



Ammonium Nitrate Security Program Technical Assessments

Sandia National Laboratories

Karmen Lappo, James Phelan, Jason Phillips, Nathan Peterson, John Parmeter, Madia Stein,
Don Gilbert, and Allen Gorby

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Name/Org: Karmen Lappo, Org. 5436 Date: 02/17/2015

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EXECUTIVE SUMMARY

In 2008, Congress amended the Homeland Security Act of 2002 and directed the Department of Homeland Security (DHS) to regulate the sale and transfer of ammonium nitrate in order to prevent its misuse in an act of terrorism. In response to this direction, the DHS created the Ammonium Nitrate Security Program (ANSP), which seeks to reduce the likelihood of a terrorist attack involving ammonium nitrate by creating a registration program for purchasers and sellers of ammonium nitrate. The DHS/ANSP proposed a regulation that includes a trigger for regulatory actions of the purchase of a threshold quantity of a material with a threshold level of ammonium nitrate in a mixture. While an abundance of technical data exists on the detonability of ammonium nitrate quantities and mixtures, aggregation of historic data sets could not eliminate all data gaps, indicating additional test data were needed for DHS/ANSP to make more informed regulatory threshold decisions.

Sandia National Laboratories performed a literature review that identified six key areas in need of technical assessments:

- · Effect of total mass on detonability
- · Effect of physical form on detonability
- · Effect of dilution on AN mixture detonability
- Effect of inorganic powder mesh size on detonability
- · Ease of AN-based fertilizer product weaponization
- Methods to assess the presence and quantity of ammonium nitrate in mixtures

Technical assessments were performed by Sandia National Laboratories with carefully controlled parametric test campaigns to determine the effects of total mass, physical form, and dilution on the detonability of ammonium nitrate based mixtures. The Federal Bureau of Investigation (FBI) sponsored technical assessments performed by Rocky Mountain Scientific Laboratory on the effect of powder type and size on detonability and those results are summarized herein. Sandia National Laboratories evaluated one fertilizer product for ease of weaponization to complement the results from previous assessments.

The parametric test campaigns did not exhaustively test all combinations under all possible test conditions, but selected those materials/conditions realistic to terrorism bomb design and/or favorable to support detonation as an appropriate and conservative measure to determine thresholds. The materials selected included:

- Ammonium nitrate—fertilizer grade and a fertilizer mixture (calcium ammonium nitrate)
- Fuels—LH , inorganic powder, and SCSH
- · Diluents-ammonium sulfate and dolomite

Fertilizer grade ammonium nitrate was selected for test formulations as a representative bulk fertilizer that could be diverted to explosive formulation and similar to material obtained from extraction of ammonium nitrate from fertilizer mixtures. One commercial ammonium nitrate based fertilizer mixture product was selected, CAN-27, as this material represents a bulk compound fertilizer that can also be

diverted to explosive formulation as well as processed to yield higher-concentration ammonium nitrate remains. While many other ammonium nitrate materials and mixtures exist, the materials selected were representative of ammonium nitrate materials that could be diverted to terrorist use.

Historic uses of ammonium nitrate/fuel explosives include three main types of fuels: liquid or solid

hydrocarbons, energetic hydrocarbons, and inorganic powder. LH was selected as the most
representative material for the liquid/solid hydrocarbons and SCSH for A Second Family of
hydrocarbons due to the historic prevalence of both.
inorganic powder explosive fuels create
special safety hazards because these formulations are much more susceptible to initiation by static
discharge. inorganic powder was selected for safety and ease in formulation preparation for
effect of total mass, effect of physical form, and effect of dilution on ammonium nitrate mixture
detonability studies. The FBI-sponsored work compared the detonability of ammonium nitrate with
multiple powders of varied shapes and sizes.
Dolomite (calcium-magnesium carbonate) was selected as a diluent because this material has agronomy value, is inert with regard to explosive chemistry, and has historically been used by the United Kingdom to limit the detonability of ammonium nitrate fuel explosives derived from fertilizer mixtures. Ammonium sulfate was also selected as a diluent because this material has agronomic value, is found fertilizer mixtures claimed to have reduced detonability when compared with pure AN, and is an active fuel with regard to explosive chemistry. An active oxidizer with regards to explosive chemistry (e.g., potassium nitrate), which also has agronomic value, was not selected for evaluation.
The controlled conditions included the following: (1) processing of the
prills or granules, (2) sufficient charge diameters (1997), (3) light confinement
with , (4) use of a booster (
), (5) , (6) use of pour density
, and (7) use of a fuel/oxidizer ratio that is derive
from historical testing.
The effect of total mass on detonability test campaign used diminishing quantities of 25 lbs, 10 lbs, 2lb and 1 lb of each of the two fertilizers with each of the three fuels (24 tests) with two duplicates, two o balanced formulations, and two baseline commercial blasting ammonium nitrate fuel oil materia for a total of 30 tests.
The effect of physical form test campaign performed four tests using the CAN-27 fertilizer mixture with
incremental improvement tactics to assess ease of detonability. The test series began with neat CAN-2
(as produced with no fuel), followed by CAN-27 (as produced) with inorganic powder, then
CAN-27 with inorganic powder, and then hot water processed CAN-27 with inorganic powder.
The effect of dilution on ammonium nitrate mixture detonability test campaign used diminishing
quantities of ammonium nitrate with a single fuel type, inorganic powder
. With
ammonium sulfate diluent, the tests began with 40% ammonium nitrate and decreased to 20%

ammonium nitrate in four tests with the proportions of ammonium nitrate, ammonium sulfate. One test was performed with the proportions of ammonium nitrate, ammonium sulfate, and proportions of ammonium nitrate, ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate and decreased to 10% ammonium nitrate, with the proportions of ammonium nitrate, ammonium nitrate
The effect of inorganic powder form and size test campaign evaluated ammonium nitrate with inorganic powder in various shapes and sizes , with a detonator or booster in a nominal diameter PVC rate stick for a total of eight tests.
The definition of explosive performance varies with the intended use of the information. The DHS/ANSP seeks to mitigate the risk of terrorist acts that can cause catastrophic damage to people, property, and critical infrastructure. As such, this work did not require a rigorous and scientific determination of detonation in each test (i.e., reaction velocity exceeds the sound speed of the material for a test configuration with a length to diameter ratio of). Multiple diagnostics provided evidence to determine whether test trials exhibited the characteristics of a detonation, including time of arrival pins, blast pressure gauges, witness plates, visual observation of post-test unreacted material, and high speed video.
The effect of total mass on detonability test results showed:
For both the ammonium nitrate and calcium ammonium nitrate with H., inorganic powder, or SCSH, the results showed that formulations with one pound of ammonium nitrate would detonate as indicated with multiple diagnostic metrics. Continued reduction of the quantity was not pursued to determine the minimum quantity that would detonate. Minimum detonable quantities for the diluted ammonium nitrate mixtures were not determined in this work.
The effect of dilution on ammonium nitrate mixture detonability test results showed:
Through sequential tests of increasing dilutions, a minimum level of 15% ammonium nitrate diluted with dolomite in a mixture containing inorganic powder detonated as indicated by multiple diagnostic metrics. Similar test series with dilutions of ammonium sulfate showed a minimum detonable level of 25% ammonium nitrate.
The effect of physical form test results showed:
Prilled CAN-27 without fuel and prilled CAN-27 with inorganic powder both showed very low magnitude and decaying reaction velocities. The simple preparation technique of cAN-27 demonstrated that the hydroganic powder as demonstrated by the sustained high velocity reaction

front increased witness plate damage, and substantial plast overpressures measured. The slightly

more difficult	CAN-27 also produced a detonable mixture with inorganic
The effect of form and size of	organic powder test results showed:
Multiple powders of	aried shapes and sizes detonated.

Methods to assess the presence and quantity of ammonium nitrate in mixtures showed:

Straightforward calculations based upon manufacturer mixture specifications provide an adequate basis to assess the ammonium nitrate level in fertilizer products. Specialized chemical analysis techniques using crystallographic analysis confirmed the presence of ammonium nitrate in an ammonium nitrate double salt based product. Publicly available information on the molecular composition of ANDOUBLE Salt used in the calculations showed that it contains 35 to 40% ammonium nitrate. Similar calculations for CAN-26 and CAN-27 showed ammonium nitrate levels of 74% and 77%, respectively.

The results of technical assessments were presented on two occasions to an interagency subject matter expert (SME) panel for review. The SME panel included members from DHS/Infrastructure Security Compliance Division, DHS/Office for Bombing Prevention, DHS/Office of Intelligence and Analysis, DHS/U.S. Secret Service, Department of Justice (DOJ)/Bureau of Alcohol, Tobacco, Firearms and Explosives, DOJ/FBI, Department of Defense (DOD)/Joint Improvised Explosive Device Defeat Organization, DOD/Combating Terrorism Technical Support Office, Office of the Director of National Intelligence/National Counterterrorism Center, and the Department of State. This technical panel determined that mixtures containing one pound AN or CAN-27 fertilizer/fertilizer mixture were detonable based on the test diagnostics and that a minimum detonable level of 10% ammonium nitrate by weight can be technically defended by DHS/ANSP, providing a small margin of safety beyond the 15% level, which showed a weak detonation. The panel stressed that the 10% value is solely a technical finding via consensus among the SME panel, and that DHS/ANSP policy decisions should also consider the threat potential and the economic impact on government/industry to determine the regulatory dilution threshold.

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1 BACKGROUND

1.1 Ammonium Nitrate Security Program

In 2008, Congress amended the Homeland Security Act of 2002 and directed the Department of Homeland Security (DHS) to regulate the sale and transfer of ammonium nitrate (AN) in order to prevent its misuse in an act of terrorism. In response to this direction, the DHS created the Ammonium Nitrate Security Program (ANSP), which seeks to reduce the likelihood of a terrorist attack involving AN by creating a registration program for purchasers and sellers of AN, as outlined in the DHS Notice of Proposed Rulemaking (NPRM) ¹. Regulatory actions are triggered for a purchase or transfer of a threshold quantity of a material with a threshold level of AN (for those materials that contain mixtures of AN). Regulatory processes covered in the proposed regulatory program include:

- · Purchaser and seller registration activities
- · Point of sale activities
- · Theft or loss reporting
- · Inspections and audits
- Adjudications and appeals
- Civil penalties

Sandia National Laboratories (SNL) was contracted to provide technical data in support of decision making for the ANSP. Many gaps exist in the historical record of research on the detonability of AN. To fulfill its role, SNL developed a focused test and evaluation program, which is described in detail in this report.

1.2 Ammonium Nitrate

Ammonium nitrate (NH_4NO_3) is a man-made chemical compound.² While technically a salt, it is more commonly referred to as an oxidizer and is classified as such for shipping by the Department of Transportation (DOT). It was first synthesized by Johann R. Glauber in 1659 and patented as an explosive ingredient in 1867.

Today, AN is used in many applications—both licit and illict. It is commonly used in agriculture as a highnitrogen fertilizer. AN has also been used as an oxidizing agent in explosives, including improvised explosive devices (IEDs), and is the main component in many mining and construction explosives. In the medical industry, AN is used to make instant cold packs. Ammonium nitrate is widely used due to its technical and cost effectiveness, and many industries, including the agriculture, mining, and medical industries, would be affected by changes in the regulation of AN.

¹ Department of Homeland Security, http://www.dhs.gov/ammonium-nitrate-security-program-notice-proposed-rulemaking, accessed on 11 Jun 2014.

² Denham, H. "Fertilizer Grade Ammonium Nitrate Detonability Study," The National Assessment Group, 2012.

1.2.1 Legitimate Uses of Ammonium Nitrate

Since World War II, AN has seen increased use as a fertilizer. It has been noted that AN-based fertilizer products are responsible for sustaining from one-third to one-half of the world's population, given its agronomic effectiveness to increase crop yield. Fertilizer is transported in solid or liquid form from its point of origin or place of manufacture. It is often transported by ocean-going tanker, river barge, railroad, over-the-road trucks, or moved through pipelines as a liquid or gas. Before arriving at a farm field, a fertilizer will likely have been transported by more than one mode of transportation.

The mining industry uses AN in simple ammonium nitrate fuel oil (ANFO) blends and in emulsions.⁵

ANFO is solid AN blended with fuel oil. Many mines prepare ANFO on site

. An emulsion is an intimate mixture of two immiscible liquids with one liquid phase dispersed uniformly throughout the second phase.⁶ Emulsion explosives are dispersions of water solutions of oxidizers in an oil medium or "water-in-oil" emulsions. It is this unique structure and the high ratio of oxidizer to fuel that give emulsion explosives their special characteristics. The primary difference from ANFO is that emulsions are typically water resistant and of higher bulk density.⁷ The popularity of AN-based blasting explosives is largely attributable to the low cost and high stability of ANFO.⁸

AN sees some use in the medical industry, primarily in first aid applications such as instant cold packs. Instant cold packs consist of two bags: one bag containing water inside a second bag containing AN, calcium ammonium nitrate, or urea. When the inner bag of water is broken by squeezing the package, the water dissolves the solid in an endothermic reaction. This reaction absorbs heat from the surroundings, quickly lowering the pack's temperature. The temperature of the solution falls to about 35 °F for 10 to 15 minutes. Instant cold packs are a convenient direct replacement for crushed ice and are often used for sports injuries.

1.2.2 Terrorist Use of Ammonium Nitrate

Many commonly available precursor chemicals (including AN, sulfur, charcoal, sugar, and urea) can be combined with other substances to produce improvised explosives.¹⁰ Of these, the one most frequently used for making explosive devices is AN, which is readily available in both its pure form and in certain

⁴ The Fertilizer Institute, http://www.tfi.org/introduction-fertilizer/global-dynamics, accessed 14 November 2014.



° Ibid.

⁹ Wikipedia, "Instant Cold Pack," http://en.wikipedia.org/wiki/Instant_cold_pack#cite_note-1.

¹⁰ U.S. Department of Justice, "Preventing the Illegal Use of Ammonium Nitrate," Report to Congress, 1998.

³ Fertilizers Europe, "Developments in EU Fertilizer Regulations," Ammonium Nitrate Nitric Acid Producers Conference, September 2013.

fertilizer blends. AN-based explosives used by terrorists have been historically employed in large quantities, such as in vehicle born improvised explosive devices (VBIEDs). As adversary knowledge and capabilities increase, a growing concern is the use of AN mixed with more energetically favorable fuels in small bomb configurations.

Terrorist attacks involving the use of AN often prompt governments to investigate ways to minimize both the accessibility and effectiveness of AN as an explosive agent. After enduring sustained AN-based bombing campaigns, countries such as Northern Ireland, South Africa, and the United Kingdom have imposed regulatory statutes to limit the public accessibility of pure AN fertilizers. As a result, products such as calcium ammonium nitrate (CAN) have gained market share as high nitrogen fertilizers. While the use of CAN and other fertilizers increases the difficulty in making AN-based detonable explosives, they can still be used by terrorists in explosive mixtures. Unfortunately, adversary groups have demonstrated their persistence by overcoming technical hurdles and finding simple methods to continue their use of dilute AN-based products in bombing attacks.

