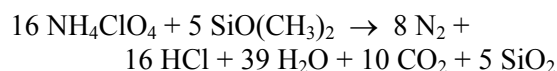
A few formulations were tried for yellow. Attempts to correct the color using barium sulfate were not successful. The mixture KY07 produces a good sodium yellow using sodium benzoate. The burn rate was an impressive 14.5 s/in. (Note that potassium perchlorate plus sodium benzoate is basically a whistle mix. A safe procedure would be to mix in the sodium ben-

zoate after the other ingredients have been combined with the silicone.)

### Ammonium Perchlorate

Ammonium perchlorate seemed to be the oxidizer most suited for use with silicone. It burns fast, acts as a chlorine donor, and has a tendency to strobe. The compositions also take fire more readily than those made with potassium perchlorate.

The simplified stoichiometric reaction of ammonium perchlorate with silicone is



giving a 83.5 to 16.5% weight ratio. If the hydrogen is not oxidized, the ratio becomes 66.5 to 33.5%. The oxidizer releases its chlorine largely as HCl gas, which is good for color generation. If PVC is added as a chlorine donor, the stoichiometric formulation is  $\text{NH}_4\text{ClO}_4$  (79.3%) and PVC (20.7%).

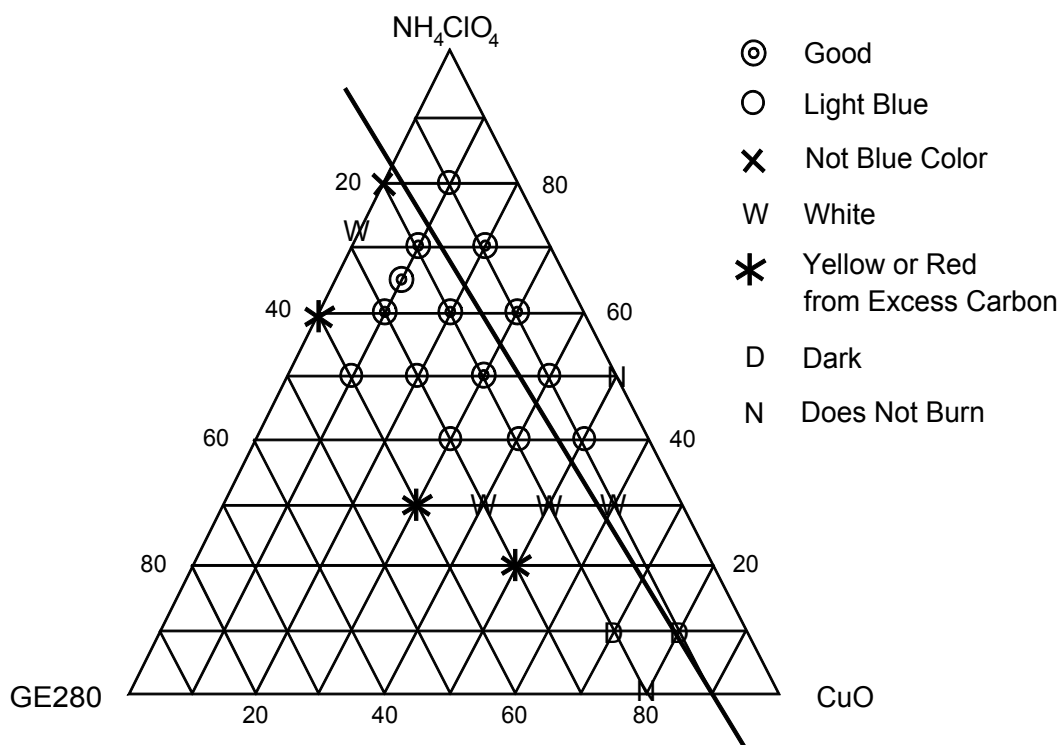


Figure 2. Blue colors using ammonium perchlorate may contain a large excess of fuel (left of sloping line).

Copper(II) oxide is used as the color agent in formulations to make blues. Copper(II) oxide also serves as a catalyst and sensitizer as summarized in Table 1. Three samples with a constant 72:28 ratio of ammonium perchlorate to silicone and a varying percent of copper(II) oxide were tested. Ignition was tested by placing a thin layer of 5FA Black Powder on the surface and igniting it quickly with a propane torch. As the copper content increased, so did the burn rate and ease of ignition.

