ISSEUD

Specification: D-0101
Revision Number: 13
Revision Date: 02/28/2023
DCN Number: 2039

SPECIFICATION
FOR
PREPACKAGED MgO BACKFILL

Prepared by
Waste Isolation Pilot Plant
Carlsbad, New Mexico

for

U. S. Department of Energy

Cognizant Engineer:	Peter Bogardus	02/21/2023
	Approved for Use	Date
Cognizant Manager:	AJ Ambrose	02/22/2023
	Approved for Use	Date



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RECORD OF REVISION

REV. NO.	REV. DATE	PAGES/SECTIONS AFFECTED	APPROVED BY
12	10/24/22	References added to Section 2.1 Added Step 3.3.1 [E] Added Step 3.3.3	DCN 1976
13	02/28/23	• Revise Attachment B & C • Reformatted entire document into current template	DCN 2039

SPECIFICATION D-0101
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1.0 SCOPE

1.1 Description of Work

1.1.1 This specification covers the definition of the pre-packaged backfill material to be emplaced in the underground areas at the Waste Isolation Pilot Plant (WIPP).

The backfill material will be Magnesium Oxide (MgO), furnished in one bulk package configuration; a super sack. The super sack shall be shipped on a support sheet and racks. The support sheet and rack are supplied by the purchaser.

2.0 APPLICABLE DOCUMENTS

The requirements of this specification have precedence over all referenced documents. Where this specification appears to conflict with the requirements of a referenced document, such conflicts shall be brought to the attention of the purchaser for resolution.

2.1 References

Reference Drawings

163-L-028-W1	Supersack Assembly and Details
163-L-028-W2	Supersack Assembly and Details

Code of Federal Regulations

Title 29, Part 1910	Occupational Safety and Health Standards
Title 30, Part 57	Safety and Health Standards-Metal and Non-Metal Underground Mines
Title 40, Part 191 Subpart B and C, Appdx. MgO	Magnesium Oxide as an Engineered Barrier
ASTM D4747/D4727M-07	Standard Specification for Corrugated and Solid Fiberboard Sheet Stock (container grade) and Cut Shapes
ISO 10058-2:2008 (en)	Chemical Analysis of Magnesite and Dolomite Refractory Products
WP 13-1	NWP LLC Quality Assurance Program Description
WP 05-WH1105	Magnesium Oxide Sample Records Management

2.2 Sustainable Procurement Expectation

Pursuant to Executive Order (EO) 13693, Strengthening Federal Environmental, Energy and Transportation Management, EO 13693, Federal Leadership in Environmental, Energy, and Economic Performance, and EO 13693, Planning for Federal Sustainability in the Next Decade, the Department of Energy (DOE) is committed to managing its facilities in an environmentally conscious manner that will sustain the natural environment and protect the health and well-being of its federal employees and contractor service providers.

Under this contract, the Contractor shall strive to perform work in a manner that sustains and promotes the natural environment and protects the health and well-being of federal employees, contract service providers and visitors using the facility. Sustainable Procurement contracting has several interacting initiatives. Among the initiatives are the following:

- The standards and requirements defined by the General Services Administration (GSA) Sustainable Facilities (SF) Tool for a list of products refer to https://sftool.gov/greenprocurement
- Recycled Content https://www.epa.gov/smm/comprehensive-procurement-guideline-cpg-program
- Bio Based/Bio Preferred https://www.biopreferred.gov/BioPreferred/
- Energy Star – energy efficiency https://www.energystar.gov/products
- TEMP – energy efficiency https://energy.gov/eere/femp/energy-efficient-products-and-energy-saving-technologies
- EPAT – office electronics https://www.epa.gov/ozone-layer-protection
- SNAP – chemical awareness/substitute chemical materials https://www.epa.gov/snap
- Water Sense – Water efficiency https://www.epa.gov/watersense
- Smart Way – Logistics management https://www.epa.gov/smartway

To the extent that the services provided by the Contractor require provision of any of the above products types, goods or services, the Contractor shall provide products in accordance with GSA guidelines. These products, goods and services shall be provided in the manner expected, unless the applicable product is not available, unable to be purchased within 150% of the non-standard product cost, or cannot be procured within a reasonable time. If the Contractor fails to meet expectations, the Contractor shall submit a Non-Sustainable Justification (NSJ) prior to procurement of the product.

