Title: Oxidizer Scoping Studies

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Oxidizer Scoping Studies

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History of Revision/Change

Revision Number	Effective Date	Pages Affected	Description of Revision/Change
0	06/29/2016	All	Original release for internal distribution only to inform the Test Plan.
1	11/03/2016	All	Rewritten for external distribution, removing redundant information, streamlining information for an external audience, and reporting relative burn rates in standardized format.
2	01/19/2017	Table 3	Plutonium perchlorate was added to table because of unsafe chemical properties.
		Table 4	Table was reformatted.
		Table 7 & Appendix B	Radioactive oxidizers with >3 waters of hydration were added to table because they are bound.
		Appendix B	Americium nitrate, curium nitrate, and neptunium nitrate were removed because they were excluded.
3	4-12-17	Pages 10-17	Tables 3 & 5: Per request of management, wording and references of some entries "harmonized" with DOE CBFO Basis of Knowledge.
		Pages 36-37	Appendix B: Appropriate oxidation state and waters of hydration have been updated to some entries.

		All	Management requests the following oxidizers identified in AK be added because it can be interpreted that they fit into the “Oxidizing solid, n.o.s” field of the Hazardous Materials Table (49 CFR §172.101): Potassium chromate Potassium dichromate Potassium iodate Potassium periodate Silver(I) oxide Silver(II) oxide Sodium dichromate dihydrate Sodium chromate The above 8 oxidizers were added to Appendices A, B, & E as well as Table 9. The following 4 oxidizers also appear in Table 10: Potassium iodate Potassium periodate Silver(I) oxide Silver(II) oxide
		All	The following 3 additional oxidizers were identified by AK at Idaho National Laboratory and added to this report: Hydroxylamine nitrate (hydroxylammonium nitrate) Ceric nitrate (cerium(IV) nitrate) Manganous nitrate (manganese(II) nitrate) The above 3 oxidizers were added to Appendix A. Cerium(IV) nitrate and manganese(II) nitrate were added to Appendix B and Table 7. Hydroxylammonium nitrate was added to Table 3.

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Oxidizer Scoping Studies

1.0 PURPOSE

The Los Alamos National Laboratory – Carlsbad Operations (LANL-CO) office was tasked by the Department of Energy's Carlsbad Field Office (DOE-CBFO), Office of the Manager to:

- Perform a review of the acceptable knowledge (AK) to identify the oxidizers and sorbents in transuranic (TRU) waste streams.
- Conduct scoping studies on the oxidizers and sorbents identified in AK review to inform planned Quality Level 1 (QL1) testing.
- Conduct a series of QL1 tests to provide the scientific data to support a basis of knowledge document for determining the criteria for: 1) accepting waste at the Waste Isolation Pilot Plant (WIPP) without treatment, 2) determining waste that will require treatment, and 3) if treatment is required, how the treatment must be performed.

The purpose of this report is to present the results of the AK review of oxidizers present in active waste streams, provide a technical analysis of the oxidizers, and report the results of the scoping study testing. This report will determine the fastest burning oxidizer to be used in the development of a *Test Plan for Preparation and Testing of Sorbents Mixed with Oxidizer found in Transuranic Waste* (DWT-TP-001). The companion report, DWT-RPT-002, Sorbent Scoping Studies, contains similar information for sorbents identified during the AK review of TRU waste streams. The results of the oxidizer and sorbent scoping studies will be used to inform the QL1 test plan. The QL1 test results will support the development of a basis of knowledge document that will evaluate oxidizing chemicals and sorbents in TRU waste and provide guidance for treatment.

2.0 SCOPE

LANL-CO personnel produced a list of oxidizers present in active TRU waste streams by comparing the chemicals listed in the AK for these waste streams to the hazardous materials descriptions and proper shipping names that are in the oxidizer hazard class or packing group of the Hazardous Materials Table (49 CFR

§172.101).[1] This AK review of active TRU waste streams produced the list of oxidizers contained in Appendix A, 49 CFR §172.101 Oxidizers Listed in the AK, hereafter referred to as the initial oxidizer list.

3.0 TECHNICAL ANALYSIS OF THE 49 CFR §172.101 OXIDIZERS LISTED IN THE AK

3.1 SYNONYM REMOVAL

The oxidizers in the initial oxidizer list have had synonyms and repeat listings removed.

3.2 NON-SPECIFIC OXIDIZER REMOVAL

From the initial oxidizer list, non-specific oxidizers were identified (**Table 1**). One entry, thallium nitrate, may represent one or both of two distinct oxidizers: thallium(I) nitrate and thallium(III) nitrate. Without additional detail, identification was not possible; therefore oxidizers identified in the AK in **Table 1** were removed from the initial oxidizer list and both thallium nitrates replaced the thallium nitrate entry. These changes are reflected in Appendix B, *Initial List of Scoping Study Oxidizers*.

TABLE 1. Non-Specific Oxidizers Identified in the AK

Non-Specific Oxidizer Listed in AK	Notes
Alkaline permanganate	Specific alkaline permanganates (potassium and sodium) remain in the oxidizer list.
Hypochlorite (unspecified)	Specific hypochlorites remain in the oxidizer list.
Nitrate	Specific nitrates remain in the oxidizer list.
Nitrites (nonspecific)	Specific nitrites remain in the oxidizer list.
Perchlorate	Specific perchlorates remain in the oxidizer list.
Permanganate	Specific permanganates remain in the oxidizer list.

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[1] U.S. Government Publishing Office (2016). 49 CFR§172.101, Electronic Code of Federal Regulations, retrieved from: http://www.ecfr.gov. This citation of the Code of Federal Regulations is in regards to the Hazardous Materials Table present within Title 49 (Transportation). Label codes 5.1 (Oxidizer) and 5.2 (Organic peroxide – also an oxidizer) were compared against the oxidizers identified in AK.

Non-Specific Oxidizer Listed in AK	Notes
Peroxide	Specific peroxides remain in the oxidizer list.
Persulfate	Specific persulfates remain in the oxidizer list.
Thallium nitrate	May represent thallium(I) nitrate, thallium(III) nitrate trihydrate, or both. Both thallium(I) nitrate and thallium(III) nitrate were added to the oxidizer list in place of “thallium nitrate.”

3.3 MIXTURES EVALUATION

Mixtures on the initial oxidizer list were removed because the individual oxidizer components of the mixtures were already present on the initial oxidizer list. The typical occurrence of oxidizers in TRU waste is not as pure constituents, but rather as mixtures. However, because of the infinite combinations possible in mixtures, the testing of mixtures was limited to two cases: 1) The oxidizer components, tested individually, to identify the most vigorous (bounding) oxidizer to recommend for the QL1 test plan, and 2) A brief examination of the potential for synergistic effects to exist between two oxidizers as discussed in **Section 5**. **Table 2** lists mixtures that were not included in Appendix B, *Initial List of Scoping Study Oxidizers*.

TABLE 2. Mixtures of Oxidizers Identified in the AK

Oxidizer Mixture Listed in AK	Notes
Aqua Regia (10% hydrochloric acid, 90% nitric acid)	Hydrochloric acid is not an oxidizer. [a] Nitric acid remains on the oxidizer list.
Glyceregia (nitric acid hydrochloric acid and glycerol)	Hydrochloric acid is not an oxidizer. [a] Glycerol is not an oxidizer. [a] Glyceregia decomposes rapidly and will not be found in the waste. [b] Nitric acid remains on the oxidizer list.
Lead hydroxide, oxide, and nitrate	Lead hydroxide is not an oxidizer. [a] Lead nitrate and lead dioxide (lead peroxide) remain on the oxidizer list.
Nitric acid-Nitradd solution [c] (nitric acid, ammonium bifluoride, acetic acid)	Ammonium bifluoride is not an oxidizer. [a] Acetic acid is not an oxidizer. [a] Nitric acid remains on the oxidizer list.
OxOut 536 (water, ammonium bifluoride, and nitric acid)	Water is not an oxidizer. [a] Ammonium bifluoride is not an oxidizer. [a] Nitric acid remains on the oxidizer list.
Plutonium nitrate + aluminum nitrate	Plutonium nitrate and aluminum nitrate remain on the oxidizer list.
Turco 4502 (contains potassium hydroxide, potassium permanganate, and potassium chromate)	Potassium hydroxide is not an oxidizer. [a] Potassium chromate is not an oxidizer. [a] Potassium permanganate remains on the oxidizer list.

[a] U.S. Government Publishing Office (2016). 49 CFR§172.101, Electronic Code of Federal Regulations, retrieved from: http://www.ecfr.gov. This citation of the Code of Federal Regulations is in regards to the Hazardous Materials Table present within Title 49 (Transportation). Label codes 5.1 (Oxidizer) and 5.2 (Organic peroxide – also an oxidizer) were compared against the oxidizers identified in AK.

[b] Vander Voort, G. (2004). Laboratory Safety in Metallography, Metallography and Microstructures, Vol 9, ASM Handbook, ASM International, p. 1081–1090.

[c] There are various formulations of Nitradd available, and the AK is not always specific regarding which products were used. However, they all involve the use of a fluoride salt and acetic acid. See, for example, an SDS for Turco Nitradd (T-4104) as provided by Henkel: http://hybris.cms.henkel.com/henkel/msdspdf?matnr=597527&country=US&language=EN

3.4 OXIDIZERS UNDER EVALUATION

Removal of the Non-Specific Oxidizers (**Table 1**) and Mixtures of Oxidizers (**Table 2**) from the initial oxidizer list resulted in the oxidizers that were evaluated in the scoping study.

4.0 OXIDIZERS EXCLUDED FROM SCOPING STUDIES

Oxidizers were further excluded from the scoping studies for a variety of reasons including unsafe properties (Sections 4.1 chemical and 4.2 radiological) or that their concentrations are extremely low in the waste (Section 4.3).

4.1 OXIDIZERS WITH UNSAFE CHEMICAL PROPERTIES

Some of the oxidizers in Appendix A, *49 CFR §172.101 Oxidizers Listed in the AK*, were not tested in the scoping studies due to unsafe chemical properties (e.g.: an explosive material).

Table 3 lists the oxidizers from Appendix A that were excluded from scoping study testing, with an explanation of the unsafe property.

Because the materials were excluded from testing does not mean that they are not of concern, but only that they were not safe to evaluate under this testing protocol. The basis of knowledge document is expected to cover how these excluded oxidizers should be treated.

