



Preparation of KNSB Propellant by
Vacuum-Evaporation Method

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Objective

The purpose of this experiment is to further study the *Krech-Nakka* method proposed for *SStS* propellant production, as detailed in the document *Propellant Development Team Proposed Investigations*, specifically Item 6c "Liquid phase sorbitol/KNO₃/H₂O mixture" (ref. http://www.sugarshot.org/downloads/pdt_investigations.pdf). In particular, this experiment attempts to produce KNSB propellant by a modified version of the "evaporation method" whereby melted sorbitol is combined with a saturated potassium nitrate/ water solution. The combined mixture is then heated under near-vacuum conditions to remove the water, producing a slurry of KNSB propellant, comparable to that produced by the traditional "melt & cast" method. This method is referred to, in this report, as *vacuum-evaporation* method.

Apparatus and materials

Sorbitol powder: 130.0 grams, "as obtained" from PVCONLY.COM

KNO₃ granules: 400 grams, "as obtained" K-Power fertilizer

H₂O: 118.1 grams (based on 338.5 gram solubility of KNO₃ in 100 ml. H₂O at 115°C.)

These quantities represent a greater amount of each constituent than what is needed to produce the target quantity of propellant (300 grams). This was done to attain better temperature control during heating, which can be difficult with small batch sizes. As well, this was done to help mitigate the adverse effect of water loss through evaporation.

Three thermostatically-controlled deep fryer units were used for heating. unit #1 for melting sorbitol, unit #2 for heating the KNO₃/H₂O solution, and unit #3 for the combined mixture. The latter was of cast aluminum construction. This unit was fitted with an EPDM-sealed Plexiglas lid with a pressure tap to provide for a vacuum line. The lid had been fitted with metal struts that spanned the rectangular opening of the deep fryer. The purpose of the struts is to prevent the walls of the deep fryer from deforming inward under negative pressure.

Unit #1 (Sorbitol) set at 260°F (130°C)

Unit #2 (KNO₃/H₂O) set at 240°F (115°C)

Unit #3 (Combined mixture) set at 260°F (130°C). Tare mass measured at 1854 grams

Temperature measurements of the KNO₃/H₂O mixture were made with a K-type thermocouple connected to a multimeter, for instantaneous readout, and to a laptop computer, for data logging at a rate of one sample per second. Readings were recorded in millivolts for later conversion to temperature. The stainless steel sheathed thermocouple sensor was mounted such that the tip was located 2 mm above the bottom surface of the unit #3 heating vessel, such that it would be immersed in the slurry. A second similar thermocouple was placed in an ice-water bath to provide a reference temperature. Calibration of the thermocouple had been checked, prior to conducting the experiment,

by immersion in boiling water. The emf reading was found to correlate exactly with published data (4.1 mV.), giving confidence in subsequent temperature measurements.

Additional temperature measurements were made with another K-type thermocouple probe connected to a second DVM with temperature measurement display capability.

A thin-walled stainless steel container was used as a transfer vessel. The purpose of the transfer vessel was to allow for accurate weighing and proportioning of melted sorbitol and KNO₃/H₂O solution prior to loading into unit #3 heating vessel..

A vacuum pump was used for evacuating the unit # 3 heating vessel such that the evaporation process would occur under near-vacuum conditions. The pump is a 120 VAC Robinair 15500, two-stage unit, rated at 5 CFM with a capability of evacuating to 40 microns.

To protect the pump, a dessicator unit and a water trap/filter were installed in the vacuum line between the heating vessel and the pump. The dessicator was filled with 1 kg of CaCl₂ granules. The mass of the dessicator unit, prior to and immediately after the test, was recorded in order to determine the amount of water absorbed and how effectively the dessicator performed.

Mass dessicator prior to test = 2252 grams.

A bourdon gauge (0-30 in.Hg vacuum) was used to measure the negative pressure within the unit #3 heating vessel. A camcorder was used to record the gauge readings over the duration of the experiment.

Three separate digital scales were used for mass measurement:

1. Weighing up to 100 grams, 0.01 gram resolution scale.
 2. Weighing from 100 to 200 grams, 0.1 gram resolution scale.
 3. Weighing from 200 to 2000 grams, 2 gram resolution scale.
- All masses stated in this report reflect these levels of resolution.

Separate silicone spatulas were used to stir the sorbitol and KNO₃/water solution.

Figures 1 through 4 illustrate the materials and apparatus used in this experiment.

Procedure and Results

Powdered sorbitol was placed unit #1 heating vessel, turned on, and allowed to melt. Full melting occurred in less than three minutes. Temperature of the melted sorbitol was measured at 125°-130° C.