3.0 PRODUCT AND DESIGN REQUIREMENTS

3.1 General Requirements

3.1.1 The materials and packaging shall conform to this specification. The packaging shall be capable of transporting the backfill material without breaking or a loss of contents.

3.1.2 The vendor shall provide a Safety Data Sheet (SDS) and a certified material composition analysis for each lot of MgO material. This submittal is only required when a new lot of MgO is introduced (Attachment A #2).

3.1.3 The vendor shall provide a SDS and flame spread, smoke generation, and decomposition product information for the super sack bag material. This submittal is only required when a new bag material is introduced (Attachment A #1).

3.2 Functional Requirements

3.2.1 Supplier filled super sacks will be handled during transportation to the WIPP, at receipt and during material handling operations on support sheets.

3.2.2 Supplier filled super sacks are to be placed on a support sheet suitable for the application.

3.2.3 The filled super sack must be able to retain its contents for a period of two years after emplacement without rupturing from its own weight.

3.2.4 The filled super sack must be able to retain a shelf life of one year of storage on the surface after the fill date.

3.3 Material Requirements

3.3.1 Backfill Material Requirements

A. The sum of magnesium oxide (MgO) plus calcium oxide (CaO) shall be a minimum of 96% +/- 2% mole, with MgO being no less than 90%. The remainder of the material shall not contain any items considered hazardous to people or the environment.

B. Backfill material shall be of a dry granular form, which shall contain less than 0.5% particles which would be retained on a ASTM E11- 04 9.5mm (3/8 inch) sieve (3/8" x down).

C. Backfill material shall be tested as outlined in Attachment B, and shall meet the temperature rise listed in the test procedure.

D. The backfill material shall have a minimum loose bulk density of 87 lb/ft3 (± 5 lb/ft^3).

E. The backfill samples will be tested by the independent lab using Attachment C and reported on Attachment D.

3.3.2 Backfill Super Sack Material Requirements

A. The super sack shall be constructed of woven polypropylene material, with a minimum weight of 8.0 ounces per square yard, coated or uncoated. Assembly shall be by normal bag fabrication methods (i.e., sewing, gluing, etc. Alternate materials and/or fabrication methods are acceptable subject to approval by NWP Engineering prior to shipment). Poly Vinyl Chloride (PVC) material is not acceptable.

B. The assembled (empty) dimensions of the super sack shall be a hexagon which is nominally 61 inches across the flats (a 61-inch inscribed circle) and nominally 25.50 inches high (47.6 ft^3). The super sack shall be constructed such that it retains its shape well enough to not deform beyond a 65-inch hexagon with 12-inch radius corners after filling and shipping.

C. The assembled super sack shall have the capacity to transport a minimum of 4,250 pounds of a material with a loose bulk density as specified in Section 3.3.1D. The super sack shall be designed to comply with the requirements of the Flexible Intermediate Bulk Container Association (FIBCA), including a safety factor of five to one (5:1) on the working load.

D. Any fill opening shall be closed to prevent leakage of material during shipping and handling. No discharge opening is required.

E. The super sack shall function as a barrier to atmospheric moisture and carbon dioxide (CO2) which is equivalent to or better than that provided by a standard commercial cement bag. If required, an independent liner may be added. The liner may be a separate part or attached to the super sack at the manufacturer's option.

3.3.3 Super Sack Inserts

A. The inserts shall be constructed of double wall – V13c, which is double walled with C & B flute glued together with a ½" total thickness and 31 ½ inch length by 19 ½ inch height.

3.4 Fabrication Requirements

3.4.1 The supplier shall provide backfill containers which comply with the requirements for super sacks as outlined in Section 3.3.2 above, depending upon the needs of WIPP. The super sack shall be filled with either 3,000 ± 50 pounds or 4,200 ± 50 pounds of backfill material as specified in Section 3.3.1 above.

3.5 Packaging and Shipping Requirements

3.5.1 Filled backfill containers shall be delivered to the WIPP site by commercial carrier. Shipment racks and containers will be provided by the purchaser. These will be in the form of stackable/collapsible racks for the super sacks (one per rack).

3.5.2 All items shall be packaged as required to provide protection from damage during shipping and handling.

3.5.3 Each individual backfill container shall be clearly labeled with the following information and a copy of the label shall be supplied to the WIPP Waste Handling Operations representative at delivery:

A. The backfill material name as it appears on the SDS.

B. All applicable hazard warnings.

C. The backfill material manufacturer's name and address. If the vendor supplying the filled backfill containers is not the backfill material manufacturer, then both the backfill material vendor's name and address, and the name and address of the vendor supplying the filled containers shall also appear on each container.