Table 1. Effect of Copper(II) Oxide.

Mixture	NH <sub>4</sub> ClO <sub>4</sub> : Silicone 72:28	CuO (%)	Ignition with 5FA	Burn Rate (s/in.)
NH4W01	100	0	0/3	14.8
NH4B01	95	5	1/2	13.8
NH4B02	90	10	2/2	13.5

The triangle diagram in Figure 2 shows the combination of ammonium perchlorate, silicone and copper(II) oxide. No chlorine donors were added. Some samples showed moderate sensitivity when hammered on an anvil; others were quite insensitive. The line drawn across the diagram represents complete oxidation and reduction per the theoretical stoichiometric reactions. Note that good colors can be found well on the fuel-heavy side of the line.

The triangle diagram in Figure 3 shows burn rate as measured in a 1" long by 5/16" diameter lance tube. There is a large area of nearly constant burn rate that roughly corresponds with blue color generation. There is probably some physical process such as melting of the oxidizer that is controlling this rate.

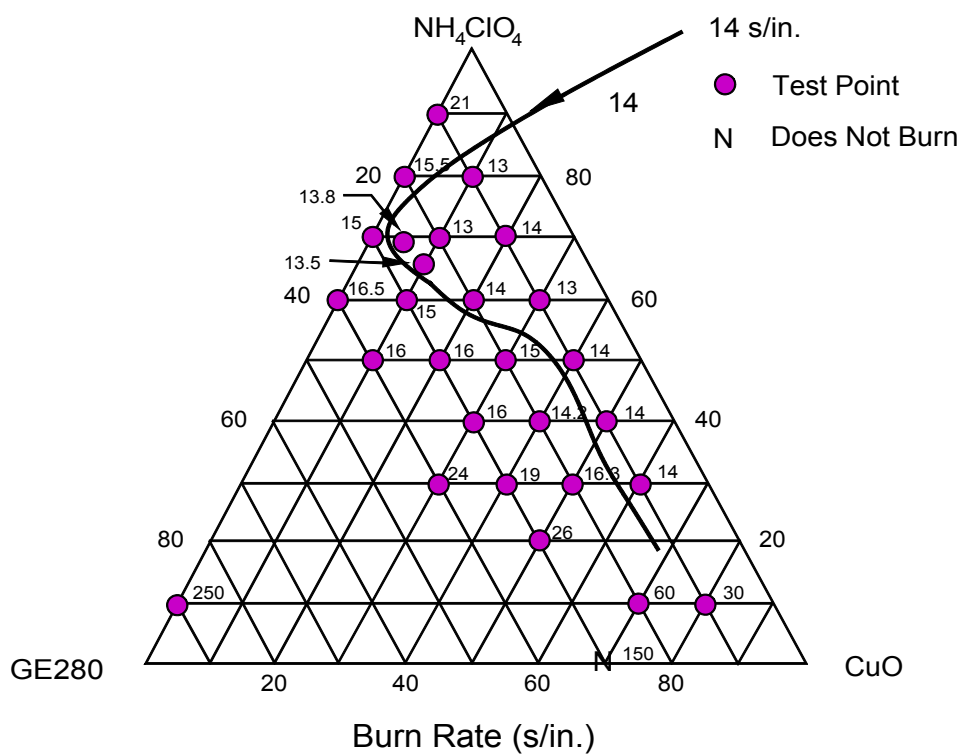


Figure 3. In the area of blue color generation, burn rate is nearly constant for a given percentage of silicone.

