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ABSTRACT

In September 2010, during cleanup and removal efforts associated with a glovebox containing a sodium-potassium alloy (NaK) cooling system, a NaK reaction event occurred. Near the end of the evolution, which was at a Materials and Fuels Complex (MFC) research facility at the Idaho National Laboratory, a 2 to 3-ounce sample of NaK was being transferred from a metal can to a polyethylene bottle so it could be sent to the disposal outlet for verification analysis. During the transfer, a reaction of the NaK with ambient air was observed in both the metal can and sample container. The potential for a reaction was identified during the planning of this activity and mitigating steps were included in the procedures. In response, the two containers were flooded with argon, closed, and observed overnight. No additional reaction was noted. The sample container and metal can were packaged into a 10-gallon drum and transferred to a permitted storage location pending disposal. While the other containers generated from the deactivation and decommissioning activity were sent off-site for disposal, the 10-gal drum remained due to concerns by a staff specialist that the material could contain potassium superoxide (KO2) as a result of the reaction event. The intent at that time was to treat the NaK in a Resource Conservation and Recovery Act (RCRA)-permitted treatment facility at MFC, instead of sending it off-site for disposal. The container was labeled, inspected, and managed as a hazardous waste.

Due to the complexities associated with safely handling and treating NaK and KO2, the container remained in storage. Treatment and disposal of the NaK and KO2 container was revisited in 2016, when researchers and chemists were consulted regarding the potential for superoxide formation and the best means to safely store and treat the container.

The facility came to realize that due to the retirements of key personnel, it needed to provide current employees operational experience with the handling and treatment of NaK. An outside company was identified and contracted to provide training and consulting services. Several Battelle Energy Alliance, Inc. (BEA) personnel then attended an alkali metals safety training class. The training course provided participants with classroom and hands–on instruction associated with:

- Hazards associated with handling and using sodium and NaK
- Proper personal protection equipment, spill control techniques, and first aid
- Common design approaches for buildings, systems, and equipment used to contain sodium and NaK
- What to expect in the event of a water reaction and how to respond
- How to properly prepare for a fire and contain/extinguish small metal fires
- A hands-on demonstration of NaK reaction and controls.

Following the training, a procedure was developed to open the container, and inspect and treat the NaK. In June 2017, facility operations personnel successfully completed the inspection and treatment of the NaK drum, eliminating the legacy liability.

INTRODUCTION

A container of sodim-potassium (NaK) with potential potassium superoxide (KO2) was generated in September 29, 2010 during a disassembly of a glovebox and NaK cooling system at a Materials and Fuels

Complex (MFC) research building, located on the Idaho National Laboratory. Workers were transferring 2-3 ounces of NaK from a metal can to a poly bottle so that it could be sent off for characterization. During transfer to the poly container, a reaction of NaK with air was observed in the metal can and poly container. This was an anticipated event. As noted in a critique report the poly bottle also generated heat resulting in a silver dollar sized melted spot on the bottle[2]. In response the two containers were flooded with argon, closed and placed in a safe condition. The workers immediately made notifications and the fire department was deployed as a precautionary measure. The containers were observed overnight with no additional reaction noted. The following day the containers were further packaged into a 10 gal drum and moved to a Resource Conservation and Recovery Act-permitted storage facility. The container was labeled and barcoded with container number MFC100296, see Figure 1. The container was transported to the storage location via a drum dolly on foot for approximately two blocks from where it was generated. According to one employee the trip took several hours because the drum heated up significantly at the slightest bump. Long cool down periods were required between short moves.

Figure 1. 10-gal container containing poly bottle with 2-3 ounces of NaK.

The work associated with the disassembly of the glovebox generated 18 containers of hazardous waste, one which was the NaK container, MFC100296. In March 2011, 17 of the 18 containers were shipped off-site for disposal, while the final NaK container remained in permitted storage due to concerns that the material could contain potassium superoxide as a result of the reaction event. Potassium superoxide is a yellow inorganic compound that is air and moisture sensitive and is a potent oxidizer that can produce explosive reactions when combined with water or organics. Based on literature information potassium superoxide can produce an impact sensitive explosive compound when combined with organic oils such as kerosene.

Discussion

The container of NaK with potential potassium superoxide remained in permitted storage for several years due to the complexities associated with safely handling and treating the material. The container was labeled, inspected and managed as a hazardous waste. In August 2016 an assessment of the permitted storage facilities was conducted and identified that the NaK container was not disposed within one year of its generation date. In accordance with the regulations containers of hazardous waste shall be disposed within one year per the storage prohibition. A teleconference was made to the Idaho Department of Environmental Quality informing them about the container.

As a result of the assessment, an operability review by MFC Environmental, Safety, and Health to ensure adequacy and compliance with applicable requirements was performed. This review concluded that the container was acceptable for storage and that the container was properly labeled and inspected per the permit. Based on review by Industrial Safety and Health several controls were put in place which included, limiting access to the locked building, isolating the container by roping and posting as "shock sensitive", see Figure 2, and suspending forklift operations pending further expert analysis.