D. The date the backfill container was filled with backfill material.

E. The weight of the super sack as indicated on the calibrated scale at the time of filing.

4.0 FIELD EXECUTION

4.1 Inspection

All shipments of backfill containers will undergo random receiving inspection at the WIPP. Containers shall be inspected for:

- Shipping damage
- Proper markings per Section 3.5.3

There are no inspections required other than receiving inspection at the WIPP, as outlined above.

5.0 QUALITY ASSURANCE REQUIREMENTS

5.1 Submittals

5.1.1 The supplier shall provide a certified material composition analysis and a SDS when introducing a new lot of MgO (Attachment A #3).

5.1.2 The supplier shall provide a SDS, flame spread, smoke generation, and decomposition information for the bag material when changing the approved bag material (Attachment A #4).

5.1.3 The supplier shall provide a certificate of compliance stating that the filled containers meet this specification with each shipment.

5.1.4 The vendor shall supply the scale calibration data, including make, model, and calibration records once every calibration period at the time of calibration (at a minimum) (Attachment A #5).

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ATTACHMENT A – DOCUMENT SUBMITTAL REQUIREMENTS

DOCUMENT SUBMITTAL REQUIREMENTS

SUBMIT DOCUMENTS PRIOR TO THE POINTS INDICATED BY THE CODE BELOW:

F – FABRICATION
T – TESTING
S – SHIPMENT
C – CONSTRUCTION/INSTALLATION
A – FINAL ACCEPTANCE

Document Requirements	See Paragraph	For Approval	For Record
1. SDS, flame spread, smoke generation, and decomposition information for Bag Material (only required for change in approved bag material).	3.1.3	F	
2. SDS for MgO Material (only required when introducing a new lot of MgO).	3.1.2	C	
3. MgO certified material composition analysis (only required when introducing a new lot of MgO).	5.1.1	C	
4. Certificate of Compliance (required for each shipment).	5.1.2		A
5. Scale calibration documentation.	5.1.4		A

ATTACHMENT B – PHOSPHORIC ACID TEMPERATURE RISE PROCEDURE

Equipment needed:

- A drying oven able to sustain a temperature of approximately 100 EC.
- Programmable furnace: Thermolyne® Model F30428C-70 or equivalent (able to ramp from 200^0 C to 810^0 C at<15^0 C/min).
- Crucibles: Glazed 10 ml High form porcelain crucibles with covers (do not use 45 ml Vyor dishes); approximate weight of the crucible plus cover is 20 grams.
- Parr Bomb (3x or more): Model 4749 acid digestion bomb
- Desiccator cabinet with viable Drierite® desiccant (capable of hosting 500^0 C crucibles)
- Vacuum filtration system using Whatman® #40 filter paper or PALL® 0.45 µm 47mm Metricel membrane filter
- An NIST-traceable partial-immersion thermometer (or electronic equivalent) able to measure temperatures from 20°C to at least 55°C, with increments of 0.1°C, for measuring MgO solution temperature rise. A second NIST-traceable full-immersion thermometer (or electronic equivalent) with increments of 1°C for measuring room air temperature. A third NIST-traceable full- or partial- immersion thermometer, depending on oven configuration (or electronic equivalent) with increments of 1o for measuring oven air temperature.
- A means of supporting the thermometer in a 400 mL beaker so that the fluid level will be at the immersion mark on the thermometer.
- An analytic balance accurate to within ± 0.01 g, calibrated with NIST traceable standards.
- A source of deionized water.
- A source of ACS Reagent Grade 85% phosphoric acid.
- 400-mL glass beakers.
- Stopwatch (optional).
- Scientific notebook (or suitable alternative which provides permanent archiving of recorded information).

Attachment B – Phosphoric Acid Temperature Rise Procedure

Procedure:

1. Mix one liter of 20% ± 0.1% phosphoric acid (by weight) from deionized water and reagent grade 85% phosphoric acid. After mixing, allow the temperature to return to within 3°C of room temperature (20° to 30°C).

2. Dry at least 60 g of as-received MgO pellets. The sample should be either dried overnight or until it is verified that the center of the MgO mass has been at approximately 100°C for at least half an hour. Verify the drying temperatures using an NIST-traceable thermometer. Pellets should be at room temperature (20° to 30°C) before being tested.

