

**U.S DEPARTMENT OF ENERGY  
CARLSBAD FIELD OFFICE**

**BASIS OF KNOWLEDGE FOR EVALUATING  
OXIDIZING CHEMICALS IN TRU WASTE**



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July 2017

**U.S. DEPARTMENT OF ENERGY  
CARLSBAD FIELD OFFICE**

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July 2017

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### CHANGE HISTORY SUMMARY

<b>Revision Number</b>	<b>Date Issued</b>	<b>Description of Changes</b>
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## ACRONYMS AND ABBREVIATIONS

AK	acceptable knowledge
AKE	Acceptable Knowledge Expert
ARC	accelerating rate calorimetry
BoK	Basis of Knowledge
°C	degrees Celsius
CBFO	Carlsbad Field Office
CCEM	chemical compatibility evaluation memorandum
CCP	Central Characterization Program
CFR	Code of Federal Regulations
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
DQO	Data Quality Objective
DSA	Documented Safety Analysis
DSC/TGA	differential scanning calorimetry and thermogravimetric analysis
EOPS	engineered organic polymer sorbent
EPA	U.S. Environmental Protection Agency
°F	degrees Fahrenheit
LANL	Los Alamos National Laboratory
LANL-CO	LANL-Carlsbad Operations
N.O.S.	not otherwise specified
NQA	Nuclear Quality Assurance
NTP	National TRU Program
ORNL	Oak Ridge National Laboratory
PNNL	Pacific Northwest National Laboratory
RCRA	Resource Conservation and Recovery Act
RFETS	Rocky Flats Environmental Technology Site
SDS	Safety Data Sheet
SPM	Site Project Manager
SRS	Savannah River Site
TRU	transuranic
WAC	Waste Acceptance Criteria
WCRRF	Waste Characterization Reduction and Repackaging Facility
WIPP	Waste Isolation Pilot Plant
wt. %	weight percent

## Units of Measurement

Å	angstrom	mL	milliliter
cm <sup>3</sup>	cubic centimeter	mL/g	milliliter per gram
g	gram	mm	millimeter
g/cm <sup>3</sup>	grams per cubic centimeter	ppmv	parts per million volume
J/g	joules per gram	psig	pounds per square inch gauge
M	moles per liter		

**NOTE: Do not use this Basis of Knowledge for evaluating oxidizing chemicals in the TRU waste remaining in the WIPP Waste Handling Building since the February 14, 2014, radiation release or the LANL Type 1 TRU waste stored at Waste Control Specialists. Refer to DOE/WIPP-17-3585.**

The latest revisions of DOE/WIPP-17-3585, DOE/WIPP-17-3589, and CBFO Form 3589-1 are available within WIPPnet at [http://bellview/cbfo/cbfo\\_docs.html](http://bellview/cbfo/cbfo_docs.html) and outside of WIPPnet at <https://sftp.wipp.energy.gov/human.aspx?r=265069120&Arg12=filelist&Arg06=255414084> with approved username and password. To obtain username and password, contact Kerry Watson or Cecil Thomas at (575) 234-7301.

## EXECUTIVE SUMMARY

The February 14, 2014, airborne radiation release in Room 7 of the Waste Isolation Pilot Plant (WIPP) underground disposal unit 7 was caused by an exothermic chemical reaction of nitrate oxidizing chemicals and organic materials in a single 55-gallon container. That event exposed hazards not previously evaluated in the WIPP safety basis. A new WIPP Documented Safety Analysis (DSA), Revision 5b, was developed that addressed potential hazards related to the airborne radiation release and evaluated and addressed new fire hazard scenarios. The revised hazards analysis within the DSA specifically included potential chemical exothermic reactions in waste containers and propagating fires. Compliance with WIPP Waste Acceptance Criteria (WAC) was credited as initial conditions to the hazards analysis, and Chapter 18 was added making the WIPP WAC compliance program a safety management program within the DSA. The WIPP DSA, Revision 5b, Chapter 18, and the WIPP WAC require the use of this document when performing the enhanced acceptable knowledge (AK) process on transuranic (TRU) waste determined to contain oxidizing chemicals.

The WIPP WAC was revised to address the conditions and requirements of DSA Revision 5b. The use of this Basis of Knowledge (BoK) by WIPP Certified Programs is specified in the WIPP WAC, Appendix H, Enhanced Acceptable Knowledge.

This document establishes criteria that must be used by the WIPP Certified Programs to evaluate TRU waste containing one or more oxidizing chemicals to determine acceptability at the WIPP as-is and to identify when additional evaluation or treatment is required. This BoK also includes options and requirements to be used by TRU waste sites when further evaluation, testing, and/or treatment are required. The U.S. Department of Energy (DOE) Carlsbad Field Office (CBFO) will review requests, plans, and technical justifications and provide written decisions prior to implementation by the TRU waste sites.

## 1.0 PURPOSE

This Basis of Knowledge (BoK) provides criteria to be used in conjunction with acceptable knowledge (AK) procedures of the WIPP Certified Programs for evaluating transuranic (TRU) waste (hereafter referred to as TRU waste or waste) with one or more oxidizing chemicals to determine acceptability at the Waste Isolation Pilot Plant (WIPP) as-is, identify when additional evaluation or treatment is required, and evaluate waste for acceptability post-treatment.

## 2.0 APPLICATION AND SCOPE

WIPP Certified Programs' AK personnel performing the enhanced AK process shall evaluate TRU waste that contains one or more oxidizing chemicals using the criteria contained in this BoK, complete the most current revision of Carlsbad Field Office (CBFO) Form 3589-1, Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria (see example in Attachment I), and include the required attachments. (For location to obtain Form 3589-1, see note above Executive Summary.) The application of these criteria is not dependent on a TRU waste site's determination that the waste does or does not exhibit the hazardous waste characteristic of ignitability due to oxidizer properties.

These criteria are based on bounding conditions developed in part to account for the potential drying of the waste from environmental conditions it can be subjected to inside the transportation packaging and after emplacement in the desiccating salt environment of the WIPP disposal units. Accounting for changes in the waste that can occur due to environmental conditions that can exist during active waste management minimizes the possibility of a radioactive particulate airborne release in the Category 2 nuclear facility until the waste is isolated from the WIPP underground ventilation air flow. If a TRU waste site's waste is outside the criteria established in this BoK, the TRU waste site has the options of:

- a) requesting the U.S. Department of Energy (DOE) CBFO evaluate information provided by the TRU waste site or Acceptable Knowledge Expert (AKE) that the unlisted oxidizing chemical is bounded by at least one of the listed oxidizing chemicals; or
- b) requesting a sorbent equivalency evaluation by CBFO to determine if the unlisted sorbent is compositionally equivalent to a tested sorbent; or
- c) performing tests using a method approved by the CBFO Manager and proposing a treatment method when necessary; or
- d) performing tests using the modified U.S. Environmental Protection Agency (EPA) method 1040 as specified in section 7 of this document, and treating with an acceptable inorganic sorbent; or
- e) treating the waste with an acceptable inorganic sorbent as described in section 5 when an unlisted sorbent is not compositionally equivalent; or



- f) treating waste previously sorbed in polyol organic sorbent with zeolites or waste sorbed in engineered organic polymer sorbent (EOPS) with zeolites or other inorganic sorbent as specified in section 6; or
- g) treating with zeolites when listed oxidizing chemical concentrations in sorbent cannot be bounded; or
- h) providing a technical justification that each container with the current waste form is sufficiently characterized to enable compliant shipment and receipt at the WIPP, and justification that the waste would not develop an unacceptable risk of a release until isolated from the WIPP underground ventilation air flow.

Based on the merits of the technical justification, the CBFO may determine that the waste is acceptable as-is, compliant with WIPP program and facility requirements, and presents no additional hazard to the facility that has not been considered and mitigated in the WIPP Documented Safety Analysis (DSA).

### 3.0 **DEFINITIONS**

**Organic Materials** - Carbon-containing compounds, which include not only hydrocarbons but also compounds with a number of other elements, including hydrogen (most compounds contain at least one carbon-hydrogen bond), nitrogen, oxygen, halogens, phosphorus, silicon, and sulfur. Organic materials are not limited to compounds produced by living organisms, but include human-made substances such as plastics and polymers. Carbon-containing compounds that are simple salts, such as carbonates, oxides, and carbides, are inorganic materials.

**Oxidizing Chemical** – A chemical that, while alone is not necessarily combustible, readily yields oxygen to cause or enhance the combustion of organic materials.

For the purposes of this BoK, an oxidizing chemical is:

- A chemical identified in this document as an oxidizing chemical; or
- A chemical or chemical mixture identified as hazard class 5.1 or 5.2 in the Hazard class or Division, or Label Codes columns of the Hazardous Materials Table in Title 49 Code of Federal Regulations (CFR) 172.101. However, a chemical with a numeric provision listed in the Special provisions (§172.102) column of the Hazardous Materials Table, that excludes it from the 49 CFR Subchapter C – Hazardous Materials Regulations is not an oxidizing chemical subject to evaluation using this BoK.

**NOTE: Safety Data Sheets (SDSs) obtained from chemical manufacturers or distributors may or may not identify that the subject chemical is a 5.1 or 5.2 hazardous material oxidizer. SDSs have not proven to be reliable sources of information. Other sources of information must be used to determine if the chemical in question is or is not an oxidizer.**

## **4.0 BASIS OF KNOWLEDGE TEST METHODS**

### **4.1 Testing of Oxidizing Chemicals and Sorbents**

Testing of oxidizing chemicals and sorbents to provide data for the BoK was focused on bounding the oxidizing hazard posed by oxidizing chemicals found in TRU waste streams. These tests were designed to provide a bounding set of results. The testing occurred in three stages:

1. A review of AK records was performed to provide a list of oxidizing chemicals and organic and inorganic sorbent materials reported in active TRU waste streams.
2. Scoping studies<sup>1,2</sup> were undertaken to a) identify the fastest-burning oxidizer, b) identify the fastest-burning EOPS, and c) determine the approximate maximum concentration of the fastest-burning oxidizer sorbed in organic sorbents that produces a non-oxidizer result. These results were used to inform experiments performed under the Los Alamos National Laboratory-Carlsbad Operation's (LANL-CO) Nuclear Quality Assurance-1 (NQA-1)-compliant program.
3. Based on these scoping studies, formal testing<sup>3</sup> was performed to determine a) the concentration of the fastest-burning oxidizer that produces a non-oxidizer result when sorbed in an organic or inorganic sorbent, b) the amount of zeolite needed to remediate either a pure oxidizing chemical or an organic sorbent with higher concentrations of the fastest-burning oxidizer, and c) the liquid holding capacity of individual inorganic sorbents.

### **4.2 EPA SW-846 Method 1040 Test**

The relative oxidizing capability of samples was measured using a modification of the EPA Test Methods for Evaluating Solid Waste, Laboratory Manual Physical Chemical Methods, SW-846 Method 1040 Test Method for Oxidizing Solids (hereafter referred to as the modified SW-846 Method 1040).<sup>4</sup> These modifications incorporate identified criteria such as moisture loss from the waste and will be discussed in detail in section 4.2.1. The SW-846 Method 1040 provides a qualitative means to measure the potential of a solid waste to increase the burning rate of a combustible substance, specifically fibrous cellulose. In this method, a 30-gram (g) conical pile of an oxidizing chemical mixed with cellulose is heated with an electrically energized wire, and the burning time of the sample is compared to a reference standard. The test method uses a set of reference standards with varying ratios of potassium bromate as the oxidizing chemical and cellulose as the fuel to categorize samples into a range of relative oxidizing

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<sup>1</sup> LANL-CO, Oxidizer Scoping Studies, DWT-RPT-001, April 12, 2017, LA-UR-16-28553.

<sup>2</sup> LANL-CO, Sorbent Scoping Studies, DWT-RPT-002, January 19, 2017, LA-UR-16-28806.

<sup>3</sup> LANL-CO, Results from Preparation and Testing of Sorbents Mixed with Potassium Nitrite, DWT-RPT-003, January 19, 2017, LA-UR-16-27276.

<sup>4</sup> Method 1040, Revision 0, February 2007, Final Update IV to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846.

strength. In the SW-846 Method 1040 test, a 3:7 ratio of potassium bromate to cellulose provides the reference standard with the longest burning time; samples with burn times longer than this reference standard or that do not ignite within three minutes are considered non-oxidizers.

#### 4.2.1 Modification to EPA SW-846 Method 1040

The SW-846 Method 1040 requires testing representative samples of wastes. Due to the heterogeneous nature of TRU waste and the variability of oxidizing chemical constituents in TRU waste across the complex, bounding conditions were defined for testing. Non-radioactive surrogates were used for testing due to inability to conduct burn rate tests on radioactive materials.

Additionally, several modifications were made to the SW-846 Method 1040 test to account for the intended use of the analytical results specific to the WIPP or to account for conditions in the laboratory. Each of these modifications is described below.

- The SW-846 Method 1040 requires drying of a sample at  $65 \pm 2$  °C for 12 hours. Instead for the modified SW-846 Method 1040, samples were dried at  $65 \pm 10$  °C until a constant mass was achieved, regardless of total drying time. This modification was made because moisture in TRU waste packages is not measured or intentionally maintained. Moisture present during packaging can evaporate from vented containers during transport and storage of waste. Because moisture can slow the burn rate of an oxidizer-fuel mixture, drying to constant weight bounds this parameter. Because drying time was removed as a parameter, control of the drying temperature was only needed to the degree to preserve the physical form of the sample, which is well below the boiling point of water. This allowed a relaxation in the control limits on the ovens.
- The SW-486 Method 1040 requires the sample to be cut, crushed, or ground so that the particle size of the sample to be tested is no larger than 0.5 millimeter (mm) (passes through a 32-mesh sieve). This could not be accomplished for the following materials: NoChar N910, NoChar N965, PIG Absorbent Mat Pad, Pig Pillows, Polyester Wipes, Universal all-purpose absorbent pillows. These samples tended to be rubbery and stick together so they would not pass through the 32-mesh sieve (NoChar N910, NoChar N965), or fibrous so that the fibers stretched resulting in a fluffy mass that could not be size-reduced further. These materials were size-reduced as much as possible, then used as is. Pads and wipes were cut into one-inch squares. Thirty gram samples were weighed out. The volumes of the samples were great enough so that a 60° glass funnel was too small to accommodate formation of the truncated cone. The samples were shaped by hand to approximate a truncated cone. The diameters of these sample bases were greater than the method target of 70 mm.

- The SW-846 Method 1040 requires each sample to be evaluated in 1:1 and 4:1 (by weight) waste to cellulose ratios. Since oxidizers by themselves or mixed with inorganic matter may not be combustible, the SW-846 Method 1040 specifies the addition of cellulose as the organic matter for testing. Many of the sorbing materials identified in active TRU waste streams are organic. During the scoping studies, it was found that adding cellulose to samples of an oxidizing chemical sorbed in EOPS or cellulose at the ratios specified by the method slowed the burn time because of the additional dilution of the oxidizing chemical. In order to present a more realistic and bounding condition, no additional fuel in the form of cellulose was added to samples of the fastest-burning oxidizer sorbed in organic sorbents. Testing a 30-g conical pile of these samples directly without adding cellulose provides a measurement of the fastest burn time. This was also the case for the remediation samples in which the strongest oxidizer was sorbed in the fastest-burning EOPS and sWheat Scoop.
- For SW-846 Method 1040, the burn rate test is repeated five times for each waste to cellulose ratio mixture. In the original SW-846 Method 1040, 100 g to 160 g of sample is mixed with cellulose to provide enough of the mixture for five tests and then 30-g aliquots are measured from this large batch for each burn test. In this study, the burn testing stations were located separately from the sample preparation area. To avoid transporting oxidizing chemicals mixed with fuel, inorganic samples and reference standards were prepared individually at the burn stations instead of measuring aliquots from a larger batch. When an oxidizing chemical had to be mixed with an organic sorbent (fuel), it was mixed in a batch and pretested in the sample preparation area and had to meet certain burn rate criteria prior to sending it to the burn stations. A Data Quality Objective (DQO) was developed to insure consistency of the samples.
- The SW-846 Method 1040 states that all tests must be conducted under standard test conditions, which include a temperature of  $20 \pm 5$  °C and a relative humidity of  $50 \pm 10\%$ . The testing was conducted in an existing facility that was temperature-controlled but not humidity-controlled. Rather than modify the facility, the following modification was made to SW-846 Method 1040: All burn rate testing of a specific sample must be conducted  $\pm 5$  °C and  $\pm 10\%$  of initial relative humidity from the conditions at which a 3:7 reference standard was last tested. (Note that temperature conditions never deviated from  $20 \pm 5$  °C, as prescribed by the method, during testing.) If conditions deviated from these ranges, a new reference standard was tested and the testing for that sample was repeated. This ensured that the sample was tested under the same environmental conditions as the reference standard.
- The SW-846 Method 1040 states that the ignition wire should be placed on a ceramic plate before the sample is added on top of the wire in a conical pile. In this configuration, there is a gap between the ceramic plate and the funnel

used to form the conical sample pile due to the ignition wire and insulator block attached to the wire. During the scoping studies, significant sample loss and loss of the conical shape due to sample leakage was observed when the ignition wire was placed before the sample was added. The SW-846 Method 1040 placement of the ignition wire was modified, for this study, to better preserve the conical pile consistency between aliquots. The funnel was inverted onto the ceramic plate with no ignition wire present, and the ignition wire was carefully pressed or slid into the cone of sample from the top or side, which better retained the cone shape and did not displace the pile base. For the samples that could not be size-reduced per the method, the samples were placed on top of the ignition wire.

