

East Building, PHH – 30

1200 New Jersey Avenue, Southeast
Washington, D.C. 20590

### Pipeline and Hazardous Materials Safety Administration

DOT-SP 9706 (ELEVENTH REVISION)

EXPIRATION DATE: April 30, 2018

(FOR RENEWAL, SEE 49 CFR § 107.109)

1. <u>GRANTEE</u>: TW Cryogenics, LLC Theodore, AL

## 2. PURPOSE AND LIMITATIONS:

- a. This special permit authorizes the manufacture, mark, sale and use of a non-DOT specification cylinder conforming with all regulations applicable to a DOT-3AA Specification cylinder, except as specified herein, for the transportation in commerce of the materials authorized by this special permit. This special permit provides no relief from the Hazardous Materials Regulations (HMR) 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 hazard and risks associated with transportation in commerce.
- 3. REGULATORY SYSTEM AFFECTED: 49 CFR Parts 106, 107 and 171-180.
- 4. REGULATIONS FROM WHICH EXEMPTED: 49 CFR §§ 173.301(a), 173.302a, and 173.304a(a) in that non-DOT specification cylinders are not authorized, except as specified herein; §§ 180.205(c), (f) and (g) and § 180.215 in that a 10-year retest period is authorized for certain gases if the cylinders are requalified by ultrasonic examination in lieu of a hydrostatic pressure test and internal visual inspection.

- 5. BASIS: This special permit is based on the application of  $\overline{\text{TW}}$  Cryogenics, LLC, dated May 13, 2014, submitted in accordance with § 107.109.
- 6. HAZARDOUS MATERIALS (49 CFR § 172.101):

Hazardous Materials Description			
Proper Shipping Name	Hazard Class/ Division	Identi- fication Number	Packing Group
Gases or mixtures of gases authorized in the HMR to be shipped in DOT-3AA Specification cylinders*	2.1, 2.2, 2.3	Various	N/A

\*The following materials may not be shipped under the terms of this special permit:

- a. Hydrogen, compressed natural gas, hydrogen sulphide, or carbon monoxide;
- b. Any gas mixture containing hydrogen sulphide or other free sulfides, or containing hydrogen or compressed natural gas;
- c. Any gas mixture containing more than 10 percent (10%) carbon monoxide;
- d. Any gas mixture containing carbon monoxide and having a dew point of minus 52EF or higher at one (1) atmosphere;
- e. Any gas or mixture of gases which does not remain in a gaseous state when contained in the cylinder at filling pressure at  $70\mathrm{EF}$ ; and
- f. Any mixture of gases, the quantity of one or more of which is capable of combining chemically with other gases in such mixture or of combining chemically with the cylinder steel so as to significantly reduce the effectiveness of the cylinder.
- q. Any gas that causes embrittlement in steel.

# 7. SAFETY CONTROL MEASURES:

- a. <u>PACKAGING</u> Prescribed packaging is a non-DOT specification steel cylinder made in accordance with Taylor-Wharton drawing 45540 July 7, 1986 on file with the Office of Hazardous Materials Special Permits and Approvals (OHMSPA) and in compliance with §§ 178.35 and 178.37 except as follows:
- § 178.35(c) Duties of inspector.
  - (1) and (2) \* \* \*
  - (3) \* \* \*
    - (v) Witness all tests except the fracture toughness test (§ 178.37(j)) provided the test is performed by a facility acceptable to the Inspector and a certified report of the test results is furnished to the Inspector by the test facility.

\* \* \*

- (ix) Verify that design qualification tests prescribed in  $\S$  178.37(d)(5) have been performed with satisfactory results.
- § 178.35(e) Pressure relief devices and protection for valves and pressure relief devices.
- [Add] Pressure relief devices must be in compliance with § 173.302(c)(1), except as follows:
  - (1) Cylinders charged with gas mixtures containing a gas toxic by inhalation, must not be equipped with any pressure relief device.
  - (2) Cylinders charged with gas mixtures containing no gas classed as toxic by inhalation, may be equipped with a combination rupture disk and fusible plug pressure relief device in compliance with CGA Pamphlet S-1.1.

§ 178.35(f) *Marking*.

Applies except using "DOT-SP 9706" in lieu of "DOT-3AA" followed by the service pressure. Test pressure must be marked following or near service pressure. The letters "TP" preceding test pressure is optional. Rejection elastic expansion (REE) in cubic centimeters (CC) must be marked near the date of test.

§ 178.37(a) Type, size and service pressure.

Prescribed packaging is a seamless steel cylinder having: a maximum service pressure of 3,600 psig; a maximum water capacity of 120 pounds; and a nominal inside diameter of 8.75 inches and a 0.230 inch minimum wall thickness.

