June 22, 2022





Pipeline and Hazardous Materials Safety Administration

DOT-SP 13220 (THIRTEENTH REVISION)

EXPIRATION DATE: 2026-05-31

(FOR RENEWAL, SEE 49 CFR 107.109)

1. <u>GRANTEE</u>: Entegris, Inc. Billerica, MA

2. <u>PURPOSE AND LIMITATION</u>:

a. This special permit authorizes the transportation in commerce of certain non-DOT specification welded pressure vessels containing certain compressed gases and liquids adsorbed onto a microporous sorbent. This special permit provides no relief from the Hazardous Materials Regulations (HMR) or the International Maritime Dangerous Goods (IMDG) Code other than as specifically stated herein. The most recent revision supersedes all previous revisions.

b. The safety analyses performed in development of this special permit only considered the hazards and risks associated with transportation in commerce.

c. No party status will be granted to this special permit.

d. This special permit serves as an "Approval" under Chapter 4.1, Section 4.1.3.7 and Chapter 7.9, Section 7.9.2 of the IMDG Code, and as a "Competent Authority Approval" as defined in 49 CFR 107.1.

- 3. <u>REGULATORY SYSTEM AFFECTED</u>: 49 CFR Parts 106, 107 and 171-180 and the IMDG Code.
- 4. <u>REGULATIONS FROM WHICH EXEMPTED</u>: 49 CFR §§ 173.302 and 173.302c in that a non-UN cylinder is not authorized, except as specified herein.
- 5. <u>BASIS</u>: This special permit is based on the application of Entegris, Inc. dated January 13, 2022, submitted in accordance with § 107.105, the public preceding thereon and the application dated April 15, 2022 submitted in accordance with § 107.109.

6. <u>HAZARDOUS MATERIALS (49 CFR 172.101)</u>:

Hazardous Materials Description				
Proper Shipping Name	Hazard Class/ Division	Identi- fication Number	Packing Group	
Adsorbed gas, n.o.s	2.2	UN3511	N/A	
Adsorbed gas, flammable, n.o.s	2.1	UN3510	N/A	
Adsorbed gas, oxidizing, n.o.s	2.2	UN3513	N/A	
Adsorbed gas, toxic, n.o.s	2.3	UN3512	N/A Hazard Zone A, B, C, & D	
Adsorbed gas, toxic, corrosive, n.o.s	2.3	UN3516	N/A Hazard Zone A, B, C, & D	
Adsorbed gas, toxic, flammable, n.o.s	2.3	UN3514	N/A Hazard Zone A, B, C, & D	
Adsorbed gas, toxic, flammable, corrosive, n.o.s	2.3	UN3517	N/A Hazard Zone A, B, C, & D	
Adsorbed gas, toxic, oxidizing, n.o.s	2.3	UN3515	N/A Hazard Zone A, B, C, & D	
Adsorbed gas, toxic, oxidizing, corrosive, n.o.s	2.3	UN3518	N/A Hazard Zone A, B, C, & D	

Hazardous Materials Description				
Proper Shipping Name	Hazard Class/ Division	Identi- fication Number	Packing Group	
Arsine, adsorbed	2.3	UN3522	N/A Hazard Zone A	
Boron trifluoride, adsorbed	2.3	UN3519	N/A Hazard Zone B	
Chlorine, adsorbed	2.3	UN3520	N/A Hazard Zone B	
Germane, absorbed	2.3	UN3523	N/A Hazard Zone A	
Hydrogen selenide, adsorbed	2.3	UN3526	N/A Hazard Zone A	
Phosphorous pentafluoride, adsorbed	2.3	UN3524	N/A Hazard Zone B	
Phosphine, adsorbed	2.3	UN3525	N/A Hazard Zone A	
Silicon tetrafluoride, adsorbed	2.3	UN3521	N/A Hazard Zone B	

7. <u>SAFETY CONTROL MEASURES</u>:

a. <u>PACKAGING</u>: Packaging prescribed is a non-DOT specification, welded pressure vessel filled with a monolith solid microporous sorbent and/or bead type sorbent onto which the gas is adsorbed. The gas remains adsorbed during transportation in essentially a solid state. The system is filled and operated at sub-atmospheric pressures and is described as a sub-atmospheric gas delivery system (SDS). The welded pressure vessel may be of cylindrical or cube design. The pressure vessel must be manufactured from cold drawn over mandrel (square shell) and/or cold deep drawn (cylindrical shell) steel. The pressure vessel must be designed and constructed in accordance with American Cap Company, Inc. drawing AC2314-C Rev-J, AC2322-E Rev-A, AC2389 Rev-F, AC2389C Rev-A, AC2919 Rev-B, AC22414 Rev-A, AC22415 Rev-A, AC2919C Rev A, AC2917 Rev B, or AC2380 Rev L on file with the Office of Hazardous Materials Safety (OHMS) and with the following specifications:

(1) Capacity: 0.4 L to 12 Liters.