- The SW-846 Method 1040 states that once the sample pile ignites, the power to the ignition wire is turned off. In the modified SW-846 Method 1040, the power to the wire was left on for 15 seconds even if ignition occurred sooner. Since ignition is not specifically defined in the SW-846 Method 1040, this modification was added to reduce the subjectivity of when ignition had taken place. If the sample ignited within 15 seconds, the ignition was turned off. If the sample did not ignite within 15 seconds, the wire remained energized until the analyst determined ignition had occurred or for at least 3 minutes, as directed in the unmodified SW-846 Method 1040.
- SW-846 Method 1040 classifies a solid waste into one of four categories of oxidizers based on the shortest mean burning time between the 4:1 and 1:1 oxidizer to cellulose samples. This allows the possibility for a non-oxidizer result to be obtained if any of the five individual burn rates used to calculate the mean burning time are shorter than the mean burning time for the 3:7 potassium bromate to cellulose reference standard. To ensure that the average burn time was in the non-oxidizer category, the following modification was made: A non-oxidizer result occurs when either the burn times for all samples and duplicate samples are greater than the 3:7 potassium bromate to cellulose reference standard, or the samples do not burn at all. If the first two aliquots of a sample or duplicate sample did not ignite within 3 minutes, the sample was labeled a non-oxidizer, and no further testing was required for the remaining aliquots for that sample or duplicate.

## 4.2.2 Quality Assurance

### 4.2.2.1 Formal Testing

Testing was performed under the LANL-CO's NQA-1-compliant Quality Assurance program, a *CBFO Quality Assurance Program Document* (CBFO-94-1012)-compliant program. Documentation of the chemicals and the measuring and test equipment (e.g., certificates of analysis, calibration, and conformance) were placed in the LANL-CO records center. All testing data were obtained using a test procedure developed from a CBFO-approved test plan.

The burn rates for individual burn rate tests performed on each sample and the mean burn rates for each series of burn rate tests were recorded on data sheets. The test plan, test procedure, test data sheets, and final data report documenting the results of testing are records maintained in the LANL-CO records center. A copy of the final data report was provided to CBFO for their records.

#### 4.2.2.2 Data Quality Objectives

SW-846 Method 1040 measures the burning times of waste mixtures, which are compared to burning times of various mixtures of potassium bromate and cellulose as reference standards to classify the waste mixtures into one of four categories. Categories I, II, and III are oxidizers, and Category IV is a non-oxidizer. Because many factors, such as particle size, reagent moisture content, room temperature, humidity, ventilation, position of the test sample in the hood, and the position of the ignition wire within the test pile, can impact the burning rates and method precision, it is essential that all steps be conducted in a consistent manner under uniform experimental conditions to obtain reliable and reproducible results. Therefore, the test plan specifies DQOs that define the acceptable level of uncertainty or variation in parameters that can affect the overall method accuracy or precision.

To ensure the environmental conditions are consistent for a set of burn rate measurements performed using the modified SW-846 Method 1040 approach, a DQO for room temperature and humidity was specified for the location of the test. All five aliquots for any sample must be tested within a range of 5 °C above or below the room temperature at which a 3:7 potassium bromate to cellulose reference standard was measured. Relative humidity of the room must remain within a range of 10% above or below the initial relative humidity at which the same 3:7 potassium bromate to cellulose reference standard was measured.

To ensure sample consistency and the proper concentrations of oxidizing chemicals and sorbents in samples, the DQO for weights of oxidizing chemicals, sorbents, and cellulose was  $\pm 0.05$  g. The DQO to establish dryness of the sample was a difference of  $\pm 2$  g between the final sample weight and the previous weight measurement. Each test sample batch was dried for 12 hours before the first weight measurement. Each test sample batch was dried for at least 4 additional hours between subsequent weight measurements. To ensure samples were dried at a consistent temperature, the DQO for drying oven temperature was  $65 \pm 10$  °C.

Finally, to ensure consistency between each burn rate measurement and to gauge the consistency of the procedures, a DQO for the range on the reference standard tests was established. For any given series of burn rates, a set of five aliquots of 3:7 potassium bromate to cellulose reference standard must be within a range of  $\pm 60$  seconds from the average result obtained, or the reference series was invalid and had to be repeated. A DQO for thermocouple measurements was also established to account for error in thermocouple readings so that the temperature of the ignition wire would not exceed 1050 °C or be less than 1000 °C.

### 4.3 Oxidizing Chemical and Sorbent Scoping Studies

Scoping studies were undertaken to identify the fastest-burning oxidizing chemical, to identify the fastest-burning EOPS, and to determine the approximate maximum concentration of the fastest-burning oxidizing chemical sorbed in organic sorbents found in TRU waste streams that produces a non-oxidizer result. These results were used to inform the formal experiments.

#### 4.3.1 Oxidizing Chemicals Scoping Study Planning

A review of the AK resulted in a list of oxidizing chemicals that were evaluated when planning the scoping studies. Some oxidizing chemicals were not tested in the scoping studies due to unsafe properties, the material by itself was unstable under the testing conditions or posed a significant possibility of violent reactions when mixed with cellulose, unsuitable physical forms (e.g., volatile liquids), or because radioactive oxidizing chemicals could not be burn-tested. These oxidizing chemicals are listed in Table 4-1.

**Table 4-1 – Oxidizers Excluded from the Scoping Study (unsafe properties and radioactive)**

Excluded Oxidizer	Reason for Exclusion	Reference
Americium nitrate	Radioactive oxidizing chemical	
Ammonium nitrate	Incidents involving explosive decomposition of aqueous solutions of ammonium nitrate have been recorded. Ammonium nitrate, with more than 0.2 percent combustible substances is an explosive. These conditions are present during scoping study testing.	a, b
Ammonium perchlorate	This powdered oxidant functions as an explosive when mixed with finely divided organic materials. These conditions are present during scoping study testing.	a, c
Ammonium permanganate	Dry ammonium permanganate is friction-sensitive and explodes at 60°C (140 °F) in air. Both of these properties are incompatible with the scoping study testing.	a
Barium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Bromine pentafluoride	This liquid oxidant may ignite or explode on contact with a combustible material, such as cellulose, as would be encountered in the scoping study testing. Reacts with all known elements, except nitrogen, oxygen, and rare gases. Gas at 41 °C (106 °F).	a, c
Bromine trifluoride	This liquid oxidant may ignite or explode on contact with a combustible material, such as cellulose, as would be encountered in the scoping study testing.	a, c
Calcium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c

Excluded Oxidizer	Reason for Exclusion	Reference
Ceric perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Chromium trioxide	Chromium trioxide may react violently upon contact with organic matter leading to ignition or explosion. It is expected that chromium trioxide would oxidize the organic sorbents that were tested in the scoping studies on contact.	a
Curium nitrate	Radioactive oxidizing chemical	
Dibenzoyl peroxide	Dibenzoyl peroxide is moderately sensitive to heat, shock, friction, or contact with combustible materials. It is known to explode when heated above its melting temperature of 103 °C (217 °F), as would be encountered in the scoping study testing.	a, c
Dibutyl peroxide (tertiary) (Di- <i>t</i> -butyl peroxide)	Decomposition of this peroxide at 165 °C (329 °F) resulted in a violent explosion. Samples will experience temperatures well beyond this during the burn rate measurements of the scoping study testing.	a, c
Europium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing. Radioactive oxidizing chemical.	a, c
Ferrous perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Gadolinium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Lead perchlorate	Metal perchlorates may react on contact with a combustible material, such as cellulose, and may become unstable in pure form upon dehydration. They may explode under exposure to heat or fire, as would be encountered in the scoping study testing.	a, c
Lithium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Magnesium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Neodymium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Neptunium Nitrate	Radioactive oxidizing chemical.	



Excluded Oxidizer	Reason for Exclusion	Reference
Perchloric acid	Perchloric acid is an oxidizing liquid. Perchloric acid has a potentially explosive reaction with combustible materials, such as those present during scoping study testing.	a
Plutonium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing. Radioactive oxidizing chemical.	a, c
Plutonium peroxide	Radioactive oxidizing chemical.	
Potassium chlorate	Contact of potassium chlorate with organic matter may result in fires or explosions, particularly if any solid materials are finely divided. These conditions are present during scoping study testing.	a, c
Potassium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c, d
Samarium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Silver perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing. Silver perchlorate is self-reactive and forms solid, explosive complexes with many hydrocarbons.	a, d
Sodium chlorate	Contact with organic matter may result in fires or explosions. It is expected that sodium chlorate will immediately react with organic sorbents on contact.	c
Sodium chlorite	Intimate mixtures of solid sodium chlorite with finely divided organic matter may be explosive and very sensitive to heat, impact or friction. These conditions will be present during scoping study testing.	a

Excluded Oxidizer	Reason for Exclusion	Reference
Sodium hypochlorite	The anhydrous solid is highly explosive and sensitive to heat or friction. These conditions will be present during scoping study testing. Most of the uses in the AK source documents show use of liquid sodium hypochlorite solutions in cooling water systems as fungicides and biocides. Some AK source documents identified sodium hypochlorite in sludge; reducing agents such as ferrous sulfamate and hydrochloric acid were also listed in the sludge. Sodium hypochlorite would not remain in the waste. One source document identified sodium hypochlorite solution used in laboratory processes for quantifying plutonium in urine. Sodium hypochlorite reacts with urea and uric acid in the urine and would not be in the waste. Sodium hypochlorite 5.25% solutions (Clorox and Clo-White bleach) were used for surface decontamination and would result in sodium chloride when the liquid evaporates. Sodium hypochlorite was included in the chemicals used in $^{238}\text{Pu}$ and $^{237}\text{Np}$ separations and would react rapidly with all metals and lower valent metal oxides. Sodium hypochlorite cannot persist in conditions found in TRU waste acceptable at the WIPP.	a
Sodium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c
Sodium permanganate monohydrate	If the combustible material is finely divided the mixture may be explosive. Sodium permanganate monohydrate spontaneously ignited when mixed with cellulose during scoping studies.	c
Sodium peroxide	Sodium peroxide when mixed with combustible materials is readily ignited by friction, heat, or contact with moisture. It may vigorously decompose under prolonged exposure to heat. These conditions will be present during scoping study testing.	a, c, d
Tetrabutylammonium perchlorate	Tetrabutylammonium perchlorate may explode under exposure to heat or fire. Contact with combustible/organic material may cause fire. These conditions are present during scoping study testing.	c, e
Tetraethylammonium perchlorate	Tetraethylammonium perchlorate may explode from heat, shock, or friction. These conditions are present during scoping study testing.	a, c
Tetramethylammonium perchlorate (Tetramethylamine perchlorate)	Tetramethylammonium perchlorate may explode under exposure to heat or fire, as would be encountered in the scoping study testing.	c
Thallium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c

Excluded Oxidizer	Reason for Exclusion	Reference
Vanadyl nitrate	Vanadyl nitrate is a very reactive oxidizer found in liquid form at room temperature. Vanadyl nitrate will ignite hydrocarbons and paper on contact. These conditions are present during scoping study testing.	a
Ytterbium perchlorate	Metal perchlorates may react on contact with combustible materials and may become unstable in pure form upon dehydration. These conditions are present during scoping study testing.	a, c

- <sup>a</sup>. Bretherick, L. (1990) Bretherick's Handbook of Reactive Chemical Hazards, 4th Ed., Boston, MA: Butterworths & Co. Ltd.
- <sup>b</sup>. U.S. Government Publishing Office (2016). 49 CFR§172.101, Electronic Code of Federal Regulations, retrieved from: <http://www.ecfr.gov>
- <sup>c</sup>. Center for Chemical Process Safety (2016). Chemical Reactivity Worksheet, Version 4.0, retrieved from: <http://response.restoration.noaa.gov/reactivityworksheet>
- <sup>d</sup>. Lewis, R. J. Sr. (1992). Sax's Dangerous Properties of Industrial Materials, 8th Ed., New York, NY: Van Nostrand Reinhold.
- <sup>e</sup>. Fisher Scientific Safety data sheet retrieved on May 10, 2016 from: <https://www.fishersci.com/shop/msdsproxy?productName=AC420141000&productDescription=TETRABUTYLAMMONIUM+PERCH+100GR&catNo=AC420141000+&vendorId=VN00033901&storeId=10652>

Table 4-2 lists the oxidizing chemicals that are not expected to be present in waste bound for the WIPP due to chemical reactivity or very low concentrations (~ 530 parts per million volume [ppmv] to below ppmv) based on AK records reviewed in February 2016. The very low concentration exclusion was applied based on source documentation for the waste stream AK revision identified in the table footnote for the subject oxidizing chemical. Concentrations of these oxidizing chemicals in the Waste Stream: OR-REDC-CH-HET, characterized in CCP-AK-ORNL-002, Rev. 4 (footnote I) must be reevaluated when the AK is revised since continued generation is projected.

**Table 4-2 – Oxidizers Excluded from the Scoping Study (identified in AK but not expected in waste, oxidizing gases, or very low ppmv)**

Withdrawn Oxidizer	Reason for Withdrawal	Reference
Antimony nitrate	Based on a synthetic feed formulation, oxidizing species of antimony are expected to be present in fission product waste streams up to 0.00003 M.	a, b, c, d, e
Bromine chloride	Bromine chloride is a very reactive oxidizer that reacts vigorously with combustible materials on contact. Liquid below 5 °C (41 °F) decomposes partly at its boiling point of 5 °C (41°F) and decomposes on contact with moisture. It is not expected to be in the waste because it would have reacted.	f
Hydrogen peroxide	Hydrogen peroxide as a ≥ 20 wt. % to ≤ 40 wt. % solution is a very reactive oxidizer normally found as a solution. Incompatible with combustible materials, strong acids, strong bases, metals, and strong reducing agents. In pure or diluted form, explosive mixtures are formed upon contact with organic compounds. When hydrogen peroxide solution is dried or left open to the atmosphere, it will decompose producing oxygen and heat. Breaks down quickly when exposed to light.	g

Withdrawn Oxidizer	Reason for Withdrawal	Reference
Nitric oxide	Nitric oxide (NO) is a gas. When nitric oxide is exposed to the atmosphere, it will decompose to form innocuous reaction products. Therefore, it is not expected to be present in the waste.	h
Nitrous acid	Nitrous acid (HNO <sub>2</sub> ) quickly disproportionates into nitric acid and nitric oxide. Therefore, nitrous acid is not expected to be present in the waste. Nitrous acid forms stable, water-soluble nitrites with Na, K, and Ag, which are included in the scoping study testing.	h
Palladium nitrate	Palladium (II) nitrate is currently only found in three waste streams (OR-CHEM-CH-HET, OR-RADP-CH-HET, and OR-SWSA-CH-SOIL), as described in AK Reports CCP-AK-ORNL-003, CCP-AK-ORNL-005, and CCP-AK-ORNL-009. According to source document P969 for CCP-AK-ORNL-005, palladium nitrate solution was shipped offsite for the production of medical sources. According to source document U406 (referenced by all three waste streams), palladium nitrate was found in small volume (40 mL) stock solutions used as a synthetic fission product feed. Palladium metal remained at approximately 0.04 wt. % in simulated fission product waste streams. Palladium metal did not dissolve in nitric acid, nitric acid with hydrofluoric acid, and hydrochloric acid. Palladium prefers to remain in metal form rather than converting to palladium nitrate salt.	c, d, e, i
Rhodium nitrate	Rhodium nitrate is currently found in one debris waste stream, OR- RADP-CH-HET. The original solution described in the AK Report, CCP-AK-ORNL-003, was at extremely low concentrations, 0.00121 M. The reprocessing of fuel separates fission products before the recovery of uranium and plutonium. Rhodium nitrate was not tested because it is a fission product that has been separated from actinides and is not in TRU waste.	c
Ruthenium nitrate <i>should have been identified as</i> Ruthenium III nitrosyl nitrate	Ruthenium nitrate does not exist. Ruthenium (III) nitrosyl nitrate is the only form of ruthenium that is nitrated. The reprocessing of mixed-oxide fuel separates fission products before the recovery of uranium and plutonium. Ruthenium (III) nitrosyl nitrate was not tested because it is a fission product that has been separated from actinides and is not in TRU waste.	c, j, k
Tellurium nitrate	Tellurium nitrate was found in only four waste streams from two sources. In the first use, it was converted to tellurium chloride by the addition of HCl. This would have removed the tellurium nitrate from the waste streams. In the second identified use, tellurium nitrate was produced in concentrations of 0.00142 M as part of a separations feed solution.	c, d, l, m
Tin nitrate	Tin nitrate is currently only found in one debris waste stream, OR-RADP-CH-HET. The original solution described in the AK Report CCP-AK-ORNL-003 was at extremely low concentrations, 0.00013 M.	c