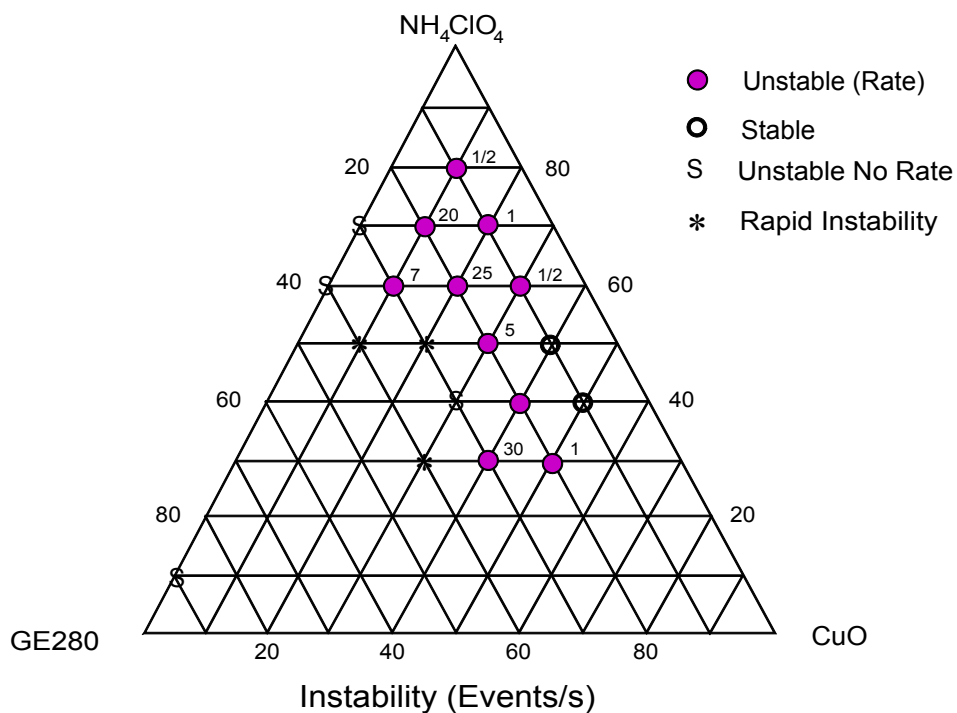


Figure 4. Somewhat strobe-like instability in open air may indicate mixtures that strobe when confined. Numbers next to points indicate strobe rate.

**Table 2. Formulations.**

Name	NH <sub>4</sub> ClO <sub>4</sub> (%)	KClO <sub>4</sub> (%)	CuO (%)	GE280 (%)	PVC (%)	Other (%)
NH4W01	72	0	0	28	0	
NH4B01	68.4	0	5	26.6	0	
NH4B02	64.8	0	10	25.2	0	
NH4B06	63	0	10	22	5	
NH4B10	20	0	50	30	0	
NH4B19	30	0	40	30	0	
KB33	0	75	5	10	10	
KB40	0	65	10	10	15	
B11(Shimizu)	0	68.5	15	0	9	Accroides resin:7.5 Starch: +5
KY07	0	70	0	15	0	Sodium benzoate:10 Charcoal, air float:5

The triangle diagram in Figure 4 shows unstable burning characteristics observed during small sample testing in lance tubes. Samples that exhibited noticeably unstable burning are marked with "S" for strobe. Where there seemed to be a strobe rate, the strobe rate is indicated by the number next to the test point. It is doubtful that any of these would pass for a blue strobe star, and no aerial shell testing was done. The strobe effect only appeared to be strong and repeatable when there was some degree of confinement, as in rocket motors. One mixture containing PVC (NH4B06) was pressed into several rockets, producing a strobe rate of 5 to 10 per second. The data on the diagram provides a rough guide for where to look for good strobe mixtures.

Some samples left a long, rigid, snake-like ash when burned in the uncured state. Data on this property is sketchy, but some mixes that behaved this way were NH4B01, NH4B10, and NH4B19. These had the drawback that large volumes of HCl gas are generated. Perhaps a mixture using potassium perchlorate, copper(II) oxide, silicone grease and a substance that evolves additional gas can be found that does not generate HCl.

Some compositions generated a large amount of smoke, while others generate relatively little. This seemed to depend on the amount of silica that remained in the slag. A good low-smoke lance may be possible.

### Mixing Methods

The following methods were found suitable for small batches. For larger amounts, appropriate precautions should be taken.<sup>[4]</sup>

The dry ingredients were sifted together two or three times through a fine screen. The silicone was then combined with the dry ingredients. Highly reactive auxiliary fuels like benzoate were added after the oxidizer was combined with the silicone.

It is important to note that silicone pyrotechnic compositions, unlike typical moist pyrotechnic mixes, burn hot and fast in the uncured "wet" state. And they can be readily ignited. It is a serious miscalculation to assume that "wet" compositions are benign until cured.