(2) Material: The steel shell is manufactured by cold deep drawing a blank from a sheet of steel meeting one of the following specifications:

(i) Carbon steel shell:

(A) ASTM Grade A-1011/A-1011M-04A, DS Type A modified, hot rolled, steel with max carbon content 0.25%; or

(B) Steel as specified in Table 1 of Appendix A to part 178 of the HMR.

(ii) Stainless steel shell: AISI 304/304L stainless steel.

(3) End Plug: Hot Rolled Steel SAE 1020 modified with max. carbon content 0.25%; or AISI 316L stainless steel bar.

- (4) Minimum sidewall thickness: Cylindrical, 0.085 inch Cube, 0.12 inch.
- (5) Maximum service pressure: 5.2 bar (75 psig).
- (6) Minimum test pressure: 21 bar (304.6 psig).
- (7) Minimum design burst pressure: 94.5 bar (1,371 psig).

(8) The pressure vessel must be manufactured in accordance with § 178.35 except as follows:

(i) § 178.35(b)(2) Inspections and verifications must be performed by a competent inspector of the manufacturer.

(ii) § 178.35(f)(1)(i) The pressure vessel must be marked "DOT-SP 13220" followed by the service pressure in lieu of marking the DOT specification number. Markings must be stamped plainly and permanently in accordance with § 178.51(n).

(9) The pressure vessel must be equipped with pressure relief devices as prescribed in § 173.301(f) for the hazardous material being transported except that compliance with Compressed Gas Association Pamphlet S-1.1 referenced in § 171.7 (compliance with paragraph 9.1.1.1 is not required). The selection of the type, location and quantity of a pressure relief device for an adsorbed gas package is based on the name of the gas in the unabsorbed state. For example, the selection of a pressure relief device for "UN3519 Boron trifluoride, adsorbed" would be based on the pressure relief device required for Boron Trifluoride, UN1008. Valve protection must be in accordance with § 173.301(h).

(10) Pressure vessels must be manufactured in accordance with American Cap Company Inc., Cylinder Manufacturing Procedure Doc. No. 10-007 and SDS Manufacturing Workflow Chart.

(11) Welding or brazing must be by automated processes. Welding procedures and operators must be qualified in accordance with CGA Pamphlet C-3. Welded pressure vessels containing the sorbent material are not heat treated after the welding or brazing operation.

(12) Tests of welds must be performed in accordance with § 178.51(l).

(13) At least one completed pressure vessel selected at random per lot of 200 or fewer must be hydrostatically tested to failure or to at least 94.5 bar (1,371 psig). The test must be performed without sorbent present. The pressure vessel must not show evidence of leakage or damage below 94.5 bar (1,371 psig). All pressure vessels used in the burst test must be destroyed.

b. <u>REQUALIFICATION</u>: Each pressure vessel must be requalified every 10 years as follows:

(1) The pressure vessel must be evacuated and filled with helium, to at least 21 bar (304.6 psig). The pressure vessel must be placed in a vacuum chamber and the chamber evacuated to a pressure sufficient for the leak detector to meet the leak test specification. The pressure vessel must be held in the chamber until the leak detector provides a "ready to test" signal and the leak rate determined. The leak detection equipment must be calibrated for each test with nominal detection limits of 1×10^{-10} atm. cc/sec. The pressure vessel must be rejected if the leak rate is greater than 1×10^{-6} atm. cc/sec.

(2) The requalifer must comply with the marking and record keeping requirements of §§ 180.213 and 180.215.

c. <u>OPERATIONAL CONTROLS</u>:

(1) Prior to the first filling, each completed pressure vessel with sorbent must be vacuum baked at a maximum temperature of $300 \,^{\circ}$ C to remove any contaminants.

(2) Prior to filling with the gas, each completed pressure vessel containing the sorbent and with the valve attached must be proof pressure tested to at least 21 bar (304.6 psig) and helium leak tested per the test method in paragraph 7.b. of this special permit.

(3) Only Entegris, Inc. or its agents authorized and trained by Entegris, Inc. may fill the pressure vessels and offer them for transportation. The authorized agents must be on file with OHMS.

(4) The pressure of each filled cylinder must be less than 101.3 kPa (14.7 psig) at 20 $^{\circ}$ C (68 $^{\circ}$ F) and must be less than 300 kPa (43.5 psig) at 50 $^{\circ}$ C (122 $^{\circ}$ F).

(5) The internal pressure at 65 °C (149 °F) of the filled cylinder must not exceed the test pressure of the cylinder.