<sup>a</sup>. Various vendors were explored for sourcing antimony (III) nitrate (Cole-Parmer, Fisher Scientific, Sigma-Aldrich, Strem Chemical, and VWR). None sell this material. A similar material was found during the search of the literature: antimony (III) oxide hydroxide nitrate (Marceau, E. et al. (1996). Synthesis and Thermal

- Decomposition of Antimony (III) Oxide Hydroxide Nitrate, *Journal of Thermal Analysis*, 46, pp.27-37).
- b. Berry, F. J. et al. (1983). Studies of Antimony Oxides Formed by Dehydration of Antimony Suspensions in Nitric acid, *Inorganica Chimica Acta*, 83(1984), pp. 167-169.
  - c. Ramirez, M. (2014). Central Characterization Program Acceptable Knowledge Summary Report for Oak Ridge National Laboratory Radiochemical Processing Research and Development Contact-Handled Transuranic Waste, Waste Stream: OR-RADP-CH-HET, CCP-AK-ORNL-003, Rev.3, Carlsbad, NM: Central Characterization Program.
  - d. Ramirez, M. (2014). Central Characterization Program Acceptable Knowledge Summary Report For Oak Ridge National Laboratory Solid Waste Storage Area 5 North 7802N Trench Area Contact-Handled Transuranic Waste, Waste Stream: OR-SWSA-CH-SOIL, OR-SWSA-CH-HET, CCP-AK-ORNL-009, Rev.1, Carlsbad, NM: Central Characterization Program.
  - e. Source Document U406, Ramirez, M. (2014). Central Characterization Program Acceptable Knowledge Summary Report for Oak Ridge National Laboratory Radiochemical Processing Research and Development Contact-Handled Transuranic Waste, Waste Stream: OR-RADP-CH-HET, CCP-AK-ORNL-003, Rev.3, Carlsbad, NM: Central Characterization Program.
  - f. Center for Chemical Process Safety (2016). Chemical Reactivity Worksheet, Version 4.0, retrieved from: <http://response.restoration.noaa.gov/reactivityworksheet>
  - g. Conner, W. V. (1993). Hydrogen Peroxide Safety Issues, RFP-4599, U.S. Department of Energy, EG&G Rocky Flats.
  - h. Budavari, S. et al. (Eds.). (1996). *The Merck Index*, 12th Ed., Whitehouse Station, NJ: Merck & Co., Inc.
  - i. Ramirez, M. (2016). Central Characterization Program Acceptable Knowledge Summary Report For Oak Ridge National Laboratory Contact-Handled Transuranic Waste from Analytical Chemistry Laboratory Operations Waste Stream OR-CHEM-CH-HET, CCP-AK-ORNL-005, Rev.3, Carlsbad, NM: Central Characterization Program.
  - j. Swain, P. et al. (2013). Separation and Recovery of Ruthenium: A Review, *Journal of Radioanalytical and Nuclear Chemistry*, 298(2), pp.781–796.
  - k. Burch, W. D. et al. (1977) LMFBR Fuel Reprocessing Program Progress Report For Period January 1 To March 31, 1977, ORNL/TM-5879, Oak Ridge, TN: Oak Ridge National Laboratory.
  - l. Ramirez, M. (2016). Central Characterization Program Acceptable Knowledge Summary Report For Oak Ridge National Laboratory Radiochemical Engineering Development Center Contact-Handled Transuranic Waste, Waste Stream: OR-REDC-CH-HET, CCP-AK-ORNL-002, Rev. 4, Carlsbad, NM: Central Characterization Program.
  - m. Joo, I. (2016). Central Characterization Program Acceptable Knowledge Summary Report for Oak Ridge National Laboratory Radiochemical Engineering Development Center Remote-Handled Transuranic Waste Stream: OR-REDC-RH-HET, CCP-AK-ORNL-500, Rev.5, Carlsbad, NM: Central Characterization Program.

### 4.3.2 Oxidizer Scoping Studies

A series of modified SW-846 Method 1040 tests were performed in which the burning times of various oxidizing chemicals were compared to the burning time of a reference standard. Potassium nitrite was determined to be the fastest-burning oxidizing chemical likely to be present in TRU waste. Scoping studies also confirmed that mixtures of two oxidizers result in an oxidizer that has an intermediate burn rate when compared under identical conditions to the burn rates of the two individual oxidizing chemicals (i.e., there is not a synergistic effect from the binary combinations of oxidizing chemicals tested). Based on these results, potassium nitrite was selected as the bound oxidizing chemical to use for testing. Potassium nitrite only bounds those oxidizing chemicals listed in Table 5-1.

### 4.3.3 Sorbent Scoping Studies

Sorbents identified for testing were mixed with an oxidizing chemical and tested to determine the fastest-burning organic sorbent and compare its effectiveness as a fuel to that of cellulose.

Waste Lock 770, a sodium polyacrylate, and Quik-Solid, a cross-linked polyacrylate, had faster average burn times than the cellulose standard. However, the average burn times of both polyacrylates were not significantly different from the average burn times of cellulose. Therefore, cellulose was chosen as the fuel in the majority of the testing in order to more closely adhere to the EPA SW-846 Method 1040. To bound the remediation portion of the formal testing, in which zeolite is added to mixtures to render them non-oxidizers, Quik-Solid, an equivalent fuel and superior sorbent, was selected instead of cellulose.

### 4.3.4 Selection of Wet Mixing vs. Dry Mixing

An earlier study on oxidizing chemicals by the Energetic Materials Research and Testing Center found that the burn rate of a sample of oxidizing chemical dry-mixed with a sorbent was slower than the same oxidizing chemical sorbed, or wet-mixed, onto the sorbent.<sup>5</sup> This trend was measured and demonstrated in the scoping studies and confirmed in the formal testing. As a result, all of the samples except the remediation of dry potassium nitrite and the reference standards were prepared by dissolving potassium nitrite and adding the solution to the sorbent. The resulting average burn time of a sample prepared in this manner will be faster and therefore more bounding than the same sample where the dry potassium nitrite powder was mixed with the sorbent. For example, zeolite (4 angstrom [Å] pore size), when wet-mixed with 35 wt. % potassium nitrite, is a non-oxidizer. When zeolite (4 Å pore size) is dry-mixed with 50 wt. % potassium nitrite, it is a non-oxidizer.

## 4.4 Oxidizer Testing

Testing was performed to determine 1) the liquid holding capacity of individual inorganic sorbents, 2) the concentration of the fastest-burning oxidizing chemical sorbed with an organic or inorganic sorbent that produces a non-oxidizer result, and 3) the amount of zeolite needed to remediate a pure oxidizing chemical or oxidizing chemical mixed with an organic sorbent.

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<sup>5</sup> G. Walsh, Results of Oxidizing Solids Testing, EMRTC FR 10-13, Energetic Materials Research and Testing Center, New Mexico Institute of Mining and Technology, Socorro, New Mexico, April 12, 2010.

#### 4.4.1 Methods for Formal Oxidizer Testing

There are three parts to the tests that were performed:

1. Liquid holding capacity tests, in which inorganic sorbent samples were mixed with potassium nitrite solutions, then subsequent burn rate testing of these samples, as described in section 4.4.1.1;
2. Preparation of carbohydrate sorbents and EOPSs with potassium nitrite solutions, then subsequent burn rate testing, as described in section 4.4.1.2;
3. Determination of the concentration of zeolite required to remediate pure potassium nitrite salt and 3:2 mixtures of potassium nitrite and sorbents, as described in section 4.4.1.3.

##### 4.4.1.1 Liquid Holding Capacity Tests and the Preparation of Inorganic Sorbent Samples

In the liquid holding capacity tests, potassium nitrite was dissolved in a near saturated solution (75 wt. %  $\text{KNO}_2$ ) of deionized water. The solution was dispensed on three or more portions of the as-received inorganic sorbent. Liquid was dispensed on the portions just until the material was fully saturated and free liquid was observed. The dispensed volume of solution was used to determine the liquid holding capacity and the amount of potassium nitrite in each portion of sorbent. These portions were then combined into a single sample, dried to constant weight, and size-reduced per the modified SW-846 Method 1040.

A safety burn rate check was required by the LANL-CO test procedure and hazard control plan before transporting samples to burn rate testing stations. The burn rate check was performed on a single 30-g conical pile at a 4:1 ratio of sample to cellulose and a single 30-g conical pile at a 1:1 ratio of sample to cellulose. If either the 4:1 or 1:1 conical burned in 90 seconds or less, a new sample was prepared using a less concentrated solution of potassium nitrite. This cycle was repeated until both ratios of the sample had a burn rate greater than 90 seconds. When both the 4:1 and 1:1 conical piles either had a burn rate longer than 90 seconds or had an incomplete burn for the safety check, then the sample was sent for burn rate testing. If the sample tested as a non-oxidizer during burn rate testing, a duplicate sample was prepared using the same concentration and amount of solution. The sample and duplicate were sent to different burn rate test stations for modified SW-846 Method 1040 testing. The oxidizing chemical concentration was reduced, as needed, when a sample or duplicate produced an oxidizer result at the burn rate test station using the modified SW-846 Method 1040 test. Results were reported at the highest potassium nitrite concentration that produced a non-oxidizer result.

Testing was curtailed on some sorbents for the following reasons:

- When potassium nitrite solution was added to Spill-X-C, which contains 10 to 20 percent fumaric acid, a reaction was observed (vigorous generation of colorless gas).
- Petroset II was immiscible and did not sorb the potassium nitrite solution.
- Ascarite, which contains sodium hydroxide, and sodium metasilicate were deliquescent at 65 °C and could not be dried to constant weight.

#### **4.4.1.2 Potassium Nitrite to EOPS and Carbohydrate Sample Preparation**

Carbohydrate and EOPS samples were also prepared in duplicate and provided to different burn rate test stations for modified SW-846 Method 1040 testing. Initially, samples of 30 wt. % potassium nitrite to 70 wt. % EOPS or carbohydrate sorbent were prepared by sorbing a potassium nitrite solution onto the sorbent, drying the mixture to constant weight, and size-reducing the sample per the modified SW-846 Method 1040. The initial ratio of potassium nitrite to EOPS was based on modified SW-846 Method 1040 test results from the scoping studies, which found that at 40 wt. % potassium nitrate some EOPSs tested as oxidizers, but all EOPSs with 30 wt. % potassium nitrite (the fastest burning oxidizing chemical) tested as non-oxidizers.

The safety burn rate check, as described in section 4.4.1.1, was performed on a single 30-g conical pile with no addition of cellulose. The  $\text{KNO}_2$  concentration was reduced as needed when 30 wt. % potassium nitrite burned in 90 seconds or less in the safety burn rate check or produced an oxidizer result when tested using the modified SW-846 Method 1040 test. Sample results were reported at the highest potassium nitrite concentration that produced a non-oxidizer result.

#### **4.4.1.3 Remediation Samples**

Three types of samples were tested to determine the amount of zeolite necessary to remediate for a non-oxidizer result: dry potassium nitrite, potassium nitrite sorbed in Quik-Solid, and potassium nitrite sorbed in sWheat Scoop.

The 3:2 potassium nitrite to Quik-Solid and 3:2 potassium nitrite to sWheat Scoop samples were prepared via wet mixing. These mixtures and pure potassium nitrite were dried to constant weight, size-reduced per the modified SW-846 Method 1040 test, and mixed with zeolite that was also dried and size-reduced (initially 40 wt. % zeolite). Each mixture was prepared and tested with both 4 Å pore size zeolite and 10 Å pore size zeolite, respectively.

For the pure potassium nitrite salt mixed with zeolite, the safety burn rate check and burn rate testing (described in section 4.4.1.1) was performed on a 30-g conical pile of 4:1  $\text{KNO}_2$  to cellulose and a 30-g conical pile of 1:1  $\text{KNO}_2$  to cellulose. For the 3:2 potassium nitrite to Quik-Solid and 3:2 potassium nitrite to sWheat Scoop samples, no cellulose was added, and the safety burn rate check was performed on a 30-g conical



pile of the sample. When a sample had a burn rate of 90 seconds or less in the safety burn rate check or produced an oxidizer result when tested using the modified SW-846 Method 1040 test, additional zeolite was added in increments of 10 wt. % until a non-oxidizer result was obtained.

#### **4.4.2 Results of the Testing**

Results of the testing were provided to CBFO in DWT-RPT-003, Results from Preparation and Testing of Sorbents Mixed with Potassium Nitrite. The criteria for waste acceptance and treatment of waste are in part based on the results of the formal testing.

### **5.0 CRITERIA FOR EVALUATING TRU WASTE WITH OXIDIZING CHEMICALS**

#### **5.1 Evaluating Oxidizing Chemicals Using CBFO Form 3589-1**

The WIPP Certified Program's AKEs must evaluate waste containing one or more oxidizing chemicals to the criteria in sections 5.1 through 5.11 using the most current revision of CBFO Form 3589-1, Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria, and include required attachments.

CBFO Form 3589-1 with attachments provides a record of the oxidizing chemical evaluation and identifies containers of waste within a waste stream or waste stream subpopulation that met the BoK criteria as-is and those requiring additional evaluation or treatment before the waste can be determined acceptable.

When the TRU waste site addresses the issues with waste previously rejected, the AKE will document actions by the TRU waste site on the checklist and attach copies of CBFO approvals of actions implemented by the sites to complete the evaluation. The checklist with attachments shall be signed and dated by the AKE and WIPP Certified Program Site Project Manager (SPM) then submitted to the BoK Review Board. The completed checklist with attachments and the BoK Review Board documented determination will complete the AK record for the oxidizing chemical review using the BoK criteria.

#### **5.2 Oxidizing Chemical Verification**

The oxidizing chemicals identified in Table 5-1 are bounded by potassium nitrite testing addressed in section 4 when determining the allowed wt. % of oxidizing chemical in EOPS (Table 5-3), inorganic sorbents (Table 5-4), and when remediating previously sorbed oxidizing chemicals (Table 6-1). Waste streams with oxidizing chemicals, whether listed on Table 5-1 or not, must be reevaluated by the AKE to determine if oxidizing chemicals are actually present in the waste. The reevaluation must focus on the process chemistry where the waste originated. Actions that could change the oxidizing chemicals during or after the process include: reducing, neutralizing, rinsing, solidifying, drying, calcining, pyrolyzing, and others. If the waste has been repackaged, actions that would have affected the waste chemistry must be identified and evaluated, and additions to the waste must be accounted for.

If the AK reevaluation determines that an oxidizing chemical is present in the TRU waste stream and not listed in Table 5-1, the TRU waste site or AKE may provide information to the CBFO Office of the Manager showing that the oxidizing chemical is bounded by at least one oxidizing chemical listed on Table 5-1. The CBFO Office of the Manager will evaluate the documented position and provide a written decision for the AK record. If the CBFO determines the oxidizing chemical is not bounded by at least one of the oxidizing chemicals listed in Table 5-1, then the TRU waste site must select an applicable option from section 2.0.

