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## Fuel-oxidizer mixtures: a lab and field study

Ryan C. Rettinger, Matthew Porter, Jeffrey Canaria, James L. Smith, and Jimmie C. Oxley

Chemistry Department, University of Rhode Island, Kingston, USA

### ABSTRACT

Improvised explosive may be as primitive as a fuel and oxidizer mixture; yet not all fuel-oxidizer mixtures are explosive. Predicting explosive potential from laboratory-scale tests is desirable to screen a large matrix of potential threats in varying concentrations. Herein the properties of various fuel/oxidizer mixtures were measured at small scale (2 g) with bomb calorimetry and on large scale (5 kg) with high speed photography for detonation velocity and with piezoelectric pressure probes for TNT air blast equivalence. Potassium nitrate (KN), potassium chlorate (KC), potassium permanganate (KMnO<sub>4</sub>), potassium iodate (KIO<sub>3</sub>), ammonium nitrate (AN), and ammonium perchlorate (AP) were prepared with sucrose (Su) and aluminum (Al) fuel. Results were compared to each other as well as predictions from Cheetah thermochemical code.

### KEYWORDS

Oxidizers; fuel-oxidizer; detonable; explosive; TNT equivalence

## Introduction

Hundreds of years ago, the field of energetic materials began with the invention of a fuel-oxidizer mixture of charcoal, sulfur, and potassium nitrate, which became known as black powder (Davis 1956). Within the last century, the fuel-oxidizer mixture of ammonium nitrate and fuel oil (ANFO) became popular as a commercial blasting agent (Marshal and Oxley 2009) and later as a terrorist tool (Marshal and Oxley 2009; Woodfin 2007). In the intervening period, the discovery of nitration resulted in a number of well-performing, high-density organic molecules – nitrate esters, nitroarenes, and nitramines. Because these molecules have become the basis of military weaponry, much effort has been expended in modeling their detonation performance. Fuel/oxidizer mixtures, when examined by the same protocols, have been termed “non-ideal” explosives because complex, diffusion-limited reaction zone chemistry rarely converts all the available energy in sufficient time to support detonation front propagation, leading to the tendency of models to over-predict the actual detonation performance. Nevertheless, it has become imperative that we understand fuel-oxidizer mixtures since their ease of clandestine creation—simply mixing a fuel and oxidizer together—has made them a common choice in illicit bombings.

We have previously reported a series of 11 oxidizers and 13 fuels examined by differential scanning calorimetry (DSC), simultaneous DSC/TGA (SDT), and by open burn. DSC is usually the first step in evaluating the energy content of an energetic formulation because the technique can use less than a milligram of material. In preparing the fuel/oxidizer DSC samples, great care was taken to make the samples homogeneous. Nevertheless, the DSC traces were difficult to interpret due to the small size of the prepared batches and the presence of multiple thermal events (Oxley et al. 2015). Herein we report a re-investigation of some selected fuel-oxidizer mixtures using isoperibol calorimetry – a Parr bomb calorimeter – recording the heat release and dynamic pressure rise of 2 g samples burning under argon.

**CONTACT** Jimmie Oxley ✉ [joxley@chm.uri.edu](mailto:joxley@chm.uri.edu) 📍 Chemistry Department, University of Rhode Island, 140 Flagg Road, Kingston, RI 02881

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Detonation of select formulations was attempted on the 10 pound-scale. (On average 5 kg of fuel-oxidizer mixtures filled the 4" id x 24" long schedule 40 PVC charge casing and were boosted with a 546 g donor charge containing PETN-based sheet explosive and C4). The event was recorded by high-speed photography and a single blast pressure transducer.

## Experimental Section

### *Bomb Calorimetry with Pressure Transducer*

The fuels chosen were sucrose from Fisher Scientific and 23  $\mu\text{m}$  steric acid-coated aluminum powder from Obron. Oxidizers were ground and sieved 100–200 mesh (150–75  $\mu\text{m}$ ). Sucrose was also ground with a commercial coffee grinder and sieved 100–200 mesh as well. Fuel/oxidizer mixtures were prepared as dry loose powders. Heat output and closed-vessel dynamic pressure records were determined using a Parr 6200 calorimeter and Parr 1108 bomb (Alloy 20), fitted with a pressure transducer (Parr 6976 pressure recording system, including a 5108A Kistler IEPE coupler, and a 211B2 Kistler piezoelectric pressure transducer with a calibrated sensitivity of 1.096 mV/psi). The Parr bomb was calibrated (i.e., 10 trials) with benzoic acid ignited with fuse wire (9.6232 J/cm) and cotton string (167.36 J) in 2515 kPa oxygen ( $\Delta H_{\text{comb}} = 26,434$  J/g) in an oxygen atmosphere. The string was in contact with both the fuse wire and sample. When electrical current was passed through the fuse wire, it sufficiently heated to cause ignition of the cotton string, which reliably ignited samples under oxygen. The fuel-oxidizer samples were ignited with a fuse wire under argon pressure (2859 kPa, 400 psig). Three to six 2 g samples of the loose pyrotechnic powder were run under each set of conditions, with the exception of potassium nitrate-sucrose doped mixtures of which only two samples each were performed. For all measurements, standard deviation was less than 4.6% for enthalpy and 10.0% for pressure. This vessel pressure represented the maximum initial pressure which the regulator could handle. It appeared to be a good balance, allowing rapid initiation of burn, and minimizing heat losses with the walls of the Parr bomb (Massey 1963). With some energetic materials, it has been observed that there is a critical pressure of ignition associated with a specified input energy (Barrett 1971; Shannon 1967). Igniting samples at a higher initial pressure is more likely to overcome the critical pressure of the sample. A National Instruments USB-6210 data acquisition card and LabView software were used to collect the pressure/time data at a rate of 10 kSa/s. This sample collection rate of 100  $\mu\text{s}$  sample intervals was resolved sufficiently so that the pressure/time records, including the rapid rise time, appear continuous on the millisecond time-scale of the burn events for these experiments.

### *Sample Preparation for Detonation Diagnostics*

Potassium nitrate (KN), potassium chlorate (KC), potassium permanganate ( $\text{KMnO}_4$ ), potassium iodate ( $\text{KIO}_3$ ), ammonium nitrate (AN), and ammonium perchlorate (AP) were prepared with sucrose (Su) fuel; in addition, KN, AP, and AN were prepared with aluminum (Al) fuel. All two-component fuel-oxidizer mixtures were prepared at a 70:30 wt:wt ratio with fuel. Later, KN-Su mixtures were adulterated with both KC and high explosive 1,3,5-trinitro-1,3,5-triazacyclohexane (RDX). Sucrose and oxidizers were prepared separately by grinding with a Vita-Mix 5000 blender and sieving each to 100–200 mesh (150–75  $\mu\text{m}$ ). Both the aluminum powder (23  $\mu\text{m}$ ) from Obron and RDX purchased from Accurate Energetic Systems were used as received. Fuel-oxidizer samples of approximately 5 kg were manually mixed in a 37.9 L (10 gal) plastic bag for about 2 min. For the detonation studies, schedule 40 clear, polyvinyl chloride (PVC) tubes of 4 inch nominal diameter (10.16 cm) were purchased from McMaster Carr in 8 foot lengths and cut to 24 inches (60.96 cm) long. PVC booster cups were assembled by gluing a 4 inch PVC sewer and drain endcap to a 4 inch PVC coupler. Into the booster cup were placed two sheets (30 g) of #2 PETN sheet explosive, which had been cut into circle shape to fit tightly into the booster cup. On top of the sheet explosive, C4

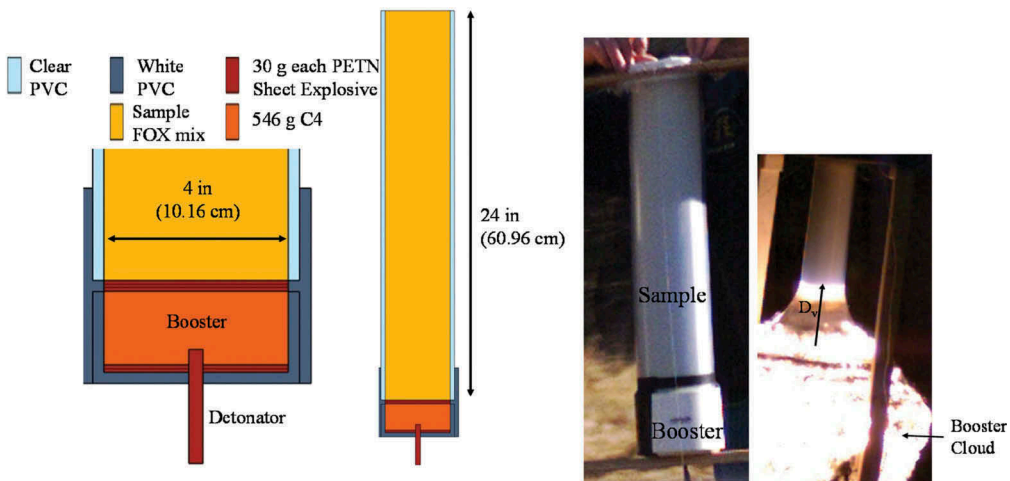
(546 g) was packed and then three more discs of PETN sheet explosive. The fuel-oxidizer mixture was added by pouring from the plastic mixing bag, using a craft paper funnel and hand-tamped to settle the loose powder. The test device was mounted in a vertical position (booster end down, [Figure 1](#)) on a wooden test stand about 1 m from the ground, and initiated by a #8 electric blasting cap.