(6) Each pressure vessel must remain in dedicated product service for its entire life.

d. <u>DISPOSAL</u>: The pressure vessels that are rejected with helium leakage rates $\ge 1 \times 10^{-6}$ and $\le 1 \times 10^{-3}$ atm. cc/sec may be repaired and offered for transportation for the purpose of disposal as follows:

(1) The pressure vessel leak is repaired in accordance with Entegris work instruction 22741, which is on file with the OHMS.

(2) The pressure vessel is evacuated to a pressure of ≤ 200 Torr (3.87 psia) at 70 °F (21 °C).

(3) The repaired pressure vessel is leak tested in accordance with Entegris work instruction 22741. There must be no hazardous gas leak detected using the leak detection equipment and procedure specified in Entegris work instruction 22741.

(4) The valve outlet is sealed with a metal cap and gasket in accordance with 173.40 (c) (3).

(5) The repaired pressure vessel is fitted with a valve protection cap meeting the requirements of §§ 173.301 (a) (12), 173.301 (h) and 173.40 (d).

(6) Pressure vessels that have been repaired for the purpose of disposal shall have their status changed to SCRAP and the corresponding scrap label attached in a durable manner.

(7) The repaired pressure vessel(s) must be overpacked in a UN 1A2 steel drum tested and marked for a PG II or higher performance level in accordance with Entegris work instruction 22741. The gross weight of the overpack must not exceed the maximum gross mass marked on the UN 1A2 drum.

(8) Only persons issued a requalification identification number (RIN) authorizing the requalification of DOT-SP 13220 pressure vessels are permitted to perform the repairs.

8. <u>SPECIAL PROVISIONS</u>:

a. Under the terms of this special permit, the grantee may only offer hazardous materials (i.e., the grantee is not authorized as a carrier).

b. A person who is not a holder of this special permit who receives a package covered by this special permit may reoffer it for transportation provided no modification or change is made to the package and it is reoffered for transportation in conformance with this special permit and the HMR.

c. A current copy of this special permit must be maintained at each facility where the package is offered or reoffered for transportation. Offerors of empty cylinders (containing no residual hazardous material) being returned for refilling are not required to have a copy of this special permit.

- 9. <u>MODES OF TRANSPORTATION AUTHORIZED</u>: Motor vehicle, rail freight, and cargo vessel.
- 10. <u>MODAL REQUIREMENTS</u>: A current copy of this special permit must be carried aboard each cargo vessel and motor vehicle used to transport packages covered by this special permit.
- 11. <u>COMPLIANCE</u>: Failure by a person to comply with any of the following may result in suspension or revocation of this special permit and penalties prescribed by the Federal hazardous materials transportation law, 49 U.S.C. 5101 <u>et seq</u>:
 - o All terms and conditions prescribed in this special permit and the Hazardous Materials Regulations, 49 CFR Parts 171-180.
 - o Persons operating under the terms of this special permit must comply with the security plan requirement in Subpart I of Part 172 of the HMR, when applicable.
 - o Registration required by § 107.601 <u>et seq</u>., when applicable.

Each "Hazmat employee", as defined in § 171.8, who performs a function subject to this special permit must receive training on the requirements and conditions of this special permit in addition to the training required by §§ 172.700 through 172.704.

No person may use or apply this special permit, including display of its number, when this special permit has expired or is otherwise no longer in effect.

Under Title VII of the Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (SAFETEA-LU)- "The Hazardous Materials Safety and Security Reauthorization Act of 2005" (Pub. L. 109-59), 119 Stat. 1144 (August 10, 2005), amended the Federal hazardous materials transportation law by changing the term "exemption" to "special permit" and authorizes a special permit to be granted up to two years for new special permits and up to four years for renewals.

12. <u>REPORTING REQUIREMENTS</u>: Shipments or operations conducted under this special permit are subject to the Hazardous Materials Incident Reporting requirements specified in 49 CFR §§ 171.15 Immediate notice of certain hazardous materials incidents, and 171.16 Detailed hazardous materials incident reports. In addition, the grantee(s) of this special permit must notify the Associate Administrator for Hazardous Materials Safety, in writing, of any incident involving a package, shipment or operation conducted under terms of this special permit.

Issued in Washington, D.C.:

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for William Schoonover Associate Administrator for Hazardous Materials Safety

Address all inquiries to: Associate Administrator for Hazardous Materials Safety, Pipeline and Hazardous Material Safety Administration, U.S. Department of Transportation, East Building PHH-13, 1200 New Jersey Avenue, Southeast, Washington, D.C. 20590.

Copies of this special permit may be obtained by accessing the Hazardous Materials Safety Homepage at <u>https://www.phmsa.dot.gov/approvals-and-permits/hazmat/special-permits-search</u>. Photo reproductions and legible reductions of this special permit are permitted. Any alteration of this special permit is prohibited.

PO: RS/TG