**Table 5-1 – Oxidizing Chemicals Bounded by Formal Testing**

Oxidizing Chemicals Bounded by Formal Testing	
Aluminum nitrate nonahydrate	Potassium iodate
Ammonium cerium (IV) nitrate	Potassium nitrate
Ammonium persulfate	Potassium nitrite
Barium nitrate	Potassium periodate
Bismuth (III) nitrate pentahydrate	Potassium permanganate
Cadmium nitrate	Potassium persulfate
Calcium hypochlorite	Praseodymium(III) nitrate hexahydrate
Calcium nitrate tetrahydrate	Rhenium nitrate hexahydrate
Cerium (III) nitrate hexahydrate	Rubidium nitrate
Cesium nitrate	Samarium(III) nitrate hexahydrate
Chromium nitrate nonahydrate	Silver nitrate
Cobalt nitrate hexahydrate	Silver nitrite
Copper nitrate trihydrate	Silver (I) oxide
Erbium (III) nitrate pentahydrate	Silver (II) oxide
Europium (III) nitrate pentahydrate	Sodium bromate
Gadolinium (III) nitrate hexahydrate	Sodium chromate
Indium nitrate tetrahydrate	Sodium dichromate dihydrate
Iron (III) nitrate nonahydrate	Sodium nitrate
Lanthanum (III) nitrate hexahydrate	Sodium nitrite
Lead (II) nitrate	Strontium nitrate
Lead peroxide	Terbium(III) nitrate pentahydrate or hexahydrate
Lithium hypochlorite	Tetrabutylammonium nitrate
Lithium nitrate	Tetrapropylammonium nitrate
Magnesium nitrate*	Thallium(I) nitrate trihydrate
Mercury (II) nitrate monohydrate	Thallium(III) nitrate trihydrate
Neodymium (III) nitrate hexahydrate	Thorium nitrate hexahydrate
Nickel (II) nitrate hexahydrate	Uranyl nitrate hexahydrate
Nitric acid	Yttrium(III) nitrate hexahydrate
Plutonium nitrate pentahydrate	Zinc nitrate hexahydrate
Potassium bromate	Zirconium(IV) nitrate pentahydrate
Potassium chromate	Zirconium oxynitrate
Potassium dichromate	

\*Magnesium nitrate is hygroscopic and deliquescent readily converting to magnesium nitrate hexahydrate when exposed to moisture. Magnesium nitrate hexahydrate is the most stable form of magnesium nitrate salt.<sup>6</sup> Magnesium nitrate hexahydrate is not regulated as a U.S. Department of Transportation (DOT) hazardous material per Special Provision 332 in 49 CFR §172.102. Testing performed by LANL-CO during the oxidizer scoping studies further supports that magnesium nitrate hexahydrate does not enhance the combustion of organic matter.

<sup>6</sup> Wheeler, R. C., Frost, G. B., "A Comparative Study of the Dehydration Kinetics of Several Hydrated Salts," Can. J. Chem., **33** (1955), 546-561.

### 5.3 Distribution of Oxidizing Chemicals Within Waste Components

TRU waste with oxidizing chemicals may consist of a single waste component or multiple waste components. Only the waste components with oxidizing chemicals require evaluation using the criteria in the BoK. Personnel performing the enhanced AK process must determine how well the oxidizing chemicals are distributed as well as the concentration of oxidizing chemicals within each waste component that contains oxidizing chemicals.

### 5.4 Neutralization of Oxidizing Acids, Bases, and Solutions

Oxidizing acids and aqueous solutions with oxidizing chemicals in containers and as free liquids separated from the solid portion of the waste generated or treated and repackaged after the issue date of the BoK shall be neutralized. Neutralization to a pH of 6 to 8 is desired as a best business practice, but the waste is acceptable at the WIPP if the oxidizing acids and aqueous solutions with oxidizing chemical have a pH measured  $>2$  to  $< 12.5$  before or after neutralization.<sup>7</sup> Waste shall be treated using methods for determining the Resource Conservation and Recovery Act (RCRA) corrosivity characteristic as specified in 40 CFR §261.22 or equivalent state regulation.

Neutralization of strong acids and bases is necessary for the following reasons:

- The reduction of acid-catalyzed and base-catalyzed chemistry reduces the effectiveness of oxidizing chemicals
- Strong acids and bases can break down the mineral structure of zeolites and other inorganic sorbents,<sup>8</sup> and the organic “backbone” of some EOPs, affecting the capacity of the sorbent and possibly rendering the oxidizing chemical treatment ineffective
- Compatibility of the waste with other waste, packaging materials, and the payload container is substantially increased
- The potential for hydrogen gas formation from corrosion processes is minimized
- The LANL-CO formal testing was conducted using neutral salts

Potentially explosive compounds can form when organic neutralizing or buffering agents such as citric acid or triethanolamine are used with oxidizing chemicals. AKEs shall identify and evaluate the use of organic neutralizing or buffering agents with oxidizing

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<sup>7</sup> The catalytic action of strong acids has long been known to be dependent on the strength of the acid. See for example the following references cited in C.A. Krause, “The Properties of Electrically Conducting Systems, including Electrolytes and Metals,” American Chemical Society Monograph Series, Issue 7, 1922 : Ostwald, *J. prakt. Chem.* **28**, 449 (1883); **29**, 385 (1884); **31**, 307 (1885). Goldschmidt and Thueson, *Ztschr. j. phys. Chem.* **81**, 30 (1913). Dawson and Powis, *J. Chem Soc.* **104**, 2135 (1913). Taylor, *Meddel K. Vet.-Akad’s. Nobclinstitut.* Vol 3, No.1 (1913); Ramstedt, *ibid.*, Vol 3, No. 7 (1915).

<sup>8</sup> G. Jozefaciuk and G. Bowanko, “Effect of Acid and Alkali Treatments on Surface Areas and Adsorption Energies of Selected Minerals,” *Clays and Clay Minerals*, Vol. 50, No. 6, 771-783, 2002.

acids and bases, and acidic and basic solutions. This evaluation applies prior to sorbing and when the organic neutralizing or buffering agents were ingredients in inorganic sorbents and EOPS products. When either case exists, testing must be performed that demonstrates that the sorbed waste does not pose a hazard when exposed to mechanical impact, spark, friction, and/or heat. Testing results shall be provided to the CBFO Office of the Manager. When testing determines the waste does pose a hazard, the TRU waste site must identify a method of treatment that will eliminate the hazard. Concurrence on the proposed method of treatment must be obtained from the CBFO Manager to assure acceptability at the WIPP following treatment.

## 5.5 Organic Sorbents

Organic sorbents and sorbing material such as rags, wipes, sorbent pads, and pillows that have been used in TRU waste can be divided into the following groups:

- EOPSSs, also known as hydrogels and super-absorbing polymers (polyacrylates, polyacrylamides, etc.)
- Polyols (organic molecules containing multiple hydroxyl functional groups, which includes synthetic and naturally occurring carbohydrates such as polyvinyl alcohol, rayon, cellulose, starch, etc.)
- Condensation polymers such as polyesters
- Hydrocarbons (polypropylene, polystyrene resins, etc.)

Mixtures of polyol materials identified in Table 5-2 and oxidizing chemicals are incompatible, and mixing them can result in adverse reaction consequences. **Polyol organic sorbents with oxidizing chemicals are not acceptable at the WIPP without treatment (see section 6).**

Potentially explosive compounds can form when organic neutralizing or buffering agents such as citric acid or triethanolamine are used with oxidizing chemicals. Treated mixtures of polyol sorbents, organic neutralizing or buffering agents, and oxidizing chemicals shall be tested to determine if the waste poses a hazard when exposed to mechanical impact, spark, friction, and/or heat. Documentation of the testing methods or protocols employed and the results of testing demonstrating that the waste does not pose a hazard shall be provided to the CBFO Office of the Manager for an acceptability determination. Acceptability determinations will be provided in writing by the CBFO Manager. When testing determines the waste does pose a hazard, the TRU waste site must identify a method of treatment that will eliminate the hazard. Concurrence on the proposed method of treatment must be obtained from the CBFO Manager to assure acceptability at the WIPP following treatment.

**Table 5-2 – Polyol Organic Sorbents that Require Treatment**

Sorbent Name	Composition listed in the Safety Data Sheet
Slikwik	100% processed corncobs (cellulose)
sWheat Scoop	100% Wheat (70-89 dry wt. % carbohydrates)
Carbohydrate sorbents not otherwise specified (N.O.S.)	A composition containing cellulose, starches, or sugars.
Polyols N.O.S	Polymeric alcohols with multiple free hydroxyl groups such as polyvinyl alcohols, and rayons

### 5.5.1 Organic Sorbents With Oxidizing Chemicals

Table 5-3 lists the wt. % of oxidizing chemical allowed when well mixed in a tested EOPS. An oxidizing chemical can be considered well mixed in the EOPS when at least one of the following criteria is met:

- EOPS was added to the liquid; or
- A known liquid is added in a volume approaching the sorbing capacity of the EOPS for that liquid; or
- Liquid is stirred or mixed with the EOPS

**Table 5-3 – Wt. % of Oxidizing Chemicals Allowed in EOPS (Previously Sorbed)**

EOPS	Composition listed in the Safety Data Sheet	Wt. % of oxidizing chemicals allowed
Aquasorb	Cross linked copolymer of acrylamide and potassium acrylate	≤ 31
NoChar N910 and A610	Thermoplastic elastomer (Copolymer of styrene, butadiene and possibly acrylates and phthalates)	≤ 30
NoChar N960 and A660	Copolymer of acrylamide	≤ 32 ≤ 40 (metal nitrates only*)
NoChar N965	Mixture containing 60% N910 and 40% N960	≤ 31
Quik-Solid	Sodium polyacrylate (lightly cross-linked)	≤ 33
Universal Polypropylene	Polypropylene	≤ 30
Waste Lock 770	Sodium polyacrylate, crosslinked	≤ 31
Waterworks SP400	Acrylic acrylate resin	≤ 32

\* Excludes silver nitrate (AgNO<sub>3</sub>) and lithium nitrate (LiNO<sub>3</sub>)

Waste shall be evaluated to determine if the oxidizing chemical concentration is below the wt. % in Table 5-3. Sum the dry weight of each of the oxidizing chemicals and divide by the cumulative sum of the weights of the sorbents and oxidizing chemicals to

yield the concentration of oxidizing chemicals in the waste. If the weights of either the oxidizing chemicals or the sorbents are not known, it may be possible to perform bounding calculations for the oxidizing chemical concentration using information such as solubility of the oxidizing chemical and the EOPS sorbing capacities for their products that are specific to the chemical(s) being sorbed (typically, information such as ionic strength and pH of the solution must be provided). Examples of how bounding calculations can be used are shown in section 5.6.1. When oxidizing chemical concentrations are not known and cannot be bounded, the TRU waste site may select an appropriate option listed in section 2.0 or the mixture can be treated as 100 wt. % oxidizing chemicals.

The addition of more organic sorbent to achieve the allowable oxidizing chemical concentration is not acceptable. When dried to constant mass, EOPSs are fuels, and potassium nitrite (the bounding oxidizer) concentrations above those listed in Table 5-3 accelerated their burn rates when tested. The sorbent scoping studies burn test showed that 40 wt. % potassium nitrate in NoChar N960 burned slower than the 3:7 potassium bromate and cellulose standard. When the TRU waste with oxidizing chemicals contains only metal nitrates (excluding silver nitrate  $\text{AgNO}_3$  and lithium nitrate  $\text{LiNO}_3$ ), 40 wt. % of oxidizing chemical concentration in NoChar N960 or A660 is acceptable.

EOPSs have been used to sorb oxidizing chemical solutions for more than 15 years without a reported self-ignition event. While EOPSs and carbohydrate sorbents are organic compounds, they react with oxidizing chemicals differently. The oxidizing chemical to fuel concentrations that produced non-oxidizer results in burn rate tests were approximately 20 wt. % less for the carbohydrate sorbents than for the EOPSs. Color changes were observed in some EOPSs during sample preparation for burn testing, indicating that some chemical changes were occurring. These potential chemical changes were represented in the samples tested and the burn rates observed for these samples were virtually the same as the burn rates for EOPS samples with no observed color changes.

Oxidizing chemical wastes sorbed in EOPSs not found in Table 5-3 are not acceptable for disposal at WIPP until the following criterion is met:

- The TRU waste site or AKE has provided information to CBFO showing that the sorbent is equivalent to one of the sorbents listed by name in Table 5-3 and requested an equivalency determination from the CBFO. A written response from the CBFO Office of the Manager documenting the CBFO's determination result will be provided to the TRU waste site and the AKE for inclusion in the AK record.

When equivalency is determined, 30 wt. % oxidizing chemical to EOPS will be the approved acceptance criteria. If the CBFO determines the EOPS is not equivalent, TRU waste sites must select an applicable option other than sorbent equivalency listed in section 2.0.

## 5.5.2 Organic Rags, Wipes, Sorbent Pads, and Pillows

There have been numerous fires across the DOE complex attributed to concentrated<sup>9</sup> and even diluted<sup>10</sup> solutions of nitric acid or nitrate salt solutions on cellulose rags resulting from decontamination activities or spill cleanup. Historically, cellulose and strong iodizing agents such as nitric acid were the second leading materials involved in spontaneous combustion events at the Savannah River Site (SRS).<sup>11-12</sup> Spontaneous combustion events involving fuel and oxidizing chemicals at the SRS typically resulted after absorbent cloth pads were used for leak or spill cleanup and then bagged for subsequent disposal.<sup>13</sup>

A white paper was written<sup>14</sup> by Gilbert and Venneman disputing the assignment of the RCRA ignitability (D001) and reactivity (D003) characteristics to the Rocky Flats “Wet Combustibles” waste stream managed at the Idaho National Environmental and Engineering Laboratory Radioactive Waste Management Complex. The D001 hazardous waste number had been assigned to the waste stream due to the presence of nitric acid soaked cotton rags and paper wipes. The authors provide a discussion negating the possibility for self-ignition of cellulosic materials and plastic (personal protective equipment, non-lead and leaded glovebox gloves, polypropylene, polyvinyl chloride, Teflon, Tygon, rubber, latex and other items) contaminated with nitric acid and resulting nitrate salts after 20 to 30 years of storage. This position is most likely valid if the salts were in contact with cellulose for the entire length of storage. They go on to speculate that cellulose nitrate would have to result from cellulose rags and wipes used in glovebox decontamination with 6 M nitric acid or possibly the formation of lead nitrate for these materials to spontaneously ignite. They also believe that spontaneous ignition would have to occur immediately after contacting cellulose with nitric acid. They did not consider other chemical pathways that could lead to self-ignition or that waste containing nitrate salts can exhibit the ignitability characteristic without igniting. They did not provide any substantiating data to support this claim other than a discussion on internet searches performed in 2001.

Defense Nuclear Facilities Safety Board (DNFSB) staff investigated a fire that occurred on May 6, 2003, inside Building 371, Glovebox 8, at the Rocky Flats Environmental

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<sup>9</sup> See for example, SRS Separations Incident report SI-86-1-11, which involved spontaneous ignition of 64 wt. % nitric acid soaked waste resulting from spill cleanup activities.

<sup>10</sup> See for example, SRS Separations Incident report SI-82-3-11, which involved spontaneous ignition of 3M (18%) Nitric acid contaminated swipes which had been used to decontaminate a sampler box and discarded without neutralization or rinsing.

<sup>11</sup> Science Applications International Corporation, “Safety Analysis-200 Area Savannah River Plant FB-Line Operations,” DPST5A-2 SUPP-9, April 1988, pg. 5-7. See AK Summary report SRS-FB-Line RH FBL.01, source document D020, “Safety Analysis-200 Area Savannah River Plant FB-Line Operations.”

<sup>12</sup> W. Durant, et al., “Adverse Experiences with Nitric Acid at the Savannah River Site,” WSRC-TR-91-22, Revision 1, June 1991

<sup>13</sup> Science Applications International Corporation, “Safety Analysis-200 Area Savannah River Plant FB-Line Operations,” DPST5A-2 SUPP-9, April 1988, pg. 359. See AK Summary report SRS-FB-Line RH FBL.01, source document D020, “Safety Analysis-200 Area Savannah River Plant FB-Line Operations,” pg 359

<sup>14</sup> K.L. Gilbert and T.E. Venneman, “A Review of U.S. Environmental Protection Agency Hazardous Waste Numbers to Selected Wastes Stored at the INEEL RWMC”

Technology Site (RFETS). DNFSB staff concluded that decontamination materials (cerium nitrate solution and rags) that might not have been neutralized and reduced were placed in the glovebox and likely contributed to the cause of the fire.<sup>15</sup> The possibility of having similar incidents at Hanford during decontamination and decommissioning work spurred Pacific Northwest National Laboratory (PNNL) and Fluor to investigate the safety impacts of a variety of decontamination methods. Scheele, et al.<sup>16</sup> examined cerium nitrate, RadPro®, ceric ammonium nitrate with Glygel organic surfactant, and Aspigel decontamination products. These decontamination products were tested with cotton rags, synthetic rags, and vacuum cleaner filters. In a set of tests, cerium nitrate was reduced then neutralized with sodium hydroxide. Cotton rags (86:14 cotton to polyester) were wetted with the resulting 1.25 M sodium nitrate solution and air dried for 24 hours. Accelerating rate calorimetry (ARC) testing conducted at 2 days and 14 days showed self-reacting exothermic behavior with thermal onset at ambient temperatures. A test performed at 114 days showed an unacceptably low thermal onset temperature (70 °C). When aged to 149 days, testing showed an increased thermal onset temperature at an acceptable range (>95 °C). Testing of these cotton rags was not continued after 149 days. Synthetic rags (80:20 polyester to polyamide) containing 1-6% (0.2 M – 1 M) sodium nitrate aged 4 days showed higher (acceptable) ARC thermal onset temperature than cotton rags, but showed exothermic behaviors at unacceptably low temperatures (≤95 °C) after 304-day aging. No further testing was done after 304 days. This report provides too few data points to draw definite conclusions. The report does not disclose how samples were maintained during the testing period. No duplicate tests were performed. This report does indicate that low initiation temperature thermal reactions may occur with low nitrate concentrations in cotton rags and possibly some synthetic rags.