Small test batches, around 10 grams, were mixed by folding the material over on itself and pressing it flat, at first with a wooden spatula, then by hand. The spatula was scraped clean once or twice to avoid losing silicone from the mix.

Larger batches, around 200 grams, were placed in a zip-closure food storage bag and kneaded. The bags with the plastic closure shuttle in the one-gallon size worked best. The mixed dry ingredients were dropped in first, then the silicone was added by wiping it from the weighing paper onto one side of the bag, keeping it away from the corners and the zip-

per. A moderate amount of air was left in the bag, so that the two sides could touch together easily in the middle. The powder was worked into the silicone (by pulling the material apart, exposing fresh silicone, and covering that area with powder) until all was dampened. After most of the powder was mixed in, the material sticking to the surface of the bag was removed by rubbing other composition across it. When all of the material was consolidated, it was removed and hand-kneaded further to complete the mixing process. Upon opening the bag, an alarming odor of ammonia was detected. This was largely due to the cure process of the silicone. (One should remain vigilant when detecting ammonia, however, as it may indicate breakdown of ammonium perchlorate and an impending disaster.)

## Rocket Construction

Preliminary work showed that ammonium perchlorate-silicone blues work well as strobe rockets. The color was good and the lift was moderate. Limited rocket building skill and experience nevertheless produced nineteen successful launches out of nineteen, with only one rocket deviating significantly from the expected trajectory. Neither formulation nor construction detail seemed to be critical to achieve success. But there is plenty of room for experimentation and improvement.

Most of the experience was with the formula NH4B06, which contains PVC. This mixture was not the result of any exhaustive optimization; rather, it was chosen from the middle of the "good" area in Figure 2, and it worked on the first try! Although PVC was added to improve color, no visible difference was found later when compared to a similar mixture that replaced the PVC with silicone. These mixtures are quite fuel-rich compared to stoichiometric oxidation-reduction. The first motor used the

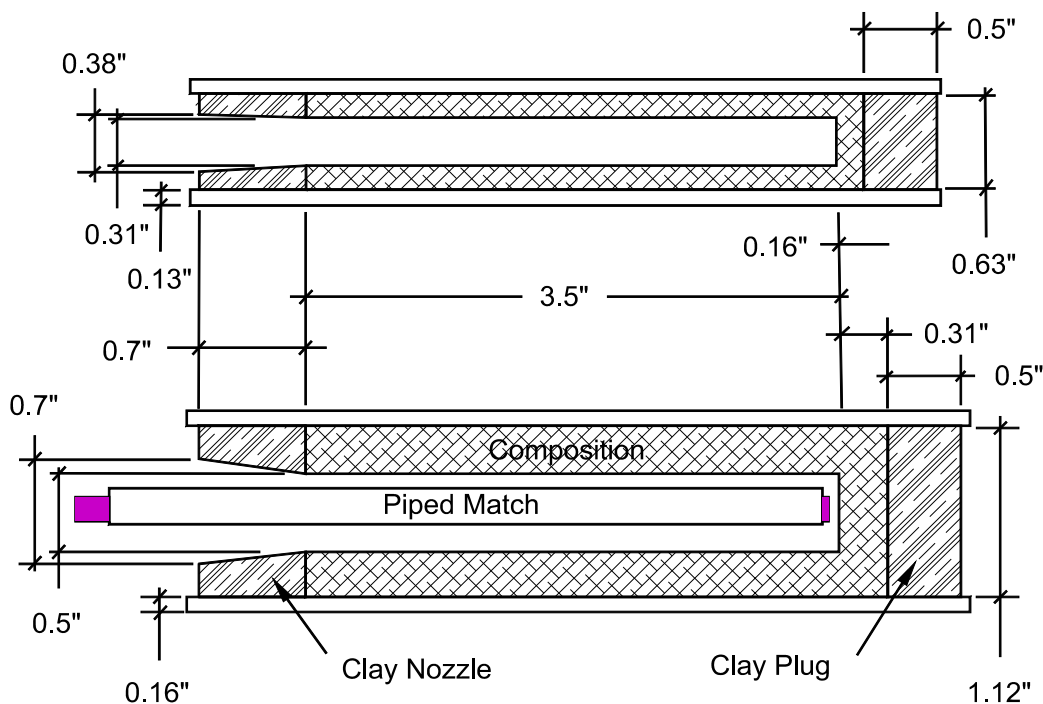


Figure 5. Sketch of rocket designs.



more expensive GE280, and the remaining used GE5000. The lower molecular weight GE280 seemed to give better lift, but the sample size (only one) was very small so this is not conclusive. Equivalent results are probably achievable using the GE5000.