§ 178.37(b) Authorized steel.

Basic oxygen or electric furnace steel of uniform quality is authorized. The steel analysis must be in conformance with the following:

#### CHEMICAL COMPOSITION IN WEIGHT PERCENT

	Ladle	Check Analysis	Tolerance
Element	Analysis	Under	Over
Carbon	.32/.36	0.01	0.02
Manganese	.60/.90	0.03	0.03
Phosphorus	.025 Max.	_	0.01
Sulphur	.010 Max.	_	0.00
Silicon	.15/.35	0.02	0.03
Chromium	.80/1.10	0.03	0.03
Molybdenum	.15/.25	0.01	0.01
Vanadium	.10 Max.	0.01	0.01
Aluminum	.01/.05	_	0.00
Copper	.20 Max.	_	0.00

Note 1: Steel shall be treated with calcium to provide the following J-K microcleanliness rating per ASTM Standard E-45, Method A.

A (Su	lfides)	B (Al	umina)	C (Si	licates)	D (Ox	ides)
Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
2.0	1.5	2.0	1.0	1.0	0.5	2.5	1.5

Certificate from the material manufacturer must certify that the material was calcium treated and must include in such certification the J-K microcleanliness rating for each heat of steel.

Note 2: Steel shall be aluminum killed and made by a fine grain deoxidation practice.

§ 178.37(d) Manufacture.

\* \* \*

- (1) Metal removal for any purpose other than removal of isolated defects and threading must be done prior to hydrostatic and ultrasonic tests. The thickness of treated areas must be measured and may not be less than the minimum prescribed thickness.
- (2) Each cylinder must be of seamless construction manufactured by the backward extrusion method with integrally formed heads and bottoms.
- (3) The thickness of the bottoms of cylinders must not be less than two times the minimum wall thickness of the cylindrical shell; such bottom thickness to be measured within an area bounded by a line representing the points of contact between the cylinder and floor when the cylinder is in a vertical position.
- (4) Shape and thickness of the cylinder bottom and sidewall adjacent to the bottom must be such that failure during the cyclic pressure test occurs in the sidewall of the cylinder.
- (5) The design authorized herein must be qualified for production by subjecting at least three prototype samples to pressure cycling tests and burst tests as follows:
  - (i) Cycle Test. The cycle test must be performed on the completed cylinder after hydrostatic test by subjecting the cylinder to successive

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hydrostatic pressurizations from the lower cyclic pressure to the upper cyclic pressure at a rate not to exceed 10 cycles per minute. Adequate recording instrumentation shall be provided if equipment is to be left unattended for any period of time. Lower cyclic pressure must not exceed 10 percent of the upper cyclic pressure. Upper cyclic pressure must be at least equal to the minimum prescribed test pressure.

- (ii) Burst Pressure Test. The burst pressure test must be performed on the completed cylinder by hydrostatically pressurizing the cylinder to destruction. Rate of pressurization must not exceed 200 psi per second.
- § 178.37(e) Welding or brazing.

Welding or brazing is prohibited.

- § 178.37(f) Wall thickness.
  - (1) Delete
  - (2) The minimum wall thickness must be such that the wall stress at the minimum specified test pressure does not exceed 67 percent of the minimum tensile strength of the steel as determined by the mechanical tests required in  $\S$  178.37(k). A wall stress of more than 92,100 psi is not permitted. The wall thickness must not be less than 0.230 inch.
  - (3) Applies except P, the minimum test pressure, must be at least 3/2 service pressure.
- § 178.37(q) Heat treatment.

The completed cylinders must be uniformly and properly heat treated prior to tests. Heat treatment of cylinders shall be as follows:

(1) The furnace shall be equipped with recording infrared pyrometers capable of determining cylinder temperature in both the austenitizing and tempering sections at the beginning and end of each soak zone. The furnace shall have fault controls providing uniform temperature in each control zone and proper function of the feed mechanism.

- (2) Each cylinder must be heated and held above the upper critical temperature (Ac3) for at least one hour per inch of thickness based on the maximum thickness of the cylinder and then quenched in a suitable liquid medium having a cooling rate not in excess of 80 percent of water. The steel temperature on quenching must be above the Ac3 temperature, but not higher than  $1700\mathrm{EF}$ .
- (3) After quenching, each cylinder must be reheated to a temperature below the transformation range but not less than 1000 F, and must be held at this temperature for at least one hour per inch of thickness based on the maximum thickness of the cylinder. Each cylinder must then be air cooled.