TABLE 3. Oxidizers Excluded from the Scoping Study Due to Unsafe Chemical Properties

Excluded Oxidizer	Reason for Exclusion	Reference(s)
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, b
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, b
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, d
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
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
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-*t*-butyl peroxide)	Decomposition of this peroxide at 165°C (329°F) resulted in a violent explosion. Samples will experience	a, c

Excluded Oxidizer	Reason for Exclusion	Reference(s)
	temperatures well beyond this during the burn rate measurements of the scoping study testing.	
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.	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
Hydroxylammonium nitrate	Hydroxylammonium nitrate in the solid form is considered unstable and possibly explosive. It is unsuitable for scoping study testing.	e
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
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.	a, c, f
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

Excluded Oxidizer	Reason for Exclusion	Reference(s)
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, g
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, g
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
Sodium hypochlorite	The anhydrous solid is highly explosive and sensitive to heat or friction. These conditions will be present during scoping study testing. There are documented uses of sodium hypochlorite, but they would not place it in TRU waste streams due to location of use or its reactive chemical nature. Sodium hypochlorite solutions were used in cooling water systems as fungicides and biocides – a use that would place it outside of TRU waste streams (INL-026 P061). Sodium hypochlorite was identified as potentially present in sludge containing reducing agents ferrous sulfamate and hydrochloric acid – conditions that would react it away (INL-001 RF-P406). 5.25% solutions of sodium hypochlorite (Clorox or Clo-White bleach) were used for surface decontamination – this would result in sodium chloride when the liquid evaporates (INL-025 I1030, INL-026 P044, and INL-001 RF-P178). In conclusion, sodium hypochlorite will not persist in conditions found in TRU waste acceptable at the WIPP and, therefore, is not expected to be found in the waste.	a, h, i, j, k, l
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	c

Excluded Oxidizer	Reason for Exclusion	Reference(s)
	spontaneously ignited when mixed with cellulose during scoping studies.	
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, g
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, m
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
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

[a.] Bretherick, L. (1990) Bretherick's Handbook of Reactive Chemical Hazards, 4th Ed., Boston, MA: Butterworths & Co. Ltd.

[b.] U.S. Government Publishing Office (2016). 49 CFR§172.101, Electronic Code of Federal Regulations, retrieved from: http://www.ecfr.gov

[c.] Center for Chemical Process Safety (2016). Chemical Reactivity Worksheet, Version 4.0, retrieved from: http://response.restoration.noaa.gov/reactivityworksheet

[d.] Center for Disease Control and Prevention NIOSH Pocket Guide to Chemical Hazards retrieved on February 27, 2017 from: https://www.cdc.gov/niosh/npg/default.html

[e.] Amrousee, Rachid et al. (2016) Hydroxylammonium nitrate (HAN)-based green propellant as alternative energy resource for potential hydrazine substitution: From lab scale to pilot plant scale-up, Combustion and Flame, 176 (February 2017), pp. 334-348

[f.] This oxidizer will again be excluded in section 4.2 due to unsafe radioactive properties.

[g.] Lewis, R. J. Sr. (1992). Sax's Dangerous Properties of Industrial Materials, 8th Ed., New York, NY: Van Nostrand Reinhold.

[h.] Bleach 101 by the Clorox Company retrieved on February 27, 2017 from: https://www.clorox.com/dr-laundry/bleach-101/

[i.] Pipeline and Hazardous Materials Safety Administration (PHMSA) Response Letter dated June 23, 2004 regarding Clo White bleach (Ref. No. 04-0122) retrieved on February 27, 2017 from: http://www.phmsa.dot.gov/portal/site/PHMSA/menuitem.6f23687cf7b00b0f22e4c6962d9c8789/?vgnextoid=2f202386d0a64110VgnVCM1000009ed07898RCRD&vgnextchannel=56acd3c1af814110VgnVCM1000009ed07898RCRD

[j.] Ramirez, M. (2013). Central Characterization Program Acceptable Knowledge Summary Report for Waste Retrieved from Designated Areas within the Subsurface Disposal Area at the Idaho National Laboratory, Transuranic Waste Streams: ID-SDA-DEBRIS, ID-SDA-SLUDGE, and ID-SDA-SOIL, CCP-AK-INL-001, Rev.12, Carlsbad, NM: Central Characterization Program.

[k.] Ramirez, M. (2013). Central Characterization Program Acceptable Knowledge Summary Report for Idaho National Laboratory Argonne Contact-Handled Transuranic Waste From Facility Maintenance and Laboratory Operations, Transuranic Waste Streams: ID-AECHEM and ID-AECHHM, CCP-AK-INL-025, Rev.2, Carlsbad, NM: Central Characterization Program.

[l.] Ramirez, M. (2013). Central Characterization Program Acceptable Knowledge Summary Report for Idaho National Laboratory Sludge Repackage Project Combined Sludge Waste, Waste Stream: ID-SRP-S3000, CCP-AK-INL-026, Rev.0, Carlsbad, NM: Central Characterization Program.

[m.] Fisher Scientific Safety data sheet retrieved on May 10, 2016 from: https://www.fishersci.com/shop/msdsproxy?productName=AC420141000&productDescription=TETRABUTYLAMMONIUM+PERCH+100GR&catNo=AC42014-1000+&vendorId=VN00033901&storeId=10652

4.2 OXIDIZERS WITH UNSAFE RADIOACTIVE PROPERTIES

Eight scoping study oxidizers could not be tested because they are radioactive. The exclusion from testing was based solely on the fact that the study did not have access to facilities to produce and burn multi-gram quantities of these radioactive materials. We are unaware of any facilities in the DOE complex that has an authorization basis that allows burning of gram quantities of radioactive materials mixed with oxidizers and combustibles. **Table 4** identifies the radioactive oxidizers that were not tested in the scoping studies.

TABLE 4. Radioactive Oxidizers

Radioactive Oxidizer	
Americium nitrate	Plutonium perchlorate
Curium nitrate	Plutonium peroxide
Neptunium nitrate	Thorium nitrate
Plutonium nitrate	Uranyl nitrate hexahydrate

4.3 OXIDIZERS WITH NON-IMPACTIVE CONCENTRATIONS

Some oxidizers identified by AK as being a possible component of the waste are expected to be present in waste bound for the WIPP in extremely low concentration. In some cases, this is due to their chemical reactivity (reactions with the ambient environment, packaging materials, and co-mingled waste). In other cases, their initial concentrations were low. These concentration levels are based on the AK evaluated for this review and are not necessarily applicable to future waste streams.

Table 5 lists oxidizers from Appendix A, *49 CFR §172.101 Oxidizers Listed in the AK*, that were withdrawn from the scoping studies because they are no longer present or were found in only a few waste streams with small anticipated quantities, so the decision was made to withdraw them from the scoping studies. The remaining oxidizers are listed in Appendix B, Initial List of *Scoping Study Oxidizers*.

TABLE 5. Other Oxidizers Withdrawn Due to Non-Impactive Concentrations

Withdrawn Oxidizer	Reason for Withdrawal	Reference(s)
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. It is a liquid below 5°C (41°F) and decomposes at 10°C (50°F). Bromine chloride decomposes on contact with moisture. It is not expected to be in the waste because it would have reacted.	f, g
Hydrogen peroxide	Hydrogen peroxide in pure form, as well as solutions such as a ≥20 wt.% to ≤40 wt.% solution, behaves as a very reactive oxidizer. It is incompatible with combustible materials, strong acids, strong bases, metals, and strong reducing agents. In pure or diluted form, explosive mixtures may be 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. Hydrogen peroxide breaks down in the presence of light. Even a relatively stable, properly stored sample of hydrogen peroxide decomposes at the rate of 0.5% per year at room temperature increasing by a factor of two for each 10°C (50°F) rise in temperature. It is not expected to be in the waste because it would have reacted.	h, i, j, k
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.	k

Withdrawn Oxidizer	Reason for Withdrawal	Reference(s)
Nitrous acid	Nitrous acid (HNO_2) 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.	k
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 CCP-AK-ORNL-005's source document P969, 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 remained at approximately 0.04 wt.% in simulated fission product waste streams. According to CCP-AK-ORNL-003's source document P1067, the palladium-containing residues did not dissolve in nitric acid, nitric acid with potassium fluoride, and hydrochloric acid. The palladium appears to remain in the insoluble residues as opposed to converting to the palladium nitrate salt by dissolving in nitric acid. The reprocessing of nuclear fuel separates fission products before the recovery of uranium and plutonium. Palladium nitrate was not tested because palladium is a fission product that would have been separated from actinides and is not in the TRU waste.	c, d, e, l
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 nuclear fuel separates fission products before the recovery of uranium and plutonium. Rhodium nitrate was not tested because rhodium is a fission product that would have been separated from actinides and is not in the TRU waste.	c
Ruthenium nitrate (should have been identified as 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 nuclear fuel separates fission products before recovery of uranium and plutonium. Ruthenium (III) nitrosyl nitrate was not tested because ruthenium is a fission product that would have been separated from actinides and is not in the TRU waste.	m, n
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, o, p

Withdrawn Oxidizer	Reason for Withdrawal	Reference(s)
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

[a.] 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.] Lide, D. R. (Ed.) (1993). CRC Handbook of Chemistry and Physics, 74th Ed., Ann Arbor: CRC Press.

[h.] Conner, W. V. (1993). Hydrogen Peroxide Safety Issues, RFP-4599, U.S. Department of Energy, EG&G Rocky Flats.

[i.] Bretherick, L. (1990) Bretherick's Handbook of Reactive Chemical Hazards, 4th Ed., Boston, MA: Butterworths & Co. Ltd.

[j.] Lewis, R. J. Sr. (2001) Hawley's Condensed Chemical Dictionary, 14th Ed., New York, NY: John Wiley & Sons.

[k.] Budavari, S. et al. (Eds.). (1996). The Merck Index, 12th Ed., Whitehouse Station, NJ: Merck & Co., Inc.

[l.] 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.

[m.] Swain, P. et al. (2013). Separation and Recovery of Ruthenium: A Review, Journal of Radioanalytical. Nuclear Chemistry, 298(2), pp.781–796.

[n.] 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.

[o.] 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.

p. 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.