### Detonation Diagnostics

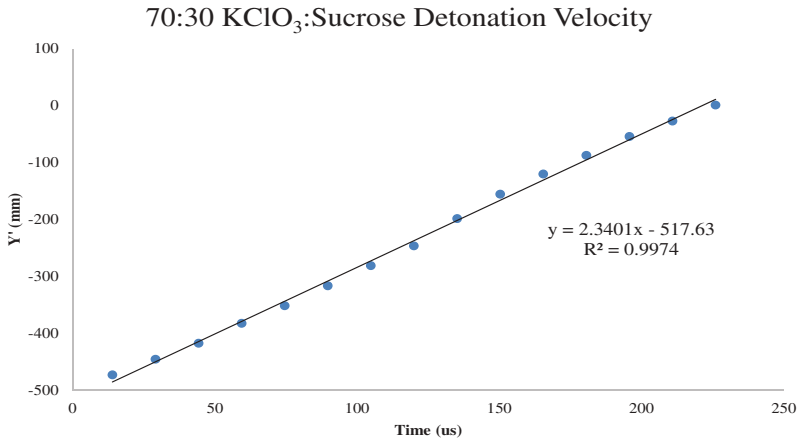
Detonation velocity was determined via high-speed camera record using a Phantom V7.11 with a frame rate of 66,019 fps, interframe time of 15.15  $\mu$ s, resolution of 160  $\times$  304 pixels, exposure of 0.4  $\mu$ s (0.29  $\mu$ s exposure for aluminum mixtures), 1 s of pre-trigger, and 1 s of post-trigger. A twisted pair of duplex wire affixed to the detonator, served as a ‘make-wire’ camera trigger. Phantom PCC 2.8 software was used to process the camera data, tracking the detonation front and using a distance scaling calibration for each record to obtain a detonation velocity. The detonation front was assumed to be the forward most position of the emitted band of light, following the contribution of the booster ([Figure 1](#)). The initiation of the booster produces a significant visual event and fireball, present even in samples that do not detonate, and is defined herein as the ‘booster cloud’ ([Figure 1](#)). After using the Phantom PCC 2.8 software to track the scaled detonation front position x-y points through time, a correction was made to the geometric coordinates to account for tilt. The detonation velocity was calculated by performing a linear regression of all angle-corrected front positions at known camera frame time intervals. The distance vs. time curves were linear ( $R^2 > 0.99$ ) for all of the samples that steadily detonated ([Figure 2](#)). Deviations from linearity were attributed to variations in packing density.

In two cases replicates were attempted with TNT and with potassium chlorate/sugar. However, since TNT was flaked and chlorate/sugar was powdered, it proved impossible to hand-pack to the same density. For the 9% (TNT) to 16% (chlorate) variations in density, there was a 6 to 17% variation in detonation velocity.

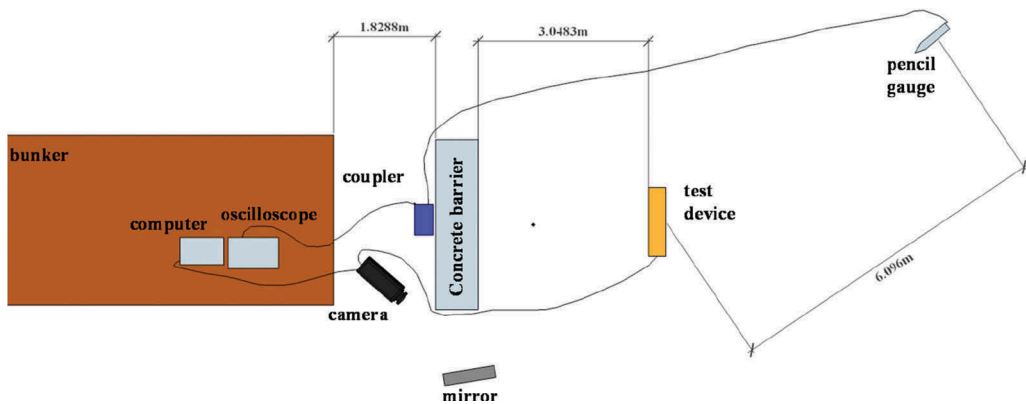
A pencil gauge pressure transducer (Kistler 6233A, 25 psi limit, calibrated sensitivity of 200 mV/psi) with coupler (Kistler 5134B, 0.05 Hz high pass filter) measured blast overpressure. Fifty foot coaxial cables connected the pencil gauge to the coupler, and coupler to a Tektronix oscilloscope. The pencil gauge was mounted 1.29 m high, positioned 6.096 m (20 ft) from the test device on a wooden stand weighted with sand bags. The Tektronix oscilloscope (model MSO4014B, max bandwidth of 100 MHz) was set with a typical



**Figure 1.** Schematic and photo of test fixture.



**Figure 2.** Detonation front tracking of tilt-corrected  $Y'$  points representing the shock front position through time. Linear slope is detonation velocity (mm/ $\mu$ s).



**Figure 3.** Original setup of camera & pressure transducer. Later filmed direct line-of-sight  $\sim$ 60 m.

sampling rate between 5–500 MSA/s; it was automatically triggered on the rising edge of the pressure signal. Figure 3 shows the overall test arena setup.

### Predictive Tools

Cheetah 7.0 from Lawrence Livermore National Laboratory (product library: jczs revision 1923 or bkws) was used to predict detonation velocity, detonation pressure, and total energy of reaction. Each mixture was evaluated with Cheetah using the density that was measured for its large scale test. It should also be noted that the product library chosen in the Cheetah software had a significant effect on the detonation energy calculation, as will be discussed later.

The blast effects calculator (BEC V5.1) was used to obtain air blast TNT equivalence from the measured peak air blast pressures (Kingery and Bulmash 1984; Swisdak 2001; Swisdak and Ward 2001). For each experiment, the BEC relation was used to calculate TNT equivalent charge mass of the booster-fuel-oxidizer-mixtures combined charge mass ( $TNT_{eq\ comb}$ ), i.e. the amount of TNT that would have achieved the same blast pressure at the same distance. To account for the booster contribution, the TNT equivalent weight (not pressure) of the booster alone ( $Booster\ TNT_{eq}$ ) was subtracted from the  $TNT_{eq\ comb}$  (Eq. (1)). An

experiment with an identical booster and sand acting as an inert charge allowed the precise TNT equivalent weight of the booster only to be calculated (1.419 kg). The booster-corrected equivalent was divided by the actual charge mass of the fuel-oxidizer mixture to yield the normalized TNT equivalence (TNT<sub>eq</sub>). Shooting the booster alone also gave a visual reference of what a ‘failure’ would look like on camera record.

$$TNT_{eq} = \left[ \frac{(TNT_{eq} \text{ comb} - \text{Booster TNT}_{eq})}{FOX \text{ Charge Mass}} \right] \quad (1)$$

## Results

### Parr Bomb Results

A Parr bomb calorimeter was used primarily to estimate the maximum available chemical potential energy from each fuel-oxidizer mixture, reported as bomb calorimeter heat of reaction (Table 1). Combustion was accomplished under argon gas instead of oxygen gas to determine energy release without any added combustion under oxygen atmosphere. As a result, the heats of reaction ( $\Delta H_{rxn}$ ) should be necessarily lower than heats of combustion, especially because many of these mixtures are fuel heavy. *The heat of reaction is intended to provide an upper-estimate of the amount of exothermic energy contribution to wave propagation in the chemical reaction zone of the detonation wave, and a lower-estimate of the amount of energy release in air blast.* Not all the chemical energy measured by calorimetry reacts in sufficient time to support the detonation wave. Non-ideal explosives like these fuel-oxidizer mixtures particularly demonstrate this deficiency. The included pressure transducer observed the pressure response as a function of time. Figure 4 shows the collected pressure traces and marks them according to whether detonation was subsequently observed in 5 kg scale tests. Pressure responses of ammonium nitrate (AN) and potassium nitrate (KN) with sucrose were significantly delayed compared to other mixtures. It is interesting to note that KN:sucrose burned only slightly slower and with slightly less energy than a similar mix which was spiked with 4% potassium chlorate (KC), but on the large scale straight KN:Sucrose mixes did not achieve steady detonation, while those with 4% KC did. A comparison of the same oxidizers with different fuels shows the best choice of fuel in terms of energy input is aluminum (dotted line) over sucrose (solid line) (Figure 4, Table 1) (Oxley et al. 2015). Figure 5 compares heat of reaction to the average rate of pressure rise, where average rate of rise was calculated as  $\Delta P/\Delta t$ . Interestingly, the ammonium nitrate-sucrose mixture (AN:Su) displayed such a long rise time (~7.6 s) that its  $dP/dt$  value was nearly zero.

### Detonation Velocity

Table 1 shows fuel-oxidizer mixtures for which detonation testing was attempted. Five of the mixtures failed to propagate detonation; of these, all exhibited at least a slightly higher-than-zero TNT air blast equivalency, but only three mixtures propagated long enough before failing to record a detonation velocity. Figure 6 provides screen captures of the detonation records. The detonation front position was assumed to be the leading edge of the bright line running ahead of the booster product expansion cloud. Figure 7 shows an enlarged picture of three example fuel-oxidizer mixtures known to be improvised explosive threats which detonated (AN:Sucrose, AN:Al, and KC:Sucrose) and one more example of one which did not detonate (KMnO<sub>4</sub>:Sucrose). Detonation was distinguished from a burn by the rapid PVC wall expansion angle immediately behind the front, e.g. AN:Al in Figure 9. The early stages of the KN:Al record suggest a steady detonation, but after some time the wall expansion angle separated from the bright front, indicating a transition to low-pressure burn (Figure 10). It could be said that this mixture was more flammable than detonable, a phenomenon known as a ‘pathological detonation’ (Sharp and Falle 2000; Sharpe 2000) for which no detonation solution is more stable or faster-moving than the burn front.



**Table 1.** Summary of Lab & Field Measurements & Cheetah Predictions. Strikethroughs under detonation velocity are velocities measured prior to failure; zero input means no observable reaction front and mixture scattered on ground. Some air blast records which did not collect or were over-saturated are marked "-". For TNT heat of detonation, a literature value (Ornellas 1982) was used. Bomb Calorimetry values are the average of 4 to 5 runs; all other entries represent individual tests. Energy of Detonation is total energy of detonation (TED).