The H₂O and approximately half the KNO₃ was placed in unit #2, stirred well, turned on, and heated. After all KNO₃ had dissolved, the remainder of the KNO₃ was added. After about 5 minutes, all KNO₃ appeared to be fully dissolved. Some frothing occurred when the boiling point was reached. The thermostat had to be adjusted downward to prevent

boiling. Temperature at boiling was measured to be between 113° – 114°C. It was found that exact temperature control of the heater was difficult, due to the high wattage of the unit in combination with the relatively small mass of water. It was eventually decided to add 35 ml H₂O to replace that lost by boiling, when it was noticed that some of the KNO₃ was precipitating (35 ml was a guesstimate as to the amount lost through evaporation).

A portion of the melted sorbitol was then poured into the pre-heated stainless steel transfer container placed onto a digital scale. Total mass of sorbitol transferred was 106 grams. A portion of the KNO₃/H₂O solution was then poured into the same container, to the tune of 252 grams. A glass stirring rod was then used to mix the contents, which were then poured into unit #3. The heater unit was then powered on, and the lid placed on top. Temperature logging was activated, and the vacuum pump powered up. The pressure rapidly dropped and was down to 30 in.Hg. (near-vacuum) within 5 minutes. The slurry temperature was initially approximately 70°C., and slowly elevated over the course of approximately 13 minutes. As soon as the slurry temperature reached 135° C, heating was ceased. and the vacuum pump turned off.

The lid was removed and the contents examined. The slurry had a very porous “spongy” appearance. There was no obvious indication of wetness or excess water in the slurry. The colour was “ivory”, quite similar to standard KNSB slurry. A spatula was then used to stir the contents and the consistency was found to be quite viscous (barely scoopable). Stirring tended to reduce the viscosity to some extent.

The heating unit and contents were then placed on the digital scale and the mass recorded. The unit was then powered up and heating reconvened, in order to drive out possible residual moisture, and to see how the viscosity of the slurry would vary with temperature. A slight sizzling sound was heard initially, indicating some residual water.

It was found that when the temperature of the slurry reached 140°C, the viscosity was minimum, and that further heating did not make any noticeable difference.. The slurry was “scoopable” but not pourable at that point. A maximum temperature of 160°C. was achieved before heating was discontinued. The colour of the slurry at that point had taken on a slight yellowish tinge. The heating unit and contents were again placed on the digital scale and the mass recorded. Five propellant strands were then made for burn rate measurements.

The transfer vessel and residual contents were also weighed.

The recorded masses were as follows:

Heating vessel & contents after initial heating: 2154 grams
Heating vessel & contents after subsequent heating: 2152 grams
Transfer container & residual contents: 83.4 grams
Transfer container, tare: 80.6 grams.

The dessicator mass was measured after the test to determine how much water was absorbed by the CaCl₂. The mass was found to be 2282 grams. The water trap was visually inspected, with no indication of water or goop present.

A sample of the propellant was ignited. Hard to ignite, but once ignited, burned fiercely and in a very stable manner, typical of conventional “melt & cast” KNSB.

Immediately after cooling, the strands were flexible. After 10 hours, the strands were found to be fully cured (rigid).

Burn rate measurement of each of the five strands was performed 48 hours after casting. To ensure even burning, the strands were painted with hi-heat aluminum paint. Three gauge marks were marked on each strand and the distance between the marks measured and recorded (L1, L2). The strands were then hot-glued vertically to a fixture and ignited at the top end using a flat strip of steel that had been heated red hot. The burn times were measured and recorded.

One interesting observation relates to the burning surface of the strands. When standard “melt & cast” KNSB is burned, numerous glowing, tiny “hot spots” are seen on the burning surface. Such hot spots were completely absent on the burning surface of these strands.

To obtain a better estimate of the residual water content, a sample of the propellant was ground to a fine granular form, weighed, spread onto a cooking sheet, then placed into a pre-heated oven set at 65°C, and allowed to remain for two hours. After this time period, the sample was allowed to cool, then re-weighed.

Sample mass, initial = 20.20 grams

Sample mass, final = 20.00 grams

Delta mass (assumed water) = 0.20 grams, or 1.0 percent of propellant mass

In order to determine if the residual water in the propellant affects the burn rate (under ambient conditions), the dried sample was re-melted at a temperature of 130°C., then formed into two strands. Unlike the strands made from the as-cast propellant, which cured after 10 hours, these strands were, curiously, still fully flexible after 24 hours. After 40 hours, the strands were still slightly flexible. The strands were then prepared similar to the earlier strands, and burn time measurements taken and recorded.