5.0 SCOPING STUDY TESTING

Scoping study testing was performed to determine the relative strength based on the burning rate of the oxidizers present in Appendix B, Initial List of *Scoping Study Oxidizers*, to that of potassium nitrate. Potassium nitrate was selected as the reference oxidizer for the scoping studies because the Energetic Materials Research and Testing Center (EMRTC) Report FR 10-13 concluded that a 1:1 potassium nitrate: cellulose mixture in a 30g conical pile burned fastest for a 1:1 mixture.[2] EMRTC had worked with LANL-CO and Washington TRU Solutions previously, and on their behalf, established the concentration at which the most oxidizing mixture of sodium and potassium nitrate salts mixed with zeolite or grout becomes a non-oxidizer.[3] This scoping study started with the information produced by EMRTC, but determined that a 4:1 potassium nitrate: cellulose mixture in a 30g conical pile burned even faster than a 1:1 mixture when following the Environmental Protection Agency (EPA) SW-846 Method 1040.[4] The 4:1 potassium nitrate: cellulose mixture was selected to become the standard to which the burning rate of the scoping study oxidizers would be evaluated.

For an oxidizer to be decidedly faster burning than potassium nitrate, it must have burned faster in a 4:1, by weight, mixture of the oxidizer: cellulose than a 4:1 mixture of potassium nitrate: cellulose. The oxidizer with the fastest measured burning rate represents a bounding condition for further exploration in a QL1 test plan (DWT-TP-001, *Test Plan for Preparation and Testing of Sorbents Mixed with Oxidizer found in Transuranic Waste*).

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[2] Walsh, G. (2010). Results of oxidizing solids testing, EMRTC Report FR 10-13, Energetic Materials Research and Testing Center, Socorro, NM: New Mexico Institute for Mining and Technology, prepared for Washington TRU Solutions, LLC.

[3] (2012). Amount of Zeolite Required to Meet the Constraints Established by the EMRTC Report FR 10-13: Application to LANL Evaporator Nitrate Salts, LA-UR-14-26860, Carlsbad, NM: LANL-Carlsbad Office Difficult Waste Team.

[4] EPA (2007). EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 1040, Test Method for Oxidizing Solids, Rev.0. Retrieved from: https://www.epa.gov/hw-sw846/sw-846-test-method-1040-test-method-oxidizing-solids

The experiments to determine the bounding oxidizers were conducted according to a procedure documented within Attachment 1 of the LANL-CO Hazard Control Plan RSO-HCP-DWT-001, Rev.4, *Scoping Tests on Oxidizers and Polymeric Sorbents*.[5] The equipment list and setup was derived from the EPA SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, Method 1040, *Test Method for Oxidizing Solids*.[6] The procedure within RSO-HCP-DWT-001 is a modification of Method 1040.

5.1 EVALUATION OF NITRATE HYDRATES

Many nitrates tend to be hygroscopic or even deliquescent. Regardless of the form purchased, the nitrates will tend to a preferred level of hydration that can vary with temperature and relative humidity. Anhydrous versions of the nitrate salts were not tested if the material typically forms hydrates because the waste is in vented containers under ambient conditions and anhydrous nitrates would hydrate during storage.

It was observed during the scoping study testing that metal nitrate hydrates containing over 3 waters of hydration are all weaker oxidizers than potassium nitrate. In fact, no nitrate-based oxidizer-cellulose mixture with a nitrate containing four or more waters of hydration would maintain combustion of cellulose without the continued external heat input provided by the ignition wire. Once the ignition wire (see Appendices C, *Oxidizer Sorbent Tests*, and D, *Method 1040 Equipment*) was de-energized, combustion (if observed) ceased almost immediately. These observations are recorded in **Table 6**.

TABLE 6. Average Burn Times of Metal Nitrate Salts with Waters of Hydration

Scoping Study Oxidizer	Formula	Relative Burn rate to the KNO_3 Standard for a 30g conical pile of selected oxidizer mixed with cellulose in a 4:1 weight ratio (smaller numbers reflect faster burning)
Aluminum nitrate nonahydrate	$Al(NO_3)_3 \cdot 9H_2O$	Did not burn
Cadmium nitrate tetrahydrate	$Cd(NO_3)_2 \cdot 4H_2O$	Incomplete burn
Calcium nitrate tetrahydrate	$Ca(NO_3)_2 \cdot 4H_2O$	Did not burn

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[5] Appendix C, *Oxidizer Sorbent Tests*.
[6] Appendix D, *Method 1040 Equipment.*

Scoping Study Oxidizer	Formula	Relative Burn rate to the KNO_3 Standard for a 30g conical pile of selected oxidizer mixed with cellulose in a 4:1 weight ratio (smaller numbers reflect faster burning)
Cerium(III) nitrate hexahydrate	$Ce(NO_3)_3 \cdot 6H_2O$	Did not burn
Copper nitrate trihydrate	$Cu(NO_3)_2 \cdot 3H_2O$	127.24%
Iron(III) nitrate nonahydrate [a]	$Fe(NO_3)_3 \cdot 9H_2O$	Did not burn
Magnesium nitrate hexahydrate	$Mg(NO_3)_2 \cdot 6H_2O$	Did not burn
Mercury(II) nitrate monohydrate [b]	$Hg(NO_3)_2 \cdot H_2O$	141.76%
Nickel nitrate hexahydrate	$Ni(NO_3)_2 \cdot 6H_2O$	Did not burn
Thallium(III) nitrate trihydrate	$Tl(NO_3)_2 \cdot 3H_2O$	147.36%
Zirconium oxynitrate hydrate [c]	$ZrO(NO_3)_2 \cdot {\sim}6H_2O$	Did not burn

[a.] Iron(III) nitrate nonahydrate could not be processed with drying and sieving. It decomposed at 65°C and unheated material was too moist to sieve; therefore, it was tested in the form it arrived in from the supplier.

[b.] Due to the hazard posed by toxic smoke, this test sample was reduced in size to a 10g conical pile instead of the 30g pile normally used.

[c.] Sigma-Aldrich as well as other vendors list zirconium oxynitrate as a generic hydrate using the notation: $ZrO(NO_3)_2 \bullet xH_2O$. As furnished, the material has approximately six waters of hydration.

An example of non-oxidizer behavior exhibited by a heavily-hydrated nitrate is shown in **Figure 1** for an aluminum nitrate nonahydrate sample. Note the red-hot ends of the wire extending from the conical pile indicating that the ignition wire is energized. This sample did not burn and the experimentalists observed no change in the appearance of the conical pile even though the ignition wire was energized until it failed on each of the three replicates.[7]

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[7] Wires failed due to thermal stress. Wires tended to fail at various times and were highly unlikely to last beyond 3 minutes.

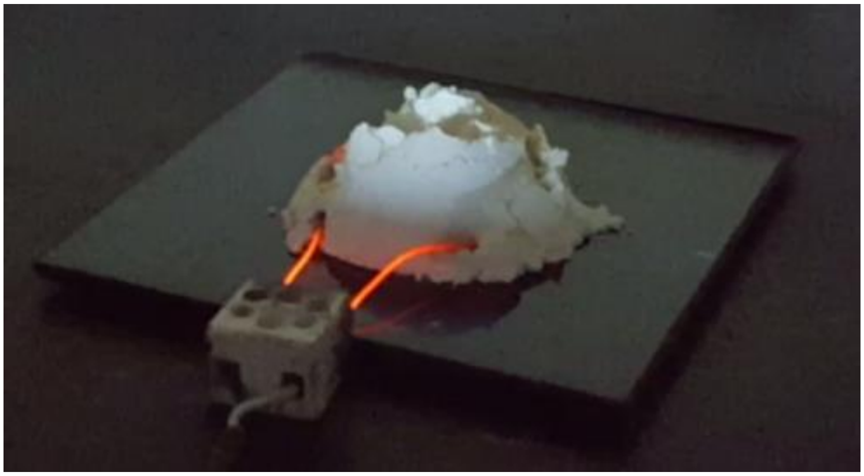

FIGURE 1. The Attempted Combustion of a 4:1, by Mass, Mixture of Aluminum Nitrate Nonahydrate and Cellulose

Scoping study tests on metal nitrate salts that do not contain waters of hydration demonstrated that they all promoted combustion (**Table 9**). Metal nitrate salts that contain more than 3 waters of hydration did not burn at all or did not burn to completion when mixed with cellulose in a 4:1 weight ratio. It was observed that the decomposition of the metal nitrate hydrates appears to require more energy input, in the form of heat, than non-hydrate metal nitrates; thus, it is harder for them to promote combustion of cellulose during the burn tests. This led to the conclusion that metal nitrate salts with more than 3 waters of hydration would not burn to completion due to a significant energy sink created by the waters of hydration present.

Furthermore, in the scoping study test series, nitrate salts were dried at 65ºC, yet the literature[8] shows that much of the dehydration takes place over 100ºC. No attempt was made to dehydrate the hydrated metal nitrates beyond the removal of sorbed water. It is characteristic of most transition metal nitrates that their dehydration cannot be achieved by heating because dehydration proceeds together with partial decomposition of the nitrate groups.[9] Heating many of these

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[8] Malecki, A. et al. (2000). Mechanism of Thermal Decomposition of d-Metals Nitrates Hydrates, Journal of Thermal Analytical Calorimetry, 60, pp.17–23.

[9] Malecka, B. et al. (2015). Thermal decomposition of d-metal nitrates supported on alumina, Journal of Thermal Analytical Calorimetry, 119, pp.1053–1061.

nitrate salts above 100ºC to remove water would have resulted in their decomposition,[10] which would invalidate the scoping test studies.

An example of an accelerated combustion (oxidizer behavior) for a nitrate without waters of hydration is shown for a silver nitrate sample in **Figure 2**. Within an average of 10.86 seconds, the entire pile was completely consumed. This image contrasts dramatically with the non-combustion of the heavily-hydrated aluminum nitrate nonahydrate in **Figure 1**.