Composition			Bomb Calorimetry					Phantom				Detonation Performance			Cheetah Prediction	
Ox 1 ID	Ox1 w	Ox2 ID	Ox2 w	fuel ID	fuel w	Charge Mass (kg)	Charge Density (g/mL)	Calorimetry Heat (kJ/g)	dt (ms)	Calorimeter dP (psi)	dP/dt (psi/ms)	Det Velocity (km/s)	Air Blast TNT Eq booster corrected	Air Blast Energy (kJ/g)	Cheetah Det Vel (km/s)	Energy of Detonation (kJ/g)
KNO <sub>3</sub>	70	-	-	Al	30	3.64	0.751	5.98	398	915	2.30	0.55	1.20	5.51	1.23	7.24
NH <sub>4</sub> ClO <sub>4</sub>	70	-	-	Al	30	3.13	0.691	9.36	79	2293	28.97	2.24	2.10	9.60	2.75	10.21
NH <sub>4</sub> NO <sub>3</sub>	70	-	-	Al	30	3.14	0.681	7.85	192	1504	7.85	2.70	2.59	11.86	3.57	9.11
KMnO <sub>4</sub>	70	-	-	Sucrose	30	5.22	1.101	2.07	507	228	0.45	<del>0.00</del>	0.10	0.46	2.12	2.83
KIO <sub>3</sub>	70	-	-	Sucrose	30	6.83	1.491	1.47	329	365	1.11	<del>0.00</del>	0.18	0.84	3.60	1.39
KNO <sub>3</sub>	70	-	-	Sucrose	30	4.71	0.973	2.81	508	534	1.05	<del>0.67</del>	0.28	1.30	3.14	2.69
KClO <sub>3</sub>	70	-	-	Sucrose	30	4.79	0.989	4.05	99	1037	10.53	2.58	1.42	6.48	3.78	3.59
KClO <sub>3</sub>	70	-	-	Sucrose	30	5.25	1.102	4.05	99	1037	10.53	3.05	1.08	4.95	4.22	3.61
KClO <sub>3</sub>	70	-	-	Sucrose	30	5.05	1.287	4.05	99	1037	10.53	2.86	-	-	4.49	3.61
NH <sub>4</sub> NO <sub>3</sub>	70	-	-	Sucrose	30	4.12	0.868	2.70	7655	222	0.03	3.49	0.85	3.91	4.60	2.66
NH <sub>4</sub> ClO <sub>4</sub>	70	-	-	Sucrose	30	4.66	0.979	4.88	97	1347	13.94	3.89	1.87	8.53	4.89	3.93
KNO <sub>3</sub>	67.9	KClO <sub>3</sub>	3.0	Sucrose	29.1	5.00	1.143	3.06	470	571	1.21	<del>4.46</del>	0.33	1.53	3.039	2.80
KNO <sub>3</sub>	67.2	KClO <sub>3</sub>	4.0	Sucrose	28.8	5.00	1.012	3.16	400	537	1.34	1.68	-	-	3.04	2.84
KNO <sub>3</sub>	63.0	KClO <sub>3</sub>	7.0	Sucrose	30.0	4.71	0.968	2.89	520	587	1.13	1.71	1.12	5.14	3.05	2.96
KNO <sub>3</sub>	35.0	KClO <sub>3</sub>	35.0	Sucrose	30.0	4.77	1.007	3.41	146	809	5.53	2.24	1.17	5.36	3.44	3.14
KNO <sub>3</sub>	69.4	RDX	1.0	Sucrose	29.6	5.00	0.910	2.93	390	581	1.49	<del>4.58</del>	0.28	1.30	3.07	2.72
KNO <sub>3</sub>	67.9	RDX	3.0	Sucrose	29.1	5.00	0.896	3.18	470	571	1.21	1.73	0.39	1.78	3.14	2.77
KNO <sub>3</sub>	66.5	RDX	5.0	Sucrose	28.5	4.25	0.884	2.93	420	609	1.45	1.76	0.88	4.01	3.21	2.82
KNO <sub>3</sub>	35.0	RDX	50.0	Sucrose	15.0	4.99	1.053	4.24	208	1139	5.48	4.80	0.96	4.41	4.83	4.02
TNT	100	-	-	-	-	3.66	0.769	4.57	-	-	-	3.84	-	-	4.34	3.42
TNT	100	-	-	-	-	4.00	0.814	4.57	-	-	-	4.24	1.00	4.59	4.50	3.47
Booster	100	-	-	-	-	0.55	-	-	-	-	-	-	0.00	0.00	-	-

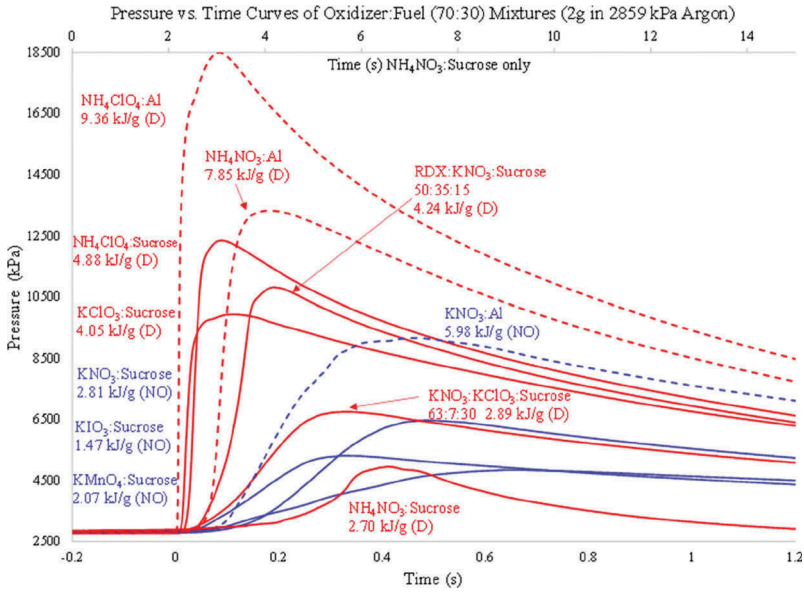


Figure 4. Pressure vs. time for burn 2 g fuel:oxidizer mixes under argon. In parentheses result of 5 kg test (D = Detonation; NO = No Detonation). Dashed lines are mixes with Al; solid lines the fuel is sucrose.  $\text{NH}_4\text{NO}_3$ :sucrose so slow that it has its own time axis.

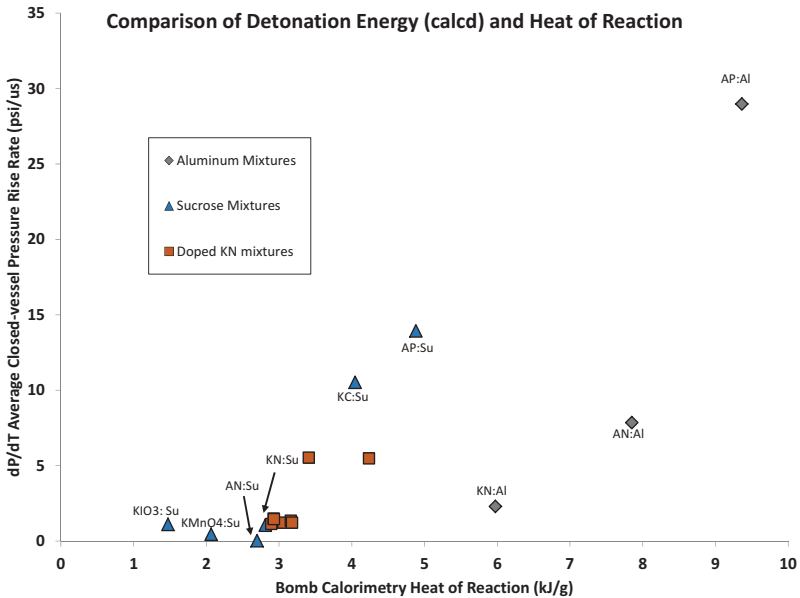
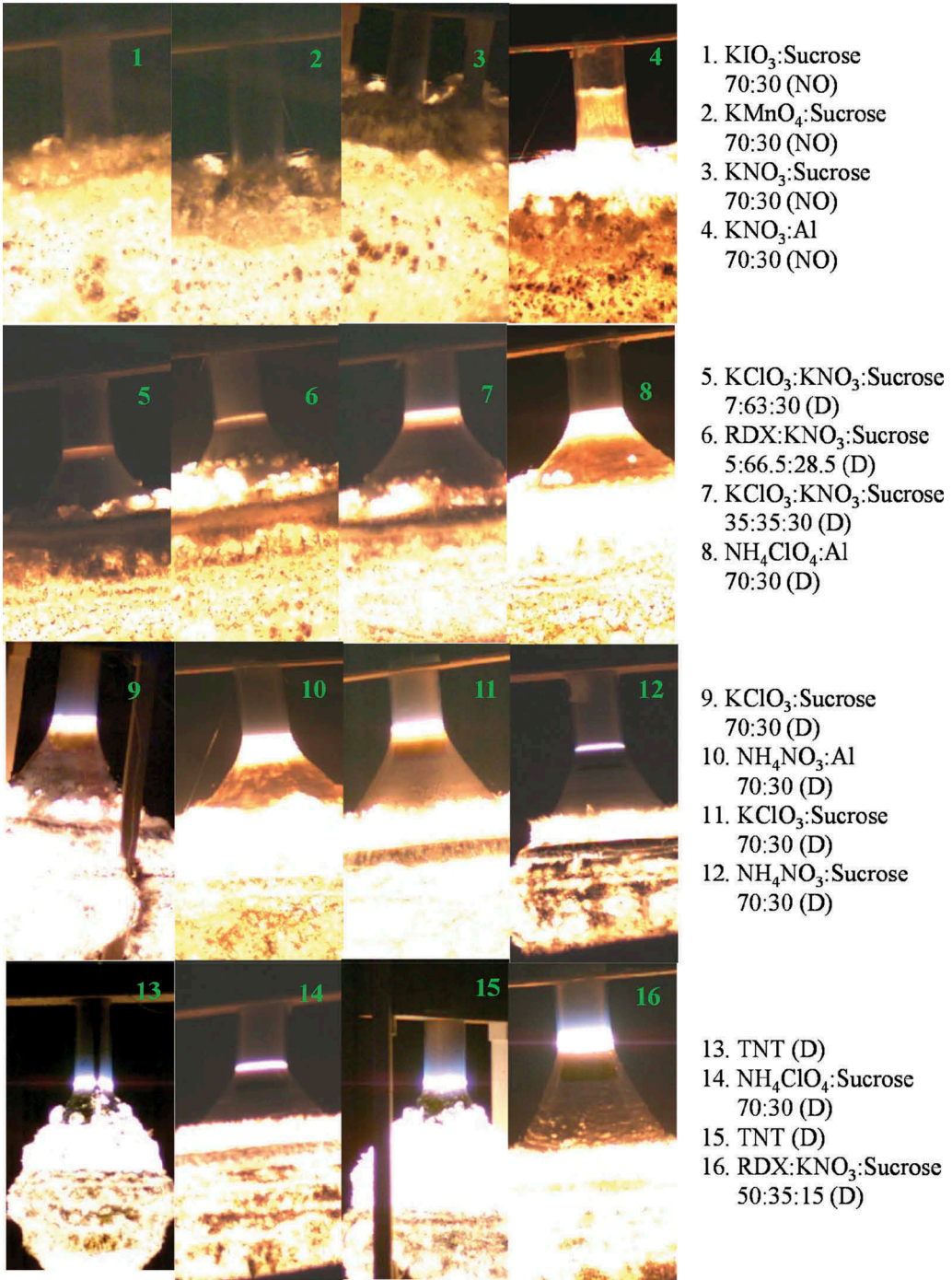


Figure 5. Comparison of two values obtained via bomb calorimetry-heat of reaction and average slope of pressure rise in closed-vessel bomb.