These rocket compositions required a hollow core to achieve good lift and a strobing effect. Otherwise, the details seemed to be non-critical. Five-eighths-inch dia. Roman candle tubes with 1/8" walls and 30 mm Ruggieri display candle tubes were used for the casings. These were built using the dimensions in Figure 5. Longer versions of the 5/8" model (up to a 4.5" long core) were constructed in an unsuccessful attempt to find the core length at which the burn rate became explosive. The results were less beautiful but more amusing because of the resulting erratic strobe rate.

Most of the rockets had clay nozzles. If the nozzle was omitted, the rocket still flew, but stopped strobing about 50 feet above the ground. The choice of nozzle material seems to be unimportant, but materials with a high sodium content might wash out the color. Powdered clay with 25% grog works well. Also up to 10% silicone can be added to the mix, which makes the nozzle less likely to break up in handling and easier to remove from the spindle. Pullout strength [the static axial force required to dislodge the nozzle from the tube] is reduced by about one-third, however.

Because of the workability of the silicone, tooling can be much simpler than for conventional rockets. The ramming force required is much lower than for dry mixes, so no outside support is required to keep the casing from rupturing. The spindle and rammer are also simpler than for black powder motors. The nozzle portion of the spindle is the only part that must be made of metal such as aluminum or brass. Otherwise, the whole tool may be made of hardwood like maple or cherry. The silicone compound is easily removed from the spindle, so essentially no taper is required above the nozzle. This makes the tools easy to build, and also results in all of the composition burning out at the same time. The spindles used in this study had brass nozzles and aluminum shafts with a di-

ameter taper of about 0.02" over the 3/2" length.

Only two ramming tools are necessary, one with a long straight bore, and the other with no bore at all. For safety, the long rammer is designed with enough clearance (about 0.06") so that it can never come in contact with the spindle, and is made of wood as an extra precaution. (Normal rocket tools, which can make contact metal-to-metal, should not be used.)

The composition was made in a food storage bag as described above. It was then broken into pieces small enough to pass freely between the spindle and the tube. This reduced the amount of composition resting on top of the spindle. Such composition can cause air voids in the finished motor or pack into the rammer. Dicing the composition with a plastic knife on wooden cutting board is an effective, but slow method. The composition remains workable for an hour or two after mixing. In fact, allowing it to sit out in the granulated state for a half an hour may aid the curing process through absorption of moisture from the air.

The nozzle is rammed without side support, and ramming is stopped when slight deformation of the tube is observed. This is done with only the nozzle former in place and the rest of the spindle removed. The clay will not consolidate above the top of the nozzle former, automatically creating a nozzle of the correct height. The tube's rotational position is marked with a pen; then it is removed from the tool for inspection and for cure if it contains silicone. The spindle portion is then screwed on, the rocket tube replaced, and the fuel rammed. This is an unorthodox method, but it does not cause problems with these motors.

Before the fuel is rammed, the outside of the case is marked at the point where the composition is to end. The thickness of the fuel above the top of the spindle is made equal to the distance between the spindle and the tube. This gives zero delay from end of lift to burn-through.

Increments of fuel are kept small to avoid trapping large air pockets between areas of consolidated composition. The material does not need to be rammed hard. It takes a certain

amount of time for the air to leak out between the grains, and ramming hard does not help. A long series of light taps drives out the air and leaves the composition solid. Composition adhering to the inside of the rammer is evidence that the increment was fully rammed. Remove this material from the rammer after each increment. When the fuel is level with the top of the spindle, add a generous increment, and switch to the solid rammer. Complete the fuel loading to the marked height, and finish off with clay at the top. Old-fashioned kitty litter (9 mesh) works when sealed after ramming with a bit of white glue. The lack of external support prevents the top clay plug from being consolidated as thoroughly as is normally done with black powder motors. This concession to safety does not appear to affect operation.