Discussion

The pressure within the heating vessel, as it varied over time, is shown in Figure 5. It can be seen that the pump was very effective in bringing the pressure down to a near-vacuum level (30 in.Hg) within approximately 5 minutes.

The temperature of the slurry, as it varied with time, is also shown in Figure 5. The “sawtooth” nature of the curve is most certainly a result of cycling of the heater

thermostat. The trend is shown in Figure 6, with the temperature eventually converging at a level of approximately 130°C., which is the temperature value at which the thermostat was set at. The complete vacuum-evaporation process took a total of just under 13 minutes to reach this point.

The mass of sorbitol/KNO₃/H₂O initially placed into the heating vessel is given by:
mass (initial) = 106 + 252 – (83.4 – 80.6) = 355 grams.

The mass after first and second heating session given by:
mass (final 1) = 2154 – 1854 = 300 grams.
mass (final 2) = 2152 – 1854 = 298 grams.

The initial mass of water in the batch given by:
mass water = 118.1/(118.1 + 130) * 252 = 57.4 grams.

Percentage of water removed after first heating session given by:
(355 – 300) / 57.4 = 0.96 or 96%

Percentage of water removed after second heating session given by:
(355 – 298) / 57.4 = 0.99 or 99%

Note that these values must be considered approximate and subject to error due to the additional water that was added to compensate for the loss through evaporation. Interestingly, this result is in good agreement with the 1% residual moisture content determined by oven-drying the granulated sample.

The amount of water trapped by the dessicator:
mass trapped water = 2282 – 2252 = 30 grams.

A small amount of water (perhaps a few grams) was seen to be present in the clear vinyl tubing that connected heating unit #3 to the dessicator. However, the sum of these accounts for only about 60% of the water removed by the first heating session. An examination of the pump oil showed it to be cloudy, suggesting the presence of moisture. (the oil in the pump was consequently drained and replaced with fresh oil).

The burn rates for the various strands were calculated from the burn times and gauge lengths, and are tabulated in Tables 1 and 2. Table 1 presents the results for the as-cast propellant strands, made from the batch of propellant produced, and Table 2 presents the results for the strands made from the sample of propellant that had been further dried in the oven.

Conclusions

The process of producing KNSB propellant by vacuum-evaporation has been shown to be feasible. Water removal was determined to be effective despite lack of stirring of the slurry. The resulting propellant had a residual moisture content of 1%, which is likely to be similar, or better, than that produced by the standard “melt & cast” method. Clearly, it would be of value to do an actual measurement of the moisture content of “melt & cast” propellant for comparison.

The time duration required to remove the water from the 300 gram sample (13 minutes) is neither quick or exceptionally slow. This amount of propellant produced in this experiment represents approximately 1/10th the mass that is expected to be produced in a practical batch size required for the *SStS* project. What is not known is how to scale up the time required for the water evaporation process for larger batch sizes. Since 15 minutes is the targeted processing time for a full-scale batch, the process would be practical only if the time required for water evaporation is essentially independent of batch size. Otherwise, some means of increasing the evaporation rate would be needed, such as might be achieved by stirring the slurry.

Temperature appears to be a good indicator of when the moisture has been fully removed from the propellant. For this experiment, the process was terminated (intentionally) somewhat earlier than that needed to drive off the moisture to an acceptable residual level (tentatively taken to be 1%). The continual monitoring of temperature appears to be a practical means of monitoring progress of the batch condition during the vacuum-evaporation process.

The viscosity of the propellant slurry was found to be greater than that typically obtained from the “melt & cast” method. It should be noted that the viscosity of “melt & cast” propellant is highly dependant upon KNO₃ particle size (and thus offers a means of controlling viscosity). Experiments performed for the *SStS* project by Serge Pipko demonstrated that the KNSB propellant produced by the evaporation method contains a large quantity of very fine particles, or more correctly, crystals. The consequence of this is a particularly viscous propellant that is “barely scoopable”. It was found that the viscosity could be reduced to some extent by heating the slurry to 140°C and by enthusiastic stirring. Heating to higher temperatures did not seem to reduce the viscosity appreciably. If this method is to be considered for the *SStS* project, it would be prudent to investigate possible means to reduce the slurry viscosity. Possibilities are the inclusion of an additive such as iron oxide (which is known to reduce viscosity) or a small change to the O/F ratio, such as 64/36.

Heating of the propellant to a temperature of 160°C. was found to be non-detrimental to the propellant and that the degree of decomposition was very slight, at least for short duration heating.