3. Weigh 300 ± 0.5 g of 20% phosphoric acid into a glass 400 mL beaker. Record the weight in the scientific notebook (or suitable alternative).

4. Place a piece of insulating cardboard on the stir plate and then place the beaker on the cardboard. Put a stir bar in the solution and initiate stirring so that a dimple about 1-2 cm deep forms in the center of the beaker.

5. Put the thermometer in the phosphoric acid to the immersion line and fix it in this position so that it cannot change during the remainder of the test.

6. Note the temperature and do not proceed until it has stabilized (e.g., does not change by more than 1 EC in two minutes). Record the temperature once it has stabilized.

7. Weigh 18.00 ± 0.1 g of pre-dried as-received MgO pellets. Record the weight in the scientific notebook (or suitable alternative).

8. Add the MgO to the phosphoric acid and note the starting time of the experiment to the nearest second. Alternatively, start the stopwatch.

9. At one-minute intervals, record the temperature in the scientific notebook (or suitable alternative). Continue taking temperature measurements until the temperature starts to fall (about 30-35 minutes).

10. Perform three replicates of this procedure.

11. To be acceptable, the average maximum temperature rise observed in the three replicates must be at least 20°C.

ATTACHMENT C – REACTIVITY (MOLE % PERICLASE + LIME) ACCEPTANCETEST PROCEDURE

Quantitative weight measurements should be performed accurately to 0.1 mg. Balances should be calibrated or cal checked daily or before use, using non-expired masses that have been cal checked against NIST traceable masses. Temperature tolerances are given in the procedure. Thermocouples with their associated meters must be calibrated or cal checked with a frequency that is not less than the manufacturer's recommendation, against non-expired NIST-traceable temperature standards.

Sample Acceptance and Chain of Custody

1. Inspect sample. The color should be white to off-white. The material should be hard and granular. There should be no signs of adulteration. The sample must come with the Analysis of Shipment sheet provided by the manufacturer.

2. If the sample is acceptable, fill out the chain of custody form. A copy of this form will be transmitted to the WIPP contract Site Technical Representative (STR) with the analysis report within 48 hours of sample acceptance.

3. If the sample is not acceptable, the sample should be rejected and the WIPP contract STR must be contacted within 2 hours of the determination to obtain another sample or arrange for a new sample to be taken.

Compositing Sample

1. Remove 10-10.01 g (weighed accurately to 0.01 g) from each of the samples that the WIPP contract STR has specified to be composited. Mix well and place into a new container, appropriately labeled, before proceeding.

HYDRATION AT 245EC

1. Place 10 ml of DI water and 1 g to 1.1 g (weighed accurately to 0.1 g) ofMgO into a Parr Acid Digestion Bomb.

2. Stir contents with a stir rod to ensure the MgO is wetted. Remove stir rod making sure that significant quantities of solid or liquid are not lost.

3. Close vessel.

4. Repeat steps 1-3 to produce 3 separate samples.

5. Place Parr Bombs into a furnace. Heat the bombs in furnace to a temperature of between 245° - 250°C.

6. Maintain furnace at 245° - 250°C for 2 hours, measured from the time when the furnace reaches its set point. One hour after the furnace reaches its set point check and record the furnace temperature.

7. Remove Parr Bombs from furnace and cool until bombs are able to be safely handled.

8. If a Parr Bomb is dry upon opening, the sample will be discarded. Replacement samples for the dry bomb(s) will be prepared if more than 1 bomb is dry upon opening.

Attachment C – Reactivity (mole % Periclase + Lime) Acceptance Test Procedure (Continued)

LOSS OF IGNITION

1. Record the sample appearance on the benchsheet. Document performance of any required sample composting as directed in the Work order Lab Notes.

2. Inspect the desiccant for signs of expiration, per the color change on the indicator. **This is a critical step**. Re-dry expired desiccant prior to use.

3. Record ID of balances, furnace, crucibles and record initial sample mass.

4. Ignite crucibles at 800 – 810 ^{0}C for 1 hour in the furnace. Crucible tare is determined prior to adding hydrated samples.

5. Allow crucibles to cool to 500 ^{o}C and transfer to desiccator. A fan may be used to facilitate quicker cooling of the desiccator.

6. Determine and record tare-mass of each crucible (previously ignited at 800 – 810 ^{o}C) as soon as the crucibles have cooled sufficiently ($Mass_{DC)}$.