L. Peppers and D. Saiki of EG&G Rocky Flats conducted differential scanning calorimetry and thermogravimetric analysis (DSC/TGA) tests of cellulose towels and wipes at RFETS in 1995. The site had become concerned about storing cellulosic towels and wipes that had been used to clean gloveboxes with nitric acid.<sup>17</sup> RFETS prepared two sets of cellulose wipes by folding each wipe and soaking it in 12M nitric acid. One set was maintained at room temperature, the other was heated to 50 °C. The wipes maintained at room temperature were soaked for 1, 7, 30, and 90 days. Those maintained at 50 °C were soaked for 10 and 31 days. The wipes were air dried after removal from the nitric acid bath then placed in a 50 °C oven for an additional 2 to 3 days. The cellulose wipes soaked at room temperature were tested and showed the lowest onset temperature at 7 days. The onset temperatures observed were 275 °C (1 day), 268 °C (7 days), 330 °C (30 days), and 335 °C (90 days). The cellulose wipes

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<sup>15</sup> H. Massie, DNFSB Staff Issue Report Memorandum to J. K. Fortenberry, October 29, 2003, re: Glovebox Fire at Rocky Flats Environmental Technology Site.

<sup>16</sup> R.D. Scheele, et al., "Thermal Stability Studies of Candidate Decontamination Agents for Hanford's Plutonium Finishing Plant Plutonium Contaminated Gloveboxes," PNNL-15410, September, 2005, Pacific Northwest National Laboratory, Richland, Washington.

<sup>17</sup> L.G. Peppers and D.M. Saiki, "Evaluation of the Reactivity/Flammability of Cellulosic Wipes Before and After Exposure to 12 Normal Nitric Acid," MST94-003, EG&G Rocky Flats, Golden, Colorado, 1995.



soaked at 50 °C had observed thermal onset temperatures of 325 °C (10 days) and 363 °C (31 days). In contrast, 100% cotton towels soaked in 12M nitric acid at room temperature for 18 days had an observed thermal onset at 140 °C. This temperature is outside of the Center for Chemical Process Safety DSC/TGA safety range for process safety published in *Guidelines for Chemical Reactivity Evaluation and Application to Process Design*. That is, the observed thermal onset temperature of 140 °C would require the 100% cotton towels to remain below 40 °C (104 °F) at all times to ensure an exothermic reaction would not occur.

Using Fourier transform infra-red spectroscopy, Peppers and Saiki identified the products of cellulose breakdown but did not observe nitrocellulose formation in the cellulose wipes. Even though carboxylation products were identified, they failed to account for the high heats of formation for these oxidation products which could lead to thermal runaways. They concluded that cellulose wipes soaked in 12 M nitric acid for up to 90 days at room temperature and 31 days at 50 °C did not present a fire hazard. These tests were focused on determining the presence or absence of nitrocellulose, without the presence of mixed acids, rather than assessing the potential for thermal runaways.

The Scheele report was challenged in 2009<sup>18</sup> by R. Marusich of **CH2MHILL** Plateau Remediation Company, who characterized Scheele's data interpretation as overly conservative. Some of the assumptions by Marusich regarding heat transfer within the waste matrix appear to be incorrect and may lead to erroneous conclusions. For example, Marusich concluded that 1/3 of the waste in a modeled drum could reach a thermal onset temperature of 70 °C, but since 2/3 of the waste did not, no exothermic reaction could occur. In addition, he assumed that drum breaching would not occur at pressures up to 38 psig (pounds per square inch gauge). These assumptions are overly optimistic from a WIPP perspective. The relevance to this BoK is 1) the lower than previously recognized concentration of oxidizing chemicals in cotton rags that may initiate an exothermic reaction, and 2) the observation of reduced thermal onset for aged nylon and polyester rags. It should be noted that reduced thermal onset for aged nylon and polyester rags was observed by Scheele in a single ARC test. However, the potential for this type of reaction is also identified in *Bretherick's Handbook of Reactive Chemical Hazards* and is attributed to transesterification of organic ester to nitrate esters.<sup>19</sup> Further characterization and study to better understand the effects of oxidizing chemicals and acids on cellulosic and synthetic towels, wipes, and other sorbent products is warranted.

Throughout the approximately 15 years of characterizing and certifying TRU waste prior to the February 14, 2014, radiological release event in the WIPP underground, no oxidizing chemical and organic material combustion events were reported to have occurred during repackaging or remediation of legacy waste at the TRU waste sites.

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<sup>18</sup> R.M. Marusich, "Analysis of the Reactivity of RadPro Solution with Cotton Rags," CHPRC-00308, CH2MHILL, Richland, Washington, August 11, 2009.

<sup>19</sup> L. Bretherick, *Bretherick's Handbook of Reactive Chemical Hazards*, 4<sup>th</sup> Ed, Boston, MA, Butterworths & Co., Ltd, 1990, pg 1672.

During nitrate waste treatment and repackaging operations at the Los Alamos National Laboratory (LANL) Waste Characterization Reduction and Repackaging Facility (WCRRF), indications of chemical incompatibility (e.g., heating and gas generation) were observed, but not acted upon. Organic neutralizing agents and carbohydrate sorbent(s) were added to nitrate waste in the WCRRF. No thermal runaways have been observed or reported in the 671 other containers with very similar waste constituents that were packaged in the WCRRF. Approximately 500 of those were sent to the WIPP, 120 to Waste Control Specialists, and the rest are in storage at LANL. Drum number 68660, determined to be the source of the 2014 radiological release, is the only container that has been visually observed as showing evidence that a significant thermal event had taken place within the container.

Requiring treatment for all legacy waste containers having rags, wipes, sorbent pads, and pillows cannot be justified without AK documentation that these specific waste components are likely to contain oxidizing chemicals.

When AK indicates the potential for nitrate contaminated rags, wipes, sorbent pads, and pillows containing cellulose, samples of the headspace gas may be collected and analyzed for hydrogen ( $H_2$ ), carbon dioxide ( $CO_2$ ) and nitrous oxide ( $N_2O$ ) to determine if a chemical oxidation reaction is occurring inside the container.

The presence of  $CO_2$  and  $N_2O$  oxidation gases is a reliable indicator that a nitrate and cellulose reaction is occurring. The converse is also true. In cases where it is known that cellulose rags or other cellulose containing sorbent materials were soaked with nitric acid or nitrate solutions prior to packaging, the absence of oxidation products in the headspace can definitively establish that the nitrate and fuel mixture has been consumed.

An approximately 3:1 ratio of  $CO_2$  to  $N_2O$  has been seen for mixtures of sWheat Scoop cat litter and nitrate salts being monitored at LANL. The observed ratio does not vary significantly with temperature, but the absolute concentrations of  $CO_2$  and  $N_2O$  are temperature dependent. A ratio of approximately 3:1  $CO_2$  to  $N_2O$  was also observed by PNNL when evaluating the reaction of cellulose with nitrate salts.

The ratio of  $H_2$  to  $CO_2$  can also be evaluated to provide additional confirmation that oxidizing chemicals are not reacting with organic constituents in the waste. The concentration of  $H_2$  will be greater than the  $CO_2$  concentration when chemical reactions are not occurring.

When headspace gas samples are collected and analyzed, the CBFO Office of the Manager will evaluate the measured concentrations of  $H_2$ ,  $CO_2$ , and  $N_2O$  gases. When the measured concentrations of chemical oxidation gases being generated are below threshold rates of concern, the CBFO can determine that containers with organic rags, wipes, sorbent pads, and pillows can be safely handled with no further sampling and analysis required. When oxidation gases are being generated at concentrations above threshold rates of concern, the TRU waste site must select an applicable option from

section 2.0. The result of this determination will be provided in writing for inclusion in the AK record.

### 5.5.3 Miscellaneous Organic Materials

Other organic wastes or materials containing oxidizing chemicals are known to exist at some of the TRU waste sites. Some of these are in the TRU waste inventory and others will enter the TRU waste inventory. An example of a homogenous organic waste is ion exchange resins with various nitrate loadings. Known materials expected to become TRU waste are spent organic solvents with nitric acid and metal nitrate salts resulting from separations processes, and nitrate salts with glycerin or sugar solutions. Ion exchange resins with oxidizing chemicals that are stabilized with portland cement are not oxidizers when the resins are well mixed in the cement and do not exceed 10 wt. % in the set cement monolith. When the cement monolith is intact based on visual observation, it is acceptable to conclude that the 10 wt. % limit for the ion exchange resins has been met. Ion exchange resins in excess of 10 wt. % tend to fracture the cement due to swelling.<sup>20</sup>

Ion exchange resins that do not meet the criteria above, organic solvents, and other miscellaneous organic materials containing oxidizing chemicals are not acceptable at the WIPP without a verifiable basis that can be used to determine the waste will be safe and compliant for receipt and emplacement in the WIPP. A verifiable basis may include the results of testing or other information that can be confirmed. Due to the potential to form mechanical impact, spark, friction, and/or heat sensitive compounds when some oxidizing chemicals and organics are mixed, the CBFO may require additional testing beyond the testing of oxidizing chemicals to determine acceptability.

## 5.6 Inorganic Materials With Oxidizing Chemicals

### 5.6.1 Oxidizing Chemicals Sorbed in Inorganic Sorbents

Table 5-4 provides the maximum concentration of oxidizing chemical acceptable at the WIPP when it is sorbed in a listed inorganic sorbent. An oxidizing chemical can be considered well mixed in the inorganic sorbent when at least one of the following criteria is met:

- Inorganic sorbent is added to the liquid; or
- A known liquid is added in a volume approaching the liquid holding capacity of the inorganic sorbent; or
- Liquid is stirred or mixed with the inorganic sorbent.

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<sup>20</sup> G. Veazey and R. Ames, "Cement Waste Form Development for Ion-Exchange Resins at the Rocky Flats Plant," LA-13226-MS, March 1997, Los Alamos National Laboratory

**Table 5-4 – Inorganic Sorbents, Allowable-Oxidizing Chemical Concentrations, and Liquid Holding Capacities**

Sorbent Name	Composition listed in the Safety Data Sheet	Wt. % of oxidizing chemicals allowed	Measured Liquid Holding Capacity (mL/g sorbent)
Absorb-N-Dry	Fuller's Earth 90-100% or Bentonite calcined 90-100% and Quartz <10%	≤ 28	0.683
Aquaset	Sodium montmorillonite	≤ 27	0.263
Aquaset II	Sepiolite	≤ 45	1.44
Aquaset II-G	Sepiolite	≤ 36	1.33
Celite S	Kieselguhr (a diatomaceous earth)	≤ 36	3.07
ChemOil-Away	Volcanic ash ≥98% organic material ≤2%	≤ 13	0.853
Drierite	Calcium sulfate	≤ 29	0.843
Oil-Dri	Bentonite 90-100%	≤ 38	1.06
Plaster of Paris	Calcium sulfate hemihydrate	≤ 24	0.931
Portland cement (when used as a dry sorbent)	Portland Cement	≤ 20 *	0.611
Spill-X-A	Magnesium oxide 60-100% Attapulgite 7-13% Sodium carbonate 5-10%	≤ 33	0.605
Totalsorb**	> 99% expanded Amorphous Alumina Silicate	≤ 36	1.51
Zeolite (10 Å pore size)	Zeolite	≤ 44	1.12
Zeolite (4 Å pore size)	Zeolite	≤ 35	1.15

\* The wt. % of oxidizing chemical allowed for portland cement does not apply to ion exchange resins with oxidizing chemicals (see section 5.5.3) and wet mixed and set cement (see section 5.10).

\*\* CBFO correspondence CBFO:ONTP:JRS:PG:17-0695:UFC 5900.00 dated June 20, 2017. Key words: BoK Approval, LA-MHD04.001, Type 1, WCS.

Waste shall be evaluated to determine if the oxidizing chemical concentration is below the wt. % of oxidizing chemical allowed in Table 5-4. Sum the dry weight of each of the oxidizing chemicals (exclude the weight of the waters of hydration) and divide by the cumulative sum of the weight of the sorbents and oxidizing chemicals (including the weight of the waters of hydration) to yield the concentration of oxidizing chemicals in the waste.

If the weights of either the oxidizing chemicals or the sorbents are known, it may be possible to perform bounding calculations for the oxidizing chemical concentration using the solubility of the oxidizing chemical and the inorganic sorbent liquid holding capacity. Examples of how bounding calculations can be used are shown below.

Example 1

The oxidizing chemical (sodium nitrate [NaNO<sub>3</sub>]), sorbent name (Aquaset II), and volume of NaNO<sub>3</sub> solution sorbed (2 gallons) are known but the concentration of NaNO<sub>3</sub> in the solution and the weight of Aquaset II are unknown.

## Step 1

Determine the weight in grams of NaNO<sub>3</sub> in the solution using NaNO<sub>3</sub> solubility of 87.6 g/100 mL in water at 20 °C and 2 gallons for the volume of solution sorbed.

$$\mathbf{NaNO_3 \text{ solubility} \times vol. \text{ sorbed} = wt. \text{ of } NaNO_3}$$

So

$$\frac{87.6 \text{ g } NaNO_3}{100 \text{ mL}} \times \frac{3785 \text{ mL}}{\text{gallon}} \times 2 \text{ gallons } NaNO_3 \text{ solution sorbed} = 6631 \text{ g } NaNO_3$$

## Step 2

Determine the weight in grams of Aquaset II required to sorb 2 gallons of NaNO<sub>3</sub> solution using the measured liquid holding capacity of 1.44 mL per gram of Aquaset II as listed in Table 5-4.

$$\frac{\mathbf{volume \text{ of } NaNO_3 \text{ solution sorbed}}}{\mathbf{measured \text{ liquid holding capacity of Aquaset II}}} = \mathbf{weight \text{ of Aquaset II}}$$

So

$$\frac{2 \text{ gallons } NaNO_3 \text{ solution} \times \frac{3785 \text{ mL}}{\text{gallon}}}{\frac{1.44 \text{ mL } NaNO_3 \text{ solution}}{\text{g Aquaset II}}} = 5257 \text{ g Aquaset II}$$

## Step 3

Calculate the wt. % of NaNO<sub>3</sub> in Aquaset II using the weight of NaNO<sub>3</sub> from Step 1 and the weight of Aquaset II from Step 2.

$$\frac{g \text{ NaNO}_3}{g \text{ NaNO}_3 + g \text{ Aquaset II}} \times 100 = \text{wt. \% NaNO}_3$$

So

$$\frac{6631 g \text{ NaNO}_3}{6631 g \text{ NaNO}_3 + 5257 g \text{ Aquaset II}} \times 100 = 56 \text{ wt. \% NaNO}_3$$

**Conclusion:** The NaNO<sub>3</sub> is at a concentration above the ≤ 45 wt. % allowed at WIPP in Aquaset II and must either be treated or have tests performed to show NaNO<sub>3</sub> at this concentration produces a non-oxidizer result.

### Example 2

Multiple concentrations of NaNO<sub>3</sub> solutions were added to the inorganic sorbent Celite S. The concentration of each individual NaNO<sub>3</sub> solution is known, however the volume of each solution sorbed is not known. The most concentrated solution sorbed was 6 molar NaNO<sub>3</sub> so it will be used to bound the NaNO<sub>3</sub> solutions sorbed. The solutions were sorbed in 4200 g of Celite S inorganic sorbent.

#### Step 1

Determine the total volume of the sorbed NaNO<sub>3</sub> solutions using the most concentrated (bounding concentration) of 6 molar NaNO<sub>3</sub> and the measured holding capacity for Celite S.

$$\text{wt. of sorbent} \times \text{measured liquid holding capacity of sorbent} = \text{volume of solution}$$

So

$$4200 g \text{ Celite S} \times \frac{3.07 \text{ mL solution}}{g \text{ Celite S}} = 12,894 \text{ mL total volume sorbed of 6 molar NaNO}_3$$

#### Step 2

Determine the weight of NaNO<sub>3</sub> solutions sorbed in Celite S using the total volume of 6 molar NaNO<sub>3</sub> solution determined in Step 1.

$$\text{total volume of 6 molar NaNO}_3 \text{ solution sorbed} \times \text{concentration of NaNO}_3 \text{ solution} = \text{bounding weight of NaNO}_3 \text{ salt in sorbed solutions}$$

So

$$12,894 \text{ mL total volume sorbed of 6 molar NaNO}_3 \times \frac{6 \text{ moles NaNO}_3}{1,000 \text{ mL}} \times \frac{85 \text{ g NaNO}_3}{\text{mole}} \\ = 6,576 \text{ g bounded weight of NaNO}_3$$

### Step 3

Calculate the wt. % of NaNO<sub>3</sub> salt sorbed in the known weight of 4200 g of Celite S using the bounded weight of NaNO<sub>3</sub> calculated in Step 2.

$$\frac{\text{g NaNO}_3}{\text{g NaNO}_3 + \text{g Celite S}} \times 100 = \text{wt. \% NaNO}_3$$

So

$$\frac{6576 \text{ g NaNO}_3}{6576 \text{ g NaNO}_3 + 4200 \text{ g Celite S}} \times 100 = 61 \text{ wt. \% NaNO}_3$$

**Conclusion:** The sorbed oxidizer is at a concentration above  $\leq 36$  wt. % wt. % allowed at WIPP in Celite S and must either be treated or have tests performed to show NaNO<sub>3</sub> at this concentration in Celite S produces a non-oxidizer result.