Following the use of AN in the Oklahoma City bombing, the U.S. Congress mandated studies to render AN inert while maintaining its efficacy for licit use. While many studies have been performed, a method for inerting AN while maintaining its economic and agronomic feasibility has not been discovered. In fact, a review of the historical records shows repeated efforts by government and industry groups to deter the illicit use of AN-based fertilizers through technical product modifications. Research has been conducted since the 1920s on AN-based complex fertilizers believed to have reduced detonability. These efforts have investigated the explosive performance of such products with various diluents. Organizing these research efforts historically reveals that funded efforts occur in response to adversary usage during the respective timeframes. As only a limited set of added diluents and fuels have been studied, several knowledge gaps exist.

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2 LITERATURE REVIEW

Research has been conducted by industry, inventors, and government both within the United States and internationally since the 1920s on rendering AN inert while maintaining its efficacy as a fertilizer. While an abundance of technical data exists on the detonability of AN quantities and mixtures, aggregation of the varied data sets could not eliminate all data gaps, indicating additional test data were needed for DHS/ANSP to make more informed regulatory threshold decisions. The literature review focused on identifying past research that support the ANSP and direct the technical assessments.

An extensive report on the numerous attempts to decrease the detonation potential of AN can be found in the report to the Technical Support Working Group (TSWG)¹² and its associated classified annex. The references that support the ANSP are organized and discussed in Appendix J, LITERATURE REVIEW SUMMARIES, in detail. Discussions include the research approach, results, and efficacy in relation to the objectives.

The sections below describe the general findings from the reviewed literature related to the following technical assessment topics:

- · Effect of total mass on detonability
- · Effect of physical form on detonability
- Effect of dilution on AN mixture detonability
- Effect of inorganic powder type on detonability
- · Ease of AN-based products weaponization
- Methods to determine presence and quantity of AN in mixtures

2.1 Effect of Total Mass on Detonability

While the literature ¹³ revealed many reports of detonability testing on AN and dilute AN-based fertilizer blends, the body of literature did not present a systematic evaluation to determine the minimum quantity of AN-based material required to make a detonable device.

2.2 Effect of Physical Form on Detonability

For improvised explosives employed by terrorists, the physical form can vary broadly with differing starting ingredients, material preparation methods, mixing ratios of oxidizer and fuel, and main charge density. While past research¹⁴ has shown that technical or fertilizer grade AN prills are effective in formulations, dilute AN based fertilizer prills seem to require processing

14 Ibid.

¹³ Several references are summarized in Appendix J. LITERATURE REVIEW SUMMARIES, J.1. Effect of Total Mass on Detonability.

produce a detonable mixture. A direct comparison of a single fertilizer type in various forms

Neat and Physically Modified in a common test configuration was not found.

2.3 Effect of Dilution on Ammonium Nitrate Mixture Detonability

Literature results are abundant in this technical focus area. The use of diluents such as dolomite, AS, and ammonium phosphate is repeatedly discussed. For example, research efforts¹⁵, such as those of Buczkowski, demonstrate the change in explosive performance as dilution is increased while maintaining oxygen balance. This effort documents a sustained detonation in mixtures of AN with dilutions as high as 40% by weight in a small charge diameter. Additional research by Kirk Yeager¹⁶ recorded a sustained detonation at 60% by weight AN dilution in a large charge diameter. While the research is plentiful, knowledge gaps exist in understanding the effects of a progressive dilution comparing AN and AN mixtures in large charge sizes relevant to the current threat environment.

2.4	Effect of Type	
Limited	research ¹⁷ has shown that variation in explosive performance	e occurs in many types of explosive
mixture	s containing various size and shape particles of inorganic	powder as the fuel.

2.5 Ease of Weaponization

Inorganic Powder -

Adversaries have demonstrated resilience in using AN-based fertilizers in terrorist attacks even after physical-chemical and/or regulatory barriers have been placed in their way. One prime example is the modified formulations of CAN-27 used by the Irish Republic Army (IRA). CAN-27 was developed to reduce the fire hazard of AN and was also found to increase the difficulty for terrorist use compared to pure AN. While techniques used to deploy pure AN and bombs are not feasible with CAN-27 simply substituted for AN, the IRA shortly devised oxidizer modifications and alternative fuels that allow the use of CAN-27 in improvised explosive mixtures. Within two years of the product's release to market, they had discovered that by altering the CAN-27 and blending it with a detonation could be achieved. Unfortunately, the proliferation of knowledge has increased due to the internet, and techniques for improvised explosive development are quickly shared among groups and individuals worldwide with malicious intentions.

¹⁵ Several references are summarized in Appendix J. LITERATURE REVIEW SUMMARIES, J.3. Effect of Dilution on AN Mixture Detonability.

¹⁷ Several references are summarized in Appendix J. LITERATURE REVIEW SUMMARIES, J.4. Effect of inorganic Powder Type on Detonability.

¹⁸ IFDC, "Study of Imposing Controls on, or Rendering Inert, Fertilizer Chemicals Used to Manufacture Explosive Materials," Submitted to U.S. Bureau of Alcohol, Tobacco and Firearms, 1997.

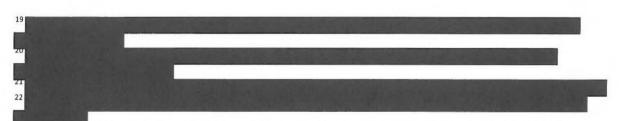
Chemical methods to increase the AN concentration of dilute AN-based commercial products for use in improvised explosives have been documented by the Air Force Research Laboratory (AFRL)¹⁹, Naval Surface Warfare Center Indian Head Division²⁰, Sandia National Laboratories²¹, and the Joint Improvised Explosive Device Defeat Organization, or JIEDDO²². Products such as CAN-27, AN/AS double salts, and a proposed concept of AN/iron sulfate have all been demonstrated to be susceptible to adversary modification through simple chemistry.

2.6 Methods to Assess the Presence and Quantity of Ammonium Nitrate in Mixtures

AN is used in AN-based composite products in both the fertilizer and mining industries and are manufactured via various processes. For example, prilling, granulation, and layering technologies may be used to develop multi-prill blends, homogeneous particle, fattened prills/granules, or double salts. The product's morphology and crystallography can vary depending on the manufacturing technique.

The ANSP defines AN to include "solid ammonium nitrate that is chiefly the ammonium salt of nitric acid and contains not less than 33 percent nitrogen by weight."²³ In addition, the DHS proposed to define AN "to include any mixture that is 30 percent or more ammonium nitrate by weight."²⁴

Several mixtures of AN have been developed for agricultural purposes and also intend to prevent their use in acts of terrorism. These mixtures do not meet the strict definition of AN in the amended Homeland Security Act (Subtitle J—Secure Handling of Ammonium Nitrate). However, opinions differ on whether these materials meet the criteria for the mixture rule. Common chemical calculations based on elemental composition can estimate the amount of AN in a mixture. More advanced analytical chemical techniques, such as x-ray crystallography, can determine the presence of AN in complex mixtures (e.g., double salts).



²³ 6 U.S.C. 488(1)(A). DHS proposes to use this definition of solid ammonium nitrate in section 31.105 of the proposed rule (FR Vol. 76, No. 149, Aug 3, 2011, Section III. A., Ammonium Nitrate Security Program, proposed rule – Discussion of Proposed Rule: Implementing Subtitle J).

²⁴ FR Vol. 76, No. 149, Aug 3, 2011, Section III. A.1., Ammonium Nitrate Security Program proposed rule – Discussion of Proposed Rule: Implementing Subtitle J, Mixture Requirement.

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3 TECHNICAL ASSESSMENTS

3.1 Materials

Additional details and images of materials used can be found in Appendix A, MATERIALS.

Table 1 provides a list of the materials used in the SNL test activities. Additional details and images of materials used can be found in Appendix A, MATERIALS.

Material Source

Ammonium Nitrate

CAN-27 (Calcium Ammonium Nitrate)

Ammonium Sulfate

Dolomite

AN Double Sall

SCSH

LH

inorganic Powder

Table 1: Materials used in Explosive Testing

3.2 Small-Scale Safety Testing (SSST)

When new explosives characterization is performed, it is imperative to understand the behavior of the explosives being prepared. It is common practice within the explosives industry and required at SNL to start with small quantity batches (e.g., 10 g) and evaluate the response of mg quantities to various energy stimuli. The mixtures are each exposed to friction, impact, electrostatic discharge, and thermal stimuli to evaluate sensitivity. After comparing test results to similar test results of a known reference explosive (pentaerythritol tetranitrate, PETN), the SNL Explosive Development Committee (EDC) safety committee reviews the data and concurs that larger batch sizes are safe to prepare. Two phases of SSST (Phase I–10 g batch size and Phase II–500 g batch size) were sequentially completed before obtaining final concurrence to produce large batches. A thorough description of the test apparatus, procedures, and results are provided in Appendix B, SMALL-SCALE SAFETY TEST (SSST) DATA. Completion of the scale-up process through safety committee review provides safety oversight to ensure the safety of the test personnel conducting work on the desired quantities for this test series.

The test results did not present high sensitivity to heat, friction, or impact. The mixtures containing powder showed sensitivity to electrostatic discharge (ESD). The same sensitivity was observed with powders by themselves. ESD is not believed to present a detonation hazard, but it was recognized that a flash hazard is possible. For this reason, all mixtures were categorized as 1.1D (Secondary Mass Detonating Explosives), and mixtures containing powder were handled as ESD sensitive.

3.3 Effect of Total Mass on Detonability

The goal of this test series was to determine the minimum quantity of AN-based fertilizers needed to create a detonable mixture when and combined with commonly available fuels. A test matrix was devised to incrementally decrease the quantity of fertilizer used until the mixture fails to sustain a detonation. Each quantity of fertilizer was mixed with one of the following fuels: Here, or SCSH. Testing of each fertilizer began by using 25 lbs and then decreased to 0.75–1.0 lb depending on the mixture.
The parameters chosen for this series were based on subject matter expert (SME) input, current known adversary manufacturing methods, and oxygen balance ratio calculations. The oxidizer/fuel mix ratio was calculated to obtain mix ratio mix ratios as the starting point for each blend. The mix ratio was then adjusted to make slightly unbalanced mixtures in some cases.
Initiation sensitivity and detonability are known to particle size in AN-based oxidizer powder explosive mixtures. For this reason, it was advantageous to use a particle size to most efficiently promote detonability in prepared blends; however, particles can present an inhalation hazard to workers. For this portion of testing, the quantities were relatively small, and safety risks were mitigated by use of remote mixing.
An optimal test configuration to evaluate detonable mass as the net explosive weight (NEW) is decreased is (which maximizes the). However, charges can be difficult to instrument for reaction front velocity propagating through the explosive. Since velocity is a primary indicator of reaction behavior, a short (length to diameter ratio = 3) cylindrical column test geometry was chosen. This configuration offered the ability to maintain a sufficient diameter to mass ratio and locations to instrument for velocity measurements of the propagating reaction front. The charge configuration utilized a centrally initiated, cylindrical, booster and maintained a constant length to diameter ratio. In addition to reaction front velocity instrumentation, each charge was instrumented to capture far field blast pressure and a thin witness plate was placed under the charge as a metric to determine reaction violence. High speed video was placed to observe the far-field blast and air shock wave.
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3.3.2 Mixture Preparation

Grinding Operations: If received in prill or granular form, oxidizer materials were to a powder form. The stone mill shown in Figure 1 was used to dolomite, and AS.

The selected dolomite adjustment will change material throughput. The stone separation increased or decreased with the stone adjustment knob.

Difference material properties were characterized to provide consistent material for testing and to accurately reproduce material properties. Particle size distributions for various stone gaps are shown in Figure 1. The particle size analyses for materials used can be found in Appendix A. MATERIALS.

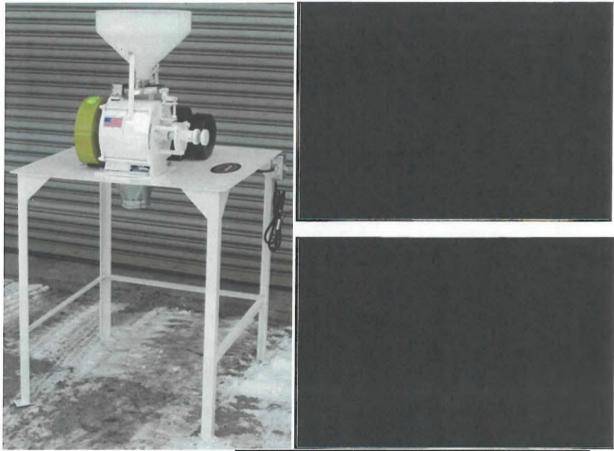


Figure 1. (Left) stone mill grinder used for material preparation

The gap between grinding stones can be adjusted from having the stones just touch to wide enough to pass prills without grinding. The fine reference in Appendix A, MATERIALS, represents the possible. The other setting plus the indicated screw adjustment.

ESD-sensitive Powder Mixtures: All of the powder mixtures presented ESD initiation thresholds below that of PETN during SSST. It is believed that these results are primarily due to the inherent ESD sensitivity of the powder. ESD testing of the neat powder resulted in sensitivities equal to or below that of the mixture. Additionally, photographs of positive reactions taken during ESD testing display brilliant white flashes/sparks, but at the same time there was little evidence of propagation or consumption of the sample material. Mixtures of AN and variously sized powders (ANP) also do not have a history of handling incidents because of ESD. To mitigate a potential flash hazard, these blends were prepared remotely with a v-shell blender. Using this method, oxidizer/additive and fuel can be added to separate compartments while the blender is in the horizontal position without making contact. Once the blender chambers are closed and secured, the operator can retreat from the explosive hazard zone and commence automated blending and charge container filling. Figure 2 shows the remote mixer and constituent placement. The charge booster was remotely placed by a robot after the charge container was filled, as shown in Figure 3.

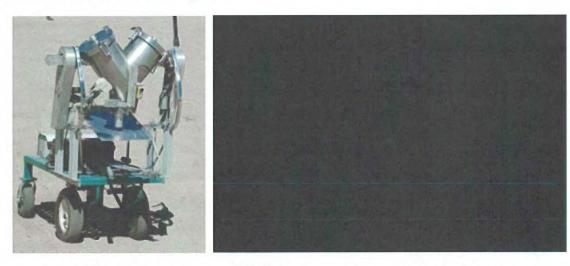


Figure 2: powder (near field) and processed AN (far field) in the v-shell blender before mixing.

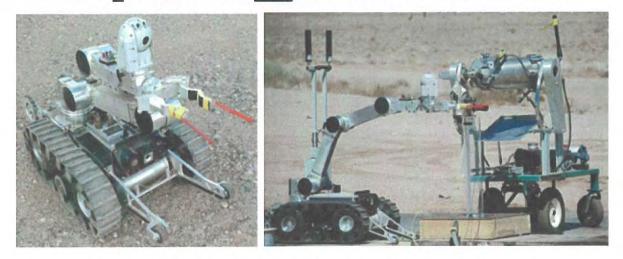


Figure 3: ANDROS robot used to place boosters in charge column.

Powder Liquid Mixture Preparation: Approval was obtained to mix all powder/liquid blends by hand or in a rotary cement mixer. After the mixture constituents were weighed (Figure 4), they were poured into a bowl or rotary mixer (Figure 5) and blended until a homogeneous blend was achieved.