#### § 178.37(h) Openings.

Openings are permitted in the cylinder head only. All openings must be threaded. Threads must be in compliance with the following:

- (1) \* \* \*
- (2) Taper threads, when used, must be in compliance with one of the following:
  - (i) American Standard Pipe Thread (NPT) type must be in compliance with the requirements of Federal Standard H-28 (1978), Section 7.
  - (ii) National Gas Taper Thread (NGT) type must be in compliance with the requirements of Federal Standard H-28 (1978), Section 7 and 9.
  - (iii) Other taper threads in compliance with other standards may be used provided the length is not less than that specified for NPT threads.
- (3) Straight threads when used must be in compliance with one of the following:
  - (i) National Gas Straight Thread (NGS) type must be in compliance with the requirements of Federal Standard H-28 (1978), Sections 7 and 9.

- (ii) Unified Thread (UN) type must be in compliance with the requirements of Federal Standard H-28 (1978), Section 2.
- (iii) Controlled Radius Root Thread (UNJ) type must be in compliance with the requirements of Federal Standard H-28 (1978) Section 4.
- (iv) Other straight threads in compliance with other recognized standards may be used provided that the requirements in (4) below are met.
- All straight threads must have at least 6 engaged threads, a tight fit, and a factor of safety in shear of at least 10 at the test pressure of the cylinder. Shear stress must be calculated by using the appropriate thread shear area in accordance with Federal Standard H-28 (1978), Appendix A5, Section 3.
- § 178.37(i) Hydrostatic test.
  - (1), (2) and (3) \* \* \*
  - Each cylinder must be tested to at least 1.5 times service pressure.
  - (5) A rejection elastic expansion limit (REE) must be developed as specified in CGA pamphlet C-5. If the elastic expansion of any cylinder, at test pressure, exceeds the limit so developed, that cylinder must be rejected.
- § 178.37(j) Toughness, ductility tests and ultrasonic examination.
  - (1) Flattening test. Between knife edges, wedge shaped, 60-degree angle, rounded to 2 inch radius; test 1 cylinder taken at random out of each lot of 200 or less cylinders. Longitudinal axis of the cylinder must be at approximately a 90-degree angle to knife edges.
  - (2) Impact tests.
    - Three Charpy impact specimens must be tested from one heat-treated cylinder taken from each 500 or less successively produced.

- (ii) Each impact specimen must be Charpy V-notch type size 10mm X 4mm or 10mm X 5mm taken in accordance with ASTM Standard A-370-77.
- (iii) Each specimen must be taken from the sidewall of the cylinder. The longitudinal axis
- of the specimen must be at 90 degrees to the longitudinal axis of the cylinder.
- (3) Hardness measurement: A hardness measurement must be performed on the cylindrical section of each cylinder after heat treatment. At a minimum, one reading must be taken in the cylindrical section approximately three inches from either the shoulder or bottom of each cylinder. The result must be recorded.
- (4) Flawed cylinder pressure test. One cylinder must be selected from each heat of steel and subjected to a preflaw pressure test. A sharp part through longitudinal flaw must be introduced into the test cylinder by a means that will not affect the mechanical or metallurgical properties of the cylinder. The flaw must be at least 2-inches long and have a depth that will cause the cylinder to fail when pressurized to not less than 90 percent and not more than 125 percent of the stress at service pressure. The cylinder must be hydrostatically pressurized to failure at a rate not in excess of 200 psi per second.
- (5) Ultrasonic Examination.

Each cylinder must be examined after heat treatment and hydrostatic proof testing by shear wave ultrasonic equipment which has been calibrated to give an indication greater than the equivalent of a 5% of wall thickness by 1" long notch. Procedures must be in accordance with Taylor-Wharton special permit E 12022. Ultrasonic test results must be recorded by individual cylinder serial numbers and maintained as long as the cylinders are in service.

- § 178.37(1) Acceptable results of tests and inspections.
  - (1) Flattening test. Flattening required without cracking to 10 times wall thickness. Maximum degree of flattening attained without cracking must be entered on the inspector's report.
  - (2) Impact tests. The Charpy V-notch impact properties for the three impact specimens which must be tested at 0.0EF or colder must not be less than the values shown below:

	Avg. value for	Min. value
Size	acceptance 3	1 specimen only
(mm)	specimens	of the three
10 X 5 or 10 X 4	13.0 ft. lbs.	10.0 ft.1bs.