### Example 3

The concentrations of sodium nitrate, potassium nitrate, and calcium nitrate in solution are not known in an evaporator liquid waste stream. The solubility of sodium nitrate is 87.6 g/100 mL of water, the solubility of potassium nitrate is 33.0 g/100 mL of water, and the solubility of calcium nitrate is 129 g/100 mL of water. The process flow sheets identify sodium nitrate and potassium nitrate solutions as the predominant feed solutions sent to the evaporator. A way to bound the concentrations of oxidizer salts in the evaporator liquid waste stream is to identify the salt with the highest solubility between the predominant feed solutions. The liquid has been sorbed in 8 gallons of Aquaset II. The density of Aquaset II (sepiolite clay) is 2.00 g/cm<sup>3</sup>.

### Step 1

Determine the weight of the Aquaset II using the volume of sorbent used (8 gallons) and the density of the Aquaset II (sepiolite, 2.00 g/cm<sup>3</sup>).

$$\text{Volume of sorbent} \times \text{Density of sorbent} = \text{Weight of sorbent}$$

So

$$8 \text{ gal. of Aquaset II} \times \frac{3785 \text{ cm}^3}{1 \text{ gal.}} \frac{2.00 \text{ g Aquaset II}}{\text{cm}^3} = 60,560 \text{ g Aquaset II}$$

Step 2

Calculate the volume of solution sorbed from the liquid holding capacity of Aquaset II (1.44 mL/g) using the following equation:

$$\text{Sorbent's Liquid Holding Capacity} \times \text{Weight of sorbent} = \text{Volume of solution}$$

So

$$\frac{1.44 \text{ mL}}{\text{g Aquaset II}} \times 60,560 \text{ g Aquaset II} = 87,206 \text{ mL solution}$$

Step 3

Calculate the weight of the bounding weight oxidizer salts in the evaporator liquid waste stream.

$$\text{Solubility of the bounding salt} \times \text{Volume of solution} \\ = \text{Bounding weight of oxidizer salts in the evaporator liquid waste stream}$$

Of the two most predominant oxidizer salts in the feed solutions, sodium nitrate has the highest solubility (87.6 g/100 mL of water); therefore, it will be oxidizer salt used for the bounding calculation.

$$\frac{87.6 \text{ g NaNO}_3}{100 \text{ mL}} \times 87,206 \text{ mL solution} = 76,392 \text{ g of NaNO}_3$$

Step 4

Calculate the bounding wt. % of oxidizer salts sorbed in 8 gallons of Aquaset II.

$$\frac{\text{g bounding oxidizer salt}}{\text{g bounding oxidizer salt} + \text{g Aquaset II}} \times 100 = \text{bounding wt. \% of oxidizer salts}$$

Or

$$\frac{76,392 \text{ g NaNO}_3}{76,392 \text{ g NaNO}_3 + 60,560 \text{ g Aquaset II}} \times 100 = 56 \text{ wt. \% oxidizer salts}$$

**Conclusion:** The sorbed oxidizer salts exceed the  $\leq 45$  wt. % allowed at WIPP in Aquaset II and must either be treated or have tests performed to show  $\text{NaNO}_3$  at this concentration in Aquaset II produces a non-oxidizer result.



## Step 5

The TRU waste site has decided to add additional Aquaset II to this waste. Calculate the additional Aquaset II that must be added and mixed to bring the bounded oxidizer salts concentration to  $\leq 45$  wt. %. Use the wt. % of oxidizing chemicals allowed value for Aquaset II from Table 5-4, and the weights in grams of Aquaset II and the bounding oxidizer salt.

$$\left( \left( \frac{100}{\text{wt. \% oxidizing chem. allowed}} - 1 \right) \times g \text{ bounding oxidizer salt} \right) - \text{initial weight of sorbent} \\ = \text{weight of additional sorbent needed}$$

So

$$\left( \left( \frac{100}{45 \text{ wt. \%}} - 1 \right) \times 76,392 \text{ g NaNO}_3 \right) - 60,560 \text{ g Aquaset II} = 32,808 \text{ g Aquaset II}$$

For components of the waste with more than one inorganic sorbent, the maximum allowed concentration of oxidizing chemicals is determined by the lowest maximum concentration of any of the components. For example, for a mixture of Drierite and Zeolite (10 Å pore size), the maximum concentration of oxidizing chemicals allowed is 29 wt. %.

Waste containing inorganic sorbents that are not found in Table 5-4 are not acceptable for disposal at WIPP until the TRU waste site or AKE has requested an evaluation that the sorbent is equivalent to one of the sorbents listed by name in Table 5-4 and received a written determination from the CBFO Office of the Manager for the AK record. If the CBFO determines that the inorganic sorbent is not equivalent, TRU waste sites must select an applicable option listed in section 2.0.

When there is inadequate information to quantitate the concentration of oxidizing chemical, the waste must be treated as 100 wt. % oxidizing chemical with an inorganic sorbent listed in Table 5-4 using the corresponding allowable oxidizing chemical concentration (see section 6). Test data are collected using a CBFO approved test plan that demonstrates that the concentration in wt. % of potassium nitrite or bounded oxidizer found in the mixture to sorbents produces a non-oxidizer result (see section 7).

**NOTE: The CBFO will not grant equivalency for Hydromatrix, Micro-Cel® E, perlite, or vermiculite. These inorganic sorbents are not effective for treating oxidizing chemicals to produce non-oxidizer results and therefore they must be treated as 100% oxidizing chemical with zeolite or another inorganic sorbent when they contain oxidizing chemicals.**

## **5.7 Mixtures of Organic and Inorganic Materials With Oxidizing Chemicals**

When components of the waste have a mixture of inorganic and organic sorbents, the maximum allowed concentration of oxidizing chemicals is determined by the lowest concentration of any of the components. For example, for a mixture of Drierite and NoChar N910 in the same container, the maximum concentration of oxidizing chemicals allowed is 29 wt. %.

The wt. % of oxidizing chemicals is calculated by summing the dry weight of each of the oxidizing chemicals and dividing by the cumulative sum of the weights of the sorbents and oxidizing chemicals. If the concentration of oxidizing chemicals in the sorbents exceeds the maximum value listed in Table 5-3 or Table 5-4, the waste can be treated by adding more of the same inorganic sorbent or with another inorganic sorbent, such that the mixture criterion in the previous paragraph is satisfied.

Sorbents that are not found in Table 5-3 or Table 5-4 are not acceptable for disposal at WIPP until the TRU waste site or AKE has requested an equivalency determination and the CBFO has determined that the sorbent is equivalent to one of the sorbents listed in Table 5-3 or Table 5-4. If the CBFO determines that the EOPS or inorganic sorbent is not equivalent, TRU waste sites must select an applicable option listed in section 2.0.

Waste containing an EOPS sorbent that is not listed on Table 5-3 that also contains an inorganic sorbent that is listed in Table 5-4 may be treated to the wt. % of oxidizing chemical allowed for the inorganic sorbent without requesting an equivalency determination as long as the oxidizing chemical wt. % in the unlisted EOPS is known. When the oxidizing chemical concentration in the mixture is greater than 30 wt. % and the inorganic sorbent's allowable oxidizing chemical concentration is less than 30 wt. %, an EOPS equivalency determination is unnecessary as long as the waste is treated with additional inorganic sorbent to meet the allowable oxidizing chemical concentration specified in Table 5-4.

When the CBFO determines the sorbent is equivalent, the CBFO Manager will issue written acceptance criteria for that sorbent and the criteria will be included in a revision to this BoK.

## **5.8 Oxidizing Chemicals That Are the Sole Component of Waste**

Oxidizing chemicals that are the sole waste component(s) (e.g., metal nitrate salts with or without free liquid) are not acceptable for disposal at WIPP without treatment.

## **5.9 Inorganic Sludges with Oxidizing Chemicals Not Mixed with Sorbents**

Inorganic sludges containing oxidizing chemicals that are not mixed with sorbents have been generated from plutonium purification and other processes. As an example, chemical salt sludges resulted from reactions that included neutralization, flocculation, co-precipitation, and evaporation of chemical salts. Inorganic sludges with up to 20 wt. % oxidizing chemical that have not been mixed with sorbent are acceptable at the

WIPP, provided liquids are not present in excess of the limit specified in DOE/WIPP-02-3122, Rev. 8.0, *Transuranic Waste Acceptance Criteria for the Waste Isolation Pilot Plant*.

### **5.10 Oxidizing Chemicals Solidified in a Cement or Grout Matrix**

Oxidizing chemical liquids and particulate containing oxidizing chemicals are sometimes fixed in an inorganic form of cement or grout for disposal. Cement or grout containing oxidizing chemical shall be considered well mixed if the process involved mixing, stirring, or other manipulation of the cement or grout paste and oxidizing chemicals prior to setting. For purposes of this BoK, wastes with oxidizing chemicals, whether listed in Table 5-1 or not, that are cemented or grouted are not considered oxidizers when the following criteria are met.

- No more than 20 percent of the set material has external dimensions less than 2 centimeters; and
- The decomposition temperature of the oxidizing chemical is equal to or greater than 350 °C <sup>21</sup>; and
- No free liquid is present.

Inorganic cements and grouts meeting these criteria prevent the oxidizing chemical they contain from being activated with heat and do not require further treatment. Oxidizing chemicals with decomposition temperatures less than 350 °C must be evaluated by the CBFO in order to determine acceptability of the waste.

### **5.11 Surfaces Contaminated With Oxidizing Chemicals**

Waste components with low porosity and impermeable surfaces that have been exposed to liquid or solid oxidizing chemicals are surface-contaminated only. These types of waste components will not exhibit oxidizing behavior regardless of the distribution of the oxidizing chemicals when there is no observable adhesion of the oxidizing chemical to the surface.

### **5.12 Soils Containing Oxidizing Chemicals**

When developing criteria for evaluating oxidizing chemicals in soils for this BoK, zeolite (4 Å nominal pore size) was evaluated and tested as a surrogate for minerals in soils. Cellulose represented organic matter and organic waste in soil.

#### **5.12.1 Oxidizing Chemicals Spilled or Released Into Soils**

Soil organic content is well below 10 wt. % at all of the TRU waste sites with most having soils with organic content below 5 wt. %. Oxidizing chemical testing using the

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<sup>21</sup> LANL- CO, "Estimation of bounding particle size that ensures a non-oxidizer result using COMSOL," February 13, 2017, LA-UR-17-21079.

modified SW-846 Method 1040 always incorporates no less than 20 wt. % cellulose when testing the oxidizing chemical burn rate in an inorganic sorbent. Therefore, oxidizing chemicals in soils resulting from spills, leaks, and discharges will have organic content below those in the 4 Å zeolite sorbent samples tested by LANL-CO. Since 35 wt. % of oxidizing chemicals is allowed in 4 Å zeolite sorbent in Table 5-4, soils with total organic content  $\leq 20$  wt. % do not require treatment when the oxidizing chemical concentration is  $\leq 35$  wt. %.

### 5.12.2 Oxidizing Chemicals in Waste Retrieved From Earthen Disposal Pits

In the case of oxidizing chemicals retrieved from disposal pits where they were likely to have been co-mingled with organic waste, the maximum organic waste concentration and maximum oxidizing chemical concentration must be accounted for, as well as the minimum concentration of inorganic matrix required to mitigate the resulting oxidizer fuel mixture. This requires a total concentration of oxidizing chemicals to be  $\leq 35$  wt. %, the total concentration of organic material to be  $\leq 20$  wt. %, and the minimum concentration of soil to be  $\geq 45$  wt. %.

TRU waste sites with burial pits where oxidizing chemical salts were disposed must remove salt crystals other than small visible crystals that are impractical to pick out from the excavated soil/waste mixture. The dissolved salt in the soils plus the remaining visible crystals cannot exceed 35 wt. %.<sup>22</sup> Crystalline oxidizing chemical salts removed from the soil/waste mixture must be treated as oxidizing chemical salts according to the concentrations listed in Table 6-1, if the WIPP is the planned disposal location. Soil/waste mixtures remaining after observable crystalline oxidizing chemical salts have been removed must never be combined with soil/waste mixtures believed to contain organic wastes.

Potentially explosive compounds can form when process waste chemicals are discarded. Organic fuels such as cutting oils, reducing agents such as zero valent metals, and unstable process chemicals such as hydroxylamine nitrate are incompatible with oxidizing chemicals and each other. The BoK does not cover these other incompatibilities. TRU waste sites must ensure that these other incompatibilities are documented, evaluated, and safely managed to ensure that the waste does not pose a hazard when exposed to mechanical impact, spark, friction, and/or heat. BoK evaluations shall not be performed on this waste until after a chemical compatibility evaluation memorandum (CCEM) has been approved by the CBFO indicating that these incompatibilities will be addressed.

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<sup>22</sup> Kimmitt, R.R., Allowable Nitrate Salt Concentration, Engineering Design File, EDF-8723, Rev. 2., Idaho Cleanup Project, Idaho Falls, Idaho.

## 6.0 CRITERIA FOR TREATMENT OF WASTE CONTAINING OXIDIZING CHEMICALS

If the waste does not pass the evaluation criteria of section 5 and must be treated, the following treatments are acceptable based on the LANL-CO formal testing.

### 6.1 Treatment With Zeolite

Table 6-1 lists the final wt. % of oxidizing chemicals and zeolites that produces a non-oxidizer result in a homogeneous treated oxidizing chemical and organic sorbent mixture.

**Table 6-1 – Final wt. % of Zeolites Required to Treat Previously Sorbed Oxidizing Chemical and Organic Sorbent Mixtures**

Mixture to Remediate	Wt. % of 4 Å to 10 Å zeolite required in a treated homogeneous mixture
Oxidizing chemical in an EOPS	≥ 50
Oxidizing chemical in polyols, polyester, polyamide, carbohydrate (e.g., cellulose, sWheat Scoop, or SlikWik)	≥ 70

The Table 6-1 final wt. % of zeolites required is a minimum value determined from 3:2 mixtures of the bounding oxidizing chemical to Quik Solid® (the fastest-burning EOPS) and sWheat Scoop® without the addition of powdered cellulose fuel. The 3:2 mixture is the most aggressive burning mixture under the modified SW-846 Method 1040 test. Any other ratio of oxidizer to fuel will produce a slower burn time. The wt. % of 4 Å to 10 Å zeolite required is the minimum wt. % of zeolite that must be achieved in the final previously sorbed waste and zeolite treated homogeneous mixture. The final zeolite concentration of the treated homogeneous mixture is determined from the as-received weight of the zeolite and the weight of the previously sorbed mixture's dry weight.

The zeolite values in Table 6-1 cannot be compared to the zeolite values listed in Table 5-3 as those were derived from targeted 30%, 20%, and 10% bounding oxidizing chemical to organic sorbent step-down tests that stopped at the highest oxidizing chemical percentage that achieved a non-oxidizer result. These test samples were also tested without the addition of powdered cellulose fuel. Only the wt. % values for oxidizing chemical and zeolites from Table 6-1 can be used when treating oxidizing chemicals previously sorbed in polyol organic sorbents. If oxidizing chemicals are sorbed in EOPS, the TRU waste site may use any inorganic sorbent listed in Table 5-4 provided the wt. % oxidizing chemical allowed is met. Table 5-3 values are used to determine if oxidizing chemicals sorbed in EOPS are acceptable without treatment and to identify the threshold value where treatment is required.

For the purpose of the BoK, the treated mixture is considered well mixed when the zeolite is distributed evenly throughout the waste. It is the responsibility of the TRU waste site to ensure that the treatment process is demonstrated to produce thoroughly blended mixtures. Additionally, it is the responsibility of the TRU waste site to ensure that the selected treatment process is compliant with their safety and regulatory requirements.

## **6.2 Handling of Zeolite**

Zeolites are believed to be compatible with the oxidizing chemicals listed in Table 5-1. However, there are some documented chemical incompatibilities and observed properties of zeolite that should be noted for the TRU waste sites that may be problematic in handling zeolites for treatment of oxidizing chemical wastes.