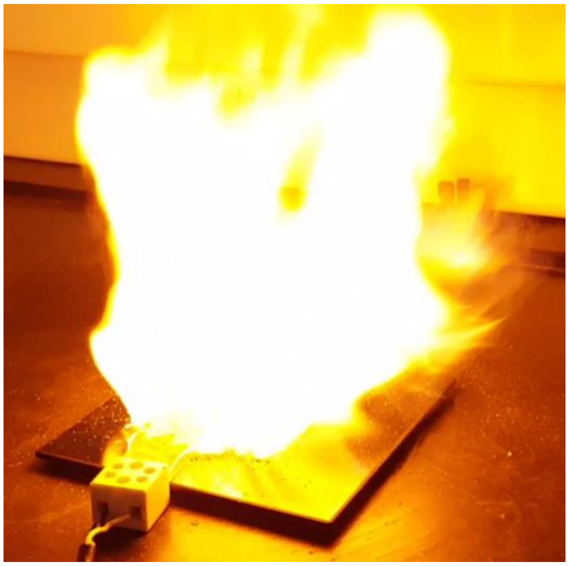

FIGURE 2. The Accelerated Combustion of a 4:1, by Mass, Mixture of Silver Nitrate and Cellulose

From this observed trend, it was decided that the remaining nitrates containing more than three waters of hydration listed in the Appendix B, Initial List of *Scoping Study Oxidizers*, would not be tested. **Table 7** identifies the nitrates that were not tested in the scoping studies. No forms of these metal nitrates will be further tested because, even if available as anhydrous salts, in the waste they will tend to the higher hydration levels shown in the *Typical Waters of Hydration* column in **Table 7**.

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[10] Dean, J. A. (1999). Lange's Handbook of Chemistry, 15th Ed., San Francisco, CA: McGraw-Hill, Inc.

TABLE 7. Metal Nitrates Containing >3 Waters of Hydration Excluded From Scoping Study Testing

Rejected Nitrate	Typical Waters of Hydration	Reference(s)
Bismuth nitrate	5	a
Chromium nitrate	9	a
Cerium(IV) nitrate	5	b
Cobalt nitrate	6	a
Erbium nitrate	5	a
Europium nitrate	5	a
Gadolinium nitrate	6	a
Indium nitrate	$4\,{}^{1}/_{2}$	c, d
Lanthanum nitrate	6	a
Manganese(II) nitrate	6	e
Neodymium nitrate	6	a
Plutonium nitrate	5	f, g
Praseodymium nitrate	6	a
Rhenium nitrate hexahydrate	6	h
Samarium nitrate	6	a
Terbium nitrate	6	a
Thorium nitrate	6	f, i
Uranyl nitrate hexahydrate	6	f, h
Yttrium nitrate	6	a
Zinc nitrate	6	a
Zirconium nitrate	5	e

[a.] Oxidizers were researched for purchase through Sigma-Aldrich (www.sigmaaldrich.com). The highest level of hydration they were available in is indicative of the form expected in the waste.

[b.] Staritsky, Eugene; Walker, Donald (1952) Optical Properties of some Compounds of Uranium, Plutonium and Related Elements, LA-1439, Los Alamos, NM: Los Alamos National Laboratory, pp. 20–21.

[c.] According to the literature (Lide, D. R. (Ed.) (1993). CRC Handbook of Chemistry and Physics, 74th Ed., Ann Arbor: CRC Press), a trihydrate is also possible; however, because waste is stored in vented containers, the waste would tend toward the higher hydration levels.

[d.] Sigma-Aldrich as well as other vendors list indium nitrate as a generic hydrate using the notation: $In(NO_3)_3 \cdot xH_2O$. As furnished, the material has approximately four and a half waters of hydration.

[e.] Budavari, S. et al. (Eds.). (1996). The Merck Index, 12th Ed., Whitehouse Station, NJ: Merck & Co., Inc.

[f.] Kulyako, Y. M. et al. (2008). Solubility of Plutonium Nitrate in Uranyl Nitrate Hexahydrate Melt, Radiochemistry, 50(3), pp. 250-252.

[g] These radioactive nitrates were reintroduced because they are bound by their waters of hydration.

[h] AK identifies this material as a hexahydrate.

[i] Morss, Lester R., et al. (2010). The Chemistry of the Actinide and Transactinide Elements, 4th Ed., Dordrecht, The Netherlands: Springer.

5.2 OXIDIZERS BOUNDED BY PREVIOUS TESTING OR REPRESENTED BY SURROGATES

One scoping study oxidizer was bounded by a combination of results obtained earlier in the scoping study and previously in the chemical literature. Two oxidizers could not be tested directly but are represented by tests conducted with chemically similar surrogates that were available. **Table 8** identifies the bounded oxidizer and the oxidizers represented by surrogates.

TABLE 8. Bounded or Surrogate Oxidizers

Bounded or Represented Oxidizer	Reason for Withdrawal	Reference(s)
Lithium hypochlorite	Lithium hypochlorite (LiClO) was bound with the test results of calcium hypochlorite ($Ca(ClO)_2$). An average burn time of 34.0 seconds for a 4:1 $Ca(ClO)_2$: cellulose mixture was observed in the scoping study tests. In the literature, a LiClO sample in a 4:1 mixture with cellulose burned in 37 seconds. In the same literature, a 1:1 LiClO sample burned in 122 seconds and $Ca(ClO)_2$ burned in 100 seconds. Therefore, lithium hypochlorite was shown to be a slower oxidizer than calcium hypochlorite and bounded by it.	a
Nitric acid	Drying sorbed nitric acid to constant mass could result in the loss of oxidizer due to evaporation. Nitrate salts likely to be produced by neutralization, such as calcium nitrate, magnesium nitrate, potassium nitrate, and sodium nitrate, are included in the scoping study list as representing nitric acid.	-
Tetrapropylammonium nitrate	Tetrapropylammonium nitrate was not readily available for purchase within the experimental timeframe of the scoping studies. Because the alkyl groups of tetrapropylammonium nitrate and tetrabutylammonium nitrate are both linear alkanes of similar chain lengths and similar chemical properties, the tetrapropylammonium nitrate is represented by the tetrabutylammonium nitrate.	-

[a] Buc, E. C. (2009). Oxidizer Classification Research Project: Tests and Criteria, Final Report, Quincy, MA: The Fire Protection Research Foundation.

5.3 Oxidizer Testing Results

The oxidizers tested in the scoping study yielded the combustion test results summarized in **Table 9**. Samples that either ignited with voltage applied to the ignition wire but did not continue burning once the wire was de-energized, did not burn to completion, or demonstrated no combustion activity were all recorded as not having burned and/or as a non-oxidizer. For samples that burned to completion, the individual burn times were recorded, and the average of 3 replicates was calculated. In each case, the chosen oxidizer's average burn rate is compared to the potassium nitrate standard's average burn rate that was measured on the same day. The complete data set is available in Appendix E, *Scoping Study Data*.

Sodium nitrate and potassium nitrate exhibited very close burn times in the testing; therefore, a second test, *Sodium nitrate (Trial #2)*, of 5 replicates was conducted. Sodium nitrate maintained a faster burn time in both trials and was decidedly a faster burning oxidizer than potassium nitrate. These data are in Appendix E, *Scoping Study Data*.

Two results in **Table 9** are not pure oxidizers, but 50-50 mixtures of a fast burning oxidizer and a slow burning oxidizer. In the first mixture, the fast-burning oxidizer was potassium permanganate (Periodic Table Group 1 permanganate) and the slow-burning oxidizer was barium nitrate (Group 2 nitrate). The second mixture included silver nitrate (Group 11 nitrate) as the fast-burning oxidizer and barium nitrate (Group 2 nitrate) as the slow-burning oxidizer. The combinations demonstrated the lack of a synergistic effect, which would have been observed if the combinations of oxidizers made the mixture burn faster than the same total mass of the fast burning oxidizer alone. No synergistic effect of combining similar or dissimilar oxidizers was observed, nor have any been found during an extensive review of the oxidizer literature. While the potential for synergistic effects cannot be completely dismissed, there are no compelling observations or information to support their existence. Therefore, it is not recommended that further synergistic effect studies be pursued.

TABLE 9. Oxidizer Relative Burn Rates [a]

Scoping Study Oxidizer	**Formula**	**Burn Rate Relative to KNO_3 Std. (smaller numbers reflect faster burn times) [b]**
Aluminum nitrate nonahydrate	$Al(NO_3)_3 \cdot 9H_2O$	Did not burn
Ammonium cerium(IV) nitrate	$(NH_4)_2Ce(NO_3)_6$	110.62%
Ammonium persulfate	$(NH_4)_2S_2O_8$	65.20%
Barium nitrate	$Ba(NO_3)_2$	421.57%
Cadmium nitrate tetrahydrate	$Cd(NO_3)_2 \cdot 4H_2O$	Incomplete burn
Calcium hypochlorite	$Ca(ClO)_2$	94.40%
Calcium nitrate tetrahydrate	$Ca(NO_3)_2 \cdot 4H_2O$	Did not burn
Cerium(III) nitrate hexahydrate	$Ce(NO_3)_3 \cdot 6H_2O$	Did not burn
Cesium nitrate	$CsNO_3$	207.29%
Copper nitrate trihydrate	$Cu(NO_3)_2 \cdot 3H_2O$	127.24%
Iron(III) nitrate nonahydrate	$Fe(NO_3)_3 \cdot 9H_2O$	Did not burn
Lead(II) nitrate	$Pb(NO_3)_2$	107.47%
Lead peroxide [c]	PbO_2	28.98%
Lithium nitrate	$LiNO_3$	45.85%
Magnesium nitrate hexahydrate	$Mg(NO_3)_2 \cdot 6H_2O$	Did not burn
Mercury(II) nitrate monohydrate [c]	$Hg(NO_3)_2 \cdot H_2O$	141.76%
Nickel nitrate hexahydrate	$Ni(NO_3)_2 \cdot 6H_2O$	Did not burn
Oxidizer mixture: 50% Barium nitrate 50% Potassium permanganate	$Ba(NO_3)_2$ + $KMnO_4$	102.47%
Oxidizer mixture: 50% Barium nitrate 50% Silver nitrate	$Ba(NO_3)_2$ + $AgNO_3$	112.67%
Potassium bromate	$KBrO_3$	44.60%
Potassium chromate	K_2CrO_4	Incomplete burn
Potassium dichromate	$K_2Cr_2O_7$	586.67%
Potassium iodate	KIO_3	62.22%
Potassium nitrite	KNO_2	16.53%
Potassium periodate	KIO_4	55.56%
Potassium permanganate	$KMnO_4$	26.76%
Potassium persulfate	$K_2S_2O_8$	131.88%
Rubidium nitrate	$RbNO_3$	57.74%
Silver(I) oxide	Ag_2O	31.58%