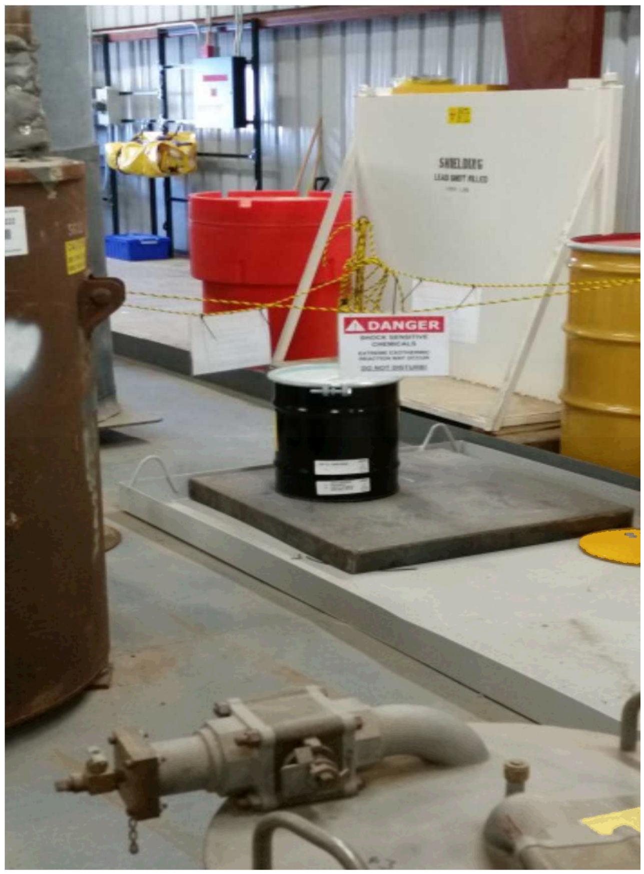

Figure 2. 10-gal container containing poly bottle with 2-3 ounces of NaK

In addition, researchers and chemists were consulted regarding the potential for superoxide formation and the best means to safely store and treat the container. Due to the retirement of key personnel, who were experienced in the handling and treatment of NaK, management decided to seek out assistance from alkali metal experts outside the laboratory. Creative Engineers Inc. (CEI) who specializes in the handling and management of alkali metals, including NaK, was contracted in September 2016 to provide training and consulting services on the safe storage, handling, and disposal of the current inventory of sodium and NaK at MFC.

Alkali Metal Experts

CEI toured MFC in October 2016 to evaluate the sodium and NaK inventory and provide safe storage, handling, and treatment and disposal options for the sodium and NaK configurations found at MFC. CEI provided a final report in November 2016, which included storage recommendations, NaK shock-sensitive safety concerns and treatment options. In regards to the NaK shock-sensitive safety concerns CEI indicated that there have been many documented incidents involving NaK and potassium superoxide that resulted in violent explosions. In most cases the explosive situation resulted from the mixing of NaK, potassium superoxide and organic material (such as mineral oil) with an applied shear force. When considering the safety implications of NaK and KO2 it is important to understand that in CEI's experience this combination alone has not been found to be explosive. It is only when organics are also present and a shear mixing force applied that the mixture exhibited explosive behavior according to all sources known to CEI[1].

Following discussions with Battelle Energy Alliance, LLC (BEA) and CEI regarding the remaining NaK container it was determined that the NaK container was likely not shock sensitive. This was primarily due to the lack of organic liquids in the container.

CEI indicated in their report that the events that occurred when preparing the poly bottle sample from the metal can are not an uncommon result when handling NaK. Often the NaK gets exposed to oxygen or small amounts of moisture and forms reaction products of oxides and hydroxides. These contaminates can react with pure alkali metal and are also hydroscopic, increasing the reactivity. Exposed or "dirty" NaK (dirty being NaK containing these mentioned impurities) can at times auto-ignite with the generation of heat very likely. The heat generated during this transfer of NaK to the storage facility was likely the result of "dirty" NaK[1].

In all instances known to CEI the organic material involved has been some type of hydrocarbon that was used to cover the NaK to prevent oxygen exposure. Although organic is often referenced, it seems hydrocarbon may be a better choice when describing the explosion cases.

Related to this incident, there was some concern that degradation of the poly bottle during the reaction and the subsequent loss of the argon blanket during the years of storage could have potentially created the case for a shock sensitive mixture since the poly bottle is organic. CEI believes this is an unlikely scenario since several organic plastics and elastomers have been tested and used in NaK service with good results. Although not specifically tested, CEI has also used polypropylene and polyethylene bottles with NaK at room temperature with satisfactory results.

The report concluded that based on CEI's opinion the NaK container could be safely inspected. The reacted NaK material was likely not shock sensitive, however, it was recommended that the drum should continue to be stored as such until a proper inspection could be made and a treatment plan developed that assumes the material was shock sensitive. It also concluded that the drum could be safely opened and inspected after the development and approval of an inspection plan.

Personnel Training Associated with Alkali Metals

Due to limited experience with handling and treating NaK, several MFC personnel were sent to an alkali metals training course offered by CEI. A two day course was provided to BEA employees on the handling of NaK. The training course provided participants with classroom and hands-on instruction associated with:

- Physical properties of alkali metals and hazards associated with handling and using sodium and NaK
- Proper personal protection equipment when handling alkali metals, spill control techniques, and first aid
- Common design approaches for buildings, systems, and equipment used to contain sodium and NaK
- What to expect in the event of a water reaction and how to respond
- How to properly prepare for a fire and contain/extinguish small metal fires

- A hands-on demonstration of NaK reaction and controls.

Some of the physical properties of NaK with a composition of 21-23 % sodium and 77-79% potassium include:

- Typically a liquid at room temperature
- Melting point -11 °C (~12.0 °F)
- Highly reactive with water and is usually stored under inert gas (i.e., argon or nitrogen).
- Fire Fighting measures use soda ash, sand, or Class D fire extinguisher
- When stored in air it can form a yellow potassium superoxide that may ignite.
- The superoxide reacts aggressively with water and organics.

In regards to the hands on training personnel were able to handle alkali metals to see and understand the properties of NaK and sodium, see Figure 3.

Figure 3. Showing NaK and Sodium

The hands on training included cleanup tips associated with small NaK spills by using damp cloths. A small amount of NaK was poured on metal tables and damp cloths were used in a swirling motion to react and clean up the NaK. Small sparks were noted from reacting the NaK, but no violent reactions were seen and cleanup of the NaK was able to be performed in a controlled environment, see Figure 4.

Figure 4. Cleanup of small spills of NaK

In addition, hands on training was done by extinguishing small fires with Class D fire extinguishers. An important aspect with the use of fire extinguishers is to use fire extinguishers which pour out the chemical instead of a spray, since it prevents from dispersing the media that is being extinguished. Also, a demonstration on how NaK reacts with water was performed. Water was sprayed in a controlled environment from a water hose. When the water mixed with the NaK it created a violent reaction and dispersed little droplets of NaK up to several feet from the source. The reaction creates excessive smoke and fire, see Figure 5.

Figure 5. NaK reaction with water

Treating of NaK at MFC

After completion of training MFC personnel developed a detailed inspection plan. The plan considered all associated risks in order to open and visually inspect the container. The inspection plan allowed for the container to be handled, transferred, opened, and inspected. The container was placed in a freezer at the storage location to ensure it would be maintained below its melting point in order to provide a more stable state for transferring the container to a separate inspection and treatment location elsewhere at MFC. After 24 hours the freezer was transferred via a forklift from the permitted storage facility to a facility where treatment of sodium and NaK is permitted. The freezer was brought into a large area enclosure tent where the container was removed from the freezer. The container was then placed into a glovebag that was sealed and inerted with argon, see Figure 6. The container was opened and the contents of the drum were inspected[3]. A small 60 ml poly bottle with a small amount of NaK was found which appeared to be approximately 2-3 ounces which coincides with what was indicated in the critique report. The poly bottle had signs of burns as noted in the critique report as well. Personnel also looked for oils or evidence of superoxides. No evidence of oils or superoxides were noted. The NaK container was then removed from the glovebag and transferred to a RCRA-permitted water wash vessel where the NaK was reacted. Due to the small amount of NaK, minimal reaction was noted.

Figure 6. Showing freezer and glovebag with container

The water wash vessel is used to deactivate ignitable and reactive hazardous waste and has an operating limit of 5 pounds of NaK or sodium. The water wash vessel has been used since 1981 and is a vertical cylindrical tank. It is approximately 10 ft in diameter and 12 ft high. The water wash vessel has a personnel and equipment access door on one side to allow for the placement of materials and the removal of any reactants. Removable floor grating is installed near the bottom of the tank to provide a working platform for personnel and equipment. There are four penetrations for spray lances to discharge water in a controlled manner and three viewing windows to observe the deactivation operations. See Figure 7 showing a picture of the water wash vessel.

Conclusions

The importance of having personnel with experience cannot be overstated. It is important that succession planning include means to retain key competencies or retain personnel proficiency in critical areas. With the loss of key personnel, who were experienced in the handling and treatment of NaK, it was decided by

MFC management to utilize external alkali metal experts to provide needed training, including hands-on experience with reactive alkali metals. This successfully restored competency and improved the comfort level for less experienced staff. After sending several operations and support staff to the training an inspection and treatment plan was prepared allowing the successful treatment of a legacy NaK container at MFC. Successful treatment of this container reduced the overall environmental liability at MFC.

REFERENCES

1. Creative Engineers, Inc. "INL Trip Report", November 18, 2016.
2. Critique Report, "Sodium Potassium (NaK) Reaction during Cleanup and Removal in MFC-789", dated September 29, 2010.
3. TSD-OI-12, NaK Container MFC100296, Dated May 22, 2017

ACKNOWLEDGEMENTS

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