LLNL-TR-465872

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Integrated Data Collection Analysis (IDCA) Program Phase II - Mixing Procedures and Materials Compatibility

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January 14, 2011

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Integrated Data Collection Analysis (IDCA) Program—Mixing Procedures and Materials Compatibility

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ABSTRACT

Three mixing procedures have been standardized for the IDCA proficiency test—solid-solid, solid-liquid, and liquid-liquid. Due to the variety of precursors used in formulating the materials for the test, these three mixing methods have been designed to address all combinations of materials. Hand mixing is recommended for quantities less than 10 grams and Jar Mill mixing is recommended for quantities over 10 grams. Consideration must also be given to the type of container used for the mixing due to the wide range of chemical reactivity of the precursors and mixtures. Eight web site sources from container and chemical manufacturers have been consulted. Compatible materials have been compiled as a resource for selecting containers made of materials stable to the mixtures. In addition, container materials used in practice by the participating laboratories are discussed. Consulting chemical compatibility tables is highly recommended for each operation by each individual engaged in testing the materials in this proficiency test.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety-testing protocols, HME, mixing methods, materials compatibility.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

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1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory small-scale safety and thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives[1]. The materials for the Proficiency Test have been selected to span the challenging experimental issues arising when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. HMEs are often formed by mixing oxidizer and fuel precursor materials, and to a large extent, the challenges are centered on the physical forms and stability of the improvised materials.

Typically, the solid-solid, liquid-liquid, or solid-liquid mixture precursors are combined shortly before use. For solid-solid mixtures, the challenges associated with producing a standardized inter-laboratory sample primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—and taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products. Table 1 shows the materials selected for use in the IDCA study.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	powder mixture
Potassium perchlorate	Charcoal	powder mixture
Potassium perchlorate	Dodecane[1]	slurry
Potassium chlorate -40 mesh	Dodecane[1]	slurry
Potassium chlorate as received	Sucrose (icing sugar mixture)[2]	powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2]	powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2]	powder mixture
Ammonium nitrate		solid
Bullseye® smokeless powder[4]		solid
Ammonium nitrate	Bullseye® smokeless powder[4]	powder mixture
Urea nitrate	Aluminum	powder mixture
Urea nitrate	Aluminum, sulfur	powder mixture
Hydrogen peroxide 70%	Cumin	sticky paste
Hydrogen peroxide 90%	Nitromethane	miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	sticky paste
Hydrogen peroxide 70%	Glycerin	miscible liquid
HMX		solid
RDX Type II Class 5		solid (standard)
PETN Class 4		solid (standard)

1. Simulates diesel fuel
2. Contains 3 wt. % cornstarch
3. Separated through a 100-mesh sieve
4. Alliant Bullseye® smokeless pistol gun powder

In addition to these issues, it is important to stress that all of these improvised explosives materials, including many of the precursors, are reactive chemicals. The precursors are often corrosive and hygroscopic, which presents difficulty in storage and handling while mixing and testing. The improvised materials are typically mixtures of oxidizers and fuels, which are generally NOT stable. For the materials in Table 1, this may cause temporal changes once the precursors are mixed, which can lead to catastrophic failure due chemical reaction[2].

All these issues affect the SSST testing. Traditional military explosives are constructed of an oxidizer and fuel in the same molecule but more importantly are chemically stable. This allows for reasonable conditions for testing. The conditions for testing improvised explosives are basically the same as for any other explosive. It is the handling methods to attain these testing conditions, and the decisions made when the testing shows high sensitivity, that are largely unknown. Hence, the IDCA proficiency study is to assist in determining the proper handling of these materials for the explosives community in general. This report focuses on mixing methods and selecting container materials for mixing, testing and storage of the improvised explosives.

Essential for this proficiency test are comparable methods, procedures and equipment. Because of the number of participants in the study, the procedures and equipment are different, so the methods and procedures must be clearly delineated and standardized when possible. For pre-testing of the materials, that is the pretreatment, mixing and handling, the procedures can be the same for each group. This report establishes a mutually agreed methodology for mixing methods to be used for the IDCA proficiency test.

The performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Air Force Research Laboratory/RXQF (AFRL-Tyndall), Naval Surface Warfare Center, Indian Head (NSWC-IHD), and Sandia National Laboratories (SNL).