The ambient burn rate of the as-cast propellant was found to be consistent, averaging 0.24 mm/second. This is slightly less than the 0.25-0.26 mm/second rate that has been measured for standard “melt & cast” KNSB. The strands made from the propellant sample that was further oven-dried were found to burn slightly (about 5%) faster than the as-cast propellant, at 0.25 mm/second. This burn rate coincides with the “melt & cast” propellant. As such, it appears that slight residual water content does have a small effect on burn rate, at least at ambient pressure. As well, the burn rate of propellant prepared by the two methods is very comparable.

Figure & Tables

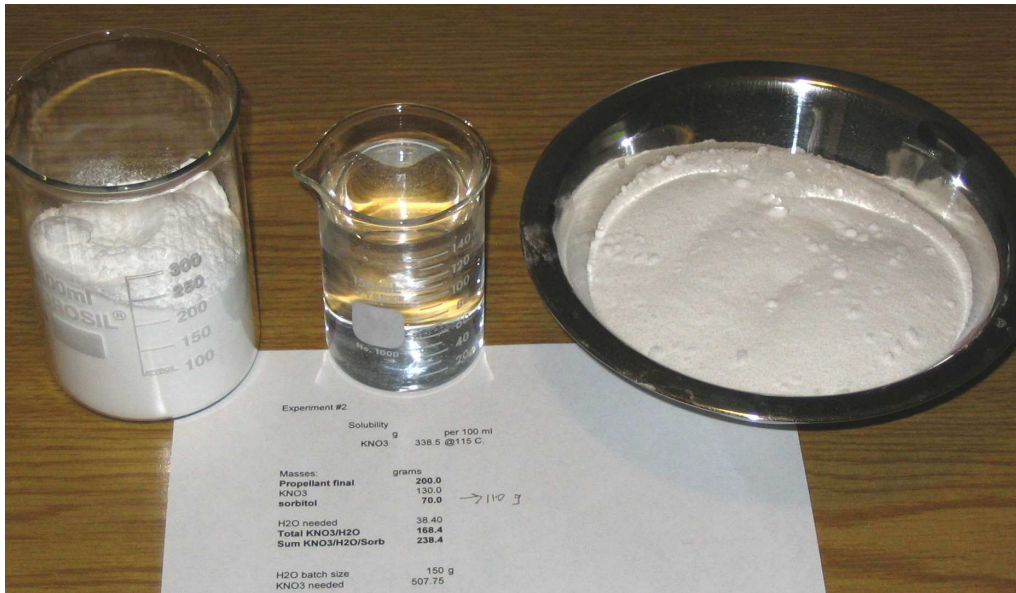


Fig. 1 - Chemicals used in experiment. Left to right: sorbitol, H₂O, KNO₃



Fig. 2 - Apparatus used in experiment.
Left to right: laptop, DVM, Heating unit #1, unit #2, unit #3, vacuum pump. Dessicator is on top of green box.

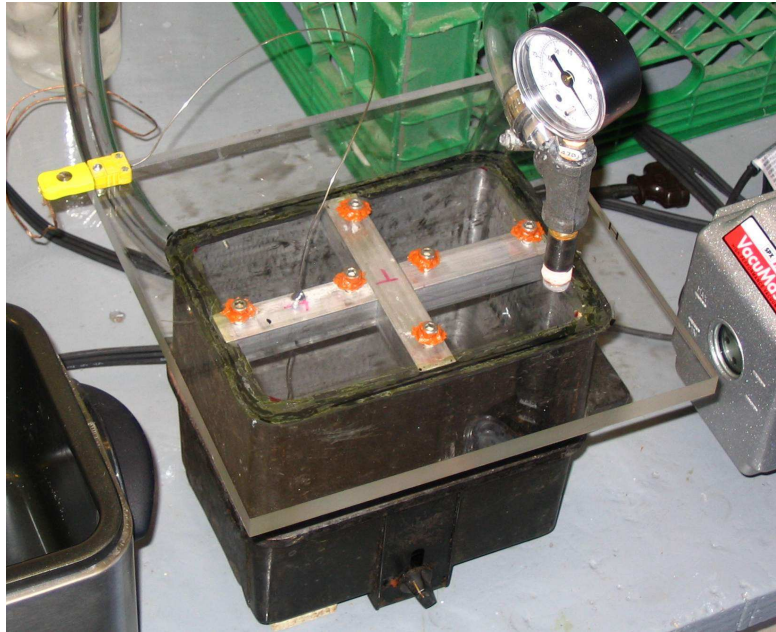


Fig. 3 - unit #3 heating vessel used for vacuum-evaporation of propellant (silver-coloured wire is the thermocouple probe)