**Blast Pressure**

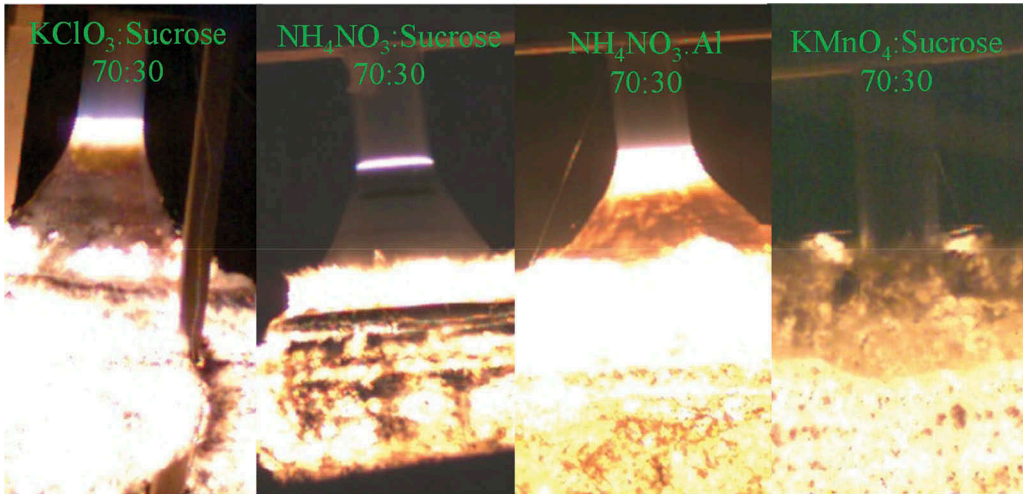
The blast records from 6 m (20ft) away are recorded in Table 1 and represent the peak air blast pressure from a pressure-time trace. An example is shown in Figure 9. The peak amplitude was the only measurement later used from the blast record although some information may be drawn from





**Figure 6.** A frame from video of each fuel-oxidizer mixture, when reaction was  $\sim 75\%$  along 2 ft pipe. Mixtures 5 to 16 show front separated from booster cloud. (D = Detonation, NO = NO Det).

the later-time section of each record. In particular, it may be possible to determine some late-time combustion reactions, which may help characterize the non-ideality of the fuel-oxidizer mixtures, but this section of the blast record is often obscured by reflections from the test environment which are difficult to subtract out from a complex combustion event. For analysis, the blast pressure was



**Figure 7.** Detonation tests: 3 steady detonations (left); and one failed (right).

first converted to TNT equivalent mass, and the booster contribution was subtracted; the remaining amount of TNT equivalent mass was divided by the actual fuel-oxidizer mixture mass to yield the TNT equivalence of the fuel-oxidizer mixture (Table 1). Later this TNT equivalence was used to infer the amount of energy released by normalizing to the heat of reaction of TNT.

### **Cheetah Thermochemical Predictions**

The Cheetah code was used to simulate the detonation parameters of each mixture tested. It uses thermochemical equilibrium to calculate the Chapman-Jouguet (CJ) state, i.e. the composition and thermodynamic states of the detonation products at a point representing a theoretical condition of stability for a 1-D detonation wave. This produces an estimate of detonation velocity and other CJ-state variables; further, the code calculates the process of an adiabatic expansion from the CJ state to the reference state (i.e. ambient pressure and temperature). After the expansion has stabilized through several iterations of chemical equilibrium, the integration of the CJ adiabat reports the total detonation energy. Results are displayed in Table 1.

### **Discussion**

The individual results from both the small scale tests and the detonation tests were successful in discriminating and ranking the mixtures. The order from each test may be slightly different, but within the dynamic range of the diagnostics, there was sufficient resolution in measurements to accurately describe the dynamic phenomena and differentiate every mixture, even when only small changes were made. Testing the reproducibility of the detonation experiments was limited by time and cost, but good agreement was seen from the triplicate measurements for potassium chlorate. Discrepancy between duplicate measurements can possibly be explained by the different experimental density from the replicates, which was not controlled. Fuel-oxidizer mixtures were chosen to examine three issues: relative detonability of the oxidizers; effect of the fuel choice on detonation performance; and sensitivity of detonation performance to small changes in composition. The booster charge was chosen to uniformly overdrive detonation across the entire cross-section of the mixture so that no initiation threshold criteria need be considered. A summary of the data is given in Table 1. Among the mixtures studied, AN and AP with sucrose fuel had the highest detonation velocity ( $D_v$ ), though density variations make it difficult to quantify the extent to which they are superior,

but without question, the AN and AP mixtures with aluminum performed the best in terms of TNT air blast equivalence.

### **Correlation of Cheetah Predictions to Measured Values**

The first comparison of our experimental data to Cheetah calculations is the comparison of isoperibol (bomb) calorimeter heat output ( $\Delta H_{\text{rxn}}$ ) to the Cheetah predicted energy of detonation. The total energy of detonation (TED) calculated from Cheetah agrees with the measured heat released in the calorimeter (Figure 10) indicating the chemical reactions in the bomb calorimeter and those simulated in the Cheetah detonation environment are similar. Bomb calorimetry results match predicted detonation energies better than other bench-scale laboratory thermal tests (e.g. DSC or TGA) probably because the temperature in the calorimeter is allowed to climb much higher than in the others tests, and the higher temperatures more closely resemble the detonation environment.

Cheetah results on fuel-oxidizer formulations with aluminum fuel were calculated assuming all aluminum reacted, which was not the case, *vide infra*. The calculated energies of combustion were only slightly higher than those of detonation. For the oxidizer mixtures with sucrose, the Cheetah calculated combustion energy was about 30% higher than detonation; and for TNT, combustion energy was approximately 400% higher than the detonation energy. This is in line with the degree to which these formulations are oxygen deficient in detonation; but when exposed to excess oxygen at longer time-scales, excess fuel burns completely in air. If the fuel-oxidizer compositions were adjusted to stoichiometric, then heat of detonation and combustion would have closely matched, but for this set of experiments, weight percent of fuel was held constant.

Observed detonation velocities correlate with Cheetah predicted detonation velocities, although all the mixtures fall below the line of agreement (Figure 11). Use of aluminum fuel did not increase the detonation velocity (neither calculated nor observed); in fact, detonation velocities of aluminum-fueled formulations were consistently lower than the sugar-fueled analogs, mostly due to decreased gas production with aluminum. The two TNT shots were performed at slightly different densities, which explain the difference in measured detonation velocities. These values agree reasonably well with previously published density vs Dv curves for TNT (Dobratz 1972), although agreement is inferred since the density is outside the published range. A line of 75% agreement seems to work well for the majority of steady detonations of fuel-oxidizer mixtures. Almost none of the mixtures achieved greater than 75–80% of their calculated detonation velocity, hence, their label as “non-ideal.” These mixtures are so slow to react and so near their critical diameters that 1-dimensional estimates (Cheetah) are particularly unreliable indicators of actual performance on the 5 kg scale. Choice of product library in Cheetah had a profound effect on detonation energy calculation. Figure 12 exhibits the range of results calculated for the potassium nitrate-sucrose mix. Based on the match to our calorimetric data, “jczs” was the library used for the data in the Table 1. The “bkws” library, whose TED measurement also closely matched the KN:Su calorimetric heat of reaction, was used for determining of percentage active aluminum discussed later; the jczs reactant library did not contain the needed entry for “inert aluminum.”

### **Measured Calorimetric Values Vs. Measured Detonation Velocity**

The bomb calorimeter measures both the overall heat of reaction ( $\Delta H_{\text{rxn}}$ ) and the time-resolved pressure response. Figure 13 shows the relationship between measured heat of reaction and measured detonation velocity. There appears to be a correlation between the heat of reaction of the sucrose-fueled mixtures and their measured detonation velocity but not for the aluminized-materials. For the sucrose formulations, the plot suggests a minimum energy ( $\sim 2.8$  kJ/g) is needed for detonation in this configuration. Assuming that is true, it was speculated that adding a species, which would contribute energy, might push low-energy formulations to detonation; this was investigated. However, complete substitution of aluminum (a high-energy fuel) for sucrose, significantly increased the heat release, but detonation velocity decreased. This effect was both predicted by Cheetah and observed in our measurements.

The pressure rise observed in bomb calorimetry was not a good indication of detonation performance. This was particularly true for the ammonium nitrate-sucrose mixtures since this detonable mixture took longer to react in the calorimeter than any other fuel-oxidizer mixture, even those, which were non-detonable under our conditions. Apparently, rapid chemical kinetics for this mixture appear exclusively at high pressures. This discrepancy is illustrated in [Figures 4 and 14](#). On the opposite end of the spectrum, AP:Su performed well in the calorimeter but produced only modestly higher detonation velocity in the field than the ammonium nitrate mixture.