Table 2. Components for testing and physical properties

Material[1]	Source	Physical State	Behavior in Atmosphere
Potassium perchlorate	Columbus	Powder	Hygroscopic
Potassium chlorate	Columbus	Powder	Hygroscopic
Sodium chlorate	Fisher	Powder	Hygroscopic
Ammonium nitrate	Fisher	Powder	Stable
Bullseye® smokeless powder[3]	Alliant	Powder	Stable
Urea nitrate	TCI America	Powder	Stable (depends on purity)
Hydrogen peroxide 70%	FMC	Liquid	Decomposes slowly
Hydrogen peroxide 90%	FMC	Liquid	Decomposes slowly
RDX Class 5	Holsten	Solid	Stable
HMX	ATK	Solid	Stable
PETN Class 4	Holsten	Solid	Stable
Aluminum	Valimet	Powder	Oxidizes rapidly in air
Charcoal	Aldrich (Darco)	Powder	Strong adsorbent
Dodecane	Alfa Aesar	Oily liquid	Stable
Icing sugar mixture	C & H	Very fine powder	Clumps in moisture
Cumin	Safeway	Seeds	Clumps in moisture when ground
Nitromethane	Fisher	Liquid	Stable
Flour (chapatti)	Laxmi	Powder	Stable
Glycerin	Fisher	Liquid	Stable
Sulfur	Sigma-Aldrich	Powder	Slowly oxidizes in air

1. Materials from Table 1
2. Responsible laboratory for purchasing/producing/distributing
3. Alliant Bullseye® smokeless pistol gun powder

2 MATERIALS

2.1 Chemicals

Table 2 lists the base materials used in this study, with the source and selected physical characteristics. In this study, most of the materials, or in some cases, where the materials are mixtures, the precursors were obtained from the same batch and distributed to the various participants. SSST is defined as less than 1-gram quantities and impact, friction and electrostatic discharge testing. Thermal testing is defined as differential scanning calorimetry (DSC).

2.2 Containers

The materials used in the study vary widely in chemical and physical properties. The choice of the compatible containers for mixing is highly dependent on the reactive and solvency properties of the specific material. In general, non-glass containers are used because of durability. However, in many cases, the manufacturer recommends glass or passivated glass as the most compatible material. Other compositions may be desirable because of the lack of durability of glass has during mixing. Due to the variety of different compatibility issues, several sources were consulted for many of the materials. Glass has the disadvantage of making sharp fragments should an explosion occur, even at the 1-gram level. Plastic is safer if compatibility allows its use.

Table 3 shows compatibilities from manufacturers and distributors of containers and similar materials. This table is meant to be a guideline for selecting the material(s) for the containers. A select few manufacturers have reference materials that are collected in a fairly easy access format. The values in the table were located by the use of eight web sources—Cole-Palmer[3], Nalgene[4], New Pig[5,6], OzoneLab[7], TAP Plastics, Inc.[8], Ultra Tech International[9], and Concord-Top Corporation[10], and allorings.com, Inc[11]. Cole-Palmer and Nalgene have interactive web sites that allow selecting chemicals and container materials for compatibility. New Pig and OzoneLab have select tables with compatibilities. allorings.com, Inc has a large table for o-ring seal compatibility. From these on-line sources, materials compatibility was search for the specific chemical of interest. Of the consulted information, only container materials that were rated in the top category, excellent, or otherwise top of compatibility, are listed.

Data for compatibility was not available for all the materials in this project. In those cases, the table indicates no data. In some cases, not all sources had data on every material. In addition, there was conflicting data for some of the materials. Also there are some materials in the table that are considered non-reactive during mixing and storage, such as the flour and cumin, so compatibility data was not obtained.

Table 3. Materials Compatibility for Containers[i,ii]