Fig. 4 - Transfer vessel with $\text{KNO}_3/\text{H}_2\text{O}$ /sorbitol mixture

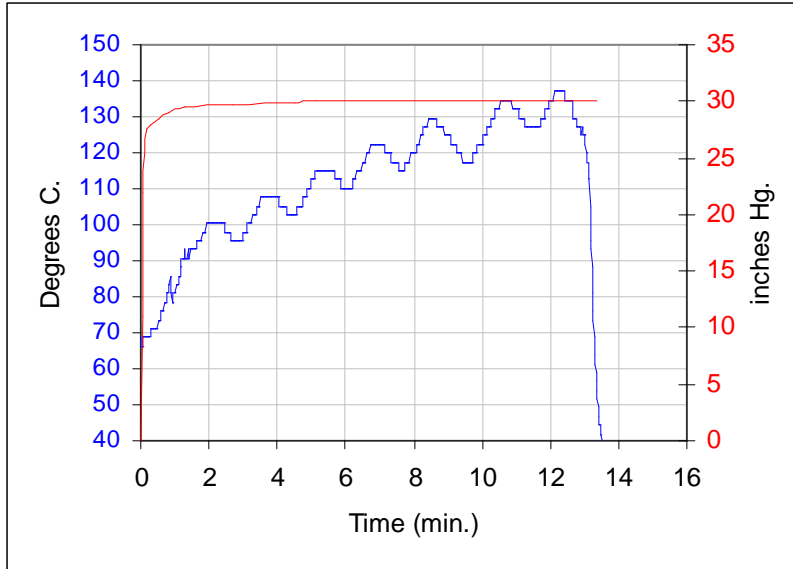


Fig. 5 - Unit #3 vacuum pressure and slurry temperature over duration of experiment

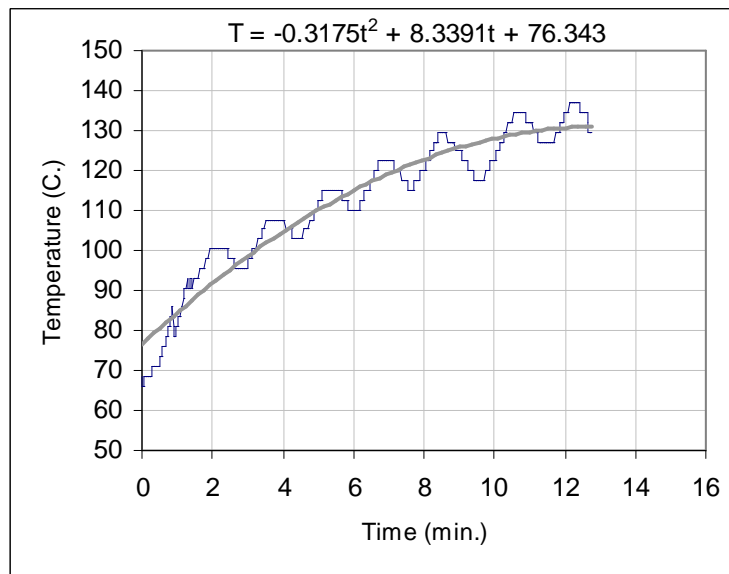


Fig. 6 - Slurry temperature with polynomial trend line fitted through curve



Fig. 7 - Propellant strands for burn rate measurements, prior to painting.



Fig. 8 – A small sample of the KNSB propellant produced by vacuum-evaporation method.

Strand	L1 (mm)	L2 (mm)	time 1 (sec.)	time 2 (sec.)	rate 1 mm/sec	rate 2 mm/sec
1	14.2	27.9	5.96	11.32	2.4	2.5
2	11.9	29.8	4.86	11.84	2.4	2.5
3	13.0	23.0	5.34	9.32	2.4	2.5
4	11.0	20.5	4.49	8.81	2.4	2.3
5	15.0	33.7	6.39	13.81	2.3	2.4
				average	2.4	2.4

Table 1 - Burn rate measurements of the as-cast propellant strands

Strand	L1	L2	time 1	time 2	rate 1	rate 2
	(mm)	(mm)	(sec.)	(sec.)	mm/sec	mm/sec
1	14.5	27.5	5.70	11.15	2.5	2.5
2	15.5	28.0	6.20	11.00	2.5	2.5
				average	2.5	2.5

Table 2 - Burn rate measurements of strands made from oven-dried sample.