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Causal Analysis of the FASB Uranium Silicide Compact Combustion Event

David E. James
T. Charles Couch

May 2013

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Level 1 Cause Analysis
ORPS Number: NE-ID--BEA-FASB-2013-0001
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Event Occurred: April 10, 2013

David E. James
T. Charles Couch

May 2013

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

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David E. Janes
Cause Analyst

5/16/2013
Date

T. Charles Couch
Research Facilities Nuclear Facility Manager

5/16/13
Date

Ronald K. Farris
Peer Review

5/21/13
Date

Philip J. Breidenbach
MFC Mission Support Director

5/16/13
Date

Mark Henry
MFC Research Division Director

5/20/13
Date

Approved by:

Riley R. Chase
Deputy Laboratory Director,
Projects, Nuclear Support and Production

5/22/13
Date

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Executive Summary

The Fuels and Applied Science Building (FASB) is a Radiological Facility, located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL), which provides space and resources for the research and development of low enrichment fuel as an alternative for research reactors, spent fuel treatment, nuclear waste research and development, and the conduct of other experimental projects.

One of the research projects involves developing accident tolerant fuels utilizing liquid phase sintering of Uranium Silicide (U_3Si_2), unreacted depleted Uranium (U), and Silicon (Si). This process involves combining powder size constituents of U_3Si_2, U, and Si, and then using a press to make what is referred to as a green compact. All powder handling and compacting is performed in the inert atmosphere of the Reduced Enrichment Research and Test Reactors (RERTR) Glovebox compartment C because of the pyrophoric nature of the constituent U_3Si_2 and unreacted depleted Uranium powders. The green compacts, approximately four grams each, are then liquid phase sintered in a furnace under a cover gas to obtain U_3Si_2 with increased density.

On April 10, 2013, three green compacts composed of U_3Si_2+U+Si powders were produced by the Principal Investigator (PI) in the inert RERTR Glovebox. Each green compact was placed in a cotton liner for protection and then placed in a small Ziploc plastic bag and labeled Sample 1, 2, and 3. All three bagged green compacts were placed in a larger Ziploc plastic bag and transferred out of the inert RERTR Glovebox and into East Hood B for decontamination prior to transferring them to a sintering furnace located in another room in FASB. Inside East Hood B, each of the samples was removed from the plastic bags and cotton liners and placed on a paper towel.

After approximately 5 minutes, at 10:00AM, Sample 1 spontaneously began to combust. The PI immediately called out to pull fire alarm and Metal-X fire extinguishing agent was applied to Sample 1 completely covering the combusting green compact (see Figure 1). Approximately 30 seconds elapsed from beginning of observed reaction to application of Metal-X.

ONLY ONE OUT OF THE THREE GREEN COMPACTS COMBUSTED

All subsequent actions taken were appropriate for this event and are detailed in the timeline for this event contained in Section 3.1.

The MFC Fire Department found no presence of fire when they entered the facility and thermal imaging indicated that the combustion event was over and did not spread from Sample 1. Radiological Controls personnel performed surveys and found no spread of contamination to personnel or the facility above established regulatory limits. MFC Industrial Hygienist verified that East Hood B was functioning as designed and flow was within specifications. FASB was released for normal access at 11:15AM.

Paired Causal Factor (Root Cause)

A3B3C06/A4B5C01

Personnel underestimated the problem by using past experience as basis
Problem identification did not identify a need for change

Past success in the production of green compacts from pyrophoric powders and exposing them to air lead to an underestimation of the hazards associated with this activity and subsequent hazard mitigation. When a new mixture was being developed it was assumed that the overarching previously successful hazard identification and mitigation would still be appropriate even though there was inherent uncertainty associated with compacted powders. When the first green compacts of the new mixture were created there were visible surface defects. Potential issues identified were discussed but did not identify a need for change.

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ACRONYMS

FASB	Fuels and Applied Science Building	NS&T	Nuclear Science and Technology
β/γ	Beta and Gamma	ORPS	Occurrence Reporting and Processing System
BED	Building Emergency Director	PFC	Permanent Field Change
cm	centimeter	PI	Principal Investigator
dpm	decades per minute	RERTR	Reduced Enrichment Research and Test Reactors
α	Alpha	Rev	Revision
HPT	Health Physics Technician	RWP	Radiological Work Permit
INL	Idaho National Laboratory	Si	Silicon
INR	Initial Notification Report	SS	Shift Supervisor
ISMS	Integrated Safety Management	TFC	Temporary Field Change
LTA	Less Than Adequate	U	Uranium (for this report specifically Depleted Uranium)
MFC	Materials and Fuels Complex	U_3Si_2,	Uranium Silicide
MSDS	Material Safety Data Sheets		
NFPA	National Fire Protection Association		

DEFINITIONS

Autoignition	Self-ignition of combustible material through chemical action (such as oxidation) of its constituents (i.e., Spontaneous Combustion).
Combustible (Material)	A material that, in the form in which it is used and under the conditions anticipated, will ignite and burn; a material that does not meet the definition of noncombustible or limited-combustible. *NFPA 1 Section 3.3.57*.
Combustion	A chemical process of oxidation that occurs at a rate fast enough to produce heat and usually light in the form of either a glow or flame. *NFPA 1 section 3.3.64*
Green	Term for compacted powders associated with powder metallurgy
Mesh Size	The number of openings in the mesh across one linear inch on a screen. < 400 mesh means that all particles would pass through a 400 mesh screen. > 400 mesh means that all particles are retained on a 400 mesh screen. < 400 mesh is equivalent to 37 microns.
Micron	One-millionth of a meter.
Pyrophoric	A chemical that spontaneously ignites in air at or below a temperature of 130°F (54.5°C). *NFPA 1 section 3.3.210.*

Causal Analysis of the FASB Uranium Silicide Compact Combustion Event

1. BACKGROUND

The Fuels and Applied Science Building (FASB) is a Radiological Facility located at the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL), which provides space and resources for the research and development of low enrichment fuel as an alternative for research reactors, spent fuel treatment, nuclear waste research and development, and the conduct of other experimental projects.

The East Room, room 103, is a radiologically clean area that contains fabrication and processing areas, as well as general work space, offices, and storage areas. The West Room, room 102, contains vent hoods and inert gloveboxes that are used for developing low enrichment fuels, passivating waste from glovebox operations, providing a clean inert atmosphere for work with corrosive and reactive materials, and testing equipment that will be used in other facilities.

One of the research projects involves developing accident tolerant fuels utilizing liquid phase sintering of Uranium Silicide (U_3Si_2), unreacted depleted Uranium (U), and Silicon (Si). This process involves combining powder size constituents of U_3Si_2, U, and Si, and then using a press to make what is referred to as a green compact. All powder handling and compacting is performed in the inert atmosphere of the Reduced Enrichment Research and Test Reactors (RERTR) Glovebox compartment C because of the reactive (pyrophoric) nature of the constituent powders. The green compacts are then sintered in a furnace under a cover gas to obtain the desired end product.

Liquid phase sintering of this material involves heating the green compact to a temperature where the Uranium and Silicon constituents enter their liquid phase while the U_3Si_2 is still in the solid phase. This allows the liquid phase Uranium and Silicon to help bind together the solid phase U_3Si_2 constituent and aids significantly in the densification of the green compact. The desired result of this process is to make a U_3Si_2 with a higher density.

Table 1. Reactive Nature of Compact Powder Composition

Material	Powder Size	Reactive Nature
Uranium Silicide (U_3Si_2)	< 400 mesh	Pyrophoric
Depleted Uranium (U)	< 300 mesh	Pyrophoric
Silicon (Si)	< 300 mesh	Combustible
	Material Form	
U_3Si_2, U, and Si mixture	Green Compact	Unknown
Uranium Silicide (U_3Si_2)	Solid	Combustible

Pyrophoric: *A chemical that spontaneously ignites in air at or below a temperature of 130°F (54.5°C). NFPA 1 section 3.3.210*

Combustible: *A material that, in the form in which it is used and under the conditions anticipated, will ignite and burn. NFPA 1 section 3.3.57*

Table 2. Liquid Phase Sintering Summary

Powder Composition (% by Weight)	Desired Result
80% U_3Si_2 + 18.5% U + 1.5% Si	Higher density U_3Si_2

Liquid Phase Anneal Temperature	1550°C

Melting Temperature	
Uranium Silicide (U_3Si_2)	1665°C
Depleted Uranium (U)	1120°C
Silicon (Si)	1410°C

1.1 Event

On April 10, 2013, three green compacts composed of Uranium Silicide (U_3Si_2), unreacted depleted Uranium (U), and Silicon (Si) powders were produced by the Principal Investigator (PI) in the inert RERTR Glovebox compartment C, which is located in FASB West Room, room102. It was identified by the PI that the three green compacts had visible surface delamination. Each green compact was placed in the cut off finger of a cotton liner for protection and then placed in a small Ziploc plastic bag and labeled Sample 1, 2, and 3. All three bagged green compacts were placed in a larger Ziploc plastic bag used as the transfer bag. The three compacts were then transferred out of the inert RERTR Glovebox and into East Hood B, a radiological fume hood, for decontamination prior to placing them in the sintering furnace which was located in the FASB East Room, room 103.

NOTE: A furnace outside of the RERTR Glovebox was planned to be used because the capability to perform liquid phase sintering of this material did not exist inside the RERTR Glovebox.

The green compacts were removed from the Ziploc plastic bags and cotton liners in the East Hood B and placed on a paper towel for contamination control in the vent hood by the PI, starting with Sample 1, then Sample 2, then Sample 3.

After approximately 5 minutes elapsed (10:00AM), the PI saw what he described as "a visible spark that originated from Sample 1," and then it began to glow red. The PI immediately called out to pull fire alarm and began to look for the Metal-X fire extinguishing agent (Class D for metal fires) inside the vent hood. The Health Physics Technician (HPT) pulled the paper towel out from under the green compacts and tossed it further into the vent hood (see Figure 1). Sample 1 ended up on a piece of duct tape in the hood (see Figure 2). The PI observed a small flame around Sample 1 (coming from the green compact) for three to four seconds.

A nearby Nuclear Science and Technology (NS&T) Technician acquired Metal-X from the HPT covering this job from a nearby bench and applied it to the glowing red compact (Sample 1), completely covering the combusting compact. Approximately 30 seconds elapsed from beginning of observed reaction to application of Metal-X.

NOTE: ONLY SAMPLE 1 COMBUSTED OUT OF THE THREE GREEN COMPACTS.

All subsequent actions taken were appropriate for this event and are detailed in the timeline for this event contained in Section 3.1. The MFC Fire Department found no presence of fire when they entered the facility and thermal imaging indicated that the combustion event was over and did not spread from Sample 1.

Radiological Controls personnel performed surveys and found no spread of contamination to personnel or the facility above established regulatory limits. Radiological surveys consisted of smears taken along the hood sash and floor area. A Maslin floor mop wipe was taken as well as wipes throughout the area. One smear taken on the Maslin floor mop and one wipe taken on the floor showed elevated levels of contamination that were below established limits (Survey Map M-20130410-47).

Table 3. Surface contamination limit values (*LRD-15001, Table 2-2*).

Radionuclide	Removable	Total (Fixed + Removable) (dpm/100 cm^2)
U-natural, U-235, U-238, and associated decay products	1,000 dpm/100 cm^2 α	5,000 alpha

Table 4. Post event survey results summary (*Survey Map M-20130410-47*)

Surveys	Results
All direct surveys	<5000 dpm/100 cm^2 β/γ <100 dpm/100 cm^2 α
Smear #3, of Maslin floor mop	<1000 dpm/100 cm^2 β/γ 63 dpm/100 cm^2 α
All other smears	<1000 dpm/100 cm^2 β/γ <20 dpm/100 cm^2 α
Wipe #2, floor in front of hood	<1000 dpm β/γ 2000 dpm α
All other wipes	<1000 dpm β/γ No detectable α

At 11:15 AM the FASB was released for normal access.

MFC Industrial Hygienist verified that East Hood B was functioning as designed and flow was within specifications. The MFC Fire Protection Engineer determined that this combustion event was within the fire hazard capabilities of East Hood B, meaning that this combustion event did not damage East Hood B and that the HEPA filters in the hood exhaust were sufficiently down streams that they were not affected by heat generated during this event. East Hood B was verified to be operating within operational parameters and prevented the spread of any potential contamination from this event.

Figure 1 shows conditions approximately 5 hours after Sample 1 combusted. The pictures have been positioned to show the relative position of Sample 1 and other items within East Hood B. The unreacted sample shown in Figure 1 did not indicate a raised temperature during any thermal scanning by the fire department, either immediately after the event during radiological surveys of the facility or when the samples were transferred back to the RERTR Glovebox.

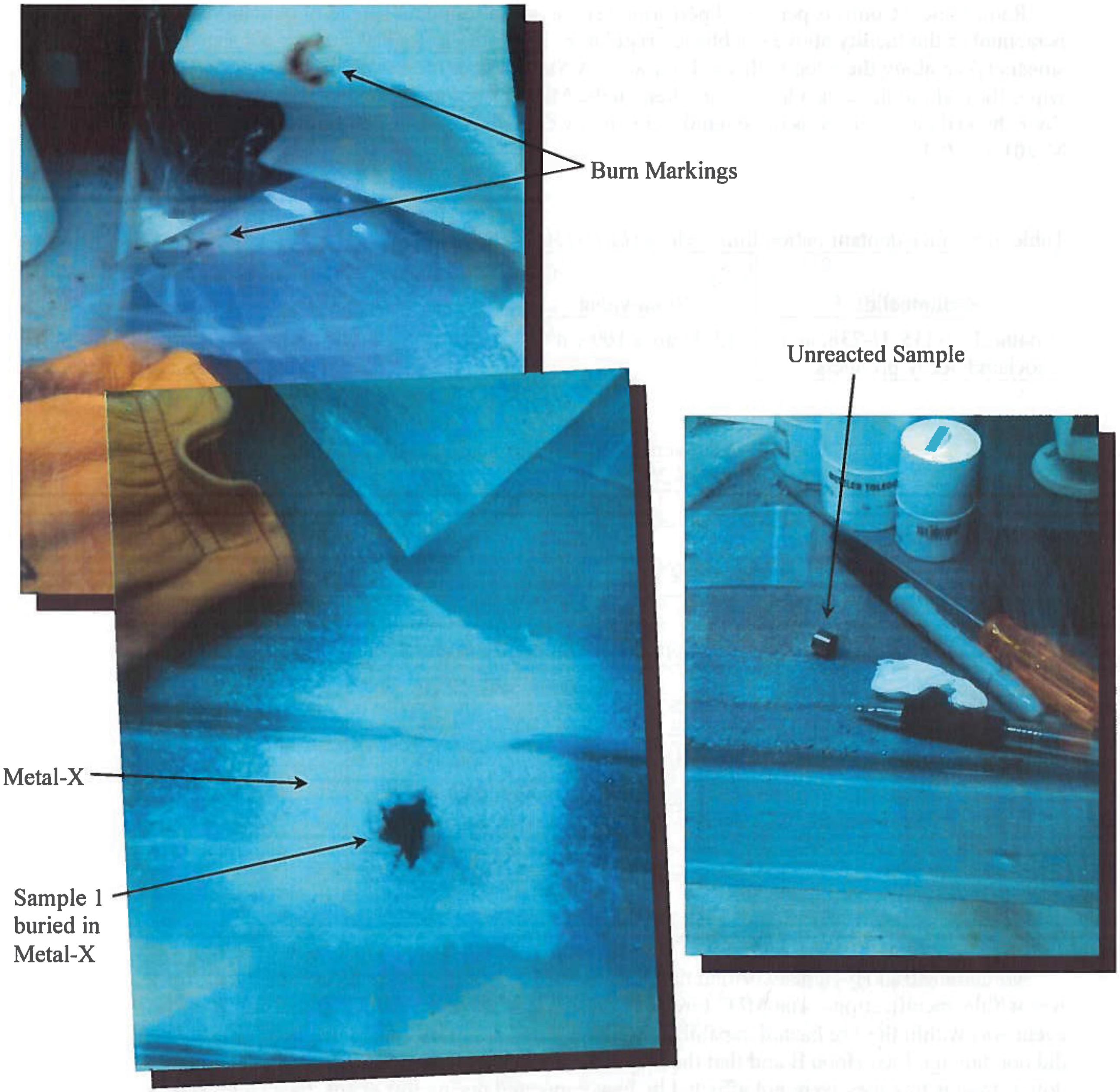


Figure 1. Post Event Pictures in Relative Position in East Hood B.

NOTE: *Photographs were taken of East Hood B at 3:30PM the day of the event through the vent hoods glass sash.*

At 6:30 PM, the compacts and METAL-X were transferred to the RERTR Glovebox compartment C and FASB returned to normal operational status. Figure 2 was taken after the remains of Sample 1 and the Metal-X were removed and the area decontaminated. This picture shows the minimal damage to the standard duct tape that was in direct contact with Sample 1 while it combusted.

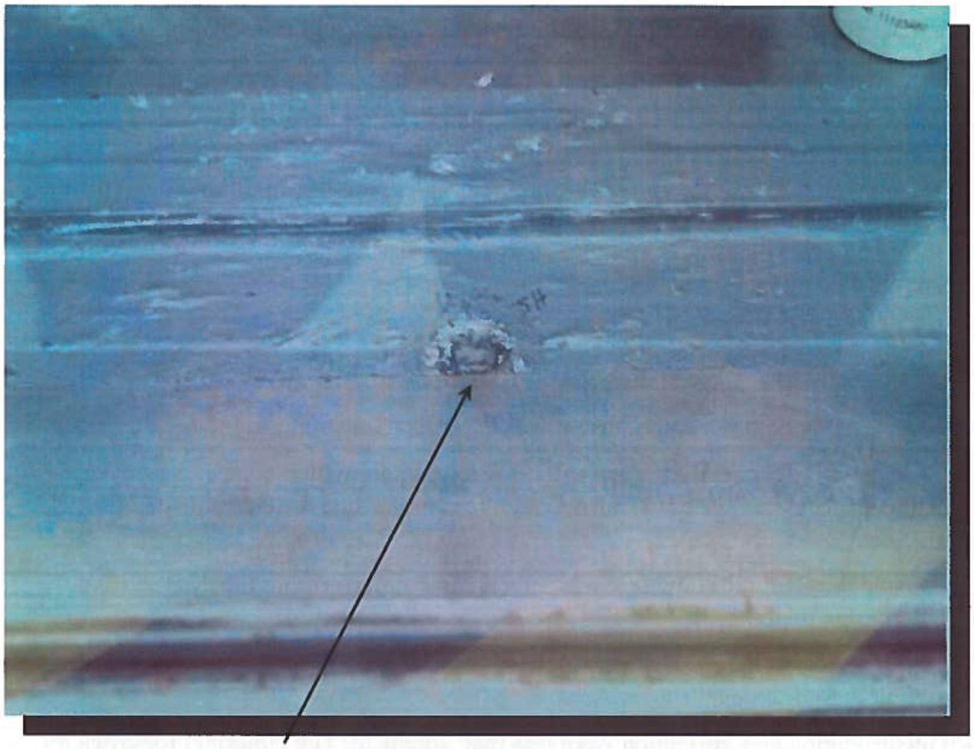


Figure 2. Minimal damage to duct tape in direct contact with Sample 1

1.2 Why Did One of Three Green Compacts Combust

1.2.1 Similarities Between Samples 1, 2, and 3

All three green compacts were created from the same batch of 80% U_3Si_2 + 18.5% U + 1.5% Si. The batch was created from previously prepared powder forms of each constituent material. Each of the constituent raw material powders was appropriately stored in separate containers inside the RERTR Glovebox compartment C.

The depleted Uranium powder was processed to the desired particle size by the process of hydride/dehydriding. This process is a method of reducing solid uranium directly into fine powder with no mechanical work necessary. A simple explanation of this process is to raise the temperature of Uranium to 275°C and add hydrogen. As a result the solid breaks apart and gains volume. At the end of the process, a vacuum is drawn to extract the hydrogen. The dehydriding process is important since Uranium Hydrides are pyrophoric.

The mixture was prepared in the RERTR Glovebox compartment C by weighing the raw materials with a scale, and then mixed by hand in a mixing jar. No binders were used in the mixture. The hand mixing duration was for one to five minutes. The mixture was then separated into three separate portions using weighing pans on a scale. The samples were poured into a die and pressed to 60,000 psig using the mechanical press located within compartment C. The density of each green compact was verified to be acceptable.

It was noted that each of the three green compacts showed some visible surface delamination around the outside diameter of the compacts. Delamination is a common surface defect that is caused mainly by spring back and friction with the die walls when the compact pressure is release and the compact ejected. In an effort to minimize compacting defects for these samples the compacting force was reduced and Zinc Sterate die lubrication was used.

The first compact completed was labeled Sample 1; the second, Sample 2; and the third, Sample 3. The samples were each placed in cotton liners and bagged as described in Section 1.1. After the samples were transferred to the East Hood B, they were removed from the plastic bags and cotton liner and placed on a paper towel. This was when the samples were first exposed to air. Sample 1 was the first out of the Ziploc bags that were sealed in the inert atmosphere of the RERTR Glovebox. The next two were removed from the plastic bags and cotton liner and placed on the same paper towel in quick succession. The samples were not touching each other on the paper towel. Approximately five minutes later, most of the similarities between the three samples stopped when Sample 1 spontaneously combusted.

1.2.2 Why Did Sample 1 Spontaneously Combust in FASB East Hood B?

1. Sample 1 spontaneously combusted because it was exposed to air in East Hood B.
2. Samples 1, 2, and 3 were transferred to East Hood B for contamination control, survey and decontamination as necessary, prior to transferring the samples to a furnace outside of the RERTR Glovebox.
3. The furnace inside the RERTR Glovebox was not capable of performing the desired liquid phase sintering of this material.
4. The desired furnace was in the FASB East Room in open air, which is in a radiologically clean room within a Radiological Buffer Area.
5. The hazard identification and mitigation were less than adequate. The material properties for this new mixture were discussed and the reactive properties of the green compacts were identified to be an unknown, but this was not included in procedures because it was incorrectly believed to be covered by existing work and hazard controls.
6. Using past experience as a basis, it was believed that this material was likely only combustible, which was stated in the procedure with mitigations listed. *(A3B3C06/A4B5C01)*

   Had this material only been combustible, which Samples 2 and 3 were, the hazard mitigation was still less than adequate. Less than adequate hazard identification and mitigation detail is provided in Section 1.3.
7. Speculate that thermal energy released from the oxidation of Sample 1 raised its temperature to the autoignition temperature associated with a defect unique to that sample or loose powder.

   These are based on the following facts:

   - Approximately five minutes elapsed while exposed to air before the event occurred
   - Surface defects were observed in all three of the samples
   - Only one of three samples spontaneously combusted
   - The other two samples were in the vent hood for about eight hours, and there was no noticeable thermal reaction as indicated by thermal imaging, and the fact that they did not combust.

   Other potential contributors:

   - It is possible that some Uranium Hydrides were still in the raw material and ended up on the surface of the compact or on the surface of the delamination surface defect
   - Hand mixing may have resulted in different constituent content in each of the three samples.

1.2.3 Why Didn't the Other Two Samples Combust?

Since Sample 1 was completely combusted, this cannot be determined from the evidence reviewed. It is possible that the other two samples oxidized to the point that the oxidation layer on the surface prevented the reaction from continuing to the point that they could spontaneously ignite.

1.3 LTA Hazard Identification and Mitigation

The FASB procedures covering this work, FASB Powder Handling, 30-07-FASB, and FASB General Laboratory Work procedure, 1356-07-FASB, covered the hazards associated with pyrophoric powders and combustible solids. However, the procedures specifically did not identify and mitigate the potential hazard associated with exposing green compacts with identified unknown reactive properties to air.

FASB Powder Handling, 30-07-FASB

The FASB Powder Handling procedure, 30-07-FASB, covers the portion of this evolution that occurred in the inert RERTR Glovebox, and states that "Powders … pressed to form a solid compact are no longer considered powder." There are no additional identified hazards or mitigations associated with removing green compacts from the inert glovebox.

Section 1.1, PROGRAM OBJECTIVES

> *The purpose of this Laboratory Instruction (LI) is to identify the hazards and mitigations associated with powder handling and processing operations in the Fuels and Applied Sciences Building (FASB). Powder handling occurs in FASB under a broad range of projects. Powdered material covered under this LI includes uranium (typically U235 and U238) and uranium alloys.*

Section 2.6, Risk and Controls

Table 5. Risk and Controls from FASB Powder Handling, 30-07-FASB, Section 2.6.

Task	Hazard	Engineering Control	Administrative Control	Personal Protective Equipment (PPE)
6. Removing powders from the glovebox.	6. Combustion/ radiological contamination.	Closed metal container	**Note: Powders** melted and cast into solid form or **pressed to form a solid compact are no longer considered powder**	Per RWP

FASB General Laboratory Work procedure, 1356-07-FASB

FASB General Laboratory Work procedure, 1356-07-FASB, covers all work in FASB that is not covered under a process specific or equipment specific procedure or manual. This procedure identifies the hazard for a combustible material, but does not cover putting a material that could spontaneously combust in air into a vent hood. Additionally, the hazard mitigations for combustible materials in East Hood B are less than adequate for the following identified issues.

- Minimizing combustible materials or a combustible material exclusion area around the working area
  - A lot of material and equipment was in the hood and not sufficiently spaced away from the working area
  - Samples were placed on a paper towel.

- No specific/marked location for Metal-X extinguisher/agent in East Hood B
  - This resulted in a 30 second delay in application of extinguishing agent
  - It was quicker to find the extinguishing agent outside of the East Hood B than the one staged inside the hood.
- Transferred material that spontaneously combusted in air, within plastic Ziploc bags and cotton liners (combustible bag not capable of withstanding a fire as can be seen in Figure 1).

Section 1.1, PROGRAM OBJECTIVES

> *The objective of this Laboratory Instruction (LI) is to identify the hazards and mitigations for general laboratory work in the Fuels and Applied Science Building (FASB). This LI pertains to general laboratory work in FASB regardless of the funding source. All program specific requirements and hazards should be addressed in a program specific LI or other work instructional or directing documents (e.g. work requests, inspection plans, etc.).*

Section 1.3, Working in hoods

> *All hoods in FASB are radiological contamination areas. Hood airflow must be within specification (and certified by Industrial Hygiene)... Work in hoods will fall under the 'low' or 'medium' hazard category (per PLN-3763 "MFC Recovery Plan") but* ***the controls in place (engineered ventilation enclosure and administrative checks) properly mitigate the risks.***

Table 6. Risk and Controls from FASB General Laboratory Work procedure, 1356-07-FAS, Section 2.17.

Task	Hazard	Engineering Control	Administrative Control	Personal Protective Equipment (PPE)
17. Work with flammable/ combustible materials (i.e., solids, liquids, and vapors).	17b. Fire hazards associated with handling flammable solids	Ensure adequate ventilation as determined by exposure assessment. Work in an inert atmosphere glovebox when the potential for generating pyrophoric dust may occur during material processing. Class ABC & D (as applicable) fire extinguishers available in the area.		Safety glasses with side shields Leather gloves recommended

All of the Engineering Controls were satisfied and the PPE used appropriately, but did not have any effect on the likelihood of this event occurring. The hazard identification and mitigation strategy in the FASB General Laboratory Work procedure, 1356-07-FASB, was not written to prevent this type of event; rather, it was written to react to the event and subsequently mitigate the effects if the event did occur.

2. SCOPE OF THIS INVESTIGATION

This investigation was commissioned to perform a Level 1 Cause Analysis of the events that led to the FASB Uranium Silicide Compact Combustion Event that occurred on April 10, 2013. The scope of this investigation was to evaluate the causes, responses, and appropriateness of mitigations associated with this event. Corrective actions have been developed, and are included in this report, to address the causes of this event as well as other issues identified during the course of this investigation. This issue and its corrective actions will be tracked in ICAMS issue number IO-026714 under source document SD-007390.

3. FACTS

3.1 Chronology of Activities

Date/Time	Action/Activity
04/10/2013 0715	FASB Shift Supervisor (SS) conducts Radiological Facilities Plan of the Day meeting, releasing work for FASB.
	The PI discussed his plan for the day: • Continuation of powder operations (30-07-FASB) • Transfer of compacts from RERTR Glovebox to East Hood B (1356-07-FASB) to prep compacts to load in furnace (1223-07-FASB).
0800	PI arrives at FASB and conducts non-documented job briefing
	PI begins working in RERTR Glovebox compartment C; • Using previously prepared U_3Si_2, U, and Si powders weighed out desired amounts of each by weight using a scale 80% U_3Si_2 + 18.5% U + 1.5% Si (by weight) • Mixed the constituents in a mixing jar by hand for several minutes (>1 but <5 min) • Separated mixture into 3 equal portions using weighing pans on a scale • Portion was poured into a die and then placed into the press • Press was used to compress the sample to 6000 psig into a green compact • Green density was measured • Compact was placed in cotton liner (the finger from a cotton liner glove), then into small Ziploc bag, the bag sealed, and labeled using a permanent marker. **NOTE:** *The first one completed was labeled Sample 1, and the process was repeated creating Sample 2 and Sample 3.*
~0945	Bagged compacts were transferred out of RERTR Glovebox compartment C into one larger Ziploc bag and transferred into East Hood B.
~0955	Compacts were removed from the Ziploc bags and cotton liners in the East Hood B and placed on a paper towel for contamination control in the vent hood by the PI, starting with Sample 1, then Sample 2, then Sample 3.
Approximately 5 minutes elapsed	
1000	**PI saw what he described as "a visible spark that originated from Sample 1" then it began to glow red.**
	PI immediately called out to pull fire alarm.
	While PI looked for METAL-X fire extinguishing agent inside the vent hood; • HPT pulled paper towel out from under the compact samples and tossed it further into the vent hood (Sample 1 ended up on a piece of duct tape in the hood (see Figures 1 and 2) • Nearby NS&T Technician acquired METAL-X from HPT covering the job from a nearby bench and applied it to the glowing red compact (Sample 1) completely covering the combusting compact. **NOTE:** *Approximately 30 seconds elapsed from beginning of observed reaction to application of Metal-X.*
	The HPT directed the PI and NS&T Technician to exit the hood and segregate themselves from other personnel.
1001	FASB Facility Operator notified the FASB SS that METAL-X had been dumped on a compact in East Hood B in response to a glowing compact.

Date/Time	Action/Activity
	FASB SS obtained radio on his way down the stairs to pull nearest fire alarm at FASB front entrance.
	FASB SS pulled manual fire pull box and FASB Forman began a building sweep for personnel accountability. FASB Foreman called 526-7777 from his cell phone to notify INL Fire Department that a pull box had been activated.
	All evacuated personnel were staged outside of FASB entrance and surveyed by HPT prior to reassembly at MFC-713. All personnel were accounted for and no contamination was found.
1005	INL Fire Department personnel responded and FASB SS briefed Incident Commander of conditions within FASB.
	FASB SS notified MFC Emergency Action Manager via radio of situation at FASB.
1008	FASB SS notified FASB Operations Manager, FASB Nuclear Facility Manager, and Safeguards' Manager of event in FASB with material left unsecured in hood. All other material in FASB was already secured.
1020	Incident Commander reported that the INL Fire Department firefighters and thermal imaging findings suggested no presence of fire.
	Nasal smears were taken on the PI and NS&T Technician with no contamination found.
	Industrial Hygienist reported that hood flow was satisfactory within specifications and hood flow meter was functioning as designed.
1030	FASB HP Supervisor briefed Radiological Controls personnel for re-entry into facility to verify radiological conditions of facility.
	Radiological Controls personnel entered FASB to survey the floor and up to the hood ledge with fire department personnel supporting with thermal activity detector.
1100	INL Fire Department personnel returned to their station at MFC.
1115	Radiological Controls personnel found no contamination (Survey M-20130410-47)
	FASB released for normal access.
1200	Meeting conducted at FASB with management and other appropriate personnel to discuss what was required to return to normal operations. • Transfer sample compacts and METAL-X from East Hood B to RERTR Glovebox.
1400	Documented pre-job brief conducted.
1415	Further discussed transfer items from East Hood B to RERTR Glovebox using procedure 30-07-FASB.
1500	Work commenced on transferring items to RERTR Glovebox in glass vials.
1510	Work was suspended to add additional work control.
1700	Temporary Field Change eCR 613562 was generated for 1356-07-FASB with appropriate level of reviews.
1800	Work resumed to return the compacts and METAL-X to RERTR Glovebox compartment C with INL Fire Department personnel using thermal imaging. No noticeable change in thermal imaging detected.
1830	Work completed to return compacts and METAL-X to RERTR Glovebox. Facility returned to normal operational status.
04/11/2013 0815	Critique was held.

3.2 Analysis

3.2.1 ISMS Core Functions

The Why Tree, Barrier Table, and Behavior Analysis techniques were used in this investigation, and are attached as Appendix A, B, and C, respectively. The results of this analysis are summarized within the context of the ISMS guiding principles below. Reviews of records, procedures, interviews with participants, and reviews of similar events were used to complete this analysis.

Core Function 1 – Define the Scope of Work

Missions are translated into work, expectations are set, tasks are identified and prioritized, and resources are allocated.

<u>This core function was fully met</u>.

The procedures and RWP covering this work had a very broad scope covering many activities, but clearly covered the scope of the work being performed.

Core Function 2 – Analyze the Hazards

Hazards associated with the work are identified, analyzed, and categorized.

<u>This core function was not fully met</u>.

The hazards associated with producing, mixing, and pressing powders into compacts were well understood and the procedures covers this.

The material properties for this new mixture were discussed and the reactive properties of the green compacts were identified to be an unknown, but this was not included in procedures because it was incorrectly believed to be covered by existing work and hazard controls. *(A3B3C06/A4B5C01)*

Core Function 3 – Develop and Implement Hazard Controls

Applicable standards and requirements are identified and agreed upon, controls to prevent/mitigate hazards are identified, the safety envelope is established, and controls are implemented.

<u>This core function was not fully met</u>.

The Hazard Identification and Mitigation associated with exposing green compacts with identified unknown reactive properties to air was not identified in procedures covering this work.

Personnel stopped and discussed this issue with subject matter experts; however, personnel still failed to recognizing that although this may be close to or within the existing scope and set of controls, that the existing set of controls may not be adequate for this identified unknown. *(A3B3C06/A4B5C01)*

The Hazard Controls implemented for a combustible solid was less than adequate to prevent this type of event or appropriately mitigate its consequence.

Core Function 4 – Perform Work within Controls

Readiness is confirmed and work is performed safely.

<u>This core function was met</u>.

Personnel performed work within the controls established in the facility procedures FASB Powder Handling, 30-07-FASB, and FASB General Laboratory Work procedure, 1356-07-FASB.

When the first green compacts of the new mixture were created, there were visible surface defects, which did not identify a need for change. There was no step to stop

Core Function 5 – Provide Feedback and Continuous Improvement

Feedback information on the adequacy of controls is gathered, opportunities for improving the definition and planning of work are identified and implemented, line and independent oversight is conducted, and, if necessary, regulatory enforcement actions occur.

<u>This core function was met</u>.

Lessons learned from previous work with pyrophoric powders and compacts were referenced before this work was initiated. Past experience with producing green compacts with different pyrophoric compositions indicated that the glovebox – vent hood – furnace route was a reliable way to sinter compacted powders. Unfortunately, using past successful experiences as a basis resulted in an underestimation of the potential hazards.

Ideally, a furnace capable of performing this work would be installed in a glovebox. Work is also underway to create alternative containers to allow the direct transfer of compacted powders from the glovebox to the furnace while maintaining an inert atmosphere.

4. CONCLUSIONS

The causes that contributed to this event are described as a Paired Causal Factor. A Paired Causal Factor is two closely related causal factors used to describe a root cause of an event and is required whenever a human error causal factor is selected. Causal Factors are the root causes that if corrected would prevent this event from recurring.

Cause codes listed are per the DOE Standard for Occurrence Reporting Causal Analysis, DOE-STD-1197-2011.

4.1 Paired Causal Factor

A3B3C06/A4B5C01:

Knowledge Based Error, Individual Underestimated the Problem by Using Past Event as Basis
Change Management LTA, Problem Identification Did Not Identify Need for Change

Past success in the production of green compacts and exposing them to air lead to an underestimation of the hazards associated with this activity and subsequent hazard mitigation. When a new mixture was being developed, it was assumed that the previously successful hazard identification and mitigation would still be appropriate even though there was uncertainty associated with this new mixture. When the first green compacts of the new mixture were created, there were visible surface defects on each of the green compacts. These potential issues were identified and discussed but did not identify a need for change.

The hazard identification and mitigation covering this work was for a wide variety of low and medium risk activities and relies on the knowledge and experience of the performers to adequately identify and mitigate hazards, as well as identify the need for change. The identified unknown reactive nature of compacted pyrophoric powders combined with the first time a new mixture was created demonstrated a lack of conservative decision making associated with this activity.

The identified unknown material properties and hazards associated with green compacts composed of pyrophoric powders was identified, but was not identified or mitigated within the procedures that cover this work.

4.2 Corrective Actions

The following corrective actions have been developed to address the root causes (causal factors) that contributed to this event.

1. Evaluate appropriate compact packaging when removing from an inert atmosphere. *(A3B3C06/A4B5C01)*

   Target Completion Date: 06/13/2013

   Actions Assigned to: Curtis Clark
   Objective Evidence: Provide documentation of completed evaluation

2. Evaluate the need for sintering furnace being placed in inert glovebox. *(A3B3C06/A4B5C01)*

   Target Completion Date: 05/31/2013
   Actions Assigned to: Tim Hyde
   Objective Evidence: Provide copy of approved evaluation

3. Evaluate and revise FASB Powder Handling, 30-07-FASB and FASB General Laboratory Work, 1356-07-FASB, for hazards identified and appropriate mitigation for work covered. *(A3B3C06/A4B5C01)*

   Target Completion Date: 05/31/2013
   Actions Assigned to: Curtis Clark
   Objective Evidence: Provide copy of approved revised procedures on EDMS

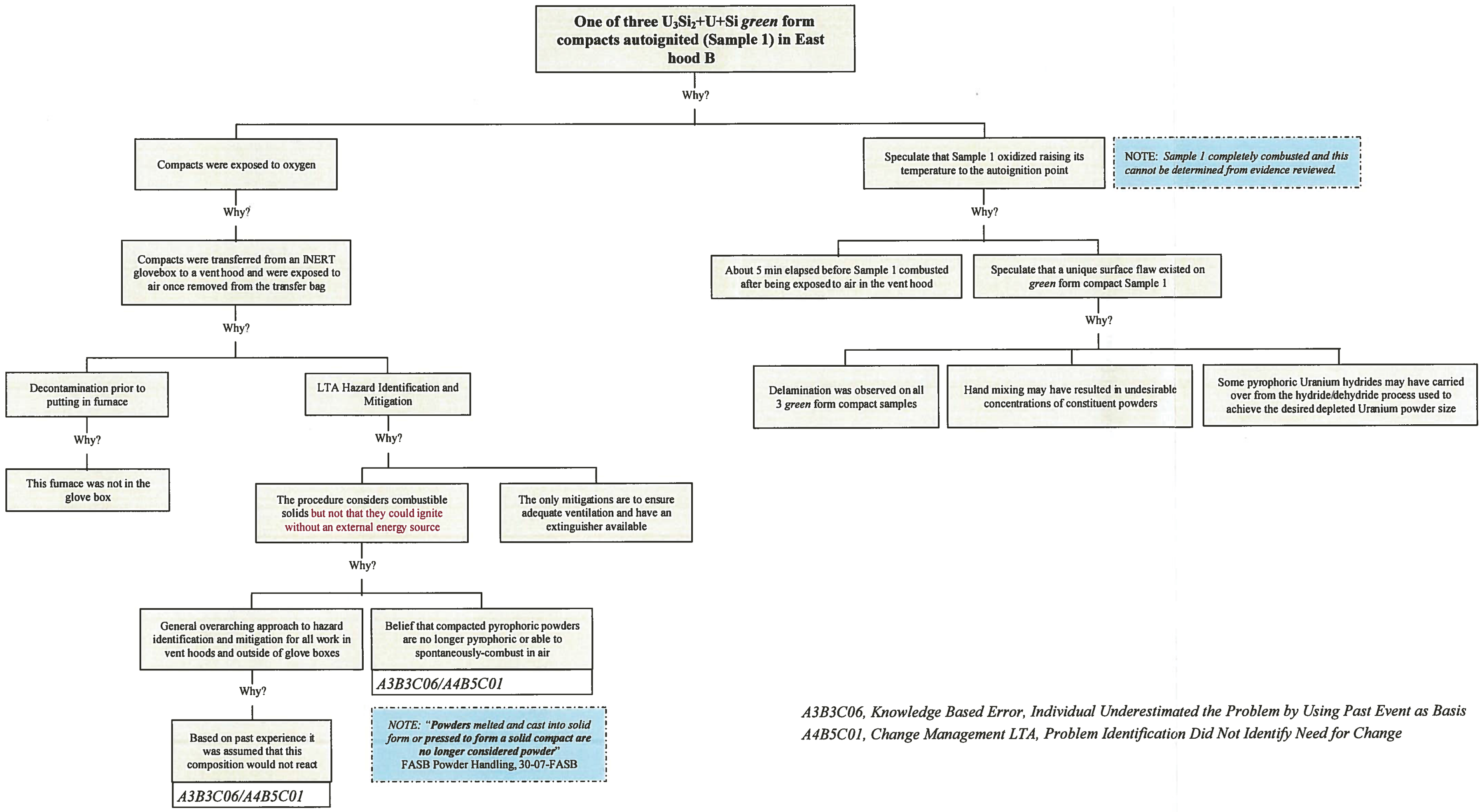
4. Develop MFC lessons learned slide. *(A3B3C06/A4B5C01)*

   Target Completion Date: 04/15/2013 - COMPLETED
   Actions Assigned to: Larry Evens
   Objective Evidence: Provide copy of lessons learned slide

5. Submit INL Lessons Learned. *(A3B3C06/A4B5C01)*

   Target Completion Date: 05/20/2013
   Actions Assigned to: Larry Evens
   Objective Evidence: Provide copy of final lessons learned

Appendix A
Why Tree



A3B3C06, Knowledge Based Error, Individual Underestimated the Problem by Using Past Event as Basis
A4B5C01, Change Management LTA, Problem Identification Did Not Identify Need for Change

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Appendix B
Barrier Analysis

NOTE: BARRIERS ARE LISTED CHRONOLOGICALLY

Barrier	Type	How did barrier perform	Why did barrier fail	How did the barrier affect the event
1. Green Compact	Engineered	Failed	• Unknown: Sample 1 completely combusted and this cannot be determined based on evidence reviewed. • Speculate that Sample 1 oxidized once exposed to air in East Hood B and was able to raise the temperature to the point that Sample 1 autoignited, most likely associated with a unique surface defect on Sample 1. Based on: - Observed surface delamination on the round side of the compact samples - Approximately 5 minutes elapsed from the time that Sample 1 was exposed to air and the time it began to combust.	• Spontaneously combusted in air without an external ignition source.
2. Procedure FASB Powder Handling, 30-07-FASB, Hazard ID & Mitigation	Admin	LTA	*(A3B3C06/A4B5C01)* • Does not cover the hazards associated with removing compacts of from the inert glovebox. *"Note : **Powders** melted and cast into a solid form or **pressed to form a solid are no longer considered powder**"*	• 30-07-FASB, section 2.6 was correct but it does not take into account the uncertainty associated with the material properties of compacted powders • Did not prevent removing these compacts from the inert glovebox.
3. Procedure FASB General Laboratory Work, 1356-07-FASB Hazard ID & Mitigation	Admin	LTA	*(A3B3C06/A4B5C01)* • Hazard identification and mitigation strategy was not written to prevent this type of event; rather, it was written to mitigate the effects if the event did occur. • Overarching hazard identification for compacted powders states that they are considered solid and failed to identify the relative uncertainty associated with green compacts and new/different recipes being compacted.	• The procedure did not take a conservative approach to handling potentially pyrophoric materials • Did not prevent removing these compacts from the inert glovebox • Did not prevent exposing the compact to air.
4. Trained personnel	Person	LTA	*A3B3C06/A4B5C01)* • Potential issues identified were discussed but did not identify a need for change. - Hazard identification for new mixture - Visible green compact flaws	• Did not prevent this event
5. Vent Hood	Engineered	By design	• Air in the vent hood was a one of the three required elements to have a fire	• Location where Sample 1 was exposed to air resulting in the event • Kept fire from spreading • Kept spread of contamination below limits
6. Metal-X Extinguishing Agent	Engineered	By design	• Did not fail, not designed to prevent initiation of event only to respond to an event	• Kept fire from spreading
	Admin	LTA	• Metal-X did not have a designated storage location in the vent hood.	• Delayed application of extinguishing agent (~30 sec) • Extinguishing agent used was from outside the vent hood

Cause Codes:

A3B3C06, Individual underestimated the problem by using past events as basis

A4B5C01, Problem identification methods did not identify need for change

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Appendix C
Behavior Table

NOTE: LATENT ORGANIZATIONAL WEEKNESS (LOW) AND LESS THAN ADEQUATE (LTA) BARRIERS ARE LISTED IN SEQUENCE FROM BARRIER ANALYSIS

LOW / LTA Barriers	Who Was Responsible	Expected Behavior	Actual Behavior	Why did the behavior make sense
Green Compact	• Performer	• Believed that this would prevent this event	• Did not prevent this event	(A3B3C06/ A4B5C01) • Belief that compacted pyrophoric powders are no longer pyrophoric or able to spontaneously combust in air • Based on past experience it was assumed that this composition would not react.
Procedures FASB Powder Handling, 30-07-FASB, and FASB General Laboratory Work, 1356-07-FASB	• Document owner • Performer	• Ensure that procedures have adequate hazard identification and mitigation • Ensure adequate review	*(A3B3C06/ A4B5C01)* • LTA Hazard Identification and Mitigation for compacted powders of Pyrophoric constituents	(A3B3C06/ A4B5C01) • Belief that compacted pyrophoric powders are no longer pyrophoric or able to spontaneously combust in air • Based on past experience it was assumed that this composition would not react.
			(A3B3C06/ A4B5C01*)* • LTA Hazard mitigation for combustible solids. The only mitigations were to ensure adequate ventilation and have an extinguisher available	(A4B5C01) • Problem identification methods did not identify need for change
Trained personnel	• Mission Manager • PI	• Identify hazards associated with new mixtures • Implement hazard mitigations into procedures	*(A3B3C06/* A4B5C01*)* • Relied on existing overarching hazard identification and mitigation	(A3B3C06/ A4B5C01) • Belief that compacted pyrophoric powders are no longer pyrophoric or able to spontaneously combust in air • Based on past experience it was assumed that this composition would not react.
Metal-X Extinguishing Agent	• Performer	• Since this material was believed to be a combustible solid, Class D fire extinguishers available in the area.	• Class D fire extinguishing agent location in East Hood B was not identified prior to starting work, delaying application of extinguishing agent during this event	• Belief that it was unlikely that this compact would combust • Inaccurate risk perception.

Cause Codes:
A3B3C06, Individual underestimated the problem by using past events as basis
A4B5C01, Problem identification methods did not identify need for change

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Appendix D
Interviewee List

MFC Research Facilities Nuclear Facility Manager (NFM)
FASB Operations Manager (OM)
FASB Shift Supervisor (SS)/BED
FASB Nuclear Facility Operator

Research Department Manager
Mission Manager
Principal Investigator
Research Scientist

Research Facilities Radiological Control Engineer
Research Facilities Senior HPT

INL Fire Marshal
MFC Fire Protection Engineer

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Appendix E
Documents Reviewed

Event Critique Report	Rev 2 04/24/2013	FASB U_3Si_2 Compact Combusts Inside of Hood
INR	04/10/2013	Rapid Oxidation of Uranium Silicide inside of Hood
ORPS Report		Number: NE-ID--BEA-FASB-2013-0001
FASB SS Log	04/10/2013	
FASB Procedures:		
30-07-FASB	Rev 3 and PFC 610632	FASB Powder Handling
1223-07-FASB	Rev 3	FASB Furnace Operations
1356-07-FASB	Rev 5 and TFC 613562	FASB General Laboratory Work
HPT Log Entries:	04/10/2013	227063, 227068, 227087, and 227112
RWP Numbers:		
MFC2013159	Rev 2	FASB – Facility management approved transfers, work in hoods, gloveboxes, bench tops and caster enclosure.
MFC2013184	Rev 1	MFC – Response to CAM Alarms, RAM Alarms and Spills
Survey Map Numbers:		
M-20130408-9		MFC787 Hood Surveys
M-20130410-47		MFC787 Emergency Response to Hood Excursion
M-20130410-68		MFC787 East Hood B Verification Survey

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Appendix F
Reference Material

Reference Material

The Safe Handling of Radioactive-Pyrophoric Materials, 1955, *L.R. Kelman, W.D. Wilkinson, A. B. Shuck, R.C. Goertz*

The Powder Metallurgy of Uranium, 1956, *Dr. H. H. Hausner, Dr. J. L. Zambrow*

Fuel Powder Production from Ductile Uranium Alloys, 1998, *C.R. Clark, M.K. Meyer, J.T. Strauss*

Handbook of Non-Ferrous Metal Powders, CH 22 – Production of Rare Metal Powders, 2008, *O.D. Neikov, S.S. Naboychenko, I.V. Murashova, V.G. Gopienko, I.V. Frishberg, D.V. Lotsko*

DOE-HDBK-1081-94, Primer on Spontaneous Heating and Pyrophoricity, 1994

Synthesis and Fabrication of Refractory Uranium Compounds, 1960, *K.M. Taylor, The Carborundum Company*

Fabrication Development for the Advanced Neutron Source Reactor, 1995, *B.W. Pace, G.L. Copeland*

Uranium Pyrophoricity Phenomena and Prediction, 2000, *M. Epstein, B. Malinovic, M.G. Plys*

Research Reactor Fuel Fabrication to Produce Radioisotopes, 2011, *A. M. Saliba-Silva, E. F. Urano de Carvalho, H. G. Riella, M. Durazzo*

Summary Report on Fuel Development and Miniplate Fabrication for the RERTR Program, 1978 to 1990, (1995), *T. C. Wiencek*

NFPA 1 Fire Code 2012 edition

Powder Metallurgy: Science, Technology and Applications, 2009, *P.C. Angelo and R. Subramanian*