Potassium perchlorate—LDPE[5], Fluorinated polymers[11], PE[9],
Potassium chlorate—LDPE[3,4,5], HDPE[4], ABS plastic[3], Buna-N (Nitrile)[3], Carbon graphite[3], Fluorinated polymers[3,4,11], CPVC[3], EPDM[3], Epoxy[3], Neoprene[3], NORYL[3], Polycarbonate[3], PE[9], PEEK[3], PP[3,4], Polyurethane[3], PPS[3], PVC[3], Ti[3], PPCO[4], PMP[4], PMX[4], PMMA[4], SAN[4]
Sodium chlorate—LDPE[4,5], HDPE[4], 304[3], ABS Plastic[3], Acetal (Delrinr)[3], Al_2O_3 Ceramic[3], CPVC[3], EPDM[3], Epoxy[3], Fluorinated polymers[3,4,11], CSPE[3], Natural rubber[3], Neoprene[3], NORYL[3], Polycarbonate[3,4], PEEK[3], PP[3,4], PPS[3], PVC[3], Ti[3], PMP[4], PMX[4], PMMA[4], SAN[4]
Ammonium nitrate—LDPE[3,4,5], HDPE[4], 304[3], 316[3],Carpenter 20[3], Cast iron[3], EPDM[3,11], Epoxy[3], Fluorinated polymers[3,4,11], NORYL[3], PP[3,4], PPS[3], PPCO[4], PMP[4], Polycarbonate[4], PVC[4], PE[9], PS[4], PMX[4], SAN[4], Tygon®[11], flourosilicon[11],
Bullseye® smokeless powder[12]—no data
Urea nitrate—no data

Hydrogen peroxide (for 50% by weight)—316[3], Al[3], CPVC[3], Fluorinated polymers[3], Hastelloy-Cr[3], Polycarbonate[3], PEEK[3], PVC[3], Ti[3], Viton®[3]
Hydrogen peroxide (for 90% by weight)—LDPE[4,5,15], HDPE[4,6], 316[3,7], ABS plastic[3], Al[3], ceramic Al_2O_3[3], CPVC[3], Epoxy[3], Fluorinated polymers[3,4], Hastelloy-Cr[3], NORYL[3], Polycarbonate[3,4], PE[9], PEEK[3], PVC[3,4], Viton®[3], PP[4], PPCO[4], PMP[4], PS[4], PMX[4], SAN[4]
MEK (for MEKP precursor)—PP[4], PPCO[4], Fluorinated polymers[4,11], TMX[4], PET[4], EPDM[11],
MEKP—Fluorinated polymers[11] (data for like materials)—HDPE (MEKP catalyst)[8]; HDPE, EVA, LDPE, PVC, DCP, and CHP (Cumene hydroperoxide)[10]
Methyl nitrate (for methanol precursor)—LDPE[3-5], HDPE[4,6], 304[3], 316[3], Acetal (Delrinr)[3], Al[3], Brass[3], Bronze[3], Buna Nitrile[3,11], Carbon graphite[3], Carbon Steel[3], Carpenter20[3], Cast iron[3], Ceramic Al_2O_3[3], Fluorinated polymers[3,4,11], CPVC[3], EDPM[3,11], Hastelloy-Cr[3], CSPE[3], natural rubber[3], NORYL[3], PEEK[3], PP[3,4], PPS[3], PPCO[4], PMP[4], PMX[4], PET[4], PVC[3], Silicone[3], Tygon®[3]
RDX Class 5—Velostat™ or Nalgene[13,16], Teflon®[14], glass[14]
HMX— Velostat™ or Nalgene[13,16], Teflon®[14], glass[14]
PETN Class 4— Velostat™ or Nalgene[13,16], Teflon®[14], glass[14]
Aluminum—stable
Charcoal—stable
Dodecane (for diesel fuel as surrogate)—LDPE[3], 304[3], 316[3], Acetal (Delrinr)[3], bronze[3], Buna N (nitrile)[3], carbon graphite[3], carpenter 20[3], Cast iron[3], Ceramic Al_2O_3[3], Epoxy[3], Fluorinated Polymers[3], Hastelloy Cr[3], Hytrelr[3], Neoprene[3], Nylon[3], Polycarbonate[3,4], PP[3,4], PPS[3], PVC[3], Tygon®[3], Viton®[3], PPCO[4], RPVC[4], PET[4], SAN[4]
Icing sugar mixture—stable
Cumin—stable
Nitromethane—LDPE[3], 304[3], 316[3], Acetal (Delrinr)[3], Al[3], Carbon graphite[3], Fluorinated polymers[3,4,11], Cu[3], Hastelloy-Cr[3], PEEK[3], PPS[3], PMP[4], PMX[4]
Flour (chapatti)—stable
Glycerine (glycerin, glycerol)—LDPE[3-5], HDPE[4,6], 304[3], 316[3], Acetal (Delrinr)[3], Al[3], Bronze[3], Buna N (Nitrile)[3,11], Carbon graphite[3], Carbon Steel[3], Carpenter 20[3], Cast iron[3], Ceramic Al_2O_3[3], Cu[3], CPVC[3], EPDM[3,11], Epoxy[3], Fluorinated Polymers[3,4,11], Hastelloy-Cr[3], CSPE[3], Hytrel[3], Natural rubber[3], Neoprene[3], NORYL[3], Nylon[3,4], Polycarbonate[3,4], PE[9], PEEK[3], PP[3,4], PPS[3], PVC[3], Silicone[3], Ti[3], Tygon®[3,11], Viton®[3], PPCP[4], PMP[4], RPVC[4], PS[4], PMX[4], PET[4], PMMA[4], SAN[4], fluorosilicon[11]
Sulfur—stable