Strong oxidizers that will attack glass will also attack zeolites. These include oxygen difluoride and other halogenated compounds that may not contain oxygen, such as fluorine, chlorine, and trifluoride. Several strong acids have been shown to break down the zeolite structure. Hydrochloric acid (HCl) can dissolve zeolites, while sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and hydrofluoric acid (HF) can cause pitting. The degree of dissolution depends on the ratio of Si-to-Al of the particular zeolite variety. Zeolites can also dissolve in strong alkaline hydroxides, such as sodium hydroxide (NaOH), but the hydroxide must be heated.

TRU waste sites handling zeolite should also be aware of its heat of immersion, which results in the zeolite heating upon being mixed with water. This was observed in the holding capacity studies as steam rising from the sample tray when the potassium nitrite solution was slowly added to the zeolite. Heats of immersion of zeolites have been reported as high as 375 J/g (joules per gram).

TRU waste sites treating waste with zeolite are encouraged to perform small-scale tests prior to initiating waste treatment with zeolite to determine if any of these properties need to be accounted for. TRU waste sites should not initiate treatment of oxidizing chemical waste unless the treatment can be performed safely and effectively.

## **6.3 Treatment of Previously Sorbed Oxidizing Chemicals and Organic Sorbents With Inorganic Sorbents Other Than Zeolites**

A TRU waste site may use an inorganic sorbent other than zeolite if test data are developed under the following condition:

- Data are collected using a CBFO approved test plan developed incorporating the criteria in section 7 to determine the wt. % of oxidizing chemical allowed for the initial waste mixture blended with the selected inorganic treatment sorbent. This is the wt. % concentration of oxidizing chemical in inorganic sorbent that produces a non-oxidizer result.

Test plans should be submitted to the CBFO Office of the Manager for approval. Approvals will be documented in writing by the CBFO Manager. Data shall be provided to the CBFO after validation by the TRU waste site. Once the data are approved by CBFO, acceptance criteria for that sorbent will be provided in writing and included in a revision to this BoK.

## **7.0 CRITERIA FOR TESTING WASTE CONTAINING OXIDIZING CHEMICALS**

Testing to determine the oxidizing chemical wt. % in sorbent, soil, or other waste that produces a non-oxidizer result shall be developed in accordance with EPA publication SW-846 entitled *Test Methods for Evaluating solid Waste: Physical/Chemical Methods, Chapter One*. The test plan must specify how the waste will be bounded if representative waste sample testing cannot be performed.

If SW-846 Method 1040, *Test Method for Oxidizing Solids*, is to be used, the method modifications specified in section 7.1 must be applied. The modifications made to the SW-846 Method 1040 test were put in place to account for the environmental conditions the waste could experience during shipment in unvented Nuclear Regulatory Commission-certified Type B packages, when received and managed in the WIPP surface storage facilities, and after emplacement in the WIPP underground until the waste is isolated from the underground ventilation air flow. DQOs will be established that are no less restrictive than those listed in section 7.2. Additional DQOs may be developed if they will add to the quality of the test results.

It is the responsibility of the TRU waste site to ensure that their testing can be implemented in compliance with site safety and regulatory requirements. The test plan shall be provided to the CBFO for review and approval. Copies of the following types of documents shall be provided to the CBFO Office of the Manager when testing is completed:

- Procurement procedures and records for purchases of quality affecting chemicals and equipment;
- Instrument calibration documents;
- Data validation and verification procedures and reports;
- Sample data sheets; and
- Data report or summary document

The CBFO Manager will issue written acceptance criteria when the results of testing are accepted. The issued criteria will be incorporated in a revision to this BoK.

Oxidizing chemicals that are likely to ignite or explode during sample preparation are not amenable to testing using the modified SW-846 Method 1040. Alternate test methods and treatments will have to be identified for these oxidizing chemicals. The CBFO will cooperate with the TRU waste sites in seeking an acceptable testing and treatment path for wastes such as these.

## 7.1 Modified Method 1040 Testing Approach

The SW-846 Method 1040 requires testing representative samples of wastes. Non-radioactive surrogates may be used for testing if a site is unable to conduct burn rate tests on radioactive materials.

Modifications shall be made to the SW-846 Method 1040 to account for the intended use of the analytical results specific to the WIPP or to account for conditions in the laboratory. The SW-846 Method 1040 modifications required or determined acceptable by the CBFO are described below.

- The SW-846 Method 1040 requires drying of a sample at  $65 \pm 2$  °C for 12 hours. The CBFO will approve modifications of the SW-846 Method 1040 to allow samples to be dried at 65 °C with temperature deviations of  $\pm 10$  °C. Drying until a constant mass is achieved is required, regardless of total drying time.
- The SW-846 Method 1040 requires the sample to be cut, crushed, or ground so that the particle size of the sample to be tested is no larger than 0.5 mm (passes through a 32-mesh sieve). Materials must be size-reduced as much as possible. If a particle size reduction to no larger than 0.5 mm is not possible, then the difference in size shall be accounted for by developing an alternative reference standard that can be compared to the SW-846 Method 1040 prepared reference standard.
- The SW-846 Method 1040 requires each sample to be evaluated in 1:1 and 4:1 (by weight) waste to cellulose ratios. Since most oxidizing chemicals alone or when mixed with inorganic sorbents are not combustible, the SW-846 Method 1040 specifies the addition of cellulose as the organic fuel for testing. Many of the sorbing materials identified in active TRU waste stream AK records are organic and are readily combustible when dried. Cellulose fuel should not be added to these samples because it will make the sample fuel rich and oxidizer lean. This condition will extend the observed burn times of the samples and it does not accurately represent the actual waste. Remediation samples containing organic sorbents shall be tested in 3:2 oxidizing chemical to organic sorbent portions without the addition of cellulose fuel.
- Per SW-846 Method 1040, the burn rate test is repeated five times for each waste to cellulose ratio mixture. Method 1040 also requires mixing 100 g to 160 g of sample with cellulose to provide enough of the mixture for five tests, and 30 g aliquots are measured from this large batch for each burn test. If these prepared sample quantities are believed to pose an undue safety risk, each 30 g sample may be prepared individually. DQOs addressing weighing accuracy and mixing times should be developed and implemented for consistency of the samples.
- The SW-846 Method 1040 states that all tests must be conducted under standard test conditions, which include a temperature of  $20 \pm 5$  °C and a relative humidity of  $50 \pm 10\%$ . If TRU waste sites are conducting testing in a facility that is



not temperature- or humidity-controlled, the following modifications may be made to the SW-846 Method 1040: Burn rate testing of a specific sample must be conducted at an initial recorded temperature with no more than  $\pm 5$  °C deviation and no more than  $\pm 10\%$  deviation from initial relative humidity at which a 3:7 reference standard was tested. If conditions deviate beyond these ranges, a new reference standard must be tested, and the testing for that sample repeated. DQOs that cover the environmental conditions must be written and monitored during testing to show the environmental conditions are not changing beyond those written in the unmodified SW-846 Method 1040.

- The SW-846 Method 1040 states that the ignition wire should be placed on a ceramic plate before the sample is added on top of the wire in a conical pile. In this configuration, there is a gap between the ceramic plate and the funnel used to form the conical sample pile due to the ignition wire and insulator block attached to the wire. The funnel shall be inverted onto the ceramic plate with no ignition wire present, and the ignition wire shall be carefully pressed or slid into the cone of sample from the top or side to better retain the cone shape without displacing the pile base. For the samples that could not be fully size-reduced per the method, the samples shall be placed on top of the ignition wire ensuring an air gap remains between ceramic plate and the ignition wire.
- The SW-846 Method 1040 states that once the sample pile ignites, the power to the ignition wire is turned off. SW-846 Method 1040 shall be modified to keep the ignition wire energized for 15 seconds, even if ignition occurs sooner, to reduce the subjectivity of when ignition takes place. If the sample ignites within 15 seconds, the ignition shall be turned off. If the sample does not ignite within 15 seconds, the wire shall remain energized until the analyst determines ignition has occurred or for at least 3 minutes, as directed in the unmodified SW-846 Method 1040.
- SW-846 Method 1040 classifies a solid waste into one of four categories of oxidizers based on the shortest mean burning time between the 4:1 and 1:1 oxidizer to cellulose samples. This allows the possibility for a non-oxidizer result to be obtained if any of the five individual burn rates used to calculate the mean burning time are shorter than the mean burning time for the 3:7 potassium bromate to cellulose reference standard. To ensure that the average burn time is in the non-oxidizer category, the following modification shall be made: A non-oxidizer result occurs when either the burn times for all samples and duplicate sample are greater than the 3:7 potassium bromate to cellulose reference standard, or the samples do not burn at all. If the first two aliquots of a sample or duplicate sample do not ignite within 3 minutes, the sample shall be labeled a non-oxidizer, and no further testing shall be required for the remaining aliquots for that sample or duplicate.

## 7.2 Formal Testing and Data Quality Objectives

The burn rates for individual burn rate tests performed on each sample and the mean burn rates for each series of burn rate tests shall be recorded on data sheets generated from the execution of a CBFO approved test plan.

Many factors, such as particle size, reagent moisture content, room temperature, humidity, ventilation, position of the test sample in the hood, and the position of the ignition wire within the test pile can impact the burn rates and method precision. It is essential that all steps be conducted in a consistent manner under uniform experimental conditions to obtain reliable and reproducible results. Therefore, the test plan shall specify DQOs that define the acceptable level of uncertainty or variation in parameters that can affect the overall method accuracy or precision.

To ensure the environmental conditions are consistent for a set of burn rate measurements performed using the modified SW-846 Method 1040 approach, a DQO for room temperature and humidity shall be specified for the location of the test. All five aliquots for any sample must be tested within a range of 5 °C above or below the room temperature at which the same 3:7 potassium bromate to cellulose reference standard is measured. Relative humidity of the room must remain within a range of 10% above or below the initial relative humidity at which the same 3:7 potassium bromate to cellulose reference standard is measured.

To ensure sample consistency and the proper concentrations of oxidizing chemicals and sorbents in samples, the DQO for weights of oxidizing chemicals, sorbents, and cellulose shall be  $\pm 0.05$  g. The DQO to establish dryness of the sample shall be a difference of  $\pm 2$  g between the final sample weight and the previous weight measurement. Each test sample batch shall be dried for 12 hours before the first weight measurement. Each test sample batch shall be dried for at least 4 additional hours between subsequent weight measurements. To ensure samples are dried at a consistent temperature, the DQO for drying oven temperature was  $65 \pm 10$  °C. Finally, to ensure consistency between each burn rate measurement and to gauge the consistency of the procedures, a DQO for the range on the reference standard tests shall be established. For any given series of burn rates, a set of five aliquots of 3:7 potassium bromate to cellulose reference standard must be within a range of  $\pm 60$  seconds from the average result obtained, or the reference series shall be invalid and must be repeated. A DQO for thermocouple measurements shall be established to account for error in thermocouple readings so that the temperature of the ignition wire does not exceed 1050 °C or fall below 1000 °C.

## 8.0 RECORDS

The following documents will be generated as necessary when implementing the BoK and shall be processed and maintained as Quality Assurance records in accordance with established CBFO records management procedures.

### CBFO records

- TRU waste site bounded oxidizing chemical request and CBFO bounded oxidizing chemical approvals with acceptance criteria
- TRU waste site sorbent equivalency requests and CBFO sorbent equivalency approvals with wt. % of oxidizing chemicals allowed
- TRU waste site test method approval requests and CBFO approvals
- TRU waste site test plans and CBFO approvals
- TRU waste site test data, CBFO review records, and CBFO issued acceptance criteria (when applicable)
- TRU waste site treatment plans and CBFO written approvals
- TRU waste site technical justification submittals and CBFO written approvals
- Completed CBFO Form 3589-1, Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria, with required attachments
- BoK Review Board's documented decision on the evaluation of oxidizing chemicals using the BoK by WIPP Certified Program's AKEs

The WIPP Certified Program AKE and SPM shall be included on electronic and hard copy distribution of CBFO responses to the above listed TRU waste site requests and submittals. The CBFO Assistant Manager for the Office of the National TRU Program (NTP), NTP Compliance Division Director, Office of Quality Assurance Director, and CBFO Waste Management Senior Technical Advisor shall be included on electronic distribution.

The BoK Review Board's documented decision on the evaluation of oxidizing chemicals using the BoK shall also be provided to the WIPP Certified Program AKE and SPM by electronic and hard copy distribution with electronic distribution to the BoK Review Board members.

### AK records

- Completed CBFO Form 3589-1, Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria, with required attachments, and the BoK Review Board's documented decision on the evaluation of oxidizing chemicals using the BoK.

## **Attachment I**

Example of CBFO Form 3589-1, Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria

**Example of CBFO Form 3589-1,  
Acceptable Knowledge Checklist for Evaluating Oxidizing Chemicals in TRU Waste Using the BoK Criteria**

Waste Stream Name: \_\_\_\_\_  
 Waste Stream Location: \_\_\_\_\_  
 AK Summary Report covering this waste stream: \_\_\_\_\_  
 AKE performing the evaluation: \_\_\_\_\_  
 Start date of the evaluation: \_\_\_\_\_  
 WIPP Certified Program SPM: \_\_\_\_\_  
 Input oxidizing chemical(s) identified by AK: \_\_\_\_\_

	Enter "Y" for yes or "N" for no	
	Y	N
Has this BoK evaluation been preceded by an approved CCEM?		
• Did the approved CCEM cover the entire waste stream?		
○ If no, did the CCEM cover a defined subpopulation of containers in the waste stream?		
• Does this BoK evaluation cover the entire waste stream?		
○ If no, identify the waste stream subpopulation covered by this BoK evaluation (list container I.D. numbers or attach list): _____		
_____		
_____		
• Are there differences between the BoK subpopulation and the subpopulation covered by the CCEM?		
○ If yes, explain differences between the BoK subpopulation and CCEM subpopulation and why they are different: _____		
_____		
_____		

Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.2 Oxidizing chemical verification</b>			
Has the AK been reevaluated to determine if identified input oxidizing chemical(s) is/are in the waste?			
<ul style="list-style-type: none"> <li>If yes, document the result of the AK reevaluation by describing each input oxidizing chemical(s) identified by AK that is not expected in the waste and why. _____            _____            _____</li> </ul>			
<ul style="list-style-type: none"> <li><b>If no, reevaluate the AK before proceeding.</b></li> <li>List the oxidizing chemical(s) in the waste based on the reevaluated AK. _____            _____            _____</li> </ul>			
Are the oxidizing chemicals listed in Table 5-1?			
<ul style="list-style-type: none"> <li>If no, record the oxidizing chemical(s) not listed in Table 5-1. _____            _____            _____</li> </ul>			
Has a bounding oxidizing chemical determination request been approved for each oxidizing chemical that is not listed on Table 5-1?			
<ul style="list-style-type: none"> <li>If yes, attach the bounding oxidizing chemical determination approval.</li> <li>If no, has the TRU waste site performed tests using a CBFO approved test plan and received acceptance criteria for the oxidizing chemical(s)?           <ul style="list-style-type: none"> <li>If yes, attach CBFO test plan approval and issued acceptance criteria (if the BoK has not been revised to include the criteria).</li> <li><b>If no, this evaluation cannot continue for affected containers until oxidizing chemical testing is completed using a CBFO approved test plan and the CBFO Manager has issued acceptance criteria for the oxidizing chemical(s).</b> <ul style="list-style-type: none"> <li><b>Attach list 5.2 of containers excluded per previous bullet.</b></li> </ul> </li> </ul> </li> </ul>			

Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.3 Distribution of oxidizing chemicals within waste components</b>			
Describe the waste component(s) containing the oxidizing chemical(s): _____			
_____			
_____			
<ul style="list-style-type: none"> <li>• Is the oxidizing chemical expected to be well distributed within the waste component? (sections 5.5.1, 5.6.1, 5.9, and 5.10)                             <ul style="list-style-type: none"> <li>○ If no, contact the CBFO Waste Management Senior Technical Advisor for assistance.</li> </ul> </li> </ul>			
<ul style="list-style-type: none"> <li>• Is the concentration of oxidizing chemical within the waste component known or bounded? (sections 5.5.1 and 5.6.1)                             <ul style="list-style-type: none"> <li>○ <b>If no, this evaluation cannot continue for affected containers until the TRU waste site selects an option listed in section 2.0 or treats the waste as 100 wt. % oxidizing chemical.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.3 of affected containers with oxidizing chemicals that are not well distributed within the waste component.</b></li> <li>▪ <b>Attach list 5.3.1 of affected containers with unknown concentrations of oxidizing chemicals within a waste component.</b></li> </ul> </li> </ul> </li> </ul>			
<ul style="list-style-type: none"> <li>• Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0,7, 5.5.1, or 5.6.1)</li> </ul>			
_____			
_____			
_____			
_____			
_____			

		Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.4 Neutralization of oxidizing acids, bases, and solutions</b>					
Was the oxidizing chemical an aqueous liquid or liquid oxidizing acid or base generated or treated and repackaged after the issue date of DOE/WIPP-17-3589? (section 5.4)					
<ul style="list-style-type: none"> <li>• If aqueous, was pH measured?</li> </ul>					
<b>If yes, list method used for measurement:</b> _____					
<b>If no, pH must be measured for affected waste before BoK evaluation can continue.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.4 of affected waste.</b></li> </ul>					
<ul style="list-style-type: none"> <li>• Was pH &gt; 2 and &lt; 12.5?</li> </ul>					
<ul style="list-style-type: none"> <li>○ If yes, neutralization or buffering is not required.</li> </ul>					
<ul style="list-style-type: none"> <li>○ <b>If no, the evaluation cannot continue until the affected waste is neutralized or the TRU waste site provides technical justification using option H of section 2, paragraph 2 of the BoK, and has received CBFO concurrence that the waste is acceptable without neutralization. Attach CBFO concurrence when applicable.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.4.1 of affected containers.</b></li> </ul> </li> </ul>					
List the neutralizing or buffering agent(s) manufacturer and product name: _____					
<ul style="list-style-type: none"> <li>• If liquid, has steel corrosion testing been performed?</li> </ul>					
<ul style="list-style-type: none"> <li>○ If yes, list method used for determination: _____</li> </ul>					
<ul style="list-style-type: none"> <li>○ <b>If no, steel corrosion test must be performed for affected containers before BoK evaluation can continue.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.4.2 of affected containers.</b></li> </ul> </li> </ul>					
<ul style="list-style-type: none"> <li>• Does the waste corrode steel at a rate &lt; 6.35 mm (0.250 inches) per year at 55 °C (130 °F)?</li> </ul>					
<ul style="list-style-type: none"> <li>○ If yes, neutralization or buffering is not required.</li> </ul>					
<ul style="list-style-type: none"> <li>○ <b>If no, the evaluation cannot continue until the affected waste is neutralized or the TRU waste site provides technical justification using option H of section 2, paragraph 2 of the BoK, and has received CBFO concurrence that the waste is acceptable without neutralization.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach CBFO concurrence when applicable.</b></li> <li>▪ <b>Attach list 5.4.3 of affected containers.</b></li> </ul> </li> </ul>					



Enter "Y" for yes or "N" for no	Y	N
List the neutralizing or buffering agent(s) manufacturer and product name: _____		
Can explosive compounds form when the neutralizing or buffering agent(s) are added to the oxidizing chemical?		
<p><b>If yes, notify the SPM, CBFO Manager and the Assistant Manager for the National TRU Program Office immediately so the TRU Waste Site can be contacted and the affected activity stopped. This BoK evaluation cannot continue until the TRU waste site provides test results demonstrating the waste does not pose a hazard.</b></p> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.4.4 of affected containers.</b></li> </ul>		
<ul style="list-style-type: none"> <li>• If organic neutralizing or buffering agent was used to neutralize or buffer previously packaged waste, did it have the potential for forming explosive compounds with the oxidizing chemical?</li> </ul>		
List the neutralizing or buffering agent(s) manufacturer and product name: _____		
<ul style="list-style-type: none"> <li>○ If yes, has testing been performed to demonstrate the waste does not pose a hazard when exposed to mechanical impact, spark, friction, and/or heat? <ul style="list-style-type: none"> <li>▪ If yes, attach CBFO approval of the testing results.</li> <li>▪ <b>If no, this evaluation cannot continue until the TRU waste site provides test results demonstrating the affected waste does not pose a hazard.</b> <ul style="list-style-type: none"> <li>• <b>Attach list 5.4.5 of affected waste</b></li> </ul> </li> </ul> </li> </ul>		
<p>When testing demonstrates the waste poses a hazard, the TRU waste site must identify a method of treatment and receive CBFO concurrence.</p> <ul style="list-style-type: none"> <li>▪ Attach the CBFO concurrence or <b>stop the evaluation of affected waste until concurrence is obtained.</b></li> <li>▪ <b>Attach list 5.4.6 of affected waste.</b></li> </ul>		
<ul style="list-style-type: none"> <li>○ If no, go to Section 5.5 questions.</li> </ul>		

Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.5 Organic sorbents</b>			
Is the oxidizing chemical sorbed in polyol organic sorbent(s)?			
<ul style="list-style-type: none"> <li>• If yes, has the waste been treated in accordance with section 6?           <ul style="list-style-type: none"> <li>○ If yes, go to Section 5.5.2 questions.</li> <li>○ <b>If no, this evaluation cannot continue for affected waste until the TRU waste site obtains approval on the proposed method of treatment from CBFO and treatment is performed.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.5 of affected waste</b></li> </ul> </li> </ul> </li> </ul>			
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0, 6, and 7) _____			
_____			
_____			
<b>Section 5.5.1 Organic Sorbents With Oxidizing Chemicals</b>			
Is the oxidizing chemical sorbed in EOPS?			
<ul style="list-style-type: none"> <li>○ If yes, is the wt. % of oxidizing chemicals allowed <math>\leq</math> the value listed in Table 5-3? (section 5.5.1)           <ul style="list-style-type: none"> <li>▪ If yes, the oxidizing chemicals at this concentration are allowed without further treatment.</li> <li>▪ <b>If no, this evaluation cannot continue until the TRU waste site selects an option listed in section 2.0 or treats the affected waste as 100 wt. % oxidizing chemical.</b> <ul style="list-style-type: none"> <li>• <b>Attach list 5.5.1 of affected waste</b></li> </ul> </li> </ul> </li> </ul>			
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.5.1 ) _____			
_____			
_____			

Enter "Y" for yes or "N" for no	Y	N
<b>Section 5.5.2 Organic rags, wipes, sorbent pads, and pillows</b>		
Does the waste include organic rags, wipes, sorbent pads, or pillows mixed with nitrate?		
<ul style="list-style-type: none"> <li>• If yes, has headspace gas analysis been performed?</li> </ul>		
<ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li>○ If yes, were other than minimal oxidation gases generated in the container?</li> </ul> </li> </ul>		
<ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li>▪ <b>If yes, this evaluation cannot continue for affected waste until the TRU waste site selects an option listed in section 2.0.</b></li> </ul> </li> </ul> </li> </ul>		
<ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li>• <b>Attach list 5.5.2 of affected waste.</b></li> </ul> </li> </ul> </li> </ul> </li> </ul>		
<ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li>Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.5.2)</li> </ul> </li> </ul> </li> </ul> </li> </ul> </li> </ul>		
_____		
_____		
_____		

EXAMPLE

Enter "Y" for yes or "N" for no	Y	N
<b>Section 5.5.3 Miscellaneous organic materials</b>		
Does the waste contain miscellaneous organic materials with oxidizing chemicals?		
List the miscellaneous organic materials with oxidizing chemicals: _____ _____		
<ul style="list-style-type: none"> <li>• Does the waste contain ion exchange resins?</li> </ul>		
<ul style="list-style-type: none"> <li>○ If yes, have the ion exchange resins been cemented or grouted to a concentration of <math>\leq 10</math> wt. % or has the monolith been verified to be intact by RTR or VE?</li> </ul>		
<ul style="list-style-type: none"> <li>▪ If yes, the ion exchange resins are acceptable at WIPP.</li> </ul>		
<ul style="list-style-type: none"> <li>▪ If no, has CBFO approved a verifiable basis showing that the ion exchange resins waste will be safe and compliant for disposal at WIPP?</li> </ul>		
<ul style="list-style-type: none"> <li>▪ If yes, attach CBFO approval.</li> </ul>		
<ul style="list-style-type: none"> <li>• <b>If no, the evaluation cannot continue for affected waste until the TRU waste site selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.5.3.1 of affected waste</b></li> </ul> </li> </ul>		
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.5.3) _____ _____ _____		
<ul style="list-style-type: none"> <li>• Does the waste contain spent organic solvents, or organic materials other than sorbents, such as glycerin or sugar solutions, that have been mixed or added to oxidizing chemicals?</li> </ul>		
<ul style="list-style-type: none"> <li>○ If yes, has CBFO approved a verifiable basis showing that the miscellaneous organic waste with oxidizing chemicals will be safe and compliant for disposal at WIPP?</li> </ul>		
<b>If yes, attach CBFO approval.</b>		
<b>If no, the evaluation cannot continue for affected waste until the TRU waste site selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.5.3.2 of affected waste</b></li> </ul>		
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.5.3)		

Enter "Y" for yes or "N" for no	Y	N
<b>Section 5.6.1 Oxidizing chemicals sorbed in inorganic sorbents</b>		
Is the oxidizing chemical sorbed with an inorganic sorbent?		
<ul style="list-style-type: none"> <li>• If yes, is the wt. % of the oxidizing chemicals allowed <math>\leq</math> the value listed in Table 5-4?</li> </ul>		
<ul style="list-style-type: none"> <li>○ If yes, the oxidizing chemicals at this concentration are allowed without further treatment.</li> </ul>		
<ul style="list-style-type: none"> <li>○ <b>If no, this evaluation cannot continue for the affected waste until the TRU waste site selects an option listed in section 2.0 or treats the waste as 100 wt. % oxidizing chemical.</b> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.6.1 of affected waste</b></li> </ul> </li> </ul>		
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.6.1)		
_____		
_____		
_____		

EXAMPLE

Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.7 Mixtures of organic and inorganic materials with oxidizing chemicals</b>			
Does the waste contain a mixture of organic and inorganic materials with oxidizing chemicals?			
<ul style="list-style-type: none"> <li>• If yes, list the inorganic or organic sorbent with the lowest wt. % of oxidizing chemicals allowed when applicable (see table 5-3 and table 5-4)</li> </ul>			
<ul style="list-style-type: none"> <li>• If no, skip to section 5.8</li> </ul>			
<ul style="list-style-type: none"> <li>• Does the combined oxidizing chemical in both sorbents exceed the wt. % of oxidizing chemicals allowed for the sorbent with the lowest allowed oxidizing chemical concentration?               <ul style="list-style-type: none"> <li>○ If yes, list the inorganic sorbent that was used to attain the allowed concentration. List the final concentration in the matrix.</li> </ul> </li> </ul>			
<ul style="list-style-type: none"> <li>• Does the mixture contain sorbents not listed in Table 5-3 or Table 5-4? Record the unlisted sorbent(s) manufacturer and product name and indicate if EOPS or inorganic sorbent: _____</li> </ul>			
<ul style="list-style-type: none"> <li>○ If yes, has CBFO approved an equivalency determination for either an unlisted EOPS or inorganic sorbent?               <ul style="list-style-type: none"> <li>▪ If yes, attach CBFO response.</li> </ul> </li> </ul>			
Check criteria used:	<input type="checkbox"/>	<ul style="list-style-type: none"> <li>▪ If no and the oxidizing chemical concentration in the mixture is &gt; 30 wt. % and the inorganic sorbent's allowable oxidizing chemical concentration is &lt; 30 wt. %, an EOPS equivalency determination is unnecessary. <b>or</b></li> <li>▪ If no and the inorganic sorbent's allowable oxidizing chemical concentration is &gt; 30 wt. %, an EOPS equivalency determination must be requested and approved.</li> </ul>	
	<input type="checkbox"/>	<ul style="list-style-type: none"> <li>• <b>This evaluation cannot continue for affected waste until the TRU waste site selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.7 of affected waste</b></li> </ul> </li> </ul>	
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.7)			

Enter "Y" for yes or "N" for no	Y	N
<p><b>Section 5.8 Oxidizing chemicals that are the sole component of waste</b></p> <p>Is the waste composed of oxidizing chemical only (e.g., a metal nitrate salt) or is a component of the waste composed of oxidizing chemical only?</p> <ul style="list-style-type: none"> <li>• <b>If yes, the affected waste is not acceptable for disposal at WIPP without treatment.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.8 of affected waste</b></li> </ul> </li> <li>• If no, proceed to section 5.9</li> </ul>		
<p><b>Section 5.9 Inorganic Sludges With Oxidizing Chemicals Not Mixed with Sorbents</b></p> <p>Is the oxidizing chemical and inorganic sludge not mixed with sorbents?</p> <ul style="list-style-type: none"> <li>• If yes: Is the weight of oxidizing chemical in the inorganic sludge <math>\leq</math> 20 wt. %</li> <li>• <b>If no: This evaluation cannot continue until the TRU waste site treats the inorganic sludge with an inorganic sorbent listed in Table 5-4 to meet the allowable wt. % oxidizing chemical concentration or selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.9 of affected waste.</b></li> </ul> </li> <li>• Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 5.6.1)</li> </ul> <hr/> <hr/> <hr/>		
<p><b>Section 5.10 Oxidizing chemicals solidified in a cement or grout matrix</b></p> <p>Is the oxidizing chemical solidified in a matrix such as cement or grout?</p> <ul style="list-style-type: none"> <li>• Was the waste well mixed? <ul style="list-style-type: none"> <li>○ If yes: 1) does greater than 20% of the set material have dimensions smaller than two centimeters; <b>and</b></li> <li>2) is the decomposition temperature of the oxidizing chemical <math>&lt;</math> 350 °C; <b>and</b></li> <li>3) does the waste contain free liquids?</li> </ul> </li> <li>▪ <b>If responses to 1, 2 or 3 above are yes, the affected waste may require treatment. Contact the CBFO Waste Management Senior Technical Advisor for assistance.</b> <ul style="list-style-type: none"> <li>○ <b>Attach list 5.10 of affected waste.</b></li> </ul> </li> <li>▪ If no, proceed to section 5.11.</li> </ul>		

Enter "Y" for yes or "N" for no	Y	N
<b>Section 5.11 Surfaces contaminated with oxidizing chemicals</b>		
Is the waste component a low porosity, impermeable surface contaminated with oxidizing chemicals?		
<ul style="list-style-type: none"> <li>• Is there observable oxidizing chemical salt build up on the surface?</li> </ul>		
<ul style="list-style-type: none"> <li>○ <b>If yes, this evaluation cannot continue for affected waste until the TRU waste site selects an option listed in section 2.0.</b></li> </ul>		
<ul style="list-style-type: none"> <li> <ul style="list-style-type: none"> <li>▪ <b>Attach list 5.11 of affected waste.</b></li> </ul> </li> </ul>		
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7 or 5.11)		
<hr/>		
<hr/>		
<hr/>		
<ul style="list-style-type: none"> <li>○ If no, proceed to 5.12</li> </ul>		

EXAMPLE



Enter "Y" for yes or "N" for no		Y	N
<b>Section 5.12 Soils containing oxidizing chemicals</b>			
Are oxidizing chemicals mixed with soil?			
<ul style="list-style-type: none"> <li>• Is the organic content in the soil &gt; 20 wt. %? (section 5.12.1)           <ul style="list-style-type: none"> <li>○ If no, is the oxidizing chemical content ≤ 35 wt. %?               <ul style="list-style-type: none"> <li>▪ If yes, no treatment is necessary.</li> <li>▪ <b>If no, this evaluation cannot be completed for affected waste until the TRU waste site selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>• <b>Attach list 5.12.1 of affected waste.</b></li> </ul> </li> </ul> </li> </ul> </li> </ul>			
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7) _____			
_____			
_____			
_____			
<ul style="list-style-type: none"> <li>• Is the waste from an earthen disposal pit? (section 5.12.2)           <ul style="list-style-type: none"> <li>○ If yes: 1) is the oxidizing chemical ≤ 35 wt. %; <b>and</b> <ul style="list-style-type: none"> <li>2) is the organic material content ≤ 20 wt. %; <b>and</b> <ul style="list-style-type: none"> <li>3) is the soil content ≥ 45 wt. %?</li> </ul> </li> </ul> </li> <li>▪ If responses to 1, 2, and 3 above are yes, no treatment is necessary.</li> <li>▪ <b>If no, this evaluation cannot be completed for affected waste until the TRU waste site selects an option listed in section 2.0.</b> <ul style="list-style-type: none"> <li>• <b>Attach list 5.12.2 of affected waste.</b></li> </ul> </li> </ul> </li> </ul>			
Describe option selected by the TRU waste site and attach CBFO approvals when applicable. (sections 2.0 and 7) _____			
_____			
_____			
_____			

Enter "Y" for yes or "N" for no		Y	N
<ul style="list-style-type: none"> <li>• Have required CBFO approvals been obtained for attachment to this checklist?                             <ul style="list-style-type: none"> <li>○ Identify all attachments to this form:</li> </ul> </li> </ul>			
<div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div> <div style="border-bottom: 1px solid black; height: 15px; margin-bottom: 5px;"></div>			
END OF CHECKLIST			
AKE:			
	Print	Signature	Date
SPM:			
	Print	Signature	Date