### ***Bomb Calorimeter vs Blast Energy***

Even though the pressure-time records from the bomb calorimeter and air blast appear similar, they are not directly comparable. The burning environment in the calorimeter was necessarily lower pressure compared to the rapid detonation chemistry. Thus, the two can represent vastly different reaction kinetics; for example, AN:Su burned particularly slow in the calorimeter, but in a shock-driven detonation environment, performed better than its  $dp/dt$  result suggested.

Peak air blast pressure measurements were converted to energy units by assuming that the TNT equivalence value for each fuel-oxidizer mixture represented a multiplier of the energy released by TNT in a detonation calorimeter (4.57 kJ/g.) (Ornellas 1982). Thus, the two y-axes on [Figure 15](#) are offset by a factor of 4.57 and a line of agreement can be drawn. Most of the data from the steadily-detonating fuel-oxidizer mixtures lie above the line of agreement. There are two likely causes for this apparent enhancement: a) TNT heat of detonation is a minimum estimate of the conversion factor to air blast energy; and b) fuel-oxidizer mixtures in this study were fuel heavy, and the excess fuel would contribute some fraction of its combustion energy to air blast peak pressure. The TNT factor is likely an underestimate because the heat of detonation (inert atmosphere) is much lower than combustion (complete burning in oxygen, greater than four times the heat of detonation). The heat of detonation (no oxygen) is likely an upper limit for the amount of energy that can be released in the detonation reaction zone contributing to wave stability and propagation velocities, but all this energy and some fraction of its 400% extra combustion energy may contribute to peak air blast pressure; i.e. the correct multiplier for air blast energy is likely higher than TNT heat of detonation, lower than the heat of combustion, and can likely change over time as more fuel burns.

### ***Doping of Potassium Nitrate-Sugar to Achieve Steady Detonation***

Potassium nitrate with sucrose fuel (KN:Su) was a particularly interesting mixture in that a non-steady detonation wave emerged for a couple of charge diameters but eventually failed. The failing velocity profile indicated a detonation velocity of 0.67 km/s, which was well below the Cheetah-predicted value of 3.14 km/s. This means that the mixture was likely very close to its critical diameter, and the detonation wave was highly curved. The failure occurred because the energy losses in the form of pressure relief waves at the charge edges eventually coalesced and disrupted the overdriven detonation condition from the booster charge. At this point, the shock wave decoupled from the chemical reaction, and the detonation wave was quenched. It is possible that increasing the charge diameter would transition this material into a detonable regime, but it seemed more interesting to alter the available energy content by doping the KN:Su with a more energetic, more explosive compound to investigate how close this borderline formulation was to forming a steady detonation.

Two strategies were employed to increase the available energy to the KN:Su mix: first, maintaining the 70:30 wt:wt KN:sucrose ratio while substituting RDX, a highly-ideal monomolecular explosive; and second, substituting potassium chlorate (KC) for portions of the potassium nitrate. When substituting RDX, as much as 50% was used, and the detonation velocity was in excellent agreement with Cheetah predictions at this high concentration of ideal explosive dopant. It was found that both 5% and 3% RDX in complementary concentrations of 70:30 KN:Su were detonable, but 1% RDX



failed similar to the unadulterated KN:Su; the threshold was therefore between 1 and 3%. The second strategy of doping KN:sucrose was to exchange a small fraction of the KN with KC, an oxidizer which successfully detonated in a mix with sucrose fuel. Following this idea introduced mixtures which were still 70% oxidizer, but of that, mixtures with 50, 10, and 5.6%, and 4.2% KC were made. In these mixtures, KC constituted 35, 7, 4, and 3% of total fuel-oxidizer mixture, and all but 3% KC detonated steadily in field tests.

In bomb calorimetry tests corresponding to field tests, samples were spiked in a similar fashion to the RDX, maintaining the 70:30 ratio of KN:Su. Examining [Table 1](#), it is notable that the doped mixtures produced little more calorimetric heat than the undoped KN:Su ([Figure 16](#)). The amount of energy at threshold percentage of KC and RDX is trivially larger than the unadulterated KN:Su mix, and Cheetah predicted energies differences are even smaller. Both the data and detonation theory indicate that energy output alone does not guarantee detonation. Gas production, diameter, density, confinement, and most importantly, the *rate* of energy release by the formulation under shock loading must be fast enough to sustain detonation. Considering all these factors affecting detonation velocity, it is interesting that the oxidizer-sucrose mixtures appear to strongly correlate with  $D_v$  ([Figure 13](#)). This is likely because they release a similar amount of gas and because their diffusion-limited reaction rates are similar.

### **Partial Aluminum Reaction**

The aluminum-fueled mixtures demonstrate substantially enhanced blast response because the air blast time scales are long enough for the aluminum to release 100% of its heat of reaction (inferred in [Figure 15](#)). However, it is well known that aluminum does not react rapidly enough to support the detonation front; hence, the provision in Cheetah to make some of the aluminum content “inert.” Because it is believed only a small fraction of the aluminum powder reacts in the reaction zone of these fuel-oxidizer mixtures, a Cheetah study was undertaken to infer the extent of reaction in the three aluminum-fueled fuel-oxidizer mixtures.

For the AN-Al mix, the experimental detonation velocity was only 2.7 km/s, compared to the Cheetah prediction, 3.57 km/s. For this investigation, it was assumed the discrepancy from prediction was exclusively attributed to partially-reacting aluminum in the reaction zone, although the kinetics of the oxidizer may also have contributed. By letting Cheetah simulate a series of virtual mixtures using its “inert” aluminum (iAl) reactant and gradually varying its ratio to “active” aluminum (aAl), the trend in Cheetah-calculated detonation velocity was fit to a multi-order polynomial. The calculated trend was numerically-solved to match the experimental detonation velocity to indicate the inferred fraction of aAl. [Figure 17](#) illustrates the Cheetah-calculated trends of  $D_v$  vs. percent aAl for the three aluminum-fueled mixtures (filled markers). The plot starts (at the right) with 30% aAl, (the 0% iAl point); note all the Cheetah-calculated values in [Table 1](#) refer to this “all-aluminum-active” point. As each line travels leftward, the concentration of aAl decreases and is replaced with iAl, keeping the ratio of oxidizer to total fuel (aAl and iAl combined) constant, as well as maintaining the experimental density for each simulation. This monotonically decreases the detonation velocity until the predicted  $D_v$  decreases to match the value measured in the field (horizontal dotted line). The fact that none of these formulations uses more than 10% active aluminum indicate that even though the bomb calorimeter and the air blast response accommodate the slow Al reactions, the dynamic shock-loaded chemical environment was only able to extract a small percentage its energy. Examining the trend lines for total energy of detonation (hollow markers) at the indicated percentage of active aluminum means these low percentages of active aluminum correspond to vastly different efficiency in the reaction zone. A summary of the inferred values in shown below the figure caption.

Increasing amount of inferred %aAl (AP < AN < KN) inversely trends ([Figure 17](#)) with the measured reaction rates (calorimetry  $dP/dt$ ) among these oxidizers (KN < AN < AP). Since AP is expected to exhibit a particularly fast reaction, less of the slow, diffusion-limited aluminum

chemistry will be able to contribute to the reaction zone and support the detonation front. Conversely, the slowest-reacting oxidizer, KN, infers the largest amount of Al reacting in the reaction zone, since the slow-burning reaction zone in KN likely provides the most time for Al to react. It should be noted that KN alone is not energetic; its decomposition is endothermic (Oxley et al. 2015). Unfueled KN should never detonate. If the inferred aluminum content is correct, the cusp of stable detonation solutions seen in Figure 17 around 4% aAl is particularly delicate for KN compared to AN or AP. The latter both contain enough energy to, at least theoretically, propagate detonation without fuel. KN must be fueled to detonate even under the best circumstances; but when the fuel is very slow-acting, KN is particularly susceptible to a terminal drop in energy output. When other Cheetah product libraries are used, the implied aAl with each oxidizer changed slightly, but the observed order remained. The trend of increased fuel consumption in the reaction zone by slower-reacting oxidizers may extend to fuels beyond aluminum.

## Ideality

To globally evaluate the degree of non-ideality of the fuel-oxidizer mixtures, a linear relation between energy release and  $(\rho_0 D_v)^2$  was assumed ( $\rho_0$  = initial density). If the Cheetah-predicted detonation velocity represented 100% of the heat of reaction energy being released inside the reaction zone, the quantity  $\eta = D_{v, \text{exp}}^2 / D_{v, \text{Cheetah}}^2$  (where  $D_v$  is the experimentally-measured detonation velocity, and  $D_{v, \text{Cheetah}}$  is the  $D_v$  predicted by Cheetah) represents an estimate of the percent of energy released in the reaction zone for that observed detonation velocity. Thus, the closer  $\eta$  is to unity (100%), the closer the formulation performed to the ideal, 1-dimensional Cheetah prediction.