i. LDPE—low density polyethylene, HDPE—high density polyethylene, ABS Plastic—acrylonitrile butadiene styrene, Buna-N (Nitrile)—co polymer of butadiene and acrylonitrile, CPVC—chlorinated polyvinyl chloride, EPDM—ethylene propylene rubbers, Epoxy—epichlorohydrin and bisphenol A resin and triethylenetetraamine hardner, Neoprene—polychloroprene synthetic rubber, NORYL—polyphenyl oxide and polystyrene, polycarbonate—poly bisphenols, PEEK—poly etherether ketone, PP—polypropylene, PPS—polyphenylene sulfide, PVC—poly vinyl chloride, PPCO—polypropylene co-polymer, PMP—polymethylpentene, PMX—polydimethylsiloxane, PMMA—poly methylmethacrylate, SAN—styrene acrylonitrile, 304—304 stainless steel, 316—316 stainless steel, Acetal (Delrin™)—polyoxomethylene, CSPE—chlorosulfinated polyethylene (for example, Hypalon®), Carpenter 20—Ni, Cr, Mo alloy stainless steel, PS—polystyrene, Hastelloy-Cr—Ni, Mo, Cr, Fe, W alloy, PET—polyethylene teraphthalate, Tygon®—polyvinyl chloride, Hytrel—copolyester elastomer, RPVC—rigid poly vinyl chloride, PPCP—polypropylene copolymer
ii. Fluoropolymers—examples include PTFE (polytetrafluoroethylene), DuPont, Teflon®; PFA (perfluoroalkoxy polymer resin), Hyflon; FEP (fluorinated ethylene-propylene); ETFE (polyethylenetetrafluoroethylene), Tefzel, Fluron; PVF (polyvinylfluoride), Tedlar; ECTFE (polyethylenechlorotrifluoroethylene), Halar; PVDF (polyvinylidene fluoride), Kynar, Solef, Hylsr; PCTFE and CTFE (polychlorotrifloroethylene), Kel-F; FFKM (perfluoroelastomer), Kalrez, Tecnoflon; FPM/FKM, Viton®, Tecnoflon; PEPE (Perfluoropolyether), Fomblin, Galden, Nafion

For solid mixtures, none of the materials have compatibility issues, so LDPE and HDPE are reasonable choices. With liquids materials, as well as their mixtures, there are some compatibility issues with hydrogen peroxide, dodecane, nitromethane, and glycerine. In these cases, the choices of containers is much more limited and care must be taken for selection. It is recommended that the individual responsible for the work consults the above chart and verifies it with the referenced web site information.

3 PROCEDURES

The first step in any of the SSST and thermal testing is formulating the test material. The components are given in Table 1 and mixing ratios are listed elsewhere.[17] In order to standardize the IDCA materials preparation, each participating laboratory is using materials from the same batch and the materials are being handled the same way until analyzed. Below are the mixing procedures decided upon as standard methods for mixing the three forms—solid-solid, solid-liquid, and liquid-liquid mixtures.

3.1 Solid-Solid Mixtures

General Procedure:

1. Weigh the oxidizer and place in a selected compatible container.
2. Weigh the fuel and add to the oxidizer in a selected compatible container.
3. Manually mix until mixture appears to be homogenous (5-10 minutes) or place a lid on container and tumble for 5-10 minutes at 30 rpm in a Jar Mill or a V-blender.
4. After 1 hour on standing at room temperature in a closed container. Submit sample for the necessary tests as required.

Note: Manually Mix <10g; Jar Mill or tumbler / V-blender >10g

3.2 Solid–Liquid Mixtures

General Procedure:

1. Weigh the oxidizer and place in a selected compatible container.
2. Weigh the fuel and add to the oxidizer in a selected compatible container.
3. Manually mix until mixture appears to be homogenous (5-10 minutes) or with a magnetic stirrer.
4. After 1 hour standing at room temperature in a closed container, submit sample for the necessary tests as required.