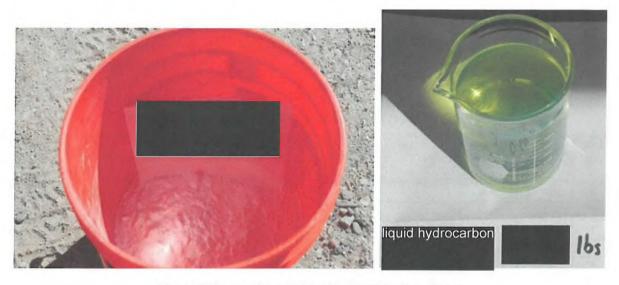


Figure 4: Measured constituents for shot 3 before mixing.

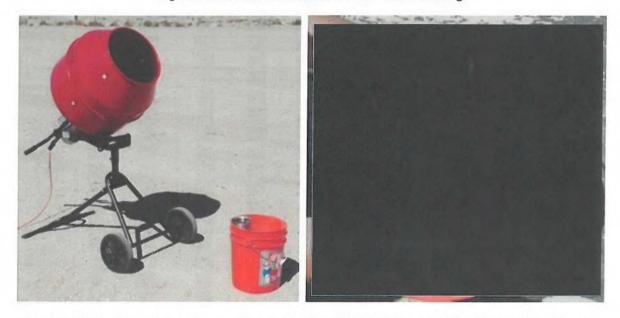


Figure 5: (Left) rotary cement mixer used to blend powder/liquid mixtures and (right) hand mixing process.

3.3.3 Test Matrix

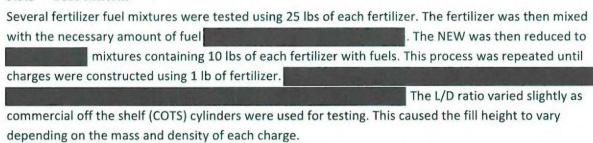


Table 2: Effect of Total Mass on Detonability Test Matrix

Shot No	Fertilizer Type	Fertilizer Mass (lbs)	Fuel Type	Fuel Mass (lbs)	Weight Percent Fuel	Charge Dia. (in.)	Booster Mass (g)	Mixture Density (g/cc)	NEW (lb)	Containment Material
1	ANFO	23.51								Acrylic
2	ANFO	23.51							0.3	Acrylic
3	FGAN	10.00	LH		0.09					Acrylic
4	FGAN	25.00	LH						TO	Acrylic
5	FGAN	2.00	LH							Acrylic
6	FGAN	2.00	LH						81	Acrylic
7	FGAN	2.00	SCSH							Polycarbonate
8	FGAN	0.75	SCSH							PVC
9	FGAN	25.00	SCSH	10-18	8.78		7.9		No.	PVC
10	FGAN	0.75	SCSH	50	2,11		500	200		PVC
11	FGAN	1.00	Inorganic Powder			69				Acrylic
12	FGAN	10.00	Inorganic Powder							Acrylic
13	FGAN	25.00	Inorganic Powder			(3)				Acrylic
14	CAN-27	2.00	Inorganic Powder	50%	200		200			Acrylic
15	CAN-27	25.00	Inorganic Powder	1		539		100		Acrylic
16	CAN-27	10.00	Inorganic Powder	8.0			-		100	Acrylic
17	CAN-27	0.75	Inorganic Powder	100		100	30	7.3		Acrylic
18	CAN-27	25.00	LH		100	10.31			200	Acrylic
19	CAN-27	10.00	LH		100					Acrylic
20	CAN-27	2.00	LH							Acrylic
21	CAN-27	2.00	LH							Acrylic
22	CAN-27	2.00	SCSH	99						PVC
23	CAN-27	0.75	SCSH		2.00					PVC
24	CAN-27	0.75	LH	55						Acrylic
25	FGAN	0.75	LH							Acrylic
26	CAN-27	10.00	SCSH							PVC
27	CAN-27	25.00	SCSH	1	250					PVC
28	FGAN	0.75	Inorganic Powder		000					Acrylic
29	FGAN	19.25	Inorganic Powder					16.0		Acrylic
31	FGAN	10.00	SCSH							PVC

3.3.4 Test Setup

Short columns were chosen for this test series. The length to diameter (L/D) ratio was held constant at approximately three. The total charge mass was reduced by decreasing the charge diameter. The total charge mass was determined by the quantity of fertilizer used (e.g., 25 lbs AN). A optimum blend was then formulated and placed in a COTS charge tube achieve an L/D near three.

Several types of instrumentation were used to evaluate the trial results. Piezoelectric pins were inserted through the charge container wall in a vertical line near the end of the charge column to measure shock time of arrival (ToA). The timing pin orientation can be seen in Figure 6. A witness plate was placed on a $2'' \times 4''$ square frame under the charge. The damage observed as on the plate is used to determine the extent of the reaction.

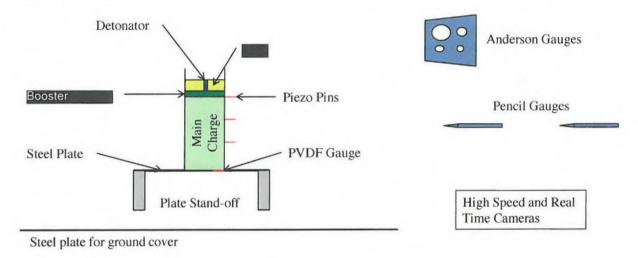


Figure 6: Effect of Total Mass test series setup (side view).

Blast pressure gauge locations were calculated and placed to scaled distance. Duplicate sensor sets were placed at 90-degree angle increments (three gauges per set). In addition, two sensors were placed at 20' from the charge consistently throughout the experiments. Anderson gauges were placed to provide an indication of blast pressure for some tests. Figure 7 provides an overhead perspective of the instrumentation setup with respect to the charge.

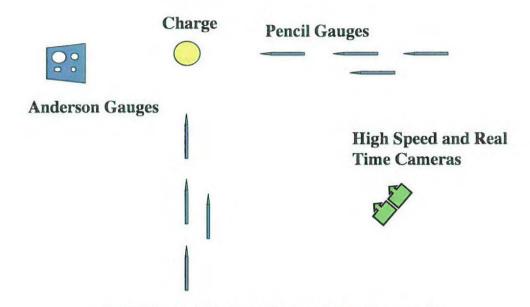


Figure 7: Effect of total mass test series setup (overhead view).

Figure 8 and Figure 9 show the actual setup. The far field pressure gauges are shown, and a close-up image provides a better vantage of the timing pins on the charge.



Figure 8: Pressure sensors placed at increasing distances from the charge.



Figure 9: Time of arrival pins are inserted into the containment wall flush with the explosive material.

Figure 10 is a diagram showing how the piezoelectric pins are constructed. As the incoming shock transmits through the crystal at the end of the pin, a conductive signal is sent to an oscilloscope. Piezoelectric pins are advantageous as they provide a prompt (nanosecond rise time) signal.

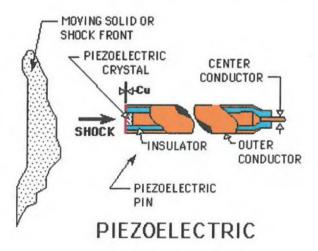


Figure 10: Piezoelectric pin diagram.²⁷

²⁷Dynasen, "Position Transducers," http://dynasen.com/product-category/position-transducers/.

3.3.5 Results

For the oxidizers tested, it was determined that a detonable mixture was achieved using only 1 lb or less with at least one of the fuels. This conclusion was reached by evaluating several metrics. First, a witness plate was placed below the charge to provide an indication of reaction violence. A hole punched through the witness plate, as in Figure 11, indicates a detonation propagated the length of the explosive column. In contrast, an undamaged or dented witness plate (Figure 12) indicates that the detonation was not sustained or a strong deflagration occurred instead of a detonation.



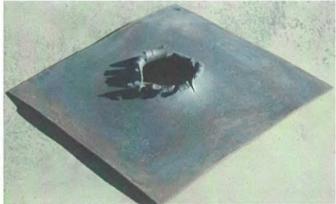


Figure 11: Witness plate indicating a sustained reaction.





Figure 12: Witness plate indicating a failing reaction.

The second metric evaluated is far field air blast pressure. The pressures recorded are compared with data from known explosives tested in the same configuration. Commercial ANFO was tested at 25 lbs in the same test configuration to serve as the series control shot. The pressures at scaled distance, Figure 13, can be evaluated to compare relative performance of various mixtures. Figure 14 provides another metric of comparison. The blast arrival times are plotted for the control shots and mixtures prepared with 25 lbs of each fertilizer with select fuels.

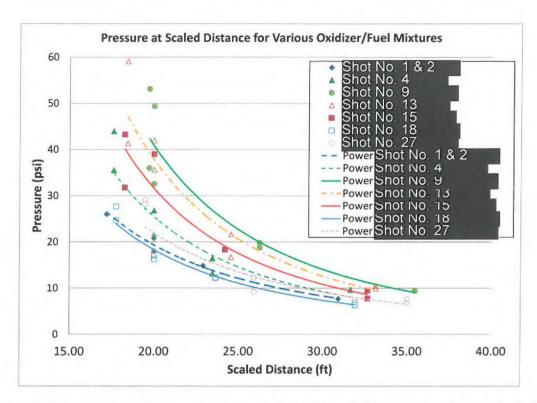


Figure 13: Pressure comparison for explosive charges derived from 25 lbs of various oxidizers mixed with multiple fuels.

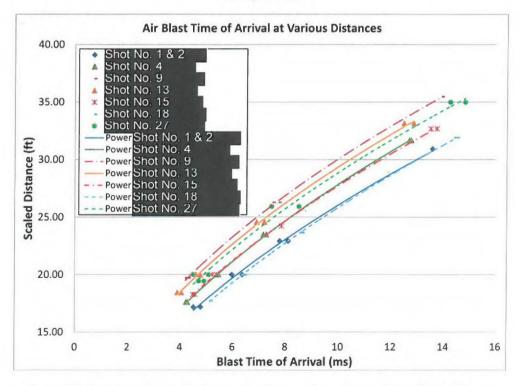


Figure 14: Blast time-of-arrival at increasing distances for various oxidizer/fuel mixtures.

The piezoelectric timing pins generate an electric signal when exposed to shock pressures. The pin placement was carefully measured relative to the charge column configuration. The timing and pin locations can be used to plot shock travel through an explosive column and to calculate inter-pin shock velocities. Figure 15 is a plot of the shock front travel through mixtures derived from 25 lbs of either AN or CAN-27 mixed with fuels. The slope of each trend line indicates the extent of explosive performance. All of the AN-based oxidizers mixed with fuels performed comparably to commercial ANFO.

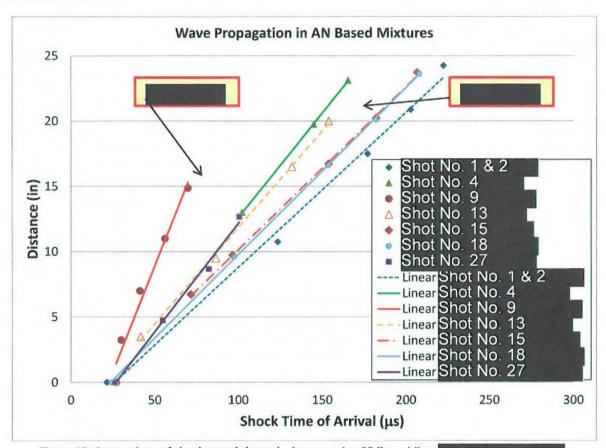


Figure 15: Comparison of shock travel through charges using 25 lbs oxidizer

Figure 16 is a plot of inter-pin shock velocities for select mixtures. Observing the inter-pin shock velocity as it progresses through an explosive charge can provide an indication of a sustained or failing detonation front. In this test series, only was observed to have decaying inter-pin shock velocities at the smallest charge quantities tested.

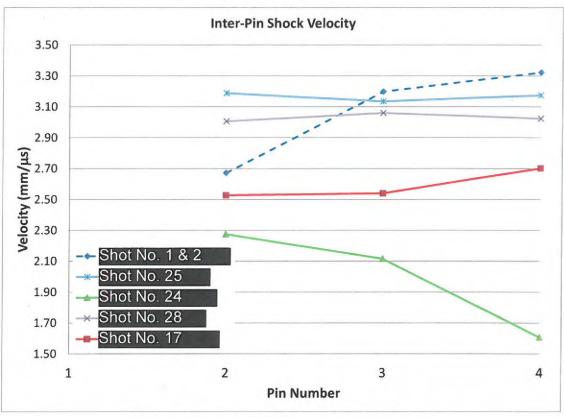


Figure 16: Inter-pin velocity for select explosive mixtures.

High speed video can be used to visually/qualitatively determine the mixture's extent of reaction.

Figure 17 through Figure 24 show a series of images comparing processed. AN with to processed with the same extracted from each video at similar times post initiation. On the left of each figure, the with the is shown, and it can be seen that the reaction fails to sustain for the same extent of time as the AN mixture on the right as time elapses. By 1.05 ms after initiation, the AN mixture is still reacting violently, while the mixture has quenched and is simply dispersing the constituents. This visualization can be used as an evaluation tool to support the determination of a sustained or failed detonation.



Figure 17: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=0 μs.





Figure 18: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=156 μs.





Figure 19: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=312 μs.





Figure 20: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=468 μs.





Figure 21: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=625 μs.





Figure 22: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=1.406 ms.





Figure 23: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=2.187 ms.





Figure 24: 0.8 lb of (left) and FGAN/ (right) respectively at approximately t=2.968 ms.

Table 3 presents the results for the effect of total mass on detonability test series. The table has been organized by fertilizer/fuel combination and decreasing charge mass. The pressures and impulse demonstrate significant energy release for all trials. The velocity and plate dent were evaluated and show that only the charge with 2.04 lbs NEW or less has a decaying reaction front. The Anderson Gauge results are not shown as they were used only for a portion of the test series and did not show conclusive results (e.g., paper torn by debris).

The test results show that all mixtures performed similarly. A "go" is indicated by the combination of reaction velocity greater than ~2.0 km/s and a hole punched through the witness plate. The testing demonstrates that ≥1 lb of AN or CAN-27 can be used to make a mixture that sustains a steady-state reaction throughout the charge length. Tests were not performed for most fertilizer/fuel combinations

containing less than 1 lb of fertilizer. The DHS/ANSP stated that testing below 1 lb of fertilizer was not warranted to inform regulatory decisions.

