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LOS ALAMOS NATIONAL LABORATORY
CARLSBAD OPERATIONS

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Results of Testing the Relative Oxidizing Hazard of Wipes and KMI Zeolite

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Results of Testing the Relative Oxidizing Hazard of Wipes and KMI Zeolite

1.0 INTRODUCTION

This report includes the results from testing performed on the relative oxidizing hazard of a number of organic sorbing wipe materials, as well as KMI zeolite. These studies were undertaken to address a need by the Los Alamos National Laboratory (LANL) Hazardous Materials Management group, which requires a material that can sorb small spills in a glovebox without creating a disposal hazard due to the potential for oxidation reactions, as requested in *Request for Testing of Wipes and Zeolite for Los Alamos National Laboratory Hazardous Materials Group (NPI-7)* (NPI-7-17-002) and *Request for Testing of Chamois Material for Los Alamos National Laboratory Hazardous Materials Group (NPI-7)* (NPI-7-17-005). This set of tests is a continuation of previous testing described in *Results from Preparation and Testing of Sorbents Mixed with* (DWT-RPT-003), which provided data for the Waste Isolation Pilot Plant's Basis of Knowledge. The Basis of Knowledge establishes criteria for evaluating transuranic (TRU) waste that contains oxidizing chemicals.

The relative oxidizing hazard of samples was determined using a modification of the U.S. Environmental Protection Agency, 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), as described in *Test Plan for Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TP-002). In this method, a 30-g conical pile of an oxidizing chemical mixed with a fuel is heated with an electrically-heated wire, and the burning time of the sample is compared to a reference standard of potassium bromate mixed with cellulose. The SW-846 Method 1040 test evaluates the relative oxidizing hazard posed by solid waste; it does not definitively determine whether a sample is an oxidizer. Scoping studies were previously undertaken to determine the bounding conditions to use for this testing. *Oxidizer Scoping Studies* (DWT-RPT-001) reported that the fastest-burning oxidizing chemical present in TRU waste is potassium nitrite (KNO_2), and *Sorbent Scoping Studies* (DWT-RPT-002) reported that the fastest-burning engineered organic polymeric sorbent is Quik Solid. They also found that wet-mixed samples (in which a solution of potassium nitrite is combined with the sorbent, which is dried before burn rate testing) burn faster than dry-mixed samples, and organic sorbents mixed with potassium nitrite burn faster without the addition of cellulose.

In these tests, the modified SW-846 Method 1040 tests were used to evaluate sorbents mixed with potassium nitrite to determine the concentration of potassium nitrite in a sorbent that produces a non-oxidizer result. Modified SW-846 Method 1040 tests were also used to determine the concentration of KMI zeolite necessary to remediate the oxidizing hazard from pure potassium nitrite and from a mixture of Quik Solid and potassium nitrite. KMI zeolite was of interest because it has previously been used by LANL in the packaging of TRU waste. The liquid holding capacity of individual sorbents was also measured.

The sorbents listed in Table 1 were evaluated with the modified SW-846 Method 1040 tests for this report. These sorbents were obtained from suppliers on the Los Alamos National Laboratory – Carlsbad Operations (LANL-CO) Qualified Supplier List (QSL) whenever possible. If sorbents were not available from QSL listed suppliers, they were obtained directly from the manufacturer or another supplier listed on the table. KMI zeolite was given to LANL-CO by the LANL Hazardous Materials Management group. Silk Lens Cloths by Royal Silk Direct Inc. were initially included for testing but were discarded when they displayed poor sorbing capability.

Table 1 – Sorbents Evaluated with Modified SW-846 Method 1040 Tests

Product Name	Primary Chemical Composition	Manufacturer/Distributor	Product #
Cheesecloth Wipers	Cellulose	Fisher Scientific	06-665-29
Hazmat Sorbent SM Pads, Premium	Polypropylene	NPS Corporation	S2-70
Kimtech Pure W4 Wipers	Polypropylene	Fisher Scientific	06-666-12
KMI Zeolite	Clinoptilolite (natural zeolite)	KMI Zeolite Inc.	n/a; provided by LANL
Microfiber Cleaning Cloth (Suede)	80% Polyester 20% Nylon (polyamide)	Photodon	360
PBI Staple Fiber	Sulfonated polybenzimidazole	PBI Performance Products	M51015
PIG BLUE Absorbent Mats	60-70% Cellulose 15-25% Polypropylene 10-15% Polyethylene 10-15% Fire retardant 10-15% Polyester	New Pig Corporation	MSD-179
Quik Solid Mats	70-100% Polyester fabric 10-30% Sodium polyacrylate	The ARK Enterprises, Inc.	QS-RP
Tanner's Select Chamois	Polypeptides (mostly collagen)	Acme Sponge and Chamois	TSX
Wool Felt	Polypeptides (mostly keratin)	National Nonwovens	WCF006