The completed motor should be removed immediately from the tool. This is easily done, except for the fact that a partial vacuum will form at the top of the spindle. If pulled too rapidly, the suction will cause large movement of the composition, ruining the motor. A large amount of spinning (clockwise) with a small amount of pulling will allow air to work its way into the space. A small hole bored through the length of the spindle or more taper would probably facilitate removal.

These motors need to cure at least one day, but three days is better, to allow the methanol and ammonia to escape. Afterwards, an electric igniter or piped match is pushed all the way to the top of the motor to achieve proper ignition. An appropriate stick for stabilization is attached before launch (see reference 6). A small amount of white glue plus two wraps of fiberglass tape works well to secure the stick. These motors will not carry large loads predictably.

## Silicones for Future Development

While only GE280 and GE5000 were used in these tests, there is a wide variety of silicone materials to choose from. These range from greases and oils to elastomers to casting resins. Each has its own manufacturing process and chemistry, and they should not be assumed to be interchangeable for pyrotechnic use. What follows are some guidelines for the development of new silicone-based compositions.

The manufacturer's Material Safety Data Sheet (MSDS), the manufacturer's technical application support engineers, and basic silicone chemistry texts should be used to gain information about the chemistry of a material. The MSDS is very useful because it gives the ingredients in a product, although some may be listed as "industry secret". The chemistry of the particular silicone should then be considered in respect to the other ingredients before mixing anything together.

Because of the possible reactions when oxidizers are combined with acids, silicones that evolve acids during cure should not be used. Do not use silicones that give off a vinegar odor. Silicones that give off weak bases like ammonia are interesting because they buffer the mixture to about pH10 during cure and can neutralize any acids that might be present in the unreacted silicone. Silicones that evolve alcohols are probably least reactive and should be sought out. (GE280 gives off both methyl alcohol and ammonia.) Those made for electronic circuit assembly are promising for future research in this respect. Avoid any silicone that, during cure, gives off amides, ketones or species with double bonds.

Silicone casting compounds are chemically more complex than other silicones. They may contain carbon-carbon bonds or other opportunities for chemical reactions, making them less stable.

Silicone greases, especially electronic or medical grades, offer the promise of a totally predictable fuel. However, they have no cure process, which means they will not serve as a strong binder. They may work in rockets in spite of this.

Gasoline resistant grades may contain side chains loaded with halogens—fluorine most likely—that can be used for color enhancement as suggested by Kosanke.<sup>[5]</sup> Note that combustion products of halogenated organic compounds can sometimes be extremely toxic.

Users unable to understand the chemistry and unequipped to handle spontaneous combustion should restrict their choices to materials with some history of use (currently GE280 and GE5000). This, however, is no guarantee of safety.

### Other Ingredients

Other fuels should be chosen carefully to avoid unwanted oxidation–reduction reactions with the oxidizers. Metals, sulfur, or nitrogen-bearing fuels are high-risk choices. Polymers such as Parlon®, PVC and polyethylene fall at the other end of the risk spectrum.

Chlorate oxidizers should be avoided. Chlorate samples made for preliminary safety testing were more sensitive to hammer blow on an anvil than perchlorates. The burn rate for potassium chlorate compositions was actually equal to or slower than that for equivalent ammonium perchlorate mixes.

Sulfates are the safest color donors but did not generate good colors in limited testing. It is unknown why these did not work; possibly there was insufficient temperature, or the metal oxides generated dissolved into the slag forming a glass. Copper(II) oxide (black—CuO) and sodium benzoate can hardly be considered safe, but they work. The copper(II) oxide used was ceramics grade (very fine powder). It is not known with which process it was made, but it was free of acid when tested with pH paper.

Chlorine donors should be restricted to polymers (PVC and Parlon®) plus totally halogenated hydrocarbons. Avoid chlorides and oxychlorides.

### Acknowledgments

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“Tinkertoys” is a registered trademark of Hasbro, Inc.; “Teflon” is a registered trademark of E. I. du Pont de Nemours and Company; and “Parlon” is a registered trademark of Hercules, Inc.

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