Table 3: Effect of Total Mass on Detonability Test Results

Shot No	Name	Charge Diameter (in)	NEW (lbs)	Peak Pressure at Scaled Distance (psi)	Impulse (psi-ms)	Average Velocity (km/s)	Witness Plate Result
2	ANFO Commercial				Street,	3.3	Hole - plate in pieces
13	processed ANIP					4.0	Hole - plate in pieces
12	processed ANIP	46.3	02.50	EXACT EX	P. Carlot	3.7	Hole - plate in pieces
29	processed ANIP		EU			3.0	Hole - small petals
11	processed ANIP				7000	3.3	Hole - plate in pieces
28	processed ANIP			Marie Sal		3.0	Hole - no petals
4	processed ANLH	000			MANYA DA	4.1	Hole - plate in pieces
3	processed ANLH					3.9	Hole - large petals
5	processed ANLH		E	E CEST	SUPERIOR DE	4.9	Hole - small petals
6	processed ANLH			203		3.1	Hole - no petals
25	processed ANLH		208	STEEL MAN	BOUND, M	3.2	Hole - no petals
9	processed AN	198		75. No. 30	CONTRACTOR OF THE PARTY OF THE	7.6	Hole - plate in pieces
31	processed AN		100			6.2	Hole - large petals
7	processed ANSCSH		BA			5.7	Hole - small petals
8	processed ANSCSH		12.00		THE RESTRICTION	5.2	Hole - small petals
10	processed ANSCSH		COM		THE RESERVE	4.4	Hole - small petals
15	processed CANIP		42)			3.1	Hole - plate in pieces
16	processed CANIP	188	1200	100000000000000000000000000000000000000		3.0	Hole - large petals
14	processed CANIP			1100000		2.8	Hole - small petals
17	processed CANIP		C.10	1 1 1 1 1 1 1 1	N. Waris	2.6	Hole - no petals
18	processed CANLH			9-0-0		3.2	Hole - plate in pieces
19	processed CANLH					3.0	Hole - large petals
20	processed CANLH		100		Man all	2.4	Hole - no petals
21	processed CANLH				Contract of the second	2.2	Dent only
24	processed CANLH				W 57-51	1.9	Dent only
27	processed CAN SCSH			2/5/6/0	E COLUMN	4.6	Hole - plate in pieces
26	processed CAN SCSH					4.1	Hole - large petals
22	processed CAN SCSH				Mark William	3.5	Hole - small petals
23	processed CANSCSH			St. California	MET SI	3.2	Hole - small petals

3.4 Effect of Physical Form on Detonability

3.4.1 Objective

The objective

The objective of this test series is to demonstrate the variation in explosive performance as a function of formulation preparation. In this test series, CAN-27 was selected as the fertilizer with the following formulations: as received, with fuel, with fuel, and enriched with fuel.

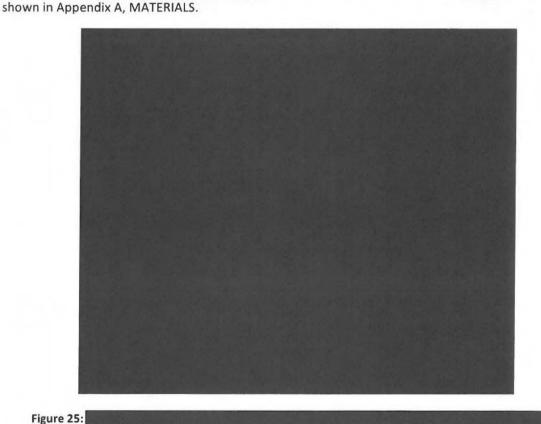
One of the simplest processing techniques is

AN from the fertilizer to remove the non-reactive ingredients to enhance the detonability.

3.4.2 Material Preparation

The explosive mixtures for this test series were processed and mixed.

The particle size distributions for processed materials are



Following processing, the individual chemicals were weighed and separated into batch size sealed bags. The proper number of batches was transported to the mixing facility for later blending. Each batch was blended and tested the following business day.

The ingredients were loaded into buckets shown in Figure 26. Mixture blending was performed using a rotary mixer shown in Figure 27. The left image in Figure 26 shows a single set of ingredients, the bucket with lid, and a filled bag bag in bucket of blended explosive. The filled bags were transported in the five-gallon buckets to the test location.



Figure 26: Weighed ingredients, mixing bucket, stored explosive mixture in bags.



Figure 27: Rotary mixer.

3.4.3 Test Matrix

The following mixtures (Table 4) were prepared for evaluation.

NEW (lbs) Oxidizer Charge Density **Form Fuel** Wt % Diameter (in) (g/cc) ANFO Commercial 1121 rocessed CAN 449 CAN 449 Prill CAN Prill 450 Processed Enriched 447

Table 4: Tests Performed during the Effect of Physical Form Series

3.4.4 Test Setup

The effect of physical form test series was performed at the Energetic Materials Research and Testing Center (EMRTC) affiliated with the New Mexico Institute of Mining and Technology Large Scale Testing Range (LSTR), which is shown in Figure 28 with the infrastructure identified.

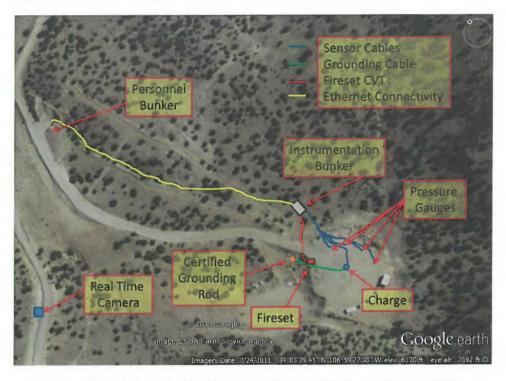


Figure 28: Overview of the EMRTC LSTR test site with infrastructure installed.

An intermediate location within 600' of the charge (between the pad and personnel bunker) was chosen to place high speed video cameras and data collection oscilloscopes. This allowed for high fidelity data capture. A communication connection was set up to the personnel bunker. In each personnel bunker, computers were set up to monitor and control the cameras and scopes from a safe distance.

Figure 29 shows the photo (left) and oscilloscope (right) instrumentation bunkers. The cameras photographed testing through Lexan windows. In addition to several high speed videos, a real time camera was placed at a distance to observe the test event.



Figure 29: Instrumentation bunkers used to house and protect high speed cameras and oscilloscopes.



Figure 30: Cameras installed in instrumentation bunker.



Figure 31: Shimadzu camera in the instrumentation bunker with long lens installed.



Figure 32: Real time camera vantage point.



Figure 33: Scopes in instrumentation bunker.

A diameter tube (cardboard) with plywood bottom was used to contain the charge material. All charge weights were 450 lbs. Piezoelectric pins were inserted through the tube side wall to be flush with the charge material. These gauges provide an electrical signal when exposed to high pressure shock wave. Knowing the separation and detected timing, the shock transit velocity through the material can be calculated. The pins were placed to begin detecting one charge diameter distance from the bottom of the charge and concentrated more intensely near the bottom. Ten pins were used at D, 0.775D, 0.55D, 0.45D, 0.35D, 0.25D, 0.15D, 0.1D, 0.05D and 0 from the bottom of the charge cavity. The pins are shown installed into the side wall of the charge container on the left in Figure 34.

To measure blast, piezoelectric far field blast gauges were placed at 75' and 100' from the charge. Two gauges were place at each distance separated by 90° . A blast gauge is shown in Figure 34, protected by a ricochet pole (which prevents debris from hitting the gauge).





Figure 34: Piezoelectric pins installed in a charge container and pencil gauge placed a distance from the charge.

Two fiducial boards (Figure 35, left) were placed 50' from the left and right of the charge. The blast and explosively projected cloud expansion can be monitored by observing how rapidly they reach the boards. In addition, a checkerboard sticker (Figure 35, right) was adhered to the charge container. As the light from the shockwave broke through the tube, a narrow focal window high speed camera was able to observe the shock front. Knowing the dimensions of the checker pattern, the shock transit velocity can be calculated by monitoring the flame front position with time. This was used as a redundant shock velocity calculation to ensure the metric was captured.



Figure 35: 3' × 3' Photometric fiducial boards were placed 50' left and right of the charge and fiducial sticker on charge container.

Any time norganic powders are involved, electrostatic discharge should be recognized as a hazard.

To mitigate unintentional ignition and flash, ESD engineering controls were installed in the test setup (Figure 36). A conductive tape was installed along the inner charge column wall to allow charge dissipation through a conductive path to a grounding rod.

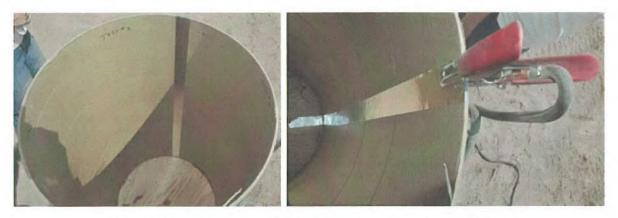


Figure 36: Conductive tape and electrical lead installed in charge container.

Jumper cables were used to connect the conductive tape and the operator to the conductive ground path (thick gauge copper wire). A conductive wrist strap was worn by the operator when handing the explosive material. In addition, cotton clothing was worn to minimize static build up. The jumper cable and ground attachment are shown in Figure 37.



Figure 37: ESD protection (lead is attached to conductive tape, wrist strap and copper wire connected to grounding rod).

Reference height markings were placed on the inside of the charge container wall in three locations around the circumference (Figure 38). The reference height was noted at each location along with a height line. The internal diameter was measured twice (90° offset) as shown in Figure 39. The measurements were averaged for use in density calculations.

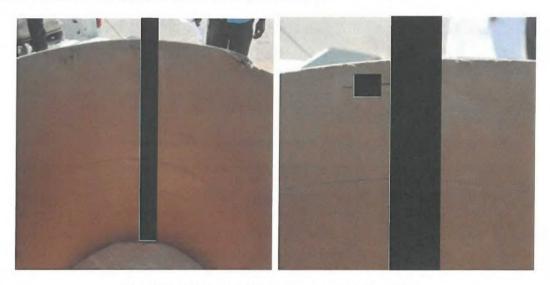


Figure 38: Reference height measurement and markings.



Figure 39: Charge inner diameter measurements.

Once the charge was filled, the explosive was leveled (Figure 40, left). All charges were pour density

. Measurements from the fill surface to each reference height marking were noted (Figure 40, right). An average of these measurements is considered the charge fill height used in bulk density calculations. The final measurement used to calculate bulk charge density is the fill mass, which was determined during charge mixing.

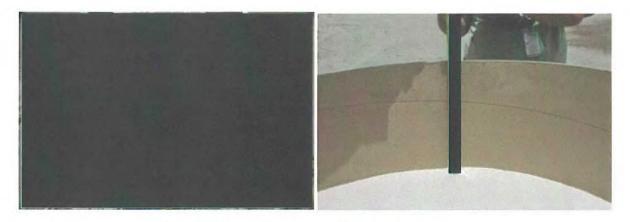


Figure 40: Charge fill leveling and fill height measurement.

3.4.5 Results

This portion of the test series serves as a demonstration that adversaries can employ simple techniques to make dilute AN-based fertilizers useful as explosives. Figure 41 contains a graph that demonstrates how the shock travels through the explosive column. The inter-pin velocity is represented on the vertical axis, and the distance the shock has traveled into the charges is represented on the horizontal axis. The magnitude of the inter-pin velocity is directly proportional to the explosive performance and damage potential. Four tests were conducted to evaluate a dilute AN-based fertilizer as received, when mixed with powder, when processed and mixed with and finally when processed to increase the AN percentage before being processed and mixed with commercial ANFO (~1100 lbs NEW) was tested as a known reference/control material for performance comparison.

The performance enhancement can be seen when is added and especially when the AN-based fertilizer is before mixing with the is. The inter-pin velocities for the as-received prilled product are very low, while those for processed enriched fertilizer mixed with are significantly higher. An additional performance enhancement is documented for the enriched fertilizer, which has been processed to increase the AN concentration. The enrichment process performed is described in Section 3.7.1.

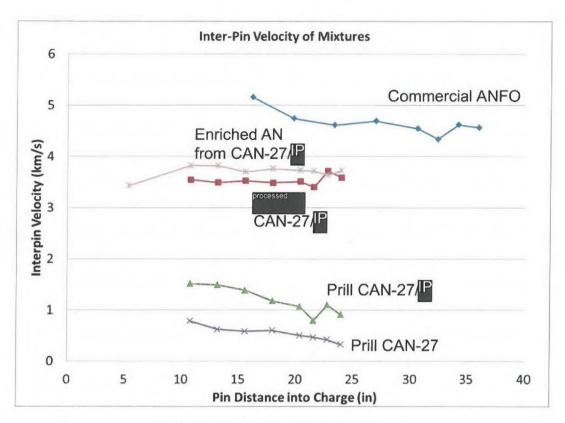


Figure 41: Inter-pin velocity for CAN-27 charges.

1/2"-thick steel witness plates were placed below each charge to evaluate the respective explosive performances. Figure 42 displays each witness plate, which demonstrates each mixture's explosive performance and damage potential.

This indicates that the fertilizer contributed little to the output and that the damage is most likely due to the energy of the booster traveling through the fill material into the plate.



Figure 42: Witness plate results for form comparison testing.

Table 5 summarizes the testing results for the Effects of Physical Form on Detonability test series. The results show that CAN-27 can be combined with powder to create explosive mixtures.

The pressure at 75' from the charge increases from 4.22 \pm 0.29 psi to 5.08 \pm 0.04 psi.

Table 5: Effect of Physical Form on Detonability Pressure and Plate Damage Results

Name	Booster Mass (lbs)	Charge Fill (lbs)	Charge Diameter (in)	Density (g/cc)	Pressure at 75' (psi)	Pressure at 100' (psi)	Witness Plate
Commercial ANFO		1121	99		14.67 ± 1.71	8.24 ± 0.30	Shattered
Enriched CAN		447		1989	8.65 ± 2.60	5.77 ± 0.26	Shattered
Processed CAN/IP		449			7.95 ± 1.19	5.08 ± 0.04	Several Pieces
Prill CAN		449		7240	6.35 ± 0.08	4.22 ± 0.29	Large Dent
Prill CAN		450	074		1.51 ± 0.01	1.01 ± 0.05	Shallow Dent
Sand + Booster		1376			0.68 ± 0.02	0.50 ± 0.02	No Dent

3.5 Effect of Dilution on AN Mixture Detonability

3.5.1 Objective

This portion of testing was performed to evaluate the detonability of mixtures containing decreasing weight percentages of AN. The goal was to determine the lowest weight percentage AN in a fertilizer blend that can be weaponized into an explosive that sustains a detonation. Taking into account cost and schedule considerations, it was determined to prepare simple blends of AN and known fertilizer additives in domestically available products, and to mix these with powder. Two common additives—AS and dolomite—were chosen for evaluation, resulting in AN/AS/P and AN/dolomite/P blends. The weight percentage of AN was reduced by 10% between trials and then refined to 5% increments near the threshold at which a reaction was not sustained. The specific mixture ratios chosen were derived from either historical testing or calculations.

Historical testing indicates that calcium carbonate or dolomite and ammonium sulfate (AS) have been repeatedly evaluated for their effectiveness in reducing detonability in AN-based fertilizer explosive mixtures. In terms of explosive chemistry, dolomite is an inert material (it has neither a surplus nor deficiency of oxygen with regard to explosive chemistry) in contrast with AS, which is considered a fuel (deficient in oxygen with regard to explosive chemistry). The study of two ternary families, AN/AS/ and AN/Dolomite, provide information on the behavior of AN when diluted with chemicals with no fuel value and minor fuel value. A ternary family containing an active oxidizer (excess oxygen with regards to explosive chemistry), such as potassium nitrate, was not evaluated during this series.

The test configuration was derived from several factors, including (1) reasonable threat quantity and size, (2) historical test parameters, (3) sufficient diameter for non-ideal explosives, and (4) the cost and time to produce charge materials for multiple trials.