7. Add 1.0 -1.1g of the sample to each Parr bomb Teflon® thimble. Stir contents, ensure that solid loss is minimized (minor loss is acceptable. Prepare each sample in triplicate.

8. Add 10ml reagent water to each Parr bomb.

9. Close vessel and install the Parr bombs in the furnace that has been equilibrated to 245 – 250 ^{o}C. Insert the temperature probe into the top of the Parr bomb so that the tip is firmly pushed against the bottom of the Parr bomb to temperature.

10. Maintain the Parr bombs in the furnace for a minimum of 2 hours. The final temperature of the Parr bomb will be approximately 240 ^{o}C. If temperature is lower, time in the furnace should be extended.

11. Record the time and temperature prior to removing the Parr bombs from the furnace.

12. Remove Parr bombs from the oven, cool until safe handling is facilitated.

NOTE: If any bomb has gone dry, the sample must be discarded. It is necessary to re-analyze any samples that have gone dry during hydration. A fan may be used to facilitate cooling. Water, ice freezer **may not** be used, as the Parr bomb would be damaged by cooling that is too rapid.

Attachment C – Reactivity (mole % Periclase + Lime) Acceptance Test Procedure (Continued)

13. Filter MgO, using vacuum filtration and Pall® 0.45µM filter. Rinse the solid sample with reagent water several times.

14. Transfer each hydrated MgO sample (**excluding** the filter paper) into a crucible that has been tared after firing at 800 °C.

15. Transfer crucibles to furnace.

16. Heat crucibles in furnace that has been equilibrated to 200 – 201 °C for an hour. Maintain crucibles in furnace at 200 – 210 °C for 1 hour. Record the temperature before removing crucibles.

17. Remove crucibles from oven, cool in desiccator (~30 minutes).

18. Record the mass of each crucible to 0.1 mg accuracy ($Mass_{200C}$).

19. Place the crucibles in the furnace (200 °C) and program the furnace to ramp, at < at 15 °C/minute to 800 – 810 °C, then hold at 800 – 810 °C for 20 minutes. Record furnance temperature.

NOTE: Some furnaces cannot exceed this ramp rate.

20. Allow furnace to cool to below 520 °C to facilitate safe removal of crucibles.

21. Transfer crucibles to desiccator and cool before weighting (~ 30 minutes). A fan may be used to facilitate faster cooling of desiccator.

22. When crucibles have cooled enough to weigh (close to ambient temperature), measure and record the masses of each crucible ($Mass_{800C}$) to0.1 mg accuracy.

23. Enter the masses *MassDC*, *Mass200C*, and *Mass800C* into cells B4 through D6, and the chemical-compositional analysis into cells B16 through C19 for this lot on the attached analysis Excel worksheet template. Transmit the worksheet to the WIPP contract STR within 48 hours of sample acceptance.

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ATTACHMENT D – SAMPLE ANALYSIS REPORT

	A	B	C	D	E	F	G	H
1								
2	**LOI results**	**Lot Number:**	s34f	**Date Shipped:**	12/12/2008			
3	**Sample ID**	**Mass DC (g)**	**Mass 200 C (g)**	**Mass 800 C (g)**	**Weight loss %**	**Notebook**	**Notebook pg**	
4	s34f	32.9	33.72	33.475	29.87804878			
5	s34f	32.9	33.72	33.475	29.87804878			
6	s34f	32.9	33.72	33.475	29.87804878			
7								
8								
9	**Average weight loss %**	29.87804878	**Notes:**	Analysis 7-27-22 to 7-29-22 by sdw				
10	**Standard deviation %**	4.35117E-15						
11								

Chemical Analysis

	A	B	C
13	**Chemical Analysis**		
14			
15		**wt %**	**uncertainty (wt %)**
16	**CaO**	1	1
17	**SiO2**	1	0.589107
18	**Al2O3**	1	0.566613
19	**Fe2O3**	1	0.04337461
20			
21	**Molecular formulas**	**Mineral Names**	**MW (g/mol)**
22	Al2O3		101.9602
23	Fe2O3		159.6922
24	SiO2		60.0843
25	Ca(OH)2	Portlandite	74.0926
26	H2O		18.01528
33			
34		**Average**	**Standard Deviation**
49	**Mole % periclase + lime**	**96.6**	**5.2**
50	**wt % periclase + lime**	95.6	1.7
51			
52			
53			