Scoping Study Oxidizer	Formula	Burn Rate Relative to KNO_3 Std. (smaller numbers reflect faster burn times) [b]
Silver(II) oxide [d]	AgO	21.05%
Silver nitrate	$AgNO_3$	44.64%
Silver nitrite	$AgNO_2$	135.80%
Sodium bromate	$NaBrO_3$	27.61%
Sodium chromate	Na_2CrO_4	Incomplete burn
Sodium dichromate dihydrate	$Na_2Cr_2O_7 \cdot 2H_2O$	147.78%
Sodium nitrate	$NaNO_3$	76.09%
Sodium nitrate (Trial 2) [e]	$NaNO_3$	92.26%
Sodium nitrite	$NaNO_2$	155.52%
Sodium permanganate monohydrate	$NaMnO_4 \cdot H_2O$	Reacted during processing
Strontium nitrate [f]	$Sr(NO_3)_2$	>739.93%
Tetrabutylammonium nitrate	$(C_4H_9)_4NNO_3$	Incomplete burn
Thallium(I) nitrate	$TlNO_3$	93.40%
Thallium(III) nitrate trihydrate	$Tl(NO_3)_3 \cdot 3H_2O$	147.36%
Zirconium oxynitrate hydrate	$ZrO(NO_3)_2 \cdot {\sim}6H_2O$	Did not burn

[a] Some oxidizer information listed here represents oxidizers that were eliminated **(***italicized***)** from further scoping study testing (7 nitrate salts with greater than 3 waters of hydration and sodium permanganate monohydrate which posed a reactivity hazard when mixed with cellulose). Data acquired during the scoping study testing were used to refine the testing parameters and generate Appendix E, *Scoping Study Data*.

[b] Samples that ignited with voltage applied to the ignition wire but did not continue burning once the wire was de-energized, did not burn to completion, or demonstrated no combustion activity are noted as “Did not burn.”

[c] Test sample size was reduced to a 10g conical pile due to hazard posed by toxic smoke from burning lead or mercury compound. The standard was also reduced to a 10g conical pile to be representative.

[d] Silver(II) oxide is actually a silver(I)-silver(III) oxide (Budavari, S. et al. (Eds.). (1996). The Merck Index, 12th Ed., Whitehouse Station, NJ: Merck & Co., Inc.).

[e] Sodium nitrate and potassium nitrate have very close burning times. Five replicates were done to verify that sodium nitrate promotes faster burning of cellulose than potassium nitrate.

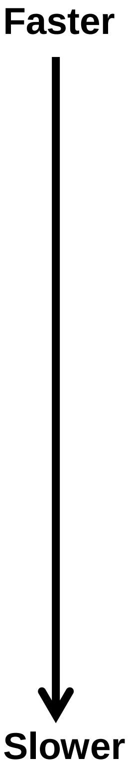
[f] The final burn times for strontium nitrate were not recorded except to note that they were greater than 180 seconds.

5.4 FAST BURNING OXIDIZERS

Table 10 summarizes the oxidizers tested that returned a faster burning rate than potassium nitrate (KNO_3) when mixed with cellulose in a 4:1 weight ratio. In each case, the oxidizer’s average burn rate was compared to the KNO_3 standard’s average burn rate recorded on the same day.

TABLE 10. Fast Burning Oxidizers

Scoping Study Fastest Burning Oxidizers	Formula	Burn Rate Relative to KNO_3 Std. (smaller numbers reflect faster burning)
Potassium nitrite	KNO_2	16.53%
Silver(II) oxide	AgO	21.05%
Potassium permanganate	$KMnO_4$	26.76%
Sodium bromate	$KBrO_3$	27.61%
Lead peroxide [a]	PbO_2	28.98%
Silver(I) oxide	Ag_2O	31.58%
Potassium bromate	$KBrO_3$	44.60%
Silver nitrate	$AgNO_3$	44.64%
Lithium nitrate	$LiNO_3$	45.85%
Potassium periodate	KIO_4	55.56%
Rubidium nitrate	$RbNO_3$	57.74%
Potassium iodate	KIO_3	62.22%
Ammonium persulfate	$(NH_4)_2S_2O_8$	65.20%
Sodium nitrate [b]	$NaNO_3$	76.09%
Thallium(I) nitrate	$TlNO_3$	93.40%
Calcium hypochlorite	$Ca(ClO)_2$	94.40%



[a] Test sample was reduced for sample and standard to 10g conical pile due to hazard posed by toxic smoke.

[b] A second trial for $NaNO_3$ had an average burn time 92.26% relative to KNO_3. This is the only oxidizer for which more than one trial was conducted. This is because there was an effort to discover the fastest burning, common nitrate salt and a second trial was desired to confirm that $NaNO_3$ had a faster average burn time than KNO_3.

Potassium nitrite (KNO_2) is the fastest burning oxidizer expected to be present in the waste streams. Therefore, it is recommended that potassium nitrite be selected as the bounding oxidizer used in the QL1 tests.

6.0 CONCLUSIONS

Potassium nitrite was identified as the bounding oxidizer within the parameters of this investigation because, of the oxidizers likely to be found in the waste streams, it promotes the fastest burning of cellulose when mixed in a 4:1 weight ratio with cellulose. Potassium bromate is recommended as the reference standard oxidizer for the QL1 study so as to more closely adhere to EPA SW-846 Method 1040.[11]

Before generalizing these results to any waste stream, careful consideration of the specific conditions present in each waste container is urged so that false confidence about the status of any particular waste is avoided.

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[11] EPA (2007). EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 1040, Test Method for Oxidizing Solids, Rev.0. Retrieved from: https://www.epa.gov/hw-sw846/sw-846-test-method-1040-test-method-oxidizing-solids

APPENDIX A

49 CFR §172.101 Oxidizers Listed in the AK

49 CFR §172.101 Oxidizers Listed in the AK
Alkaline permanganate
Aluminum nitrate nonahydrate
Americium nitrate
Ammonium cerium (IV) nitrate
Ammonium nitrate
Ammonium perchlorate
Ammonium permanganate
Ammonium persulfate
Antimony nitrate
Aqua Regia (10% hydrochloric acid, 90% nitric acid)
Barium nitrate
Barium perchlorate
Bismuth nitrate
Bromine chloride
Bromine pentafluoride
Bromine trifluoride
Cadmium nitrate
Calcium hypochlorite
Calcium nitrate
Calcium perchlorate
Ceric perchlorate
Ceric nitrate
Cerium nitrate
Cesium nitrate
Chromium nitrate nonahydrate
Chromium trioxide
Cobalt nitrate
Copper nitrate
Curium nitrate
Dibenzoyl peroxide
Dibutyl peroxide (tertiary)
Erbium nitrate
Europium nitrate
Europium perchlorate

49 CFR §172.101 Oxidizers Listed in the AK
Ferrous perchlorate
Gadolinium nitrate
Gadolinium perchlorate
Glyceregia (nitric acid hydrochloric acid and glycerol)
Hydrogen peroxide
Hydroxylamine nitrate
Hypochlorite (unspecified)
Indium nitrate
Iron (III) nitrate nonahydrate
Lanthanum nitrate
Lead nitrate (lead II nitrate)
Lead perchlorate
Lead peroxide
Lithium hypochlorite
Lithium nitrate
Lithium perchlorate
Magnesium nitrate
Magnesium perchlorate (anhydrous)
Manganous nitrate
Mercuric nitrate (mercury II nitrate)
Neodymium nitrate
Neodymium perchlorate
Neptunium nitrate
Nickel nitrate hexahydrate
Nitrate
Nitric acid
Nitric acid - Nitradd solution
Nitrites (nonspecific)
Nitric oxide
Nitrous oxide
OxOut 536 (water, ammonium bifluoride, and nitric acid)
Palladium nitrate
Perchlorate
Perchloric acid

49 CFR §172.101 Oxidizers Listed in the AK
Permanganate
Peroxide
Persulfate
Plutonium nitrate
Plutonium nitrate + aluminum nitrate
Plutonium perchlorate
Plutonium peroxide
Potassium bromate
Potassium chlorate
Potassium chromate
Potassium dichromate
Potassium iodate
Potassium nitrate
Potassium nitrite
Potassium perchlorate
Potassium periodate
Potassium permanganate
Potassium persulfate
Praseodymium nitrate
Rhenium nitrate hexahydrate
Rhodium nitrate
Rubidium nitrate
Ruthenium nitrate
Samarium nitrate
Samarium perchlorate
Silver(I) oxide
Silver(II) oxide
Silver nitrate
Silver nitrite
Silver perchlorate
Sodium bromate
Sodium chlorate
Sodium chlorite
Sodium chromate

49 CFR §172.101 Oxidizers Listed in the AK
Sodium dichromate dihydrate
Sodium hypochlorite
Sodium nitrate
Sodium nitrite
Sodium perchlorate
Sodium permanganate
Sodium peroxide
Strontium nitrate
Tellurium nitrate
Terbium nitrate
Tetrabutylammonium nitrate
Tetrabutylammonium perchlorate
Tetraethylammonium perchlorate
Tetramethylammonium perchlorate (Tetramethylamine perchlorate)
Tetrapropylammonium nitrate
Thallium nitrate
Thallium perchlorate
Thorium nitrate
Tin nitrate
Turco 4502 (contains potassium hydroxide, potassium permanganate, and potassium chromate)
Uranyl nitrate hexahydrate
Vanadyl nitrate
Ytterbium perchlorate
Yttrium nitrate
Zinc nitrate
Zirconium nitrate
Zirconium oxynitrate

APPENDIX B

Initial List of Scoping Study Oxidizers

*Some oxidizers previously excluded due to unsafe radioactive properties remain on this list because they are bounded as nitrate salts with greater than 3 waters of hydration