3.5.2 Material Preparation

The blends were prepared using the same method employed for the Effect of Physical Form test series. Figure 25 to Figure 26 show the processing, weighing, mixing, and storage steps. For this series, simple blends of pre-processed and powdered ingredients were blended to create AN/AS/P and AN/dolomite/P for 1400 lbs net explosive weight charges. The AN/P ratio was constant for each blend; as the AN weight percentage decreases in a given blend, so does the P weight percentage. The AN and AS were purchased in prill and granular forms, respectively, and processed before mixing. The dolomite was ordered pre-processed from the supplier, and all powder used for this series was powder. Commercial ANFO was used to provide a control test for comparison.

Figure 43 shows the individual ingredients pre-measured in preparation for blending.



Figure 43: Chemicals measured for blending.

3.5.3 Test Matrix

Table 6 shows the mixtures that were prepared and tested.

Table 6: Mixtures Prepared and Tested for the Effect of Dilution on AN Mixture Tests

Test No.	Oxidizer	Form	Additive	Dilution	Fuel	wt %	NEW (lbs)	Density (g/cc)
5	AN	processed	AS	40/60	IP	-	EA	
6	AN	processed	AS	40/60	IP			
8	AN	processed	AS	30/70	IP		The same	200
10	AN	processed	AS	25/75	IP	151		
9	AN	processed	AS	20/80	IP		183	
12	AN	processed	Dolomite	60/40	IP	0.50	447	(6.9)
13	AN	processed	Dolomite	50/50	IP		70	200
15	AN	processed	Dolomite	40/60	IP	100	19	
16	AN	processed	Dolomite	30/70	IP		200	0.00
17	AN	processed	Dolomite	25/75	IP			
18	AN	processed	Dolomite	20/80	IP		SUR	
19	AN	processed	Dolomite	15/85	ΙP	800		800
20	AN	processed	Dolomite	10/90	IP			
11	ANFO	Commercial	-	-	FO			
21	-	-	Sand	-	-			

3.5.4 Test Setup

Due to range, personnel, and equipment availability, portions of the testing were completed at the EMRTC LSTR site (Figure 28) and a second location. The secondary location, Multi-Bay Test Facility (MBTF) Range, was used, and the setup is shown in Figure 44. At each range, the instrumentation package was consistent.

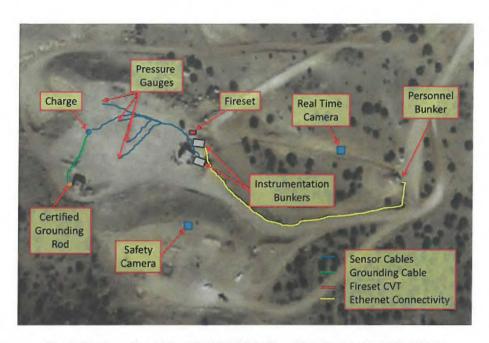


Figure 44: Overview of the EMRTC MBTF site with infrastructure identified.

A vertical cardboard tube was placed at a height of burst. The charge mass was sufficient to obtain a fill height of approximately one charge diameter. A booster, was used to initiate each charge, and . Charges were instrumented to determine the inter-pin reaction front velocity and far field blast pressure. In addition, a thin witness plate was placed under the charge. High speed and real time video were used to capture the test events.

High speed video was used to capture a test overview and the reaction front traveling through the charge. Real time video was used to observe a wide angle view of each test trial. Pressure gauges were placed at 90° offset at two distances (75′ and 100′) from the charge to measure the free field blast pressure. Figure 45 shows a wide angle image of the test pad. The charge container and stand are near the center of the image with a fiducial board to the right. Pencil gauges and ricochet poles are in the right-hand side of the image.



Figure 45: Test setup overview at EMRTC MBTF Site.

Two time-of-arrival measurement mechanisms were employed to detect the reaction front as it traveled through the test material. Piezoelectric and shorting pins were placed along the charge column side wall in parallel lines (separated by approximately 3") along the charge wall and are shown in Figure 46. As the reaction front passes by each pin, an electrical signal is sent to an oscilloscope and later used to calculate inter-pin velocities. A witness plate was placed below each charge. A visual inspection of the plate after a given trial provides an indication of the reaction violence.



Figure 46: Effect of Dilution on AN Mixture test stand and height of burst.

The test charges were slightly different than the Effect of Physical Form charges. The charges were chosen to match historical testing performed in the late 1990s.

Photometric images of the charge are used to track the flame front displacement. The fiducial sticker square size is known, so the light is tracked in reference to the sticker in the calibration image shown on the left below. The flame front location and frame time are used to determine an approximate average inter-frame velocity. The shock behavior over time can be plotted for comparison to other test trials. Figure 47 show a time lapsed image sequence. The flame edge is marked and the software calculates the location.

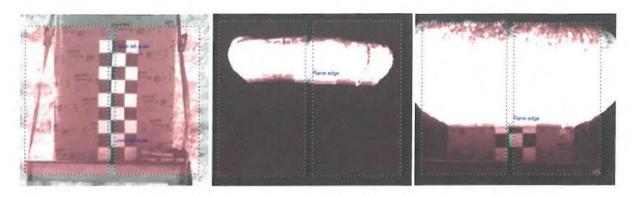


Figure 47: Photometric flame front tracking for velocity calculation.

3.5.5 Results

Data was evaluated from each test trial to determine whether the material detonated. The speed of sound in the materials tested was not measured, so direct assignment of the inter-pin reaction velocity as a detonation velocity is not claimed in comparison to this specific technical metric. Complementary data metrics were also used to evaluate indications of a detonation. The inter-pin velocities (absolute and trend throughout column), far field pressure as compared to the pressure produced by the booster with the same mass of an inert material (sand), high speed video tracking of the expanding blast front, inspection for unconsumed material after each test, and witness plate damage were all considered. After data review, it was apparent that a given trial either exhibited all the signs of a sustained reaction or had several indications of a failing reaction front. Table 7 shows the results of the Effect of Dilution test series.

Table 7: Effect of Dilution Test Series Summary Results

Test No.	Oxidizer	Form	Additive	Dilution	Fuel	wt %	Detonation
5	AN	processed	AS	40/60	IP		No
6	AN	processed	AS	40/60	IP		Yes
8	AN	processed	AS	30/70	IP		Yes
10	AN	processed	AS	25/75	IP		Yes
9	AN	processed	AS	20/80	IP		No
12	AN	processed	Dolomite	60/40	IP		Yes
13	AN	processed	Dolomite	50/50	IP		Yes
15	AN	processed	Dolomite	40/60	IP		Yes
16	AN	processed	Dolomite	30/70	IP	3	Yes
17	AN	processed	Dolomite	25/75	IP		Yes
18	AN	processed	Dolomite	20/80	IP		Yes
19	AN	processed	Dolomite	15/85	IP		Yes
20	AN	processed	Dolomite	10/90	IP		No
11	ANFO	Commercial	-	-	FO		Yes
21	-	-	Sand	-	-	-	

3.5.5.1 AN/AS/P Test Series

For the AN/AS/P test series, piezoelectric pins were used to detect reaction front time of arrival on the charge column. Figure 48 is a graphical representation of the reaction front traveling through the charge column. Starting from the top of the charge column, the velocity between consecutive pins was calculated. The left side of each trace represents the pin near the top of the column, and the witness plate is at on the horizontal axis. Each trace is labeled as a fertilizer mixture that is blended with a respective amount of by weight. The results show sustained inter-pin velocities for three mixtures (40/60 w/P), 30/70 w/P), and 25/75 w/P) above 2.0 km/s. The 40/60 AN/AS blend mixed with P by weight and the 20/80 blend w/P both failed to sustain a reaction and left unconsumed material on the test pad.



Figure 48: Inter-pin velocities for AN/AS/P mixtures tested.

Additional metrics considered in determining the go/no-go criteria included the blast pressure measured at distances from the charge and high speed video blast expansion analysis. In Figure 49, the pressures are graphically displayed for each trial. A comparison can be made to the pressure measured during a trial using a complete sand fill rather than an energetic mixture. It should be noted that even if the mixture failed to sustain a reaction, the pressures observed were well above that of a booster in a complete sand fill. For example, the 58/42 AN/AS/III test resulted in unconsumed material and still produced average pressures of 6.53 psi and 4.53 psi as compared to 0.68 psi and 0.50 psi at 75′ and 100′ respectively for the booster in a sand filled test column.

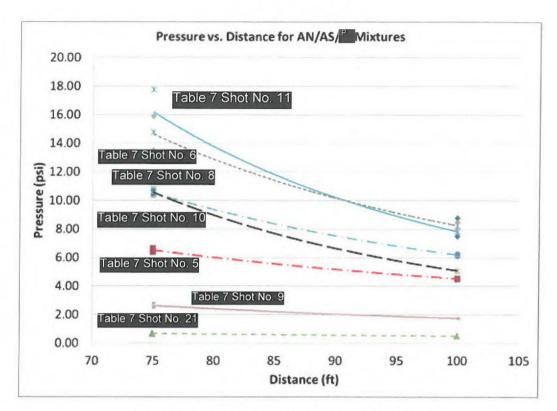


Figure 49: Blast pressure vs. distance for AN/AS/P mixtures.

In the AN/AS/P tests, the lowest AN weight percentage blend resulting in a "go" was the 29.7/70.3 blend. The first "no-go" was observed for the 24/76 blend. This particular ternary blend is interesting in that AS is an active fuel that participates in the reaction. While a mixture of AN/AS or AN/AS/P can be obtained, the ternary ratios tested tend to be fuel rich due to the large percentages of AS with respect to AN. Past testing reported a successful detonation using a fertilizer blend of AN/AS near 40/60 when mixed with weight percent powder. From this starting point, the AN weight percentage was incrementally decreased while maintaining a constant AN/P ratio.

Both calculated explosion enthalpy (ΔH) and oxygen balance (OB) have been identified as key parameters in determining detonability. In this case, the OB ratio can be defined as:

$$OB = \frac{moles \ of \ oxygen \ in \ the \ formulation}{moles \ of \ oxygen \ required \ to \ convert \ all \ Reactants} \ to \ Products$$

Based on this definition, a balanced formulation has an OB value of 1.00, a fuel rich formulation has an OB value > 1.00, and a fuel lean formulation has an OB value < 1.00.

The data in Table 8 indicate that neither the calculated ΔH value nor the OB alone is an accurate predictor of detonability for AS formulations. Considering ΔH first, it can be seen that the 29.7/70.3 formulation detonates with a ΔH of 487 cal/g, while the $\frac{58}{42}$ formulation does not detonate with a ΔH of 611 cal/g. At the same time, the $\frac{24}{76}$ formulation does not detonate with an OB of $\frac{41}{100}$, while the $\frac{100}{100}$ /C formulation $\frac{100}{100}$ 0 does detonate with an OB that is much farther from

Table 8: Results of Studies for Tested // AS/ Formulations and Binary Formulations of ANIP and AN/AS

Formulation /AS	Data Source	Go/No Go	OB Ratio	ΔH, cal/g
48/52	Commercial ASN ²⁹	Go	<1	832
46/54	Present Study	Go	<1	785
35.2/64.8	Present Study	Go	<1	591
29.7/70.3	.7/70.3 Present Study		<1	487
24/76	Present Study	No Go	<1	383
58/42	Present Study	No Go	<1	611
70/30	Kast ³⁰	Go	>1	572
60/40	Kast ³⁰	Go	>1	558
50/50	/50 Kast ³⁰		~1	497
¹⁰⁰ /0	K. Yeager ³¹	Go	>1	954
¹⁰⁰ /0	K. Yeager ³²	Go	<1	1138

Careful inspection of Table 8 reveals that formulations that detonate despite oxygen balances that are far from optimum tend to have high ΔH values, while those that detonate despite relatively low ΔH values tend to have oxygen balances that are closer to optimum. For example, the food /0 formulation detonates despite an OB value of only 1 because it has a high ΔH value of 1138 cal/g. The 29.7/70.3 formulation detonates despite a ΔH value of only 487 cal/g because it has a relatively favorable oxygen balance of 1.

³⁰ Kast, H. "Ueber Explosible Ammonisable." Zeitschrift fur das gesamte Schiess- und Sprengstoff-Wesen, Vol 21, Pg, 205-209, 1926.

³¹ Yeager, K. Phone conversation confirming detonation occurred for AN binary mixtures. ³² *Ibid*.

(see Appendix K, DETONATION

PARAMETER DEVELOPMENT) is

Table 9 provides a list of these formulations ranked by adjusted energy content or detonation parameter. It can be seen that the adjusted energy content is cal/g or higher for the nine formulations that are known to be detonable. These results suggest that this adjusted energy content is, to a good approximation, a useful parameter for predicting detonability of AS/ formulations. An approximate rule of thumb that can be proposed is that an formulation is expected to be detonable if its adjusted energy content is > 340 cal/g, and non-detonable if it is < 340 cal/g. It should be emphasized that such a rule can only be approximate, because other factors such as charge density, particle size, booster size, confinement, etc., will impact detonation performance. Note that the detonation velocity decreases as the adjusted energy content decreases, as would be expected for any valid parameter relating to detonation.

Table 9: Collection of AN/AS, AN/III and ANIII / AS Test Results from Historical and Present Testing

ANIE / AS Formulation	ОВ	ΔH (cal/g)	Go/ No Go	ΔH x OB (fuel-rich) ΔH x [2-OB] (fuel-lean) (cal/g)	Detonation Velocity (km/s)	Detonation Pressure @ 75' (psi)	Detonation Pressure @ 100' (psi)
Commercial ANFO			Go	>340	THE STATE	(AUMPIA)	
48/52 (Commercial ASN)	>1	954	Go	>340			
100 /0 (Yeager)	<1	832	Go	>340			
46/54 (Present Study)	<1	785	Go	>340			E CO
60/40 (Historic)	>1	558	Go	>340			
100 /C (Yeager)	<1	1138	Go	>340			
70/30 (Historic)	>1	572	Go	>340			
50/50 (Historic)	~1	497	Go?	>340			
35.2/64.8 (Present Study)	<1	591	Go	>340			Territor 1
29.7/70.3 (Present Study)	<1	487	Go	>340	1-21-11	6.4.3.9	Charles of
58/42 (Present Study)	<1	611	No Go	<340			
24/76 (Present Study)	<1	383	No Go	<340			Part of the
Booster + Sand			No Go	0		Minister 1	

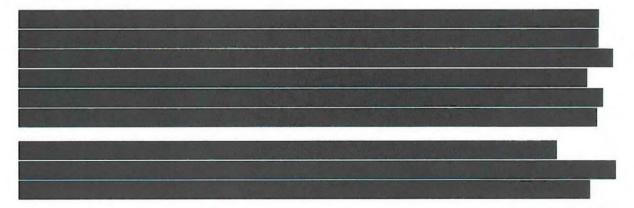
Figure 50 shows witness plates that demonstrate the damage imparted in a given test trial. As a reference, a witness plate damaged by commercial ANFO is provided first. Following are witness plates from mixtures that sustained and failed to sustain a reaction. A noticeable difference is observed when the reaction front fails before reaching the plate. The plate stays intact rather than being punctured or shattered.