Note: Manually Mix or magnetic stirrer <10g; magnetic stirrer >10g

3.3 Liquid-Liquid Mixtures

General Procedure:

1. Weigh the oxidizer and place in a selected compatible container.
2. Add the fuel to the oxidizer and stir with a magnetic stir bar for 10-15 minutes until a homogenous mixture is observed. Monitor the temperature of the mixture with a thermocouple.
3. Allow mixture to stand for 1 hour at room temperature in a closed container. Submit sample for necessary tests as required.

Note: Manually Mix or magnetic stirrer <10g; magnetic stirrer >10g

4 DISCUSSION

Materials. Compatibility of the materials to be studied with the mixing and storage containers is a critical issue for this project. Unlike standard military explosives, several of the materials in this study are mixtures containing highly reactive oxidizers with fuels. These oxidizers can be reactive with a variety of materials, so extreme care must be taken to pick a suitably inert container. The recommendations listed in Table 3 are derived from the various reference sources and are considered the best or to have suitable inertness. There are many more materials that can be used, but have not quite the same inert rating. For example, with 90% concentrated H_2O_2, 304 stainless steel is considered a reasonable material to use from a couple of sources but a better materials is 316 stainless steel.[3,6] In order to avoid this confusion only the top categories were cited as compatible.

Velostat™ is material that has been used for handling explosive because the design of the material minimizes static build up. It is a polyolefin that is impregnated with conductive carbon[18]. Unfortunately, some of the participating laboratories have had problems with compatibility with Velostat™ with some of the IDCA test materials. Caution must be taken in using containers made from this material because polyolefins, such as polypropylene, are not completely inert.

Each participant handles selected materials the following way:

- IHD—Typically uses Nalgene containers to date for the H_2O_2 mixtures; Velostat™ containers for RDX, HMX, PETN.
- LLNL—Typically uses glass or Teflon® containers.
- AFRL—stores 70% peroxide mixes in LDPE Nalgene bottles short term (less than 12 hours); Nalgene LDPE bottles for some 90% mixes. (Note: seal integrity is an issue with Nalgene bottles when working with H_2O_2: 1) the seal is tight enough so that the bottles pressurize as the peroxide decomposes, and 2) the cap/bottle seal generates a shear action as one tightens/loosens the cap.)
- LANL- Typically uses Nalgene and Velostat™ containers.

Mixing methods. There is some flexibility in the mixing methods by the choice of mixing equipment. The important point is to adequately mix the materials in a safe manner, paying attention to material compatibility, impact and shock sensitivity. Some of the materials in the IDCA study have impact sensitivity, so methods that apply too much pressure or friction need to be avoided. For others, build-up of static charge can be an issue, particularly with fine powders. Manually mixing refers to hand mixing the components without the aid of a mechanically assisted mixer. A recommended procedure is to use a spatula made with a compatible material. A Jar Mill is essentially any size cylindrical container that can be rolled or tumbled. It is recommended that the container have baffles or some type of protrusion that will assist displacement of the materials from the walls of the container. A V-mixer automatically does this, but there can be dead spots. Plastic Jar Mill containers can be improvised into having baffles. See the Appendix for an example of this using a heat gun.

The list of materials being studied by the IDCA for the proficiency test have a wide variety of compositions and physical properties, and therefore the need for the three different mixing procedures—solid-solid, liquid-liquid, and solid-liquid. Even with these categories, the methodologies described here are guidelines and no doubt some adaptation will be required on a per case basis. Each of these categories has challenges for adequate mixing. Mixing two solids, such as $KClO_4$ and Al has the challenge that all solid mixtures have, separation due to differences in particle sizes. Mixing of solid and liquids have the challenge of inhomogeneous wetting of the solid preventing even dispersion. In addition, some of the H_2O_2 mixtures with food materials, such as H_2O_2 and cumin, have been documented to change significantly over time.[19] Mixing of two liquids presents problems when there is immiscibility. For this reason, H_2O_2 at a concentration of 90% was selected with nitromethane.

5 CONCLUSIONS

Three mixing procedures have been agreed upon for the IDCA proficiency test—solid-solid, liquid-liquid, and solid-liquid. Hand mixing is recommended for quantities less than 10 grams and Jar Mill mixing is recommended for quantities over 10 grams. Consideration must also be given to the type of container used for the mixing. These materials highly depend upon reactive properties of the mixture. Consulting chemical compatibility tables is highly recommended for each operation.

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ABREVIATIONS, ACRONYMS, INITIALISMS

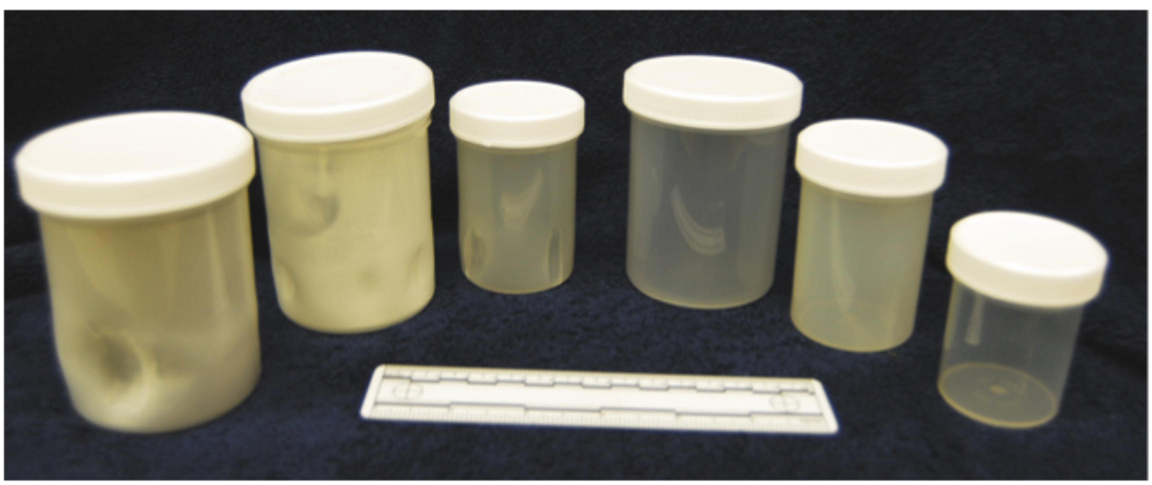
AFRL	Air Force Research Laboratory
AN	ammonium nitrate
D	detonation velocity
D-BREIE	Data-Base of Range Evaluated Improvised Explosives
EGDN	ethylene glycol dinitrate
HDPE	High density polyethylene
HME	homemade explosives
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
H_2O_2/F	hydrogen peroxide/fuel

IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division
$KClO_4$	Potassium Perchlorate
LANL	Los Alamos National Laboratory
LDPE	Low density polyethylene
LLNL	Lawrence Livermore National Laboratory
MEKP	methyl ethyl ketone peroxide
NSWC-IHD	Navel Surface Warfare Center—Indian Head Division
PETN	pentaerythritol tetranitrate
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
SNL	Sandia National Laboratories
SO/F	solid oxidizer/fuel
SSST	small-scale safety and thermal testing
TATP	triacetone triperoxide
UN	urea nitrate

APPENDIX A

Purchasing Jar Mills containers with appropriate baffles or internal stirring components for mixing can be expensive and possibly not available from major vendors in all materials and sizes. However, plastic containers can be easily modified to give lightly intrusive baffles using a solid metal object and a heat gun.

Figure A-1 shows some examples of these modifications of polypropylene containers. The color is due to the containers once had mixtures in them that had S as a component. The containers were modified by heating



with a laboratory heat gun and then deforming with the closed end of a 9/16 to 1/2 inch combination wrench.

Figure A-1. Jar Mills modified using a hot solid and heat gun. The containers on the left are used with S residue in them. The containers on the right are the same corresponding size containers that have not been modified. Containers are made of polypropylene.

ACKNOWLEDGMENTS

This work was performed by the Integrated Data Collection Analysis (IDCA) Program, a five-lab effort supported by Los Alamos National Laboratory, Lawrence Livermore National Laboratory, Sandia National Laboratories, the Air Force Research Laboratory, and Indian Head Division, Naval Surface Warfare under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division. Los Alamos National Laboratory is operated by Los Alamos National Security, LLC, for the United States Department of Energy under Contract DE-AC52-06NA25396. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. This work was performed under the auspices of the U. S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The Air Force Research Laboratory (AFRL/RXQF) and Indian Head Division, Naval Surface Warfare (NSWC IHD) also performed work in support of this effort. The work performed by AFRL/RXQL and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.

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