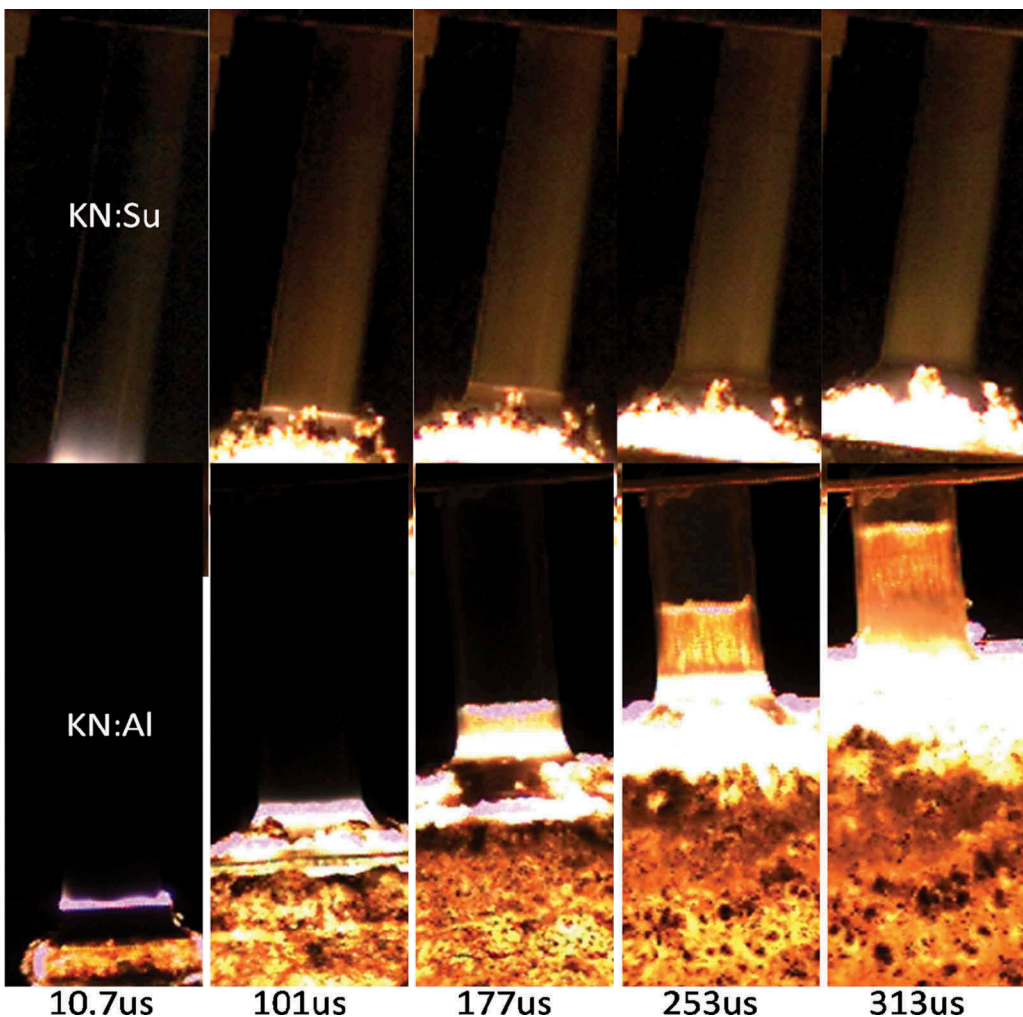
From the plot in Figure 18, it can be seen that a cutoff value around that of the bomb calorimetric heat of reaction for KN:Su (2.8kJ/g) is at the threshold between mixtures which are highly non-ideal and those closer to ideal. An interesting observation about this plot is that oxidizers tested with different fuels seem to remain similar in quantity of  $\eta$ . KN:Su and KN:Al differ in  $\eta$  by 15%, but AP:Al/AP:Su differ by only 1% and AN:Al/AN:Su by only 3%. The KN:Su mixture doped with 50% RDX indicated a nearly ideal mixture. Bomb calorimetry indicated that the added RDX did not appreciably increase the available energy content of the mixture; however, the added RDX apparently gave the mixture access to 100% of its available energy inside the chemical reaction zone. Most fuel-oxidizer mixtures only access 50–60% of their energy in the reaction zone. This is in line with the 75% line of agreement in Figure 11.

An efficiency term regarding air blast is calculated, air blast efficiency (ABE), by normalizing the inferred air blast energy (Figure 15) to the heat of reaction for each mixture. The ABE represents what fraction of the heat of reaction was released in air blast. Figure 19 illustrates the tendency of fuel-oxidizer mixtures to react inefficiently inside the reaction zone ( $\eta$ , x-axis), but their blast response often reflects 100% or more of their inert-atmosphere energy release (ABE, y-axis).

When the 50% RDX-doped KN-Su mixture, which demonstrated ideal reaction zone behavior, is examined in air blast efficiency (far right, Figure 19), no excess energy beyond its heat of reaction was observed, similar to TNT. This indicates that more ideal explosives do not experience a significant increased blast response from excess fuel combustion at this distance; at further distance, the energy of air blast may approach the heat of combustion for all mixtures. Even mixtures which failed to detonate, namely KN:Su,  $\text{KMnO}_4$ :Su, and  $\text{KIO}_3$ :Su, each released significantly higher fractions of their bomb energy in the conservative air blast results (46, 22, and 57%, respectively) compared to their ideality inside the reaction zone (4.55, 0, and 0%, respectively) inferred from Figure 19. A more comprehensive study completing the matrix of possible oxidizer-fuel combinations will reveal whether this is a global truth or just coincidence with these few samples.

## Pathological Detonation

The KN:Al data point is odd because the mixture exhibited a detonation to burn transition. The mixture could be said to be more flammable than detonable. The video record is quite clear about this transition. Initially, the shock-driven reaction propagated as the other fuel-oxidizer mixtures, but eventually a strangely turbulent, slightly dimmer flame front began to separate from shock front position indicated by the flaring of the PVC casing walls at the shock front, which lagged behind the burn front. This burning region was much cooler than the detonation front, as seen by the color temperature and light intensity in the frames captured by the Phantom camera (Figure 8(b)). After some induction time, the PVC casing finally burst, but all at once rather than progressively, further distinguishing KN:Al from the other fuel-oxidizer mixtures. Although it has been sparsely observed, certain complicated reaction kinetics and wave dynamics lead to very interesting and non-intuitive detonation instabilities, of which this low-density configuration of KN:Al is a prime example. These instabilities, or rather competing eigenvalues of wave stability conditions, have been named pathological detonations and represent only marginal cases of explosive science (Sharp and Falle 2000; Sharpe 2000). We believe the



**Figure 8.** (a).KN:Su started with a linear detonation velocity (note dim orange line emerging from booster cloud), but quickly failed. Later this formulation successfully detonated after being spiked with <5% KC or RDX. (b). KN:Al Pathological detonation in KN:Al started as an overdriven detonation (note PVC wall expansion angle in early frames), later transitioned to burn.

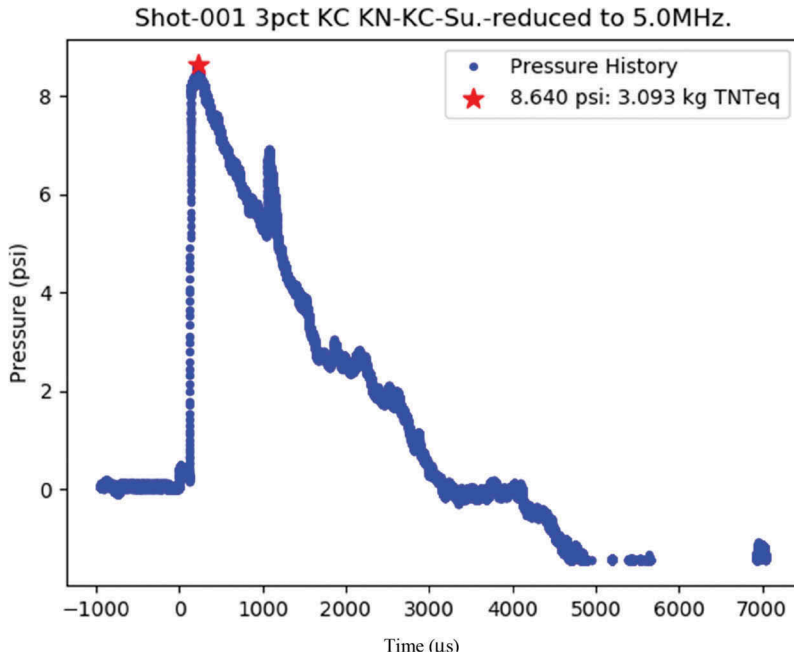


Figure 9. Typical blast pressure trace. Pressure presents as sharp rise to a maximum; perturbations later may indicate combustion dynamics are obscured by ground reflections and other complications. A python algorithm reduces sampling frequency to a user-defined 5 MHz, locates the maximum value and calculates TNT equivalent mass without a booster correction using the blast effects calculator formula. A booster correction is made later.

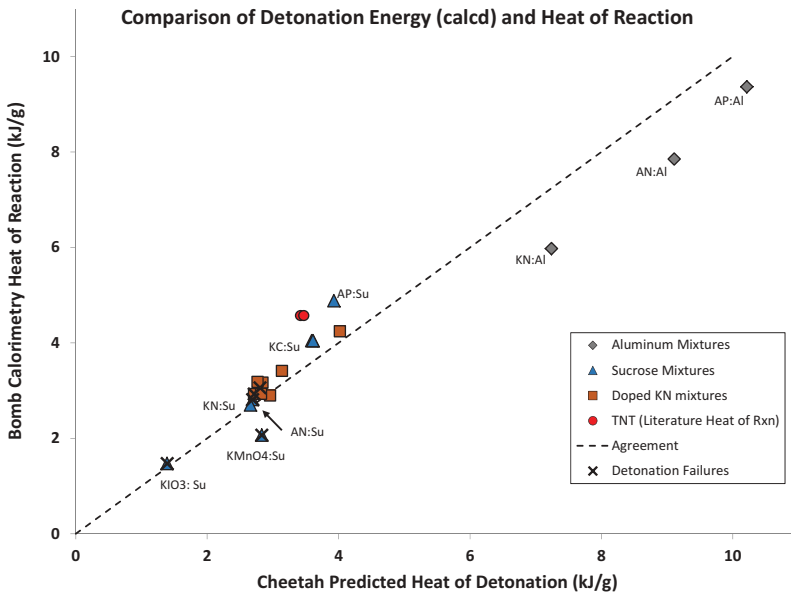


Figure 10. Correlation between  $\Delta H_{rxn}$  and Cheetah predicted total heat of detonation.

most contributing factors to the pathological condition in the case of 70:30 KN:Al are the extremely low experimental density (which encourages very fast convective burn rates and discourages high detonation velocities), the low gas production of aluminized fuel-oxidizer compositions, and the light confinement. All these factors retard the speed of the detonation in the traditional sense, giving opportunity for the



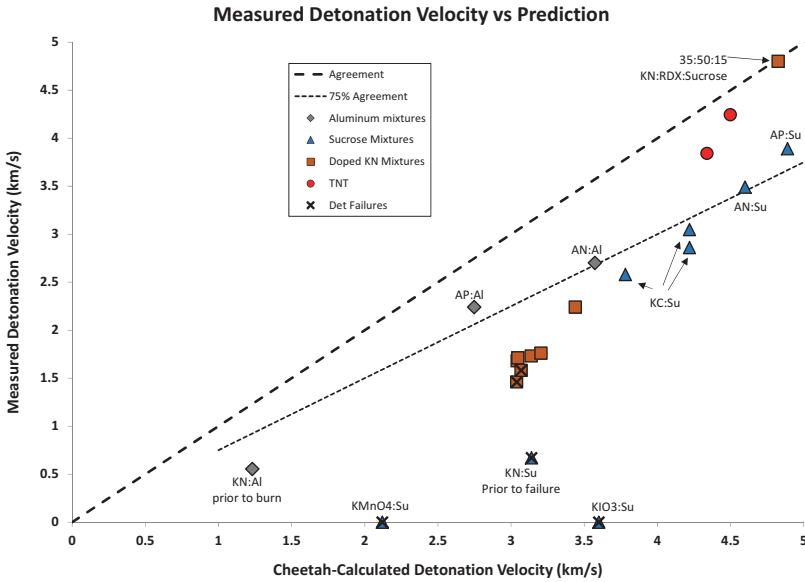


Figure 11. Detonation velocities (km/s) observed vs Cheetah calculated.