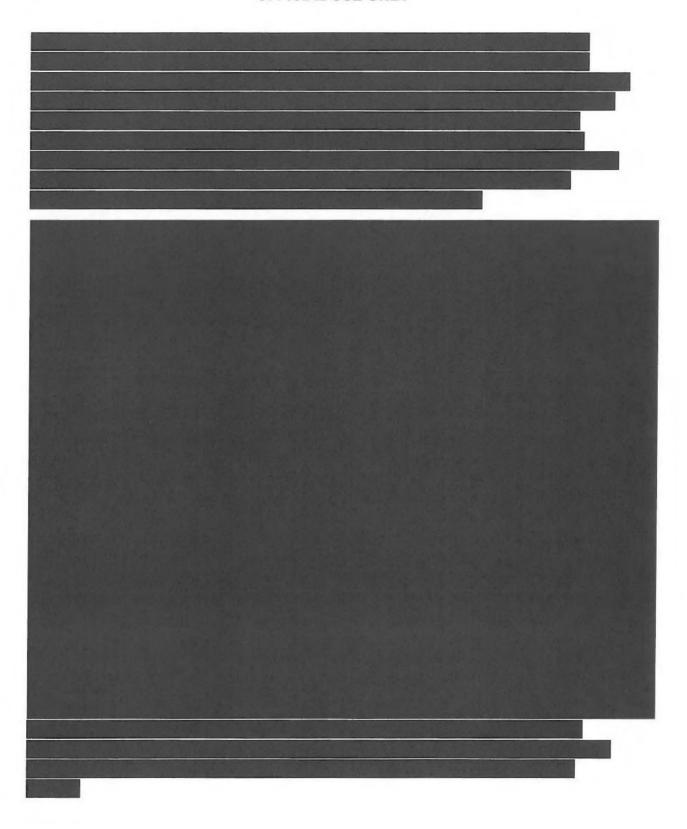




Figure 50: Witness plate damage from (top) commercial ANFO, (lower left) AN/AS (35.2/64.8) w/P and (lower right) AN/AS (24/76) w/P .

Reaction front tracking with high speed video serves as a redundant velocity measurement method. The reaction front location is tracked relative to a fiducial sticker placed on the front of the charge container (see Figure 47). Knowing the inter-frame time, the inter-frame velocities can be calculated. The extracted data from high speed video can be found in Appendix G, PHOTOMETRICS TEST REPORT.





3.5.5.2 ANIP/Dolomite Test Series

Table 10 presents a summary of the dolomite ternary mixtures test results. Commercial ANFO and a booster in sand are provided for comparison at the high and low ends of the table. Other than the commercial ANFO, the table is organized by decreasing detonability parameter.

Table 10: Data Summary for | Dolomite | Ternary Mixtures

/dolomite (wt %)	ОВ	ΔH, cal/g	Result	Pressure 75 ft, psi n=2	Pressure 100 ft, psi n=2	Detonation Velocity km/s n=6	ΔH x OB (if fuel-rich) ΔH x [2-OB] (if fuel-lean) cal/g
Commercial ANFO	1.00	1198	Go	0.00		Fr TRA	
64.76/35.24	1.00	1204	Go				
55.05/44.95	1.00	1023	Go				
45 /55	1.00	835.9	Go		markey.		
34.4 <mark>/65.6</mark>	1.00	638.2	Go				103
29.2/70.8	0.98	537.5	Go	0.00	STORY	544.00	
23.4/76.6	1.00	435.4	Go		CONTRACT OF STREET	DE TON	
17.8/82.2	1.00	333.1	Go		MARIN		100
12/88	1.00	222.9	No Go		519.27		
Sand + booster	N/A	0	No Go		MEST		

Both piezoelectric and shorting pins were used to evaluate the reaction front progression through tested mixtures. Figure 52 is a graphical display of the calculated inter-pin velocities for each data, a distance of into the charge represents the distance at which the steel witness place was located. The points on the left of each curve represent the pins nearest the detonator/booster. The reaction is progressing through the mixture from left to right on each data trend. While the inter-pin velocities become less consistent for mixtures with lower percentages of AN by weight, it is noted that the velocity remains near or above 1.8 km/s throughout the column for mixtures containing 15% AN by weight or more. The 10/90 AN/dolomite blend was the only trial in which a velocity decay occurred and pressures fell below a level for which data could be collected. This is indicated by the lack of data for pins at the end of the charge column.

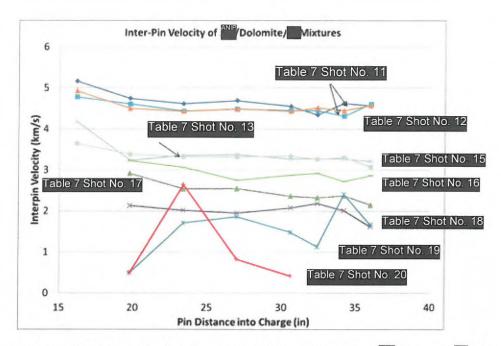


Figure 52: Piezoelectric time of arrival pin calculated inter-pin velocities in //dolomite/mixtures.

Considering the importance of observing the reaction front transit behavior, two methods of time-of-arrival capture were employed on the charge column. Figure 53 displays a comparison of calculated inter-pin velocities from both the piezoelectric and shorting pin data sets. Each test trial has two curves associated with it. The pairs are both color- and symbol-matched. The piezoelectric pins are marked by solid lines and shorting pins by dashed lines. While there are slight variations in each instrumentation method results, the general curve trends for each trial are consistent.

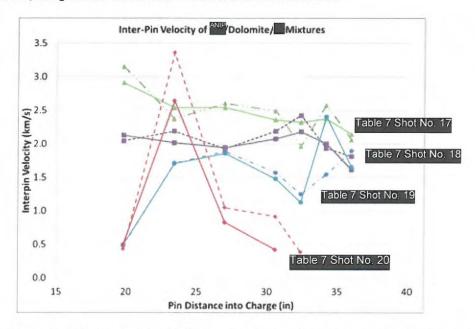


Figure 53: Comparison of piezoelectric and shorting pin calculated inter-pin velocities for low AN percentage mixtures.

The witness plate damage was documented for multiple trials (Figure 54). The results show that a reaction was sustained for the length of the charge column for all mixtures except the 10/90 AN/dolomite blend with . A failing reaction front is indicated by a failure to punch a hole through the plate and unconsumed material (presumed to be original blend that failed to participate) present after a given trial. Significant plate damage in combination with a sustained reaction front, pressures that exceed the pressure produced by a booster alone, and high speed video evidence of rapid reaction product expansion are all factors that exhibit an explosive detonation.

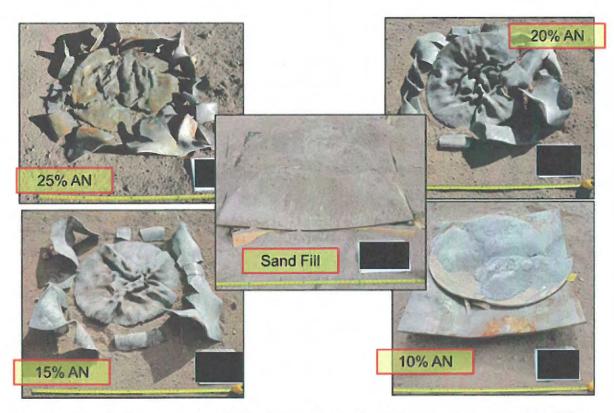
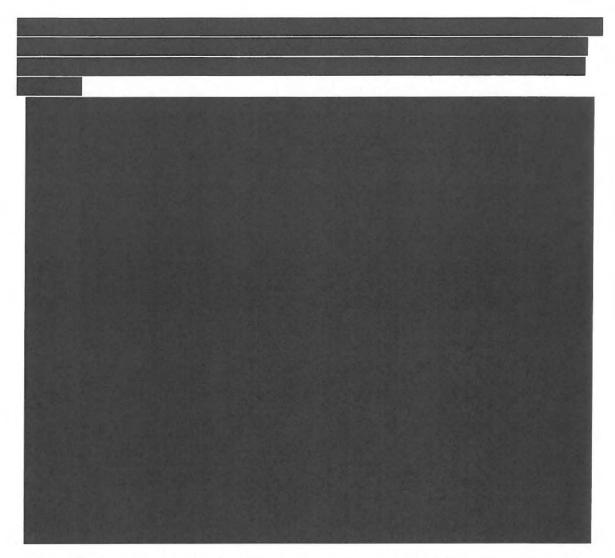


Figure 54: Witness plate damage for AN/dolomite blends containing 10-25% by weight AN mixed with and a sand fill.



High speed video results can be evaluated to compare test results and gain further insight into a given mixture's reaction violence. Presented below is a series of figures comparing two test trials, one that sustained a reaction and one in which the reaction failed and left unconsumed material. The charge is located in the center of each image, and two fiducial boards (black and white checkered plywood panels) can be seen located 50' to the left and right of the charge. The reaction products' expansion can be tracked with respect to the fiducial boards.

In Figure 56, the illumination intensity, shortly after initiation, is drastically higher for the lower image, which is also indicative of a detonation.





Figure 56: High speed video comparison at t = 273 μ s post detonation for (top) $\frac{24}{76}$ $\frac{4}{100}$ AS and (bottom) $\frac{64.76}{35.24}$ $\frac{4}{100}$ ANF/dolomite mixtures respectively.

Figure 57 presents comparison images farther along into the respective reactions. In the upper image, the mixture that failed to sustain a detonation is no longer illuminated, which indicates the reaction has quenched. In contrast, the reaction in the lower image is still progressing, and the reaction products have expanded to a significantly larger volume.





Figure 57: High speed video comparison at t = 5 ms post detonation for (top) 24 /76 ANIE /AS and (bottom) 64.76 /35.24 ANIE /dolomite mixtures respectively.

Figure 58 is a comparison of the same two experiments near the time when the expansion products in the lower image have reached the fiducial boards, 50' to the left and right of the charge placement. In contrast, at the same time interval post detonation, the mixture failing to sustain a reaction shows a product expansion not much larger than the previous time increment. The difference in the reaction violence between the trials is a valuable metric for determining explosive behavior or the lack thereof.





Figure 58: High speed video comparison at t = 10 ms post detonation for (top) $\frac{24}{76}$ AS and (bottom) $\frac{64.76}{35.24}$ AS and (bottom)

Additional detailed high speed video analysis for select individual trials and trial comparisons are provided in Appendix E, EFFECT OF DILUTION ON AN MIXTURE TEST DATA.

3.6 Effect of Inorganic Powder Type on Detonability
Rocky Mountain Scientific Laboratory (RMSL), under contract to the FBI, conducted a detonability study
of AN/IP using various shapes and sizes commonly used in industry. 33 A range of shapes and
particles sizes was chosen for evaluation in mixtures with processed AN.
Optical and scanning electron microscope (SEM) images at various magnifications were taken so the
powder sizes, shapes, and textures could be documented and compared. Figure 59–Figure 64 display th
optical images taken. A full array of SEM powder images can be viewed in Appendix F, EFFECT OF
Inorganic Powder TYPE DATA.

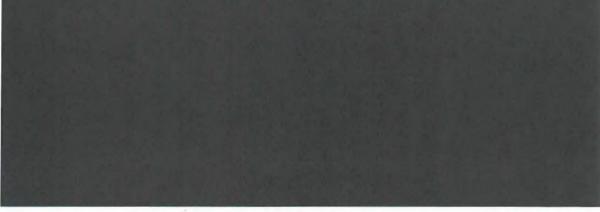
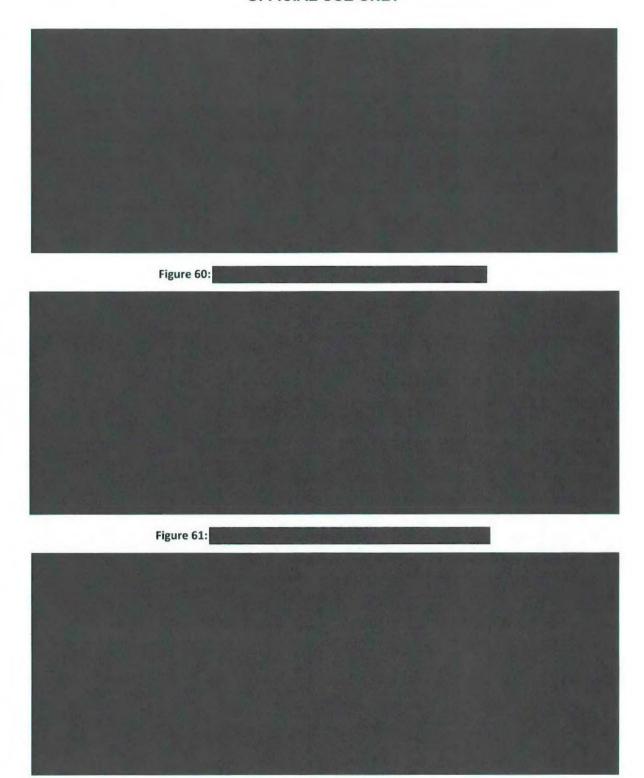
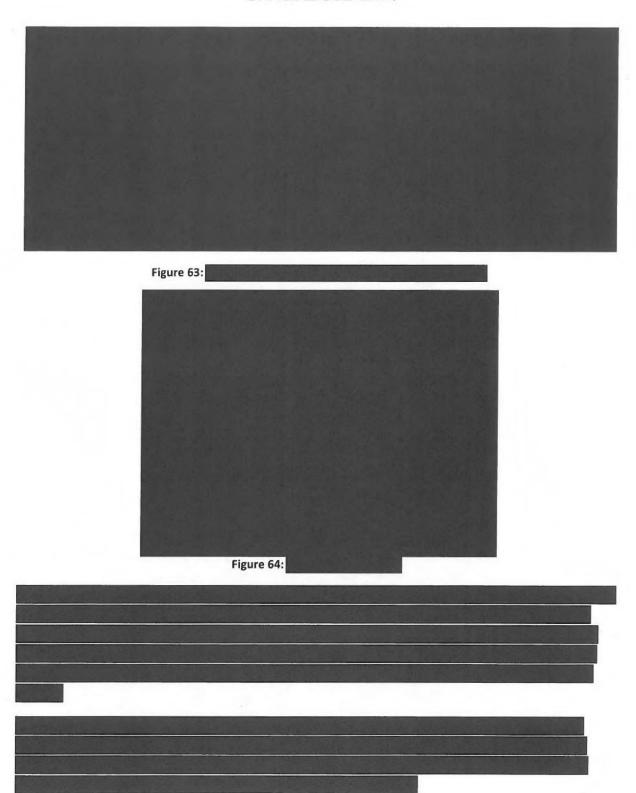


Figure 59:

33



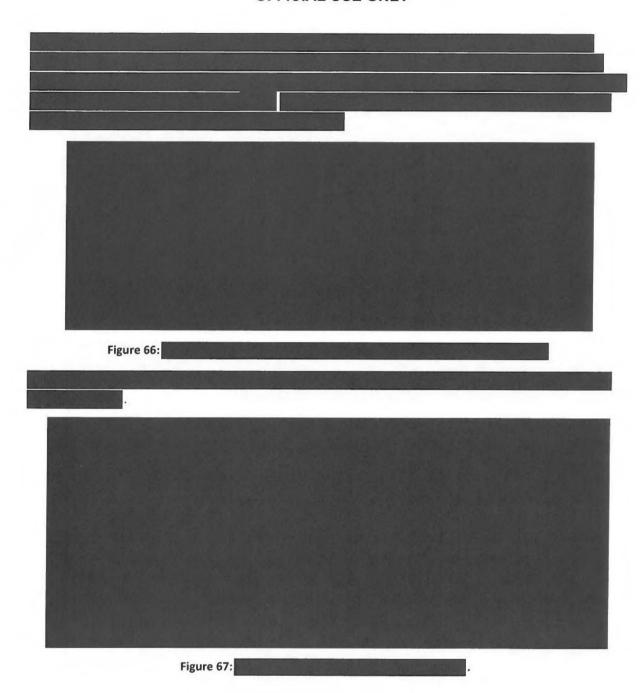


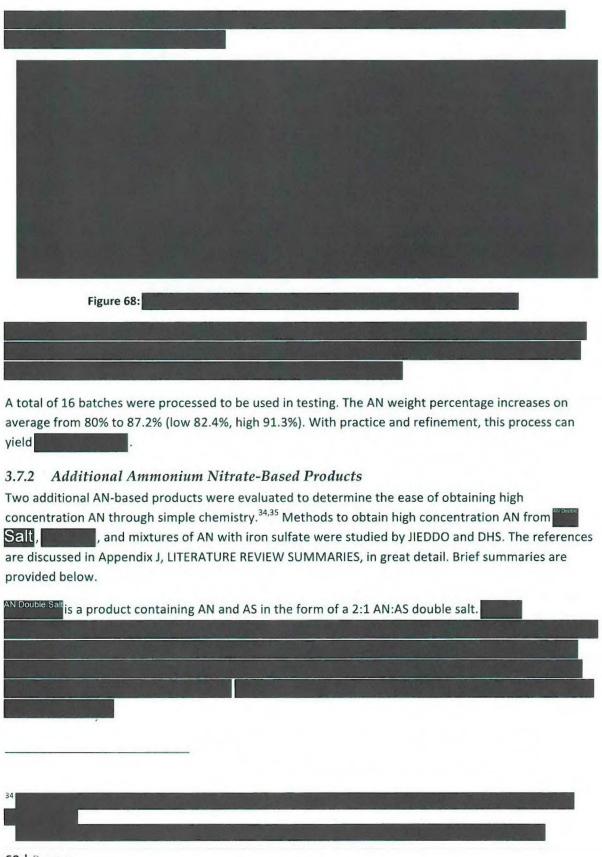
Further details on the testing conducted and associated results can be found in the FBI/RMSL report.

