

U. S. DEPARTMENT OF ENERGY
WASTE ISOLATION PILOT PLANT

SPECIFICATION D-0101
Prepackaged MgO Backfill

Table of Contents

<u>Section</u>	<u>Title</u>	<u>Page</u>
1.0	SCOPE	1
2.0	APPLICABLE DOCUMENTS	1
2.1	References	1
3.0	PRODUCT AND DESIGN REQUIREMENTS	1
3.1	General Requirements	1
3.2	Functional Requirements	2
3.3	Material Requirements	2
3.4	Fabrication Requirements	3
3.5	Packaging and Shipping Requirements	3
4.0	FIELD EXECUTION	4
4.1	Inspection	4
5.0	QUALITY ASSURANCE REQUIREMENTS	4
5.1	Submittals	4

Attachments	Number of Pages
A Document Submittal Requirements	1
B Phosphoric Acid Temperature Rise Procedure	2
C Reactivity (mole % Periclase + Lime) Acceptance Test Procedure	2
D Sample Analysis Report	3

SPECIFICATION D-0101 RECORD OF REVISION

ECO/REV#/DATE	PAGES AFFECTED	REVISION DESCRIPTION
8613 Rev. 0 5-23-97	All	Specification Created
8852 Rev. 1 12-18-97	2	Revised 3.3.1.3; deleted CAA test and reaction time, added reference to Attachment 1.
	5	Added 3.5.3.4 to require marking date of fill on sacks. Deleted text and title of 5.1 and 5.1.1 requiring a QA program complying with NQA-1, moved 5.2 Submittals to 5.1.
	10 & 11	Added Attachment 1, specifying the reactivity test equipment and procedure.
9724 Rev. 2 03-08-00	2, 3, 4, 6, Attachment A & B	Reduced the min bulk density to 87 lb/ft ³ , increased the super sack height from 24.5 to 25.5 inches, and increased the super sack weight from 4,100 to 4,200 lb.
9753 Rev. 3 04-04-00	2	Clarify material requirements.
10,182; Add. 1 Rev. 4 12-18-02	All	Remove minisacks - approved by EPA All sections reformatted Figure 3.2 deleted since no longer referenced. Subsequent figures re-numbered.
10874; Rev. 5 10-31-03	Cover page	Changed company name to Washington TRU Solutions LLC and changed Cog Eng
11168, Rev. 6 12-16-04	1 4	Removed WP 13-1 section references. Updated 3.5.3, added 3.5.3E, and 5.1.4.
11282, Rev. 7 05/09/05	1 through 7	Updated 1.0 Scope. Updated 3.2.3, 3.3.1B, 3.3.1D, 3.3.2E, and 4.1. Delete Figures 3.1, 3.2, and 3.3.
12137, Rev. 8 02/11/09	2, 3, 6 through 12	Changed weight for super sacks in § 3.4.1, changed name on Attachment B, and added Attachments C and D.

1.0 SCOPE

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

The codes, specifications, and standards referred to by number or title form a part of this specification. They are not furnished with the contract documents.

Code of Federal Regulations:

Title 29, Part 1910
(29 CFR 1910) Occupational Safety and Health Standards

Title 30, Part 57
(30 CFR 57) Safety and Health Standards - Metal and Non-Metal Underground Mines

Waste Isolation Pilot Plant, Washington TRU Solutions LLC

WP 13-1, Washington TRU Solutions LLC Quality Assurance Program Description

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 an Material Safety Data Sheet (MSDS) 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.
- 3.1.3 The vendor shall provide an MSDS 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.

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.

3.3 Material Requirements

3.3.1 Backfill Material Requirements

- A. The sum of magnesium oxide (MgO) plus calcium oxide (CaO) shall be a minimum of 95%, 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 (\pm 5 lb/ft³).

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 WTS 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 ft³). 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.

3.4 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 MSDS.
 - 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 filling.

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 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 an MSDS when introducing a new lot of MgO.
- 5.1.2 The supplier shall provide an MSDS, flame spread, smoke generation, and decomposition information for the bag material when changing the approved bag material.
- 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 - Document Submittal Requirements

DOCUMENT SUBMITTAL REQUIREMENTS			
SUBMIT DOCUMENTS PRIOR TO THE POINTS INDICATED BY THE CODE BELOW:			
F – FABRICATION T – TESTING S – SHIPMENT		C – FILLING CONTAINERS A – FINAL ACCEPTANCE	
Document Requirements	See Paragraph	For Approval	For Record
1. MSDS, flame spread, smoke generation, and decomposition information for Bag Material (only required for change in approved bag material).	3.1.3	F	
2. MSDS 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

Equipment Needed:

1. A drying oven able to sustain a temperature of approximately 100°C.
2. 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 1° for measuring oven air temperature.
3. 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.
4. An analytic balance accurate to within ± 0.01 g, calibrated with NIST traceable standards.
5. A plastic-coated magnetic stir bar 1-3 inches in length and a stir plate to drive it.
6. A source of deionized water.
7. A source of ACS Reagent Grade 85% phosphoric acid.
8. 400-mL glass beakers.
9. Stopwatch (optional).
10. Scientific notebook (or suitable alternative which provides permanent archiving of recorded information).