Initial List of Scoping Study Oxidizers
Aluminum nitrate nonahydrate
Ammonium cerium (IV) nitrate
Ammonium persulfate
Barium nitrate
Bismuth(III) nitrate pentahydrate
Cadmium nitrate tetrahydrate
Calcium hypochlorite
Calcium nitrate tetrahydrate
Cerium(III) nitrate hexahydrate
Cerium(IV) nitrate pentahydrate
Cesium nitrate
Chromium nitrate nonahydrate
Cobalt nitrate hexahydrate
Copper nitrate trihydrate
Erbium(III) nitrate pentahydrate
Europium(III) nitrate pentahydrate
Gadolinium(III) nitrate hexahydrate
Indium nitrate hydrate
Iron (III) nitrate nonahydrate
Lanthanum(III) nitrate hexahydrate
Lead(II) nitrate
Lead peroxide
Lithium hypochlorite
Lithium nitrate
Magnesium nitrate hexahydrate
Manganese(II) nitrate hexahydrate
Mercury(II) nitrate monohydrate
Neodymium(III) nitrate hexahydrate
Nickel(II) nitrate hexahydrate
Nitric acid
Plutonium nitrate pentahydrate
Potassium bromate
Potassium chromate
Potassium dichromate

Initial List of Scoping Study Oxidizers
Potassium iodate
Potassium periodate
Potassium nitrate
Potassium nitrite
Potassium permanganate
Potassium persulfate
Praseodymium(III) nitrate hexahydrate
Rhenium nitrate hexahydrate
Rubidium nitrate
Samarium(III) nitrate hexahydrate
Silver(I) oxide
Silver(II) oxide
Silver nitrate
Silver nitrite
Sodium bromate
Sodium chromate
Sodium dichromate dihydrate
Sodium nitrate
Sodium nitrite
Strontium nitrate
Terbium(III) nitrate hexahydrate
Tetrabutylammonium nitrate
Tetrapropylammonium nitrate
Thallium(I) nitrate
Thallium(III) nitrate trihydrate
Thorium nitrate hexahydrate
Uranyl nitrate hexahydrate
Yttrium(III) nitrate hexahydrate
Zinc nitrate hexahydrate
Zirconium(IV) nitrate pentahydrate
Zirconium oxynitrate hydrate

APPENDIX C

Oxidizer Sorbent Tests

Attachment 1 – Oxidizer Sorbent Tests

Calibration of Balance:

Follow the procedure for calibration of the balance. After zeroing the balance record the weight of three certified weights (1 g, 50 g, and 1 kg, 3x per weight), record the balance readings in the laboratory notebook. If the balance readings for a single weight are varying greater that 1 wt % of the certified weight, re-calibrate the balance.

Make up Bulk 4:1 Oxidizer Cellulose Material for Screening Tests:

The oxidizer to be tested needs to have a particle size no larger than 0.5 mm (pass through a 32-mesh sieve using the Roto-Tap set to at least 45 seconds). If the sample needs to be processed to reach the required particle size, place the sample in the mill and lightly grind the material until it passes through the required 32-mesh sieve. At least twelve hours prior to testing place the required amount of oxidizer salt (greater than 85 grams) in the 65 °C oven and at least four hours prior to testing place the required amount of cellulose in the 105 °C oven for drying (greater than 25 grams of cellulose). Once drying is complete, store the oxidizer and cellulose in separate desiccators and cool to ambient temperature until they are to be used (within 24 hours). If more than 24 hours elapses, the drying cycle must be repeated. Samples are prepared individually as needed for each burn test.

> **Note –** Prior to testing an oxidizer that has not already been evaluated, prepare a small (4 gram oxidizer to 1 gram cellulose) sample. Work this sample so that it is thoroughly mixed using friction and stirring. If this sample sparks, flames or flashes upon mixing and handling, cease testing this oxidizer and follow the process outlined in Section 6.0 Waste Disposal, for disposing of any portion of remaining oxidizer: cellulose mixture.

1) Weigh 24.0 ±. 0.1 g of oxidizer into a labeled sample jar (adjusting the mass of sample to account for water of hydration, if necessary).
2) Record the mass of the oxidizer used in the laboratory notebook.
3) Weigh 6.25 ± 0.05 g (excess to allow for loss during transfer) of cellulose into another labeled sample jar.
4) Record the mass of the cellulose used in the laboratory notebook.
5) Transfer the oxidizer and the cellulose into the fume hood and add the cellulose to the oxidizer sample container mixing thoroughly without placing hands over the mixing container.
6) Mix the oxidizer and cellulose until it is a homogenous mixture (appears to be all one uniform color).

7) Transfer the cellulose sample jar out of the fume hood and weigh and record the cellulose left in the container, noting any adjustments to the cellulose mass used in the sample.
8) Proceed to burn test this mixture (see Run Burn Tests section within this attachment).

Conduct Cellulose and Engineered Organic Polymer Sorbent (EOPS) Comparison Tests

The oxidizer (potassium bromate) to be used in these tests needs to have a particle size no larger than 0.5 mm (pass through a 32-mesh sieve using the Roto-Tap at 45 second intervals). At least twelve hours prior to testing place the potassium bromate in the 65 ± 2 °C oven and place the EOPS in the 105 °C oven for drying. At least four hours prior to testing, place the cellulose in the 105 °C oven for drying. Once drying is complete, store the oxidizer and cellulose in separate desiccators until they are to be used (within 24 hours). If more than 24 hours elapses, the drying cycle must be repeated. Make up a batch sample containing 3:2 potassium bromate to cellulose and EOPSs as follows:

1) Weigh 24.0 ± 0.1 grams of potassium bromate into a labeled sample jar.
2) Record the mass of the potassium bromate used in the laboratory notebook.
3) Weigh 6.25 ± 0.1 g (excess to allow for loss during transfer) of cellulose or EOPS into another labeled sample jar.
4) Record the mass of the cellulose used in the laboratory notebook.
5) Transfer the potassium bromate and cellulose or EOPS into the fume hood and add the cellulose or EOPS to the oxidizer sample container mixing thoroughly without placing hands over the mixing container.
6) Mix the potassium bromate and the cellulose or EOPS until it is a homogeneous mixture.
7) Transfer the cellulose or EOPS sample jar out of the fume hood and weigh and record the cellulose or EOPS left in the container, noting any adjustments to the cellulose or EOPS mass used in the sample.
8) Proceed to burn test this mixture (see Run Burn Tests).

Preparation of Saturated Oxidizer Solution and Test for Sorbing Capacity

Once the strongest oxidizer has been identified, the following tests will be performed to determine the sorbent capacity of example inorganic and organic sorbing materials. Zeolite will be used as the inorganic sorbing material and Nochar N960 will be used to represent EOPS. Ensure the oxidizer: sorbent sample is < 500 µm particle size by grinding and sieving with a 35 mesh sieve prior to analysis.

1) Weigh and record the weight of a labeled Erlenmeyer flask.
2) Add ~400 mL of distilled water to the Erlenmeyer flask.
3) Weigh and record the weight of the water added to the Erlenmeyer flask.
4) Add 140 grams of potassium nitrate (or a greater mass of salt depending on the solubility of the salt being used) to the water.
5) Weigh and record the amount of salt added to the solution.
6) Take the solution to boiling with stirring.
7) Cool the solution once it has reached the boiling point.
8) Weigh and record the weight of the filter and the receiving flask that will be used for removal of excess salt from the solution at room temperature.
9) Dry the filter.
10) Weigh and record the weight of the filter with the excess salt present.
11) Determine the amount of salt left on the filter.
12) Weigh and record the weight of the solution and the volume of the solution in the receiving flask.
13) Determine the concentration of the salt in the solution at room temperature adjusting for salt lost on the filter.
14) Weigh approximately 50 grams of inorganic sorbing material into a suitably sized beaker or 30 grams of organic sorbing material into a suitable sized beaker to allow for a volume increase of 2x that of the original dry organic sorbing material.
15) Record the weight of the beaker and sorbing materials.
16) For inorganic sorbing media (zeolite): Add 40 mL of saturated salt solution into the sorbing matrix.
    For organic sorbing media (Nochar N960): Add 120 mL and check for free liquids by gently turning the beaker on its side to verify all of the liquid has been sorbed by the media.
17) Continue to add aliquots of solution until free liquid is observed.
18) Record the volume of solution added and the final weight of the mixture.
19) Repeat 1 through 18 in triplicate taking the final solution volume to the point that no free liquid is observed (e.g. 5 mL less than the last aliquot where free liquid was observed in step 16).
20) Transfer the bulk "sample" into portions in pre-labeled evaporator dishes or into a pre-labeled tray.
21) Weigh and record the weight of the sample dishes or tray and dry the sample in a 105 °C oven.
22) Weigh the sample(s) after at least 12 hours of drying and then repeat drying for at least 4 hour intervals until a constant weight is achieved (within 0.5 wt % of the sample mass after 12 hours of drying).

23) Cool and store the sample(s) in a desiccator before weighing and keep in the desiccator until used, if the sample will be used within 24 hours of drying.
24) If more than 24 hours have elapsed from the time of drying, dry the sample for at least four hours and repeat drying until a constant weigh is achieved.
25) Prior to performing burn tests on samples containing inorganic sorbents, dry cellulose fibers in a layer < 25 mm in thickness at 105 °C for four hours and cool and store in desiccator until required (use within 24 hours) if cellulose will be used.
26) For inorganic sorbing media (zeolite): Run burn tests on a 30 g pile of sorbent in a 4:1 oxidizer: sorbent sample to cellulose ratio..
27) For organic sorbing media (Nochar N960): Run burn tests on a 30 g pile of the oxidizer: sorbent sample.

Obtain Preliminary Conditions Data and Compare Conditions between Burn tests:

Note: Tests must be run when ambient conditions are the following:
Temperature: initial temperature ± 5 °C
Relative Humidity: initial relative humidity ± 10 %

1) Record the time of the measurements.
2) Prior to burn tests, record the relative humidity and the temperature in the work area.
3) Record the time, relative humidity and temperature between burn test series.

Note - When oxidizer salts are being compared, the reference standard will be potassium nitrate. Otherwise, a 3:7 potassium nitrate reference standard is used.

4) If the conditions are outside of the range provided above (temperature changed more than ± 5 °C, or relative humidity is greater than ± 10 % from the original starting conditions), the potassium nitrate salt mixture or a 3:7 potassium bromate reference standard must be tested and the results recorded before testing can proceed.
5) Ensure the ignition wire temperature is 1000 °C but no greater than 1050 °C prior to performing the burn tests and unplug the wire from the variac before setting the wire in the sample.
6) Record the temperature of the wire in the laboratory notebook.

Run Burn Tests

Burn tests will be run according to the 1040 Oxidizer Test Method section 11.2 (or equivalent).

1) If a reference standard will be run, ensure the particle size of the potassium bromate is between 150 and 300 µm before using in the burn tests by passing through a 48-mesh sieve and collecting what does NOT pass through a 100-mesh sieve.
2) Ensure the particle size of the oxidizer salt: sorbent sample or the oxidizer salt is < 500 µm before using in the burn tests by passing it through a 500 µm sieve.
3) Prepare a 30 ± 0.1 g aliquot of 3:7 standard reference by weighing 9.00 ± 0.1 g of potassium bromate and 21.25 ± 0.1 g of cellulose and transferring the cellulose to the potassium bromate in the fume hood with mixing.
4) Transfer the 30 g mixture of the 3:7 potassium bromate: cellulose reference standard into the 60 ^{0} glass sample funnel.
5) Position the ceramic tile in a fume hood approximately 20 cm (8 in.) from the front of the hood in an area of laminar airflow. Air flow across the test sample should be minimal (approximately 0.5 – 0.7 m/s), but sufficient to prevent the escape of fumes or smoke into working areas.
6) Place the sample on the ceramic tile so the ignition wire can be placed onto the sample pile so that the wire is centered.
7) Position the ignition wire into the sample pile carefully to avoid displacing the pile base.
8) Measure the burning time of the 3:7 potassium bromate: cellulose reference standard by plugging in the ignition wire to the variac and turning on the power and timing the burning event.

Note – If the sample shows no sign of burning after 5 seconds, turn the wire back on for a total of 3 minutes and record the results as > 3 minutes if the sample has not been consumed in that time.

9) Record the burn time in the laboratory notebook.
10) Ensure the ignition wire is off at 5 seconds after it is energized.
11) Verify the variac is switched off, the light is off on the variac and the ignition wire has been unplugged.
12) Repeat steps 5 through 12 for the remaining aliquots to be analyzed.
13) Weigh out five 30 g ± 0.1 g aliquots of 1:1 nitrate:sorbent sample to cellulose (15 g nitrate:sorbent sample and 15 g cellulose) or 4:1 nitrate:sorbent sample to cellulose.(24 g nitrate:sorbent sample to 6 g cellulose) as required for the specific tests being performed.

14) Perform the burn test as described in steps 5 through 12 above for all of the sample aliquots.
15) At the completion of burn tests for each set of aliquots, record the room temperature and relative humidity and the time the measurement was taken in the laboratory notebook.

Waste Disposal:

Upon completion of a burn test, dispose of the sample, the ignition wire and other sample solids in room waste containers (except for the chromium, lead and silver oxidizer salt wastes and ash). Rinse water from cleaning glass ware will be collected in a carboy and transferred to CEMRC for testing and disposal. Dispose of the ash and residue from lead, silver and chromium nitrate testing in separate containers identified as RCRA hazardous waste. Collect solutions from calcium hypochlorite and sodium chlorate in separate one liter bottles that are labeled according to contents.

The hazardous waste stream that will be generated will consist of liquids and solids containing residues of barium nitrate (D005), cadmium nitrate (D006), chromium nitrate and potassium chromate (D007), lead nitrate and lead peroxide (D008), mercuric nitrate (D009), and silver nitrate (D011) which are Resource Conservation and Recovery Act (RCRA) hazardous wastes. RCRA waste will be collected in containers marked “hazardous waste” and the constituents and hazardous waste codes will be written on each container. These containers will be transferred to CEMRC for disposal.

APPENDIX D

Method 1040 Equipment

EPA SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, Method 1040, *Test Method for Oxidizing Solids*

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

6.1 An ignition source -- An inert metal wire of platinum or nickel that can be electrically heated to approximately 1000 °C and formed into the shape shown in Figure 1 (e.g., a 30 ± 1 cm in length, 0.6 ± 0.05 mm in diameter, 22 guage, ARCOR nickel/chromium heating wire, PN 66258-102, from Fisher Scientific, or equivalent)

6.2 Low-heat conducting, non-combustible, impervious ceramic tile or equivalent material, of sufficient size to support a 70-mm diameter test sample -- This tile will be used as a platform to conduct the burning rate tests for the reference substances and waste mixtures.

NOTE: A complete burn mold for this test (Figure 2) is available from Associated Design and Manufacturing Co.; Lorton, Virginia, 22079.

6.3 A thermocouple -- To measure the temperature of the ignition wire (e.g., an Omega thin wire thermocouple, PN P13R-010, or equivalent). Instead of a thermocouple, an infrared thermometer may be used.

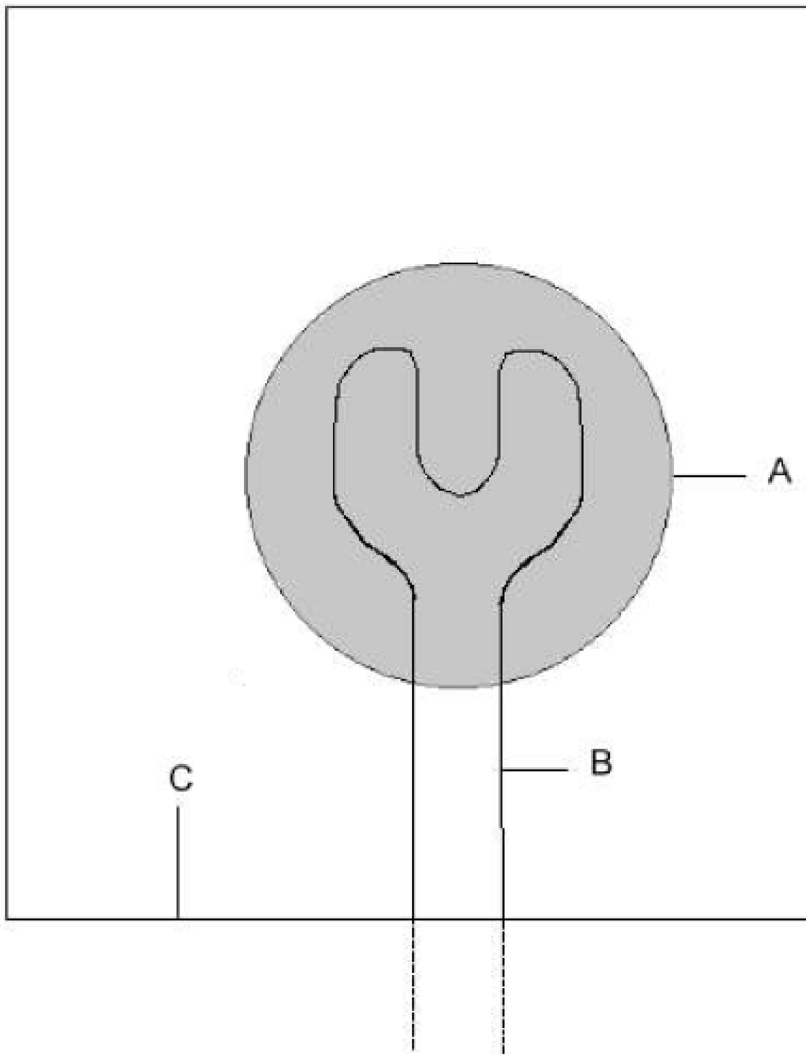
6.4 A calibrated thermometer to measure room temperature.

6.5 Standard sieves, 0.150-mm, 0.300-mm, and 0.500-mm (Tyler screen scale equivalent of 100-, 48-, and 32-mesh respectively).

6.6 A 60 ° glass funnel, sealed at the narrow end, with an internal diameter of 70 mm -- This funnel is used to form the reference standards and waste mixtures into truncated conical piles with a 70-mm base.

6.7 Stopwatch capable of measuring 0.1 sec increments.

TEST PLATE AND IGNITION WIRE
(not to scale)



A = Outline of the base of the 70-mm conical pile

B = Approximate position and shape of the ignitor wire

C = Ceramic tile or other non-combustible material, approximately 150 mm on each side

FIGURE 1. Ignition Wire Assembly[12]

The final burn station setup prior to energizing the ignition wire for the combustion test will resemble the picture in **Figure 2.**

FIGURE 2. Burn Test Station Setup

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[12] EPA (2007). EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 1040, Test Method for Oxidizing Solids, Rev.0. Retrieved from: https://www.epa.gov/hw-sw846/sw-846-test-method-1040-test-method-oxidizing-solids

APPENDIX E

Scoping Study Test Data

APPENDIX E

Burn Times on 4:1 (by mass) oxidizer to cellulose

Oxidizer	Oxidizer Formula	Date	Ambient Relative Humidity (%)	Ambient Temperature (°C)	Burn Time 1 (seconds)	Burn Time 2 (seconds)	Burn Time 3 (seconds)	Burn Time 4 (seconds)	Burn Time 5 (seconds)	Burn Time Average (seconds)	KNO_3 Std. Ambient Relative Humidity (%)	KNO_3 Std. Ambient Temperature (°C)	KNO_3 Std. Burn Time 1 (seconds)	KNO_3 Std. Burn Time 2 (seconds)	KNO_3 Std. Burn Time 3 (seconds)	KNO_3 Std. Burn Time 4 (seconds)	KNO_3 Std. Burn Time 5 (seconds)	KNO_3 Std. Average Burn Time (seconds)	Sample Burn Time Relative to KNO_3 Std. (%) (smaller is faster)
Aluminum nitrate nonahydrate	$Al(NO_3)_3 \cdot 9H_2O$	1/28/2016	26.6	21.1	DNB	DNB	DNB	NM	NM	-	26.6	21.1	41.72	36.47	29.62	NM	NM	**35.94**	-
Ammonium cerium(IV) nitrate	$(NH_4)_2Ce(NO_3)_6$	3/22/2016	40.0	15.2	22.47	21.35	20.78	NM	NM	**21.53**	40.0	18.9	15.50	19.78	23.12	NM	NM	**19.47**	**110.62%**
Ammonium persulfate	$(NH_4)_2(SO_4)_2$	6/3/2016	43.7	22.6	25.06	23.69	22.75	NM	NM	**23.83**	43.7	22.1	31.88	37.69	40.09	NM	NM	**36.55**	**65.20%**
Barium nitrate	$Ba(NO_3)_2$	4/21/2016	36.4	19.1	149.00	153.31	136.00	NM	NM	**146.10**	36.4	19.1	33.94	35.15	34.88	NM	NM	**34.66**	**421.57%**
Cadmium nitrate tetrahydrate	$Cd(NO_3)_2 \cdot 4H_2O$	3/16/2016	25.9	22.4	ICB	ICB	NM	NM	NM	-	25.9	22.4	30.50	60.00	39.75	NM	NM	**43.42**	-
Calcium hypochlorite	$Ca(ClO)_2$	5/20/2016	66.4	19.5	41.03	30.25	30.65	NM	NM	**33.98**	60.3	21.4	27.69	45.60	34.69	NM	NM	**35.99**	**94.40%**
Calcium nitrate tetrahydrate	$Ca(NO_3)_2 \cdot 4H_2O$	3/16/2016	25.9	22.4	DNB	DNB	NM	NM	NM	-	25.9	22.4	30.50	60.00	39.75	NM	NM	**43.42**	-
Cerium(III) nitrate hexahydrate	$Ce(NO_3)_3 \cdot 6H_2O$	1/27/2016	33.1	18.9	DNB	DNB	DNB	NM	NM	-	33.4	19.9	20.28	30.10	57.22	NM	NM	**35.87**	-
Cesium nitrate	$CsNO_3$	3/17/2016	28.1	19.9	58.03	51.37	41.88	NM	NM	**50.43**	28.1	19.9	17.63	32.79	22.56	NM	NM	**24.33**	**207.29%**
Copper(II) nitrate trihydrate	$Cu(NO_3)_2 \cdot 3H_2O$	4/22/2016	43.6	19.0	42.50	41.97	33.44	NM	NM	**39.30**	31.4	22.1	24.60	37.00	31.07	NM	NM	**30.89**	**127.24%**
Iron(III) nitrate nonahydrate	$Fe(NO_3)_3 \cdot 9H_2O$	1/29/2016	25.1	24.1	DNB	DNB	DNB	NM	NM	-	NM	NM	NM	NM	NM	NM	NM	-	-
Lead(II) nitrate	$Pb(NO_3)_2$	1/28/2016	26.6	21.1	35.44	40.38	40.04	NM	NM	**38.62**	26.6	21.1	41.72	36.47	29.62	NM	NM	**35.94**	**107.47%**
Lead peroxide#	PbO_2	5/4/2016	28.8	21.9	14.78	12.25	9.00	NM	NM	**12.01**	28.8	21.9	46.78	29.06	48.50	NM	NM	**41.45**	**28.98%**
Lithium nitrate	$LiNO_3$	6/7/2016	43.1	21.4	12.47	15.22	16.69	NM	NM	**14.79**	44.6	21.8	38.63	36.90	22.28	32.46	31.06	**32.27**	**45.85%**
Magnesium nitrate hexahydrate	$Mg(NO_3)_2 \cdot 6H_2O$	1/26/2016	29.9	19.1	DNB	DNB	DNB	NM	NM	-	29.9	19.1	29.22	34.00	33.91	NM	NM	**32.38**	-
Mercury(II) nitrate monohydrate#	$Hg(NO_3)_2 \cdot H_2O$	5/4/2016	28.8	21.9	73.93	60.25	42.09	NM	NM	**58.76**	28.8	21.9	46.78	29.06	48.50	NM	NM	**41.45**	**141.76%**
Nickel(II) nitrate hexahydrate	$Ni(NO_3)_2 \cdot 6H_2O$	3/3/2016	24.9	22.5	DNB	DNB	NM	NM	NM	-	24.9	22.5	20.00	20.63	20.53	NM	NM	**20.39**	-
Oxidizer mixture: 50% Barium nitrate 50% Potassium permanganate	$Ba(NO_3)_2 + KMnO_4$	5/6/2016	29.2	20.9	23.57	23.50	29.56	NM	NM	**25.54**	28.7	22.4	16.00	23.93	34.85	NM	NM	**24.93**	**102.47%**
Oxidizer mixture: 50% Barium nitrate 50% Silver nitrate	$Ba(NO_3)_2 + AgNO_3$	5/17/2016	45.2	20.3	21.60	27.90	26.37	NM	NM	**25.29**	45.0	20.4	17.31	20.25	29.78	NM	NM	**22.45**	**112.67%**
Potassium bromate	$KBrO_3$	3/16/2016	25.9	22.4	17.00	20.59	20.50	NM	NM	**19.36**	25.9	22.4	30.50	60.00	39.75	NM	NM	**43.42**	**44.60%**
Potassium chromate	K_2CrO_4	3/22/2016	40.0	15.2	ICB	ICB	NM	NM	NM	-	40.0	18.9	15.50	19.78	23.12	NM	NM	**19.47**	-
Potassium dichromate	$K_2Cr_2O_7$	3/16/2017	39.2	22.1	154.00	210.00	164.00	NM	NM	**176.00**	43.6	21.8	28.00	32.00	30.00	NM	NM	**30.00**	**586.67%**
Potassium iodate	KIO_3	3/16/2017	43.6	21.8	22.00	14.00	20.00	NM	NM	**18.67**	43.6	21.8	28.00	32.00	30.00	NM	NM	**30.00**	**62.22%**
Potassium nitrite	KNO_2	5/17/2016	46.1	19.0	3.81	4.19	3.13	NM	NM	**3.71**	45.0	20.4	17.31	20.25	29.78	NM	NM	**22.45**	**16.53%**
Potassium periodate	KIO_4	3/16/2017	43.6	21.8	18.00	18.00	14.00	NM	NM	**16.67**	43.6	21.8	28.00	32.00	30.00	NM	NM	**30.00**	**55.56%**
Potassium permanganate	$KMnO_4$	3/11/2016	29.5	20.6	10.16	9.91	11.56	NM	NM	**10.54**	29.5	20.6	25.00	19.18	74.00	NM	NM	**39.39**	**26.76%**
Potassium persulfate	$K_2S_2O_8$	4/22/2016	43.6	19.0	38.85	48.38	59.12	NM	NM	**48.78**	35.8	22.0	24.63	49.35	NM	NM	NM	**36.99**	**131.88%**
Rubidium nitrate	$RbNO_3$	5/20/2016	60.3	21.4	19.69	16.91	25.75	NM	NM	**20.78**	60.3	21.4	27.69	45.60	34.69	NM	NM	**35.99**	**57.74%**
Silver(I) oxide	Ag_2O	3/22/2017	33.9	22.7	9.00	7.00	8.00	NM	NM	**8.00**	34.6	21.9	26.00	28.00	22.00	NM	NM	**25.33**	**31.58%**
Silver(II) oxide	AgO	3/22/2017	36.2	22.9	5.00	6.00	5.00	NM	NM	**5.33**	34.6	21.9	26.00	28.00	22.00	NM	NM	**25.33**	**21.05%**
Silver nitrate	$AgNO_3$	3/17/2016	25.8	21.6	9.85	9.57	13.16	NM	NM	**10.86**	28.1	19.9	17.63	32.79	22.56	NM	NM	**24.33**	**44.64%**
Silver nitrite	$AgNO_2$	6/2/2016	44.3	21.5	38.28	35.16	46.62	NM	NM	**40.02**	43.3	21.1	24.72	34.82	28.87	NM	NM	**29.47**	**135.80%**
Sodium bromate	$NaBrO_3$	4/22/2016	34.1	21.4	8.25	8.97	8.37	NM	NM	**8.53**	32.6	22.1	24.60	37.00	31.07	NM	NM	**30.89**	**27.61%**
Sodium chromate	Na_2CrO_4	3/16/2017	39.2	22.1	ICB	ICB	ICB	NM	NM	-	43.6	21.8	28.00	32.00	30.00	NM	NM	**30.00**	-
Sodium dichromate dihydrate	$Na_2Cr_2O_7 \cdot 2H_2O$	3/16/2017	33.6	22.5	44.00	42.00	47.00	NM	NM	**44.33**	43.6	21.8	28.00	32.00	30.00	NM	NM	**30.00**	**147.78%**
Sodium nitrate	$NaNO_3$	1/27/2016	33.4	19.9	28.06	32.28	21.53	NM	NM	**27.29**	33.4	19.9	20.28	30.10	57.22	NM	NM	**35.87**	**76.09%**
Sodium nitrate (Trial 2)	$NaNO_3$	6/7/2016	44.6	21.8	34.47	23.75	29.25	30.06	31.32	**29.77**	44.6	21.8	38.63	36.90	22.28	32.46	31.06	**32.27**	**92.26%**
Sodium nitrite	$NaNO_2$	3/8/2016	27.0	20.9	29.25	25.31	28.28	NM	NM	**27.61**	27.0	20.9	13.10	22.41	NM	NM	NM	**17.76**	**155.52%**
Strontium nitrate	$Sr(NO_3)_2$	3/17/2016	28.1	19.9	159.00	>180	>180	NM	NM	**>180**	28.1	19.9	17.63	32.79	22.56	NM	NM	**24.33**	**> 739.93%**
Tetrabutylammonium nitrate	$(C_4H_9)_4NNO_3$	6/1/2016	44.0	20.9	ICB	ICB	NM	NM	NM	-	44.8	21.2	46.82	22.59	40.03	NM	NM	**36.48**	-
Thallium(I) nitrate	$TlNO_3$	6/1/2016	44.0	20.9	15.72	38.31	48.19	NM	NM	**34.07**	44.8	21.2	46.82	22.59	40.03	NM	NM	**36.48**	**93.40%**
Thallium(III) nitrate trihydrate	$Tl(NO_3)_2 \cdot 3H_2O$	6/3/2016	47.6	21.3	67.94	34.78	58.87	NM	NM	**53.86**	43.7	22.1	31.88	37.69	40.09	NM	NM	**36.55**	**147.36%**
Zirconium(IV) oxynitrate hydrate	$ZrO(NO_3)_2 \cdot xH_2O$	1/29/2016	25.1	24.1	DNB	DNB	DNB	NM	NM	-	NM	NM	NM	NM	NM	NM	NM	-	-

Legend

🟨	KNO_3 std.
🟥	Faster than KNO_3 std. under same conditions
🟦	Relative burn times
DNB	= Did Not Burn
ICB	= Incomplete Burn
NM	= Not Measured
#	= Oxidizer and standard burned in 10g piles

$$\textbf{Sample Burn Time Relative to } KNO_3 \textbf{ Std. (\%)} = 100\% + \frac{\underline{\text{(Sample Avg. Burn Time - } KNO_3 \text{ Std. Avg. Burn Time)}} \times 100}{KNO_3 \text{ Std. Avg. Burn Time}}$$