2.0 PURPOSE

This report fulfills a request from the LANL Hazardous Materials Management Group to evaluate the oxidizing hazard of sorbent wipes for potential use in a glovebox setting and the remediation of the oxidizing hazard of samples by adding KMI zeolite.

The purpose of this report is to document: 1) the liquid holding capacity of the sorbing materials listed in Table 1; 2) the concentration of potassium nitrite in these sorbents that produced a non-oxidizer result; 3) the concentration of KMI zeolite necessary to remediate the oxidizing hazard from potassium nitrite and a 3:2 mixture of potassium nitrite to Quik Solid, and 4) the effect of rinsing on the oxidizing hazard of potassium nitrite sorbed onto cheesecloth.

3.0 SCOPE

The scope of this report is limited to the data generated from the performance of *Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TST-002). The work described in this report was performed under the Los Alamos LANL-CO Quality Assurance Program, a Department of Energy – Carlsbad Field Office *Quality Assurance Program Document* (CBFO-94-1012) compliant program. While this report may inform the Basis of Knowledge, it does not discuss the criteria for which waste can be xxx.

4.0 DEFINITIONS, ACRONYMS, AND REFERENCES

4.1 Definitions

Burn rate –	The burn rate is the time, in seconds (s), from the application of electrical power to the ignition wire until the main reaction (e.g., flame, incandescence, or smoke) ends.
Average burn rate –	The average burn rate is the average of up to five individual burn rates from 30-gram (g) replicates of the same sample or duplicate.
Non-oxidizer result –	A non-oxidizer result occurs when the burn rates for all five replicates of a sample and five replicates of a duplicate are slower than the daily potassium bromate:cellulose reference standard or are incomplete burns.
Sorbent –	A sorbent is a material used to absorb or adsorb liquids.

4.2 Acronyms and symbols

g	-	gram(s)
KNO_2	-	potassium nitrite
LANL	-	Los Alamos National Laboratory
LANL-CO	-	Los Alamos National Laboratory - Carlsbad Operations
mL	-	milliliter(s)
L	-	liter(s)
QSL	-	Qualified Supplier List
TRU	-	transuranic
wt. %	-	weight percent
Å	-	Angstrom

°C - degrees Celsius

4.3 References

CBFO-94-1012:	*Quality Assurance Program Document*
DWT-RPT-001:	*Oxidizer Scoping Studies* (LA-UR-16-28553)
DWT-RPT-002:	*Sorbent Scoping Studies* (LA-UR-16-28806)
DWT-RPT-003:	*Results from Preparation and Testing of Sorbents Mixed with Potassium Nitrite* (LA-UR-16-27276)
DWT-TP-002:	*Test Plan for Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical*
DWT-TST-002:	*Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical*
LCO-QP6-2:	*Controlled Document Review and Approval*
LCO-QP17-1:	*Record Management*
NPI-7-17-002:	*Request for Testing of Wipes and Zeolite for Los Alamos National Laboratory Hazardous Materials Group (NPI-7)*
NPI-7-17-005:	*Request for Testing of Chamois Material for Los Alamos National Laboratory Hazardous Materials Group (NPI-7)*

SW-846 Method 1040: *Test Method for Oxidizing Solids*

5.0 APPROACH

There were four goals of the testing: 1) to measure the liquid holding (sorption) capacity of various sorbents, 2) to determine the maximum concentration of potassium nitrite that can be mixed with each sorbent and produce a non-oxidizer result, 3) to determine the minimum concentration of zeolite necessary to mix with an oxidizing sample to produce a non-oxidizer, and 4) to determine whether cheesecloth wipes soaked in a potassium nitrite solution could produce a non-oxidizer result through rinsing with deionized water. Further details of the testing procedure are described in *Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TST-002).

5.1 Sorption capacity

The sorption capacity of each material was measured by dispensing potassium nitrite solution onto a 10- to 50-g sample of wipes or zeolite until free liquid was just observed. At least three replicates were made for each material, and the results were averaged. Wipes were first cut into 1-inch squares.

5.2 Oxidizing chemical concentration

Once the sample's sorption capacity was reached with potassium nitrite solution, the sample was dried at 65 °C to a constant weight. After drying, the KMI zeolite sample was ground and sieved for testing. Dry samples were burn rate tested with the modified Method 1040 test. Five 30-g replicates were tested for each sample; if any replicate had a faster burn time than the average burn time of the daily potassium bromate reference standard, the sample was considered to have an oxidizer result. In that case, a new sample was made with the concentration of the potassium nitrite solution reduced by 10 wt. %. If the material produced a non-oxidizer result, a new sample was made with the concentration of the potassium nitrite solution increased by 10 wt. %, in order to find the highest possible concentration of potassium nitrite that a sample could contain and test with a non-oxidizer result. A duplicate sample was made at the highest non-oxidizing concentration of potassium nitrite with the same amount of sorbent and potassium nitrite solution as the original sample. The duplicate was burn rate tested in a different burn station with different analysts to account for the effect of environmental factors and subjectivity in determining burn rates.

5.3 KMI zeolite remediation

Previous testing (DWT-RPT-003) had determined the concentration of synthetic Sigma Aldrich 4 Å and 10 Å zeolite necessary to remediate an oxidizing sample to produce a non-oxidizer result. In the current testing protocol, KMI zeolite, with an intermediate pore size of 4-7 Å (variable), was similarly tested. KMI zeolite was added in predetermined concentrations to two different oxidizing samples to determine the amount necessary for remediation.

In one test, size-reduced KMI zeolite was dried, then mixed with potassium nitrite that had also been size-reduced and dried. In the second test, Quik Solid was mixed with a potassium nitrite solution such that the ratio of potassium nitrite to Quik Solid in the dried sample would be 3:2. Once that mixture was dried, it was size-reduced and mixed with size-reduced, dried zeolite.

For the burn rate testing, cellulose was added to the pure potassium nitrite sample in 4:1 (sample to cellulose) and 1:1 ratios. Because Quik Solid acts as a fuel, the Quik Solid sample was burn rate tested without the addition of cellulose. If an oxidizer result was obtained through burn rate testing, the concentration of the zeolite in the sample was increased by 10 wt. %. If the sample produced a non-oxidizer result, a duplicate sample was made, which was burn rate tested in a different location with different analysts.

5.4 Cheesecloth rinsing

The effect of rinsing a cheesecloth wipe saturated with potassium nitrite solution was examined to determine whether rinsing with deionized water would be an effective method of mitigating the oxidizing hazard. In these experiments, near-saturated (75 wt. %) potassium nitrite solution was dispensed on approximately 50 grams (6 wipes) of uncut cheesecloth wipes until the sorption capacity of the cheesecloth was reached. For one test, the cheesecloth was soaked in 1 L of deionized water for 1.33 minutes, then wrung out just until no longer dripping. For another test, the cheesecloth was soaked in 1 L of deionized water for 30 minutes before wringing. In the last test, the cheesecloth was soaked in 3 L of deionized water for 2 minutes before wringing. The samples were then dried, cut into 1-inch squares, and burn rate tested.

6.0 DATA AND RESULTS

6.1 Sorption capacity

The results of the sorption capacity measurements are shown in Table 2, listed from most to least sorbing.

6.2 Oxidizing chemical concentration

The maximum concentration of potassium nitrite that each sample contained after drying that produced a non-oxidizer result is shown in Table 3. Two samples were capacity-limited; Hazmat Sorbent SM Pads and Kimtech Pure W4 Wipers produced non-oxidizer results even when mixed with a near-saturated (75 wt. %) potassium nitrite solution to their sorption capacity. KMI zeolite produced a non-oxidizer result at 34 wt. % KNO_2, which was similar to the results for synthetic 4 Å and 10 Å zeolite at 37 wt. % and 49 wt. % KNO_2, respectively (DWT-RPT-003).

Samples that produced oxidizer results when mixed with 5 wt. % potassium nitrite solutions to their sorption capacity were not tested at lower concentrations of potassium nitrite solutions (Table 4). This includes cheesecloth, Quik Solid Pads, and PIG BLUE Absorbent Mats. Note that, excluding the two polypropylene samples that were capacity-limited, the materials with the highest sorption capacity (Quik Solid Pads, PIG BLUE Mats, cheesecloth, wool, microfiber) produced oxidizer results with lower concentrations (5 or 15 wt. % KNO_2) of potassium nitrite solution. While sorbents with a high sorption capacity are preferable for minimizing volume of the final waste product, when they are used to sorb solutions containing oxidizing chemicals, the oxidizing chemical becomes concentrated in the sample, which is then more likely to produce an oxidizer result.

Table 2 – Sorption Capacity of Sorbents

Sorbent	Amount of sorbent (g)	Sorbed solution (mL)	Liquid holding capacity (mL/g)	Average liquid holding capacity (mL/g)
Quik Solid Pads	50.03	850	17.0	20.2
	10.02	222	22.2	
	29.98	645	21.5	
PIG BLUE Absorbent Mats	49.89	483	9.7	9.7
	50.03	481	9.6	
	50.00	490	9.8	
Wool Felt	49.95	424	8.5	8.4
	50.03	419	8.4	
	50.02	421	8.4	
Hazmat Sorbent SM Pads, Premium	20.00	150.0	7.5	7.6
	20.01	159.4	8.0	
	20.02	149.6	7.5	
Cheesecloth Wipers	50.01	272.4	5.4	5.6
	50.02	285.2	5.7	
	50.00	281.4	5.6	
Kimtech Pure W4 Wipers	50.03	225.0	4.5	4.7
	50.03	238.6	4.8	
	49.97	240.0	4.8	
Microfiber Cleaning Cloth (Suede)	50.02	200.0	4.0	3.9
	50.02	182.0	3.6	
	49.97	198.0	4.0	
Tanner's Select Chamois	50.01	134.0	2.7	2.9
	49.96	146.5	2.9	
	50.08	160.0	3.2	
PBI Staple Fiber	50.05	105.9	2.1	2.3
	50.00	116.1	2.3	
	50.00	117.9	2.4	
KMI Zeolite	50.01	23.6	0.47	0.51
	50.02	26.3	0.53	
	50.00	26.1	0.52	
	50.01	27.0	0.54	

Table 3 – Maximum Non-oxidizing Concentration of KNO_2 Mixed with Sorbents

Sorbent	Maximum concentration of KNO_2 in dry sample to test with non-oxidizer result (wt. %)	KNO_2 wt. % solution used to make sample	Sample average burn rate relative to reference standard	Duplicate average burn rate relative to reference standard
Hazmat Sorbent SM Pads, Premium	90	75	Incomplete burns	Incomplete burns
Kimtech Pure W4 Wipers	85	75	Incomplete burns	Incomplete burns
PBI Staple Fiber	40	25	Incomplete burns	Incomplete burns
KMI Zeolite	34	65	4:1 - Incomplete burns 1:1 - 239%	4:1 - Incomplete burns 1:1 - Incomplete burns
Tanner's Select Chamois	32	15	Incomplete burns	Incomplete burns
Wool Felt	30	5	Incomplete burns	Incomplete burns
Microfiber Cleaning Cloth (Suede)	17	5	128%	Incomplete burns

Quik Solid Pads and PIG BLUE Absorbent Mats had some burn rate testing performed previously (DWT-RPT-003). Previously, PIG BLUE Absorbent Mats produced a non-oxidizer result at 32 wt. % KNO_2. In the current set of tests, the original sample produced a non-oxidizer result at 33 wt. % KNO_2, but the duplicate burn rate tests were slightly faster than the daily reference standard, making this sample an oxidizer. Because PIG BLUE Absorbent Mats have a high liquid holding capacity, this test was at the lowest concentration of potassium nitrite solution used. In the previous study, Quik Solid Pads produced an oxidizer result at 35-36 wt. % KNO_2; the five sample replicates were all slower than the reference standard, and four of five duplicate replicates were slower than the reference standard, but one replicate with a burn rate faster than the daily reference standard leads to an oxidizer result. In the current study, Quik Solid Pads produced an oxidizer result at 49 wt. % KNO_2; this sample was made using 5 wt. % KNO_2 solution, and lower concentrations were not tested.

Table 4 – Samples that Tested as Oxidizers at 5 wt. % KNO_2 Solution

Sorbent	Concentration of KNO_2 in dry sample (wt. %)	KNO_2 wt. % solution used to make sample	Sample average burn rate relative to reference standard	Duplicate average burn rate relative to reference standard
Cheesecloth Wipers	22	5	65%	n/a
Quik Solid Pads	49	5	55%	n/a
PIG BLUE Absorbent Mats	33	5	143%	93%

6.3 KMI zeolite remediation

As shown in Table 5, both pure potassium nitrite and the 3:2 mixture of potassium nitrite and Quik Solid produced non-oxidizer results at a concentration of 60 wt. % KMI zeolite. These results are similar to those from previous testing (DWT-RPT-003), in which KNO_2 produced a non-oxidizer result at 50 wt. % 4 Å zeolite and 60 wt. % 10 Å zeolite. Previously, the 3:2 mixture of potassium nitrite and Quik Solid produced a non-oxidizer result at 50 wt. % zeolite for both 4 Å and 10 Å zeolite, which is lower than what was found for KMI zeolite.

Table 5 – Minimum Non-Oxidizing Zeolite Concentrations

<table>
<tr><th rowspan="2">Concentration of KNO_2 (wt. %)</th><th rowspan="2">Concentration of Quik Solid (wt. %)</th><th rowspan="2">Concentration of KMI zeolite (wt. %)</th><th colspan="2">Sample burn rates relative to reference standard</th><th colspan="2">Duplicate burn rates relative to reference standard</th></tr>
<tr><th>4:1 sample:cellulose</th><th>1:1 sample:cellulose</th><th>4:1 sample:cellulose</th><th>1:1 sample:cellulose</th></tr>
<tr><td rowspan="5">40</td><td rowspan="5">0</td><td rowspan="5">60</td><td>incomplete burn</td><td>191%</td><td>incomplete burn</td><td>234%</td></tr>
<tr><td>incomplete burn</td><td>183%</td><td>incomplete burn</td><td>222%</td></tr>
<tr><td>incomplete burn</td><td>217%</td><td>incomplete burn</td><td>217%</td></tr>
<tr><td>211%</td><td>193%</td><td>incomplete burn</td><td>279%</td></tr>
<tr><td>incomplete burn</td><td>171%</td><td>incomplete burn</td><td>217%</td></tr>
<tr><td rowspan="5">25.5</td><td rowspan="5">14.5</td><td rowspan="5">60</td><td colspan="2">incomplete burn</td><td colspan="2">incomplete burn</td></tr>
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</table>

6.4 Cheesecloth rinsing

All three cheesecloth rinsing experiments produced an oxidizer result. No significant difference in burn rates was observed between a 1.33-minute soaking time and a 30-minute soaking time. Because it is implausible to rinse cheesecloth in volumes of water greater than 3 L while working in a glovebox, higher volumes of water were not tested. These experiments were conducted as scoping studies and did not follow the procedure provided in *Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TST-002).

7.0 DATA QUALITY

All samples reported have passed the Data Quality Objectives for standard burn rate time (± 60 seconds), weight (± 0.05 g for measurements of sample constituents in sample preparation and samples portions during burn rate tests, and ± 2 g for dryness), temperature (± 5 ^{0}C for environmental conditions, between 1001 to 1049 ^{0}C for ignition wire temperature, and ± 10 ^{0}C for oven temperature), and relative humidity (± 10 % of initial conditions). All data reported in Tables 3 and 5 have produced non-oxidizer results (defined by each replicate producing non-oxidizer results for both samples and duplicate samples), either burning at burn rates greater than the 3:7 potassium bromate to cellulose reference standard under identical relative humidity and temperature or producing incomplete burns. Samples prepared to generate these results were prepared in accordance with the test procedure *Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TST-002). All data generated as part of sample preparation and burn rate tests have been verified, and the results of this verification are documented in document review forms in accordance with *Controlled Document Review and Approval* (LCO-QP6-2).

8.0 RECORDS

All data sheets generated from implementation and execution of *Preparation and Testing of Sorbents Mixed with an Oxidizing Chemical* (DWT-TST-002) were submitted to the LANL-CO Record Center in accordance with *Record Management* (LCO-QP17-1). The tables in this report are summaries of these data sheet records and have been verified for accuracy during data verification and technical review of this document.