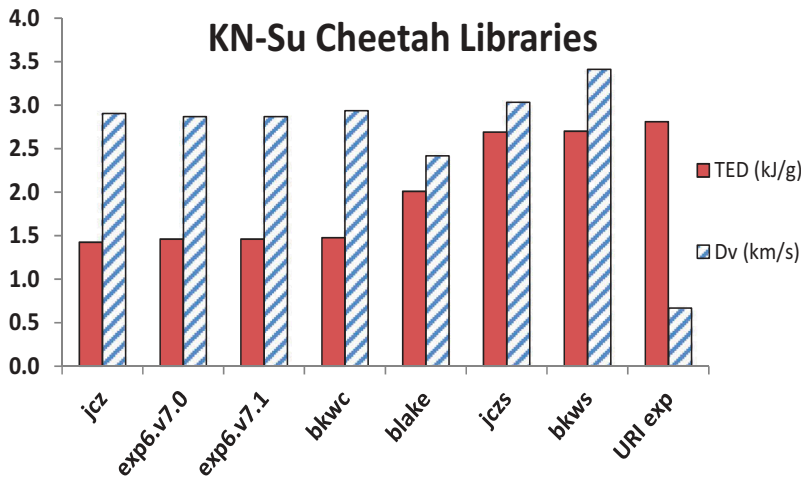


Figure 12. Cheetah library comparisons for potassium nitrate-sucrose. None of the predictions for detonation velocity (blue) matched observed values; thus, the library was chosen by closest match to energy release (red) experimentally observed (URI exp).

very high-energy, low-pressure combustion kinetics to overtake the otherwise slow but steady detonation. Further experiments at different diameters and with slightly different component ratios, as well as good charge density control and embedded pressure gauges, would be a good start to understand the complex combustion/detonation environment in this improvised, non-ideal material. It is also not clear how important the role of the included air may be at these low densities.

### Conclusions

Data presented here clearly illustrate the disparity that may be observed between the performance of an explosive in terms of blast versus detonation velocity. Measurement or calculation (Cheetah) of

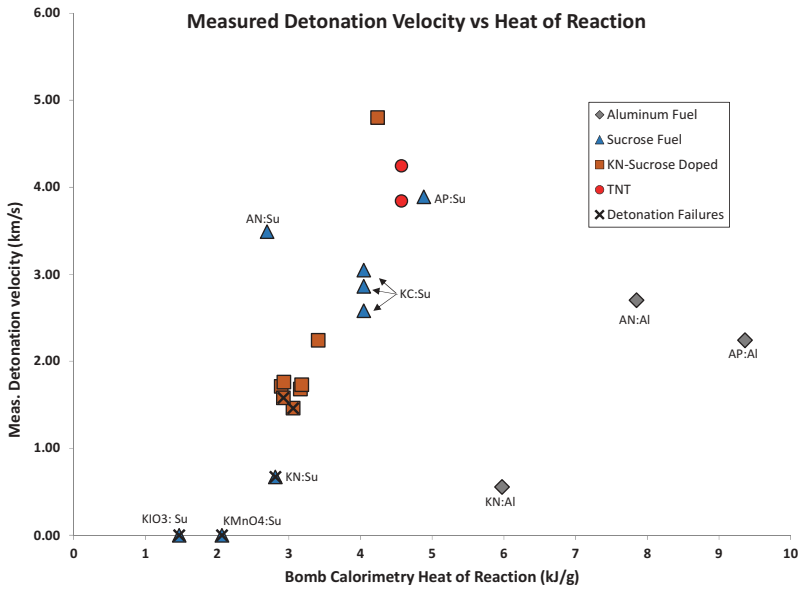


Figure 13. Measured detonation velocities vs calorimetric heat of reaction. There appears to be a trend for sucrose-fueled mixtures but not followed for aluminized.

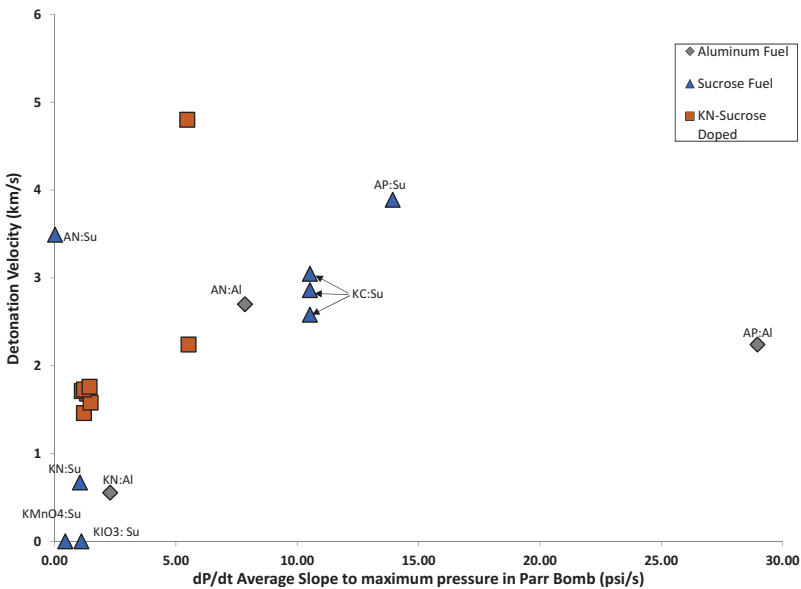
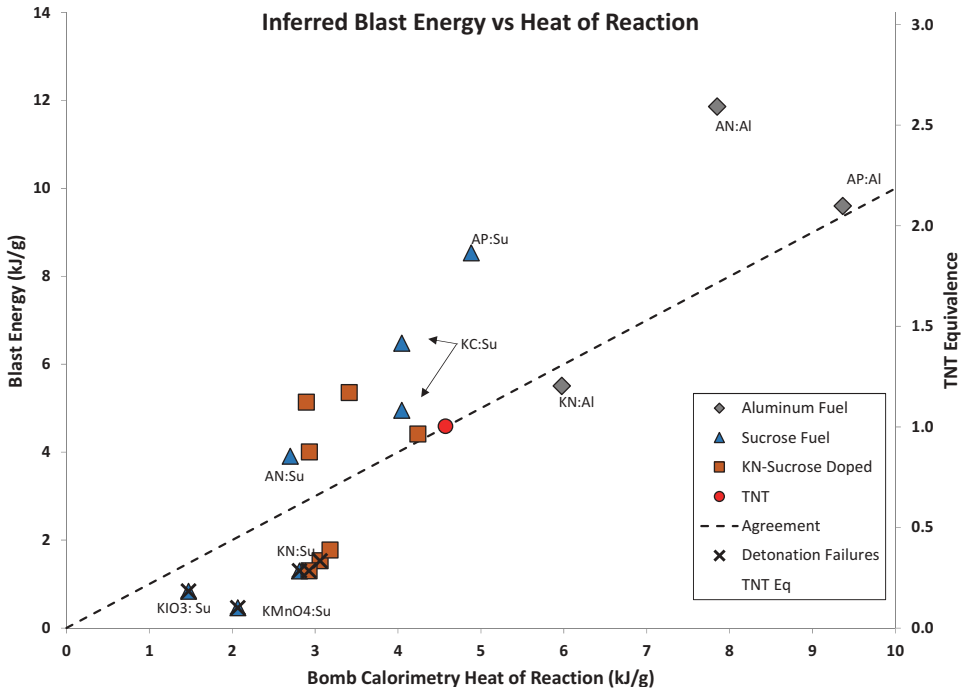


Figure 14. Correlation average closed-vessel pressure rise rate with measured detonation velocity. Note AN:Su displayed near-zero dP/dt, but showed a strong detonation velocity; conversely, AP:Al burned rapidly in the calorimeter.

heat of reaction (oxygen-free) is a useful first step in determining whether a formulation is potentially detonable. It appears there is a minimum specific energy (~2.8 kJ/g) which a formulation must possess to be detonable. However, examination of Table 1 clearly shows that some materials with relatively high reaction energy (i.e. KN:Al) do not support traditional steady detonations, while others with borderline energy (i.e. AN:Su) do. Clearly, any small-scale test or model must take into account the rate of reaction as well as total gas and energy release, but there is evidence to indicate



**Figure 15.** Air blast energy (left y-axis) and  $TNT_{eq}$  (right y-axis) vs. bomb calorimetry heat of combustion. Line of agreement illustrates which mixtures infer more or less energy in air blast than in calorimetry. Even mixtures which failed to detonate show non-zero air blast energy release.

that higher heats of reaction indicate a higher degree of ideality, i.e., access to a larger fraction of the available chemical energy to support the shock.