3.7 Ease of Weaponization

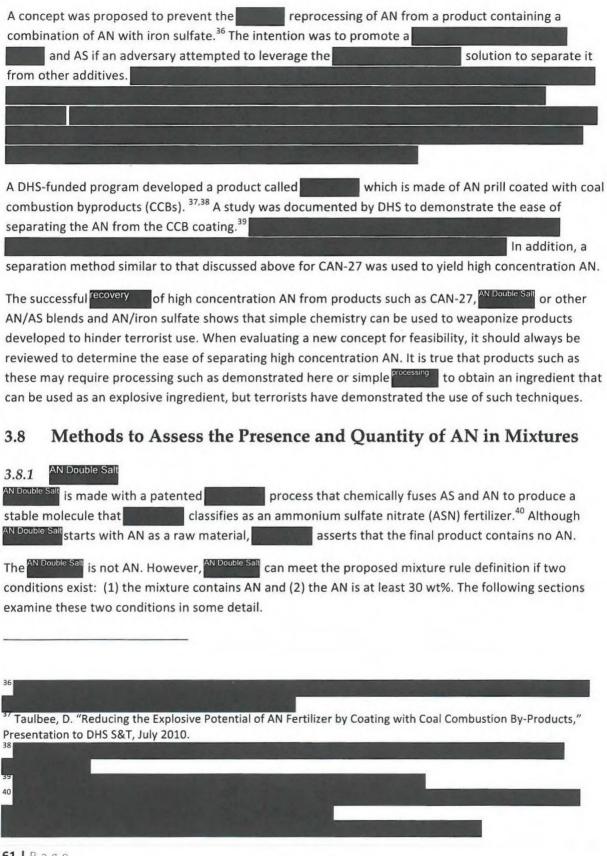
on East of Weaponing		
Typical methods for weaponization	on of AN fertilizers and mixtures are	e mechanical processes
a	nd simple chemistry (e.g.,	Adversaries long ago
realized that AN-based fertilizers	can be processed to	AN. Products such as
calcium ammonium nitrate, or CA	AN, are composed of AN,	
		, separation of the chemicals can be
	nufactured as a homogenous prill/g eight percentages have been proce	granule. Below are two examples of essed.
3.7.1 Calcium Ammonium I	Vitrate	
or calcium carbonate. The weight typically 75–79% by weight AN. Product specifics	percent AN is tailored to yield 27% can be found in Appendix A, MATE	
equipment used to process the C	AN-27.	
	The state of the s	
- 1- 1- 1- 1- 1- 1- 1- 1- 1- 1- 1- 1- 1-		
14 3 4 3 4 3 5		

Figure 65: Equipment used to process CAN-27.





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3.8.1.1 Does AN Double Salt Contain Ammonium Nitrate?

The detailed description of the patented invention of AN Double Salt describes a process to make an ammonium sulfate nitrate *composite* material composed primarily of AS and the double salt 2AN·AS (two ammonium nitrate molecules co-crystalized with one molecule of ammonium sulfate; a 3AN·AS also exists). A composite material is defined as a solid material composed of two or more substances having different physical characteristics and in which each substance *retains its identity* while contributing desirable properties to the whole. Composite material examples include concrete, fiberglass, and wood. By the assertion in the patent and the modern definition of a composite, both 2AN·AS and AN Double Sall contain AN if there are crystals of AN in the composite matrix. Scientific crystallographic examination of the provides evidence for whether AN crystals exist in AN Double Sall or other similar products

Scientific literature reports on the crystal structures of the two double salts of ammonium nitrate and ammonium sulfate (2AN·AS and 3AN·AS) using x-ray diffraction and crystallographic modeling studies on a single crystal of the 2AN·AS salt provided by the company .⁴³ The material has a total nitrogen content (26 wt.%N), ammonical nitrogen content (19.5 wt.% NH₄-N), and nitrate nitrogen content (6.5% NO₃-N) similar to the material.

This work shows that the double salts of ASN include alternating layers of cations (ammonium) and anions (nitrate and sulfate). Figure 69 shows the molecular arrangement of the cations and anions, where the AN and the AS compounds *retain their identity* and are intermixed in layers due to extensive hydrogen bonding between oxygen on the nitrate and sulfate ions (Figure 70). The crystallographic research on 2AN·AS indeed shows that AN and AS are present together in the crystal structure. The fact that AN retains its identity in 2AN·AS means that 2AN·AS contains AN. Since Contains 2AN·AS, contains AN.

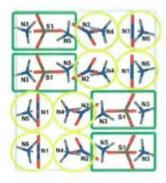


Figure 69: 2AN·AS crystal structure (green = AS; yellow = AN). 2AN·AS salt, perpendicular view to the *ab* face, 293K.

⁴¹ Highsmith, R.E., Kweeder, J.A., & Correale, S.T. (2002). U.S. Patent 0095966A1.

⁴² Meriam-Webster's 11th Collegiate Dictionary, Ver 3.0, 2003.

⁴³ Montejo-Bernardo, J.M., S. Garcia-Granada and A. Fernandez-Gonzalez (2010). "Structures of relevant ammonium salts in fertilizers." *Acta Crysallographica*. Section B, 66, 358-365.

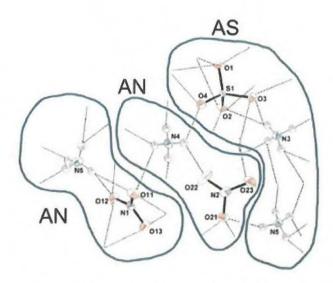


Figure 70: 2AN-AS bond structure

SNL performed similar x-ray crystallographic studies on a sample of ANDouble Sall. The granules were gently ground with agate mortar and pestle to make a randomly orientated sample mount. The sample was analyzed using Bruker 'D8 advance' diffractometer equipped with a Cu ($K\alpha_1 \lambda = 1.54060 \text{ Å}$) x-ray tube. Seven consecutive scans were averaged to produce a high quality, low noise XRD pattern. The detection limit for AN and AS was 0.4 wt. %. The ANDOUBLE Sall diffraction pattern was processed using extreme value analysis (EVA) software package, and quantitative Rietveld refinement was completed following the overall procedure recommended by Montejo-Bernardo, et. al., 2011.

Two crystalline phases were identified in the synthetic mascagnite ($(NH_4)_2SO_4$). Figure 71 shows molecular models of the 2:1 ammonium nitrate sulfate double salt (left) and ammonium sulfate (right). Rietveld refinement was used to quantify the amount of each phase, and the best fit result (Rwp = 15.01 %) is consistent with 70.60 wt. % of 2:1 ammonium nitrate sulfate and 29.41 wt. % of mascagnite.

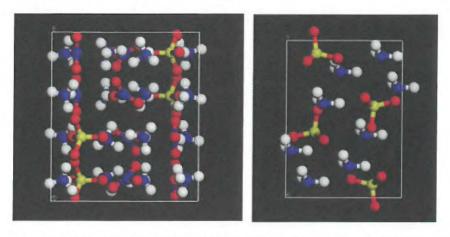


Figure 71. Left - 2:1 salt 2NH₄NO₃(NH₄)₂SO₄. Right - Mascagnite, syn (NH₄)₂SO₄

This work shows that no new molecular structures with covalent bonds are created in 2AN·AS. Ionic bonds between the ammonium cation and the nitrate anion, and the two ammonium cations and the sulfate anion, are necessary to satisfy fundamental chemistry rules for electroneutrality. In the AN ionic bond, the ammonium ion transfers an outer shell electron to the nitrate ion to form a stable electron configuration for the AN salt. This occurs similarly with two ammonium cations and one sulfate anion for the AS salt. Salts are formed through ionic bonds between cations and anions due to electrostatic attraction forces, and the salts form crystal lattice structures as solids. Since there are only NH_4^+ , NO_3^- and $SO_4^{2^-}$ ions in $2AN\cdot AS$, the NH_4^+ must share electrons with NO_3^- , which creates the AN ionic bond. The same occurs between NH_4^+ and $SO_4^{2^-}$ ions, creating AS. The fact that $2AN\cdot AS$ contains both AN and AS means that $2AN\cdot AS$ is a mixture. In fact, NO_3^- is a polycrystalline material containing both $2AN\cdot AS$ and $2AN\cdot AS$ and $2AN\cdot AS$ is a mixture is excess $2AN\cdot AS$ as described by the manufacturing process.

3.8.1.2 How Much AN Is in AN Double Salt?

The patent states that "The composites of the invention are formed by reacting ammonium sulfate with ammonium nitrate in a molar ratio of about 0.9:1 to about 1.1:1 in the presence of a small amount of water in a narrow range of temperatures and then cooling to solidification at a sufficiently rapid rate to prevent macroscopic segregation of the reaction products." A more detailed description of the method states that the steps include: (a) charging materials comprising AS particles, AN, and water to a melting device, wherein the molar ratio of AS to AN is about 0.9:1 to about 1.1:1 and the water is more than 2 wt.% to about 10 wt. % of the charged materials; (b) melting the AN and dissolving at least a portion of the AS particles at a temperature of about 180°C to about 210°C; (c) reacting the charged materials at a temperature of about 180°C to about 210°C; and (d) solidifying the product at a cooling rate of at least about 100°C/min. 45

The AN Double Sall patent claims the AN fuses 46 with the AS, the result being a "double salt" matrix with two AN molecules for each molecule of AS. This fusion is not a chemical reaction creating a new chemical structure. The production process describes melting the AN (mp = 170°C) and adding AS (mp = 235°C); however, not much is known about the solubility of AS in molten AN. The amount of water (2 to 10%) is insufficient to dissolve the AS and in excess to dissolve the AN. Nevertheless, the AN Double Sall is a fusion of AN and AS, containing both 2AN·AS and AS.

fact sheet⁴⁷ on ANDOUDLE Salt states that it is a dry solid fertilizer with an N-P-K-S rating of 26-0-0-14, containing 6.5% NO₃-N, 19.5% NH₄-N (for a total of 26% N) and 14% S. Elemental composition calculations performed herein confirm the fact sheet nitrate nitrogen, ammonical nitrogen and total nitrogen values for both 0.9:1 and 1.1:1 molar ratios (Table 11). In addition, when the %S is calculated as %SO₃ as in the material, the ANDOUBLE Sall has the same sulfur content as the

⁴⁶ Fuse: to become blended or joined by or as if by melting together (Merriam Webster's Dictionary).

Table 11: Weight Percent of N and S in AN Double Sa

0.9:1 AS to AN molar ratio		1.1:1 AS to AN molar rati	
7.04%	%NO ₃ -N in AN	6.21%	%NO ₃ -N in AN
7.04%	%NH ₄ -N in AN	6.21%	%NH ₄ -N in AN
14.08% %N _{tot} in AN		12.43%	%N _{tot} in AN
12.67%	%NH ₄ -N in AS	13.67%	%NH ₄ -N in AS
19.71%	%NH ₄ -N in SN26	19.88%	%NH ₄ -N in SN26
26.75%	%N _{tot} in SN26	26.10%	%N _{tot} in SN26
14.5%	%S	15.6%	%S
36.2%	%SO ₃	39.0%	%SO ₃

Elemental analysis of the 2AN·AS molar ratio shows that it contains 54.8% AN and 45.2% AS and a total nitrogen content of 28.76% (Table 12).

Table 12: Molecular Composition of AN and AS in 2AN-AS

%AN	%AS	% N in 2AN*AS	
54.8%	45.2%	28.76%	

Table 13 shows how much excess AS is in AN Double Sall. This calculation has two constraints, that the molar ratio of 2AN·AS is 2:1 and the total AS:AN ratio is 0.9:1 to 1.1:1. These results show the percent of 2AN·AS is 65 to 73% and the excess AS is 27% to 35%, which is consistent with the patent claim that the invention contains about 14 wt.% to about 35 wt.% AS; about 60 wt.% to 85 wt.% 2AN·AS and about 0 to 5% combined 3AN·AS and AN.

Table 13: Quantity of Extra AS Is in AN Double Salt

0.9:1 AS:AN molar ratio		1.1:1 AS:AN molar ratio	
1	mol AN	1	mol AN
0.5	mol AS	0.5	mol AS
54.8%	% AN	54.8%	% AN
45.2%	% AS	45.2%	% AS
0.4	mol AS	0.6	mol AS
26.6%	% AS	35.2%	% AS
73.4%	3.4% %2AN·AS		%2AN·AS

Lastly, an elemental analysis of the AND bouble sall shows that it contains 35 to 40% AN and 60 to 65% AS, which meets the second condition for the ANSP mixture rule that stipulates inclusion of any material that contains at least 30% AN.

Table 14: Weight Percent of AN and AS in AN Double Sa

0.9:1 AS:AN molar ratio		1.1:1 AS:AN molar rati	
40.2%	% AN	35.5%	% AN
59.8%	% AS	64.6%	% AS

3.8.2 CAN-26 and CAN-27

Calcium ammonium nitrate, or CAN, was developed by fertilizer manufacturers fifty years ago to be a non-detonable alternative to pure AN. However, terrorists and insurgents have found simple methods to process CAN to make an explosive formulation. Since CAN contains AN, this section examines the amount of AN in CAN for regulatory considerations.

CAN is produced by mixing molten concentrated AN solution with various types of calcium carbonate or dolomite. manufactures CAN-26 using pure calcium carbonate (CaCO₃) derived from rock phosphate through the UHDE process. manufactures CAN-27 using dolomite [CaMg(CO₃)₂]. The calcium carbonate remains a solid crystal regardless of the source due to the high melting point of this material (e.g., calcite mp = 1339°C). The number in the CAN nomenclature represents the total nitrogen content of the formulation including both nitrate and ammoniacal (e.g., CAN-27 contains 27% total nitrogen). Table 15 shows the results of calculating the amount of AN in CAN-26 and CAN-27 based on the total nitrogen values. While similar at about a 3:1 mixing ratio of AN to calcium carbonate, the CAN-27 contains slightly greater AN by weight than the CAN-26.

Table 15: Ammonium Nitrate Level in CAN-26 and CAN-27

Material	CAN-26	CAN-27
N (%)	26%	27%
AN (%)	74%	77%
Calcium Carbonate (%)	26%	
Dolomite (%)		23%

4 SUBJECT MATTER EXPERT PANEL REVIEW

4.1 Objectives

Two interagency technical SME panel data reviews were held to review progress, recommend changes to the test plans, and assess the technical data obtained during the Effect of Total Mass, Effect of Physical Form, and Effect of Dilution on Detonability test series. Since the DHS/ANSP proposed rule included a threshold approach to trigger regulatory actions, the DHS/ANSP would benefit from technical assessments that could delineate a distinctive detonation/no-detonation threshold for quantity and level thresholds. However, a distinctive detonation threshold does not occur with non-ideal explosives, such as improvised AN-based explosives. Non-ideal explosives are those formulations that do not instantaneously transition from unreacted material to reacted material, which are more characteristic of specially designed, high performance military explosives. At a detonability threshold, the reaction front can decelerate and sometimes cease completely, leaving unreacted material behind.

These low velocity reactions, or deflagrations, may not scientifically be defined as a detonation. The scientific definition of a detonation is a condition where the chemical reactions proceed at a velocity that is greater than the speed of sound in the unreacted material. These require very detailed assessments of the speed of sound in the unreacted material, which is dependent on the materials in the formulation and the configuration of the test itself. Also, test conditions cannot control for every process that effects detonability, so the test result at or near the threshold is stochastic, where there is an unknown probability that the test will detonate one time and deflagrate another time (for comparison, the low cost of a SSST determines a go/no-go threshold). For this effort, it was neither practical nor necessary to make scientific determinations of detonations or statistically define the detonation threshold. The objective of the technical assessments was to determine the thresholds that would support the DHS/ANSP goal to prevent the misuse of AN in an act of terrorism. Therefore, several metrics were used to determine the reaction violence during testing that shows behavior indicative of a detonation.

Test results presented to the SME panel focused on the following indications of a detonation:

- Stability of reaction front transit rate—sustained velocity or a decaying velocity over time
- Witness plate damage—hole, dent, small fragments, or lack of damage and the distance fragments are thrown from the experimental setup location
- Charge material—whether unconsumed material (other than inert species such as sand or dolomite) remains after experiment
- Blast pressure—magnitude compared to known explosives in similar quantities or to a booster detonated in a charge column of inert material (e.g., sand)

The SME panel decided that test trials distinguished as a "go" showed a sustained velocity through the test column, a hole punched in the witness plate, blast pressures that exceed the pressure observed for a booster in sand (effect of mixture tests only), and no residual oxidizer or fuel. A "no-go" is when the reaction fails to consume all of the charge material and the inter-pin velocity decays, falling below 1 km/s.

4.1.1 Factors Affecting Detonability Thresholds

The SME panel recognized that the SNL testing represents only a small subset of mixtures and test conditions. The following factors were discussed that could affect the technical thresholds determined in the test campaigns.

Fertilizer Form: Testing included simple blends of pre-processed and powdered components/ingredients. Actual products containing co-prilled/granulated products or formulated double salts with the same ingredients and constituent ratios were not used in testing and might show different results. It was not practical to test every fertilizer product.

Fuel Type:	
lf.	or is used in place of powder, the threshold would be expected to
increase, but if S	
	tio: The technical assessments focused on mixtures with the same fertilizer/fuel mixing
ratio.	
	The threshold could decrease if the fuel ratio is adjusted to produce a more fuel
lean mixture.	
Particle Size and	Shape:

Fertilizer Additive: Many fertilizer products containing AN contain additional additives. In terms of explosives chemistry, these additives can be either a fuel, inert, or an oxidizer. This work utilized AS, which is a fuel, and dolomite, which is inert. Other additives that are oxidizers were not investigated (e.g., potassium nitrate). This work showed that for AS, the mixtures became over fueled as the AS weight percent increased. As a result, the explosive performance degraded rapidly for high AS weight percent mixtures. Dolomite is inert and does not participate in the energetic mixture, acting as a simple diluent. The testing demonstrated that dolomite did not cause the AN/P in the blend to arrest until the was 90% by weight in the blend. If an oxidizer such as potassium nitrate, sodium nitrate, or calcium nitrate were evaluated as an additive in place of dolomite or AS, the dilution threshold would likely reduce. The testing performed does not account for the presence of any reactive oxidizers such as nitrate salts. As these would be active participants in the chemical reaction, any blend utilizing them would be anticipated to be more reactive than AN mixed with either inert fillers or fuels.

Ammonium Nitrate-based Explosive Performance: The explosive performance for these types of mixtures can vary from 3–5% in the best of situations (when all variables are kept constant). This means that repeating tests can result in varied output (e.g., the 10/90 AN/dolomite blend with could sustain detonation or the 15/85 blend could fail).

Diameter: When evaluating explosive mixtures, it is imperative for the charge diameter to be above the
effective diameter of the mixture. During testing, a diameter was chosen that is threat reasonable and
matched historical testing. For non-ideal explosives such as these, charge diameter is a dominant factor
in charge performance.
At the
large dilution tested, further testing would be required to determine if the failure to sustain detonation
is solely an effect of the chemical mixture. It is possible that the 10% AN by weight mixture would
sustain detonation if the charge diameter were increased. Testing at larger diameters may
decrease the dilution threshold.
Booster: The booster size can influence whether a mixture sustains a detonation.
A booster can be up to of the charge weight without being
considered to be over-boosted. It is possible that if the booster weight were increased, more dilute
mixtures could be made detonable.
Confinement: Increasing the confinement increases explosive performance.
Charge Density: The mixtures were all tested at poured density
. The density of such mixtures can affect both the initiation sensitivity and performance.

4.2 SME Panel Recommendations

Given all the factors that influence detonation, precise thresholds for variations in AN-based improvised explosive formulations do not exist. The SME panel discussed additional studies to include test replicates, other parametric test variations, and other instrumentation that could improve the determination of thresholds to better inform the DHS/ANSP regulatory decisions but determined that the combined results from the current test campaigns complement the historic literature and are sufficient.

It should be noted that the SME panel was adamant that the technical interpretations of the historic literature and the SNL test results are not DHS/ANSP policy recommendations, rather a technical consensus from the panel. DHS/ANSP decisions on regulatory thresholds should be based on a combination of factors to include, but not limited to, historic and current SNL test results, historic and current trends in terrorist use of AN-based explosives, and the impact to the regulated industries.

4.2.1 Effect of Total Mass on Detonability

The Effect of Total Mass on Detonability test results were reviewed by the SME panel in November 2013. The SME panel determined that all mixtures tested performed comparably. All mixtures tested (listed below) performed equal to or better than commercial ANFO.

ANLH—processed AN with LH
 ANIP—processed AN with D powder
 ANSCSH—processed AN with SCSH
 CANLH—processed CAN-27 with LH
 CANIP—processed CAN-27 with D powder
 CANSCSH—processed CAN-27 with SCSH

Both oxidizers, fertilizer grade AN and CAN-27, could be transformed into detonable formulations with one or more fuels in quantities of 1 lb or more. The SME panel stated that the data supported a 1 lb AN point of sale threshold quantity.

4.2.2 Effect of Physical Form on Detonability

The SME panel confirmed that processing can be used by adversaries to improve the detonability of dilute AN-based fertilizers. The processing technique used to increase the AN purity for various products will vary.

4.2.3 Effect of Dilution on Ammonium Nitrate Mixture Detonability

The SME panel was briefed on the Effect of Dilution on Ammonium Nitrate Mixture Detonability test results in January 2014. The SME panel determined that the minimum AN quantity of 25/75 AN/AS and 15/85 AN/dolomite simple blends mixed with powder both sustained reactions consistent with a detonation. The lowest AN simple blend contained as little as 15% by weight AN. The SME panel concurred that a dilution threshold of 10% AN by weight can be technically defended since the 15% by weight AN did not fail. The SME panel concurred that given the inherent variability in AN-based explosive performance and the limited testing performed, the 10% threshold is defensible to provide a small safety margin.

4.2.4 SME Panel Identified Knowledge Gaps

The following knowledge gaps were discussed by the SME Panel.

- It is unknown if homogeneous materials will perform similarly to the simple blends used in the dilution test series.
- It is unknown how commercial products with low AN weight percentages with additional
 oxidizers will perform in detonation testing. It can be expected that fertilizer products containing
 large percentages of other oxidizers (potassium nitrate) as additives will complement AN to
 promote a detonation.

- It is recognized that there are many factors that affect detonability. A full parametric
 assessment of all of these factors is of scientific interest, but would be prohibitively expensive.
- The effect of total mass for low-weight percentage AN mixtures is not known. It is expected that the effect of total mass for low AN weight percent mixtures will be larger than pure AN.
- It is unknown whether AN/AS mixtures near 40/60 are detonable when processed and boosted.
- The detonability limits of binary AN mixtures to better define the threat space in AN based ternary blends are as yet unknown.

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5 SUMMARY

In 2008, Congress amended the Homeland Security Act of 2002 and directed the DHS to regulate the sale and transfer of AN in order to prevent its misuse in an act of terrorism. In response to this direction, the DHS created the ANSP, which seeks to reduce the likelihood of a terrorist attack involving AN by creating a registration program for purchasers and sellers of AN. The DHS/ANSP proposed a regulation that includes a trigger for regulatory actions by a purchase of a threshold quantity of a material with a threshold level of AN in a mixture. While an abundance of technical data exists on the detonability of AN quantities and mixtures, aggregation of historic data sets could not eliminate all data gaps, indicating additional test data were needed for DHS/ANSP to make more informed regulatory threshold decisions.

SNL performed a literature review that identified five key areas in need of technical assessments:

- · Effect of total mass on detonability
- · Effect of physical form on detonability
- · Effect of dilution on AN mixture detonability
- Effect of powder mesh size on detonability
- Ease of AN-based fertilizer product weaponization
- Methods to determine the presence and quantity of ammonium nitrate in mixtures

Technical assessments were performed by SNL with carefully controlled parametric test campaigns to determine the effects of total mass, physical form, and dilution on the detonability of AN-based mixtures. The FBI sponsored technical assessments on the effect of powder mesh size on detonability which have been summarized in this report. SNL evaluated one fertilizer product for ease of weaponization to complement the results from previous assessments. The parametric test campaigns did not exhaustively test all combinations under all of possible test conditions, but selected those materials and conditions realistic to terrorism bomb design and/or favorable to support detonation as an appropriate and conservative measure to determine thresholds.

The definition of explosive performance varies with the intended use of the information. The DHS/ANSP seeks to mitigate the risk of terrorist acts that can cause catastrophic damage to people, property and critical infrastructure. As such, this work did not require a rigorous and scientific determination of detonation in each test (i.e., reaction velocity exceeds the sound speed of the material for a test configuration with a length to diameter ratio of 8:1). While detonations are more powerful events, violent explosions can also cause significant effects to people, property and infrastructure. Multiple diagnostics provided evidence to determine whether a formulation detonated, including time of arrival pins, blast pressure gauges, witness plates, and high speed video.

The effect of total mass on detonability test results showed:

For both the AN and CAN with H, powder, and SCSH, the results showed that formulations with 1 lb of AN would detonate as indicated with multiple diagnostic metrics. Continued reduction of the quantity was not pursued to determine the minimum quantity that would detonate. Minimum detonable quantities for the diluted ammonium nitrate mixtures were not determined in this work.

The effect of dilution on AN mixture detonability test results showed:

Through sequential tests of increasing dilutions, a minimum level of 15% AN diluted with dolomite in a mixture containing an optimum amount of powder sustained a reaction exhibiting characteristics of a detonation. Similar test series with dilutions of ammonium sulfate showed a minimal detonable level of 25% AN.

The effect of physical form test results showed:

Neat CAN-27 without fuel was not detonab	le, and CAN-27 with 🔯 powder detonated with a
decaying detonation velocity. The simple p	reparation technique of processing CAN-27
demonstrated that processing	prilled CAN-27 fertilizer before combining with
powder transforms the mixture into a de	etonable explosive blend. The slightly more difficult
concentration of CAN-27 before use	in mixtures with powder showed further explosive
performance enhancement over the processed	CAN-27/P mixture.

The effect of form and size of powder test results showed:

Multiple powder	s of varied sizes	and shapes	etonated.	BAR DE
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Methods to assess the presence and quantity of AN in mixtures showed:

Calculations from manufacturer mixture specifications provide an adequate basis to assess the AN level in fertilizer products. Specialized chemical analysis techniques using crystallographic analysis confirmed the presence of AN in an AN double salt based product (AN Double Sall). Publicly available information on the molecular composition of AN Double Sall used in the calculations showed that it contains 35 to 40% AN. Similar calculations for CAN-26 and CAN-27 showed AN levels of 74% and 77%, respectively.

The results of technical assessments were presented on two occasions to an interagency SME panel for review. The SME panel included members from DHS/Infrastructure Security Compliance Division, DHS/Office for Bombing Prevention, DHS/Office of Intelligence and Analysis, DHS/U.S. Secret Service, Department of Justice (DOJ)/Bureau of Alcohol, Tobacco, Firearms and Explosives, DOJ/FBI, Department

of Defense (DOD)/Joint Improvised Explosive Device Defeat Organization, DOD/Combating Terrorism Technical Support Office, Office of the Director of National Intelligence/National Counterterrorism Center, and the Department of State. This technical panel determined that all mixtures with 1 lb of fertilizer/fertilizer mixture were detonable based on the test diagnostics and that a minimum detonable level of 10% AN by weight can be technically defended by DHS/ANSP, providing a small margin of safety beyond the 15% level, which showed a weak detonation.

Given all the factors that influence detonation, precise thresholds for variations in AN-based improvised explosive formulations do not exist. The SME panel discussed additional studies to include test replicates, other parametric test variations, and other instrumentation that could improve the determination of thresholds to better inform the DHS/ANSP regulatory decisions but determined that the combined results from the current test campaigns complement the historic literature and are sufficient.

It should be noted that the SME panel was adamant that the technical interpretations of the historic literature and the SNL test results are not DHS/ANSP policy. DHS/ANSP decisions on regulatory thresholds should be based on a combination of factors to include, but are not limited to, historic literature and current SNL test results, historic and current trends in terrorist use of AN-based explosives, and the impact to the regulated industries.

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