Procedure:

1. Mix one liter of 20% \pm 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.

ATTACHMENT B

Phosphoric Acid Temperature Rise Procedure

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°C 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.

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) Acceptance Test 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.

1 SAMPLE ACCEPTANCE AND CHAIN OF CUSTODY

- 1.0 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.
- 2.0 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.
- 3.0 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.

2 COMPOSITING SAMPLES

- 1.0 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.

3 HYDRATION AT 245°C

- 1.0 Place 10 ml of DI water and 1 g to 1.1 g (weighed accurately to 0.1 g) of MgO into a Parr Acid Digestion Bomb.
- 2.0 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.
- 3.0 Close vessel.
- 4.0 Repeat steps 1-3 to produce 3 separate samples.
- 5.0 Place Parr Bombs into a furnace. Heat the bombs in furnace to a temperature of between 245 - 250°C.

Attachment C

Reactivity (mole % Periclase + Lime) Acceptance Test Procedure

- 6.0 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.
- 7.0 Remove Parr Bombs from furnace and cool until bombs are able to be safely handled.
- 8.0 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.

4 LOSS OF IGNITION

- 1.0 Have crucibles available that have been previously ignited for 1 hour at 800-810°C and placed in a desiccator.
- 2.0 Remove hydrated MgO samples from furnace in Section 3. Cool until the material can be safely handled.
- 3.0 Filter MgO using vacuum filtration and Whatman 40 (or equivalent) filter paper. Rinse the solid sample with DI water several times.
- 4.0 Remove ignited crucibles from desiccator and weigh each crucible to an accuracy of 0.1 mg. Record crucible mass ($Mass_{DC}$).
- 5.0 Place each hydrated MgO sample (without the filter paper) into an individual crucible from step 4.
- 6.0 Place crucibles into a furnace.
- 7.0 Heat crucibles in furnace to 200-210°C, maintain at 200-210°C for 1 hour.
- 8.0 Remove crucibles from oven.
- 9.0 Allow crucibles to cool (~ ½ hour) in a desiccator.
- 10.0 Measure and record the crucibles mass ($Mass_{200C}$) to 0.1 mg accuracy.
- 11.0 Place crucibles back into furnace kept at 200°C.
- 12.0 Heat furnace to 800-810°C at a rate not more than 15°C/min.
- 13.0 Hold furnace at 800-810°C for 20 minutes. Check and record furnace temperature.
- 14.0 Allow furnace to cool to a temperature at which the crucibles can be safely removed (about 500°C).
- 15.0 Remove crucibles from oven, allow to cool (~ ½ hour) in desiccator.
- 16.0 Measure and record crucible masses ($Mass_{800C}$) to 0.1 mg accuracy.
- 17.0 Enter the masses $Mass_{DC}$, $Mass_{200C}$, and $Mass_{800C}$ into cells B4 through D6, and the chemical-compositional analysis into cells B16 through C19 for this lot on the attached Excel analysis worksheet template. Transmit the worksheet to the WIPP contract STR within 48 hours of sample acceptance.

Sample Analysis Report

LOI results

Lot Number:

Date Shipped:

<u>Sample ID</u>	<u>Mass DC (g)</u>	<u>Mass 200 C (g)</u>	<u>Mass 800 C (g)</u>	<u>Weight loss %</u>	<u>Notebook pg</u>
1	32.9	33.72	33.475	29.8780488	<input type="text"/>
2	32.9	33.73	33.475	30.7228916	
3	32.9	33.72	33.475	29.8780488	

Average weight loss % 30.15966304 Notes:

Standard deviation % 0.48777021

Sample Analysis Report

Chemical Analysis

	wt %	uncertainty (wt %)
CaO	1	1
SiO2	1	0.5089107
Al2O3	1	0.0566613
Fe2O3	1	0.04337461

Molecular formulas	Mineral Names	MW (g/mol)
Al2O3		101.9602
Fe2O3		159.6922
SiO2		60.0843
Ca(OH)2	Portlandite	74.0926
H2O		18.01528
Mg(OH)2	Brucite	58.31968
MgO	periclase	40.3044
CaO	lime	56.0774

Attachment D

Sample Analysis Report**Mole fraction periclase +
lime**

	Average	Standard Deviation
Mole % periclase + lime	97.9	5.8
wt % periclase + lime	96.9	3.3