In general, fuel-oxidizer mixtures which exhibited a rapid rise to peak pressure in lab-scale calorimetry detonated on the large scale; **Figure 4** marks the bomb calorimetry traces to reflect the large-scale detonation result- (D) for full detonation, (NO) for failure to detonate. Those mixtures which reached peak pressure more slowly did not detonate at the large scale, with the exception of AN:Su which burned so slowly that it required its own axis (top of **Figure 4**). AN-sucrose apparently has an extremely pressure-dependent decomposition rate law, making low-pressure (30 atm) burn-time tests a poor indicator of reaction rate in the shock-loaded environment for this mixture. AP:Su and AP:Al burned extremely fast in the bomb calorimeter, yet marginally outperformed other fuel-oxidizer mixtures in terms of reaction zone ideality compared to other fuel-oxidizer mixtures with similar heats of reaction. Regardless of fuel choice (sucrose or aluminum), oxidizers tended to perform to a similar degree of ideality, e.g., AN:Al and AN:Su were of similar ideality “ $\eta_p$ ” as well as AP:Su and AP:Al.

The potassium nitrate-sucrose mixture exhibited low heat release in the bomb calorimeter, as well as slow pressure rise times, and it did not steadily detonate on the 5 kg scale. This mixture did show a temporary and failing detonation wave before quenching (**Figure 8(a)**), unlike the potassium permanganate and potassium iodate mixtures, which failed to produce any reaction light throughout the high-speed record. In spite of this, they released some fraction of their energy to air blast, contributing 22% ( $KMnO_4:Su$ ) and 57% ( $KIO_3$ ) of their respective heats of reaction to air blast energy (**Figure 18**), while typical detonating fuel-oxidizer mixtures were capable of contributing an inferred air blast energy 120–180% of their heats of reaction. We attribute this greater than 100% contribution to the discrepancy of heat of reaction versus heat of combustion when excess fuel could combine with oxygen. It should be noted that in the analyses the largest source of error was from the field data where exact replicates prove impossible to perform versus lab-scale studies where variations in enthalpy were less than 5%.

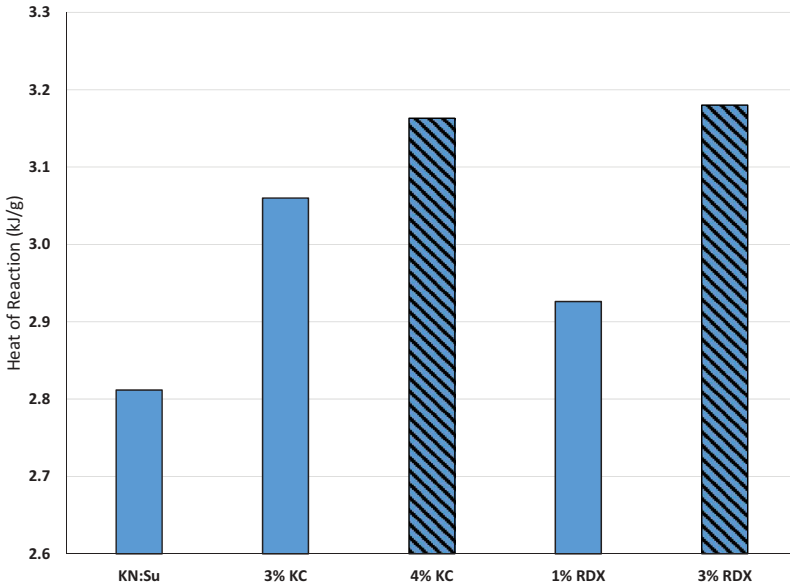


Figure 16. Thresholds for steady detonation of KN:Su doped fuel-oxidizer mixtures. Solid bars indicate unsteady detonations which eventually failed. Striped bars indicate steady detonation.

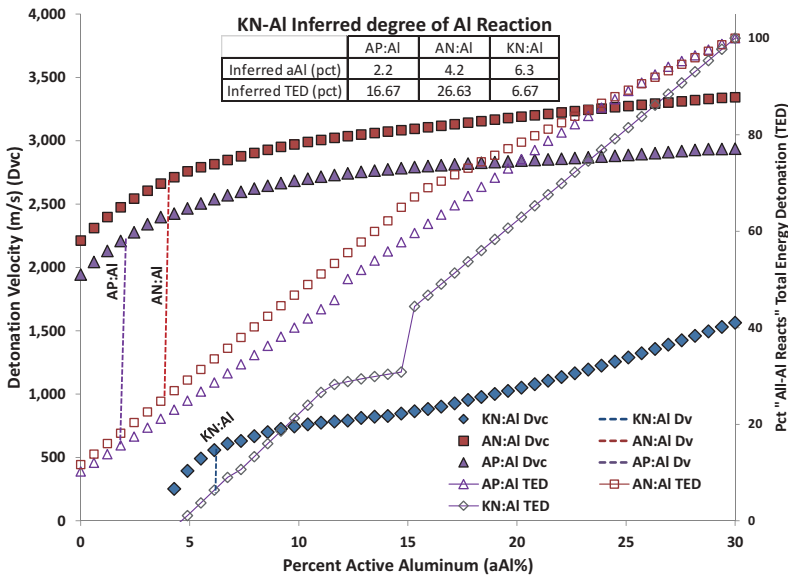
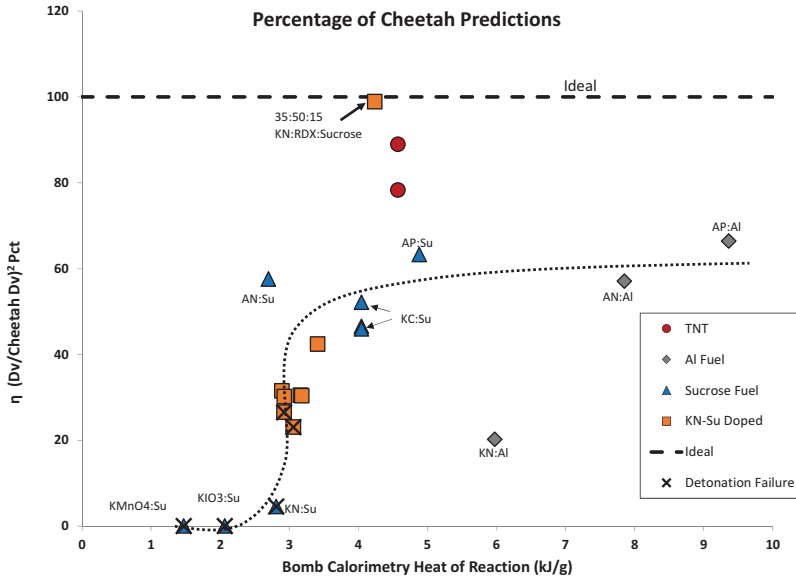
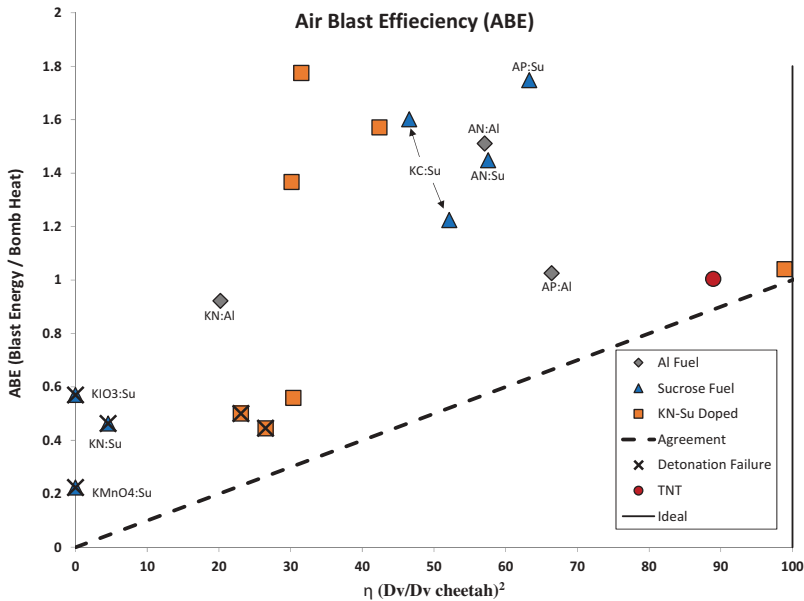


Figure 17. Cheetah calculated Dv and TED based on fraction of aluminum active in AP:Al, AN:Al, & KN:Al. Intersection of Cheetah Dv trend with experimental Dv infers active Al (x-axis); the percent all aAl TED is inferred by intersection of TED trend (hollow markers) with active Al.

The substitution of aluminum for sucrose dramatically increased the energy released in the calorimeter and the inferred air blast energy. The detonation velocity decreased significantly with



**Figure 18.** Ideality of fuel-oxidizer mixtures inferred from Cheetah calculations and detonation measurements. Most mixtures release between 50–60% of predicted detonation energy (Cheetah). Threshold mixtures which eventually failed or barely remained stable inferred <25% of total energy release in the reaction zone.



**Figure 19.** Reaction zone efficiency ( $\eta$ ) vs air blast efficiency (ABE).

aluminum fuel but to about the same degree as predicted by Cheetah. Only a small fraction of the energy released by aluminum oxidation supports the detonation front (Cooper 1994); it was determined by Cheetah analysis that less than 7, 5, and 3% of the included 30% aluminum fuel reacted in the reaction zone of KN:Al, AN:Al, and AP:Al, respectively, which represents approximately 7, 27, and 17% of their respective “all-aluminum-reacts” ideal energy release. Judging by the pressure rise rate in the bomb calorimeter, KN:Al may have consumed more aluminum because its slower reaction kinetics

allowed time for more aluminum to contribute energy in the reaction zone than the faster reacting ammonium nitrate or perchlorate. The mixture (KNO<sub>3</sub>:Al) did not detonate in the traditional sense; rather it transitioned to a fast burn. The rate recorded in Table 1 was measured prior to the burn, but the burn velocity was marginally faster (0.70 km/s), as judged by video record (Figure 8(b)).

The potassium nitrate-sucrose mixture was prodded into detonation by spiking it with 3 wt% RDX or 4 wt% potassium chlorate. Both chemicals added trivial amounts of chemical energy but gave the mixtures access to much larger percentages of their reaction heat in the reaction zone. KN:Su indicated only 4.6% of its available 2.8 kJ/g inside the reaction zone, but the 3% RDX and 4% KC-doped mixtures appeared to release 30.9 and 30.5% of their available energy in the reaction zone- a significant increase in ideality. Other steadily detonating fuel-oxidizer mixtures appear to have access to 50–60% of their energy in the reaction zone.

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