- (3) Hardness measurement. The tensile strength equivalent of the hardness number obtained may not be more than 182,000 psi; Rc 40 (Brinell 371). When the result of a hardness test exceeds the maximum permitted, two or more retests may be made; however, the hardness number obtained in each retest may not exceed the maximum permitted.
- (4) Flawed cylinder pressure test. The failure must be by leakage without crack extension, or by plastic fracture (fully ductile fracture with visible evidence of bulging.
- (5) Mechanical tests.
  - (i) Tensile strength at least 155,000 psi and not more than 175,000 psi.
  - (ii) Elongation at least 12 percent for gauge length 2 inches with width not over 1 2 inches.
- (6) Ultrasonic examination. Any cylinder having a discontinuity indication greater than the equivalent of a 5 percent of wall thickness by 1 inch long notches must be rejected. Any cylinder with rejectable ID indication shall be scrapped and destroyed.

- (7) Cycle tests. Cylinders subjected to design qualification cycling tests must withstand at least 10,000 cyclic pressurizations without distortion or failure. All cycle tests must be performed using water as the pressurizing medium.
- (8) Burst tests. Cylinders subjected to design qualification burst tests must withstand a pressure of at least 2.25 times the service pressure without failure. Failure must initiate in the sidewall in a longitudinal direction, and the cylinder must remain in one piece.
- § 178.37(n) Rejected cylinders.
  - (1) Lot definition. In this special permit, a "lot" means a group of cylinders successively produced and having the same:
    - (i) Size and configuration;
    - (ii) Specified material of construction;
    - (iii) Process of manufacture and heat treatment;
    - (iv) Equipment of manufacture and heat treatment;
    - (v) Conditions of time, temperature and atmosphere during heat treatment.
    - (2) The lot size may not exceed 200 cylinders, but any cylinder processed for use in the required destructive testing need not be counted as being one of the 200. If any lot in an already accepted heat fails any of the qualification tests as prescribed in §§ 178.37(d), 178.37(j) or 178.37(k) of this special permit, that lot may be subjected to one repeated heat treatment. Any lot subjected to second heat treatment is considered as equivalent to new heat and therefore must meet, at a minimum, the requirements specified below. If any lot subjected to a second heat treatment fails any of the prescribed mechanical tests, the cylinders from that lot may not be used under the terms of this special permit.

- (3) Lot re-qualification:
  - (i) The second heat treatment procedure must be in accordance with that prescribed in § 178.37(q) of this special permit.
  - (ii) The lot is identified in the heat treatment records appropriately indicating repeated heat treatment.
  - (iii) Flattening tests: Acceptable lot must pass all prescribed tests per §§ 178.37(j)(1) and 178.37(l)(1) of this special permit.
  - (iv) Impact tests: Acceptable lot must pass all prescribed tests per §§ 178.37(j)(2) and 178.37(l)(2) of this special permit.
  - (v) Hardness measurements: Acceptable lot must pass all prescribed tests per §§ 178.37(j)(3) and 178.37(l)(3) of this special permit.
  - (vi) Flawed cylinder tests: At a minimum one cylinder must pass the tests prescribed per §§ 178.37(j)(4) and 178.37(l)(4) of this special permit.
  - (vii) Mechanical tests: Acceptable lot must pass all prescribed tests per  $\S\S$  178.37(j)(5), 178.37(k) and 178.37(l)(5) of this special permit.
- (4) Cylinder re-qualification:
  - (i) Metal removal for any purpose is not permitted.
  - (ii) Each cylinder must be subjected to hydrostatic test to 3/2 times rated service pressure as prescribed in § 178.37(i)(4) of this special permit.
  - (iii) Elastic and plastic expansion at the proof test pressure must not exceed rejectable limits as prescribed in § 178.37(i) (5) of this special permit.

(iv) Following hydrostatic proof testing, each cylinder must pass ultrasonic examination as prescribed in  $\S$  178.37(j)(5) of this special permit.

## b. <u>TESTING</u> -

- (1) Five-year requalification Each cylinder must be requalified for use every five years in accordance with § 180.209 as prescribed for DOT-3AA cylinders or by ultrasonic examination. Cylinders requalified after having been subjected to the action of fire, must be reported to OHMSPA prior to being placed back in service.
- (i) Hydrostatic pressure test Each cylinder must be tested in accordance with § 180.205(g) except
  - (A) Each cylinder must be tested to at least 3/2 times the marked service pressure.
  - (B) A rejection elastic expansion (REE) limit must be developed as specified in CGA Pamphlet C-5.
- (ii) Ultrasonic Examination As an alternative, each cylinder may be requalified by ultrasonic examination in accordance with DOT-SP 12022 or by a retest facility approved by OHMSPA for ultrasonic examination of these type cylinders.
  - (A) The retest results for the ultrasonic examination must be submitted to OHMSPA annually.
  - (B) A cylinder that has been exposed to fire or to excessive heat (temperatures of 1000°F or greater) may not be retested by ultrasonic examination.
- (2) Ten years requalification Cylinders made under this special permit may be requalified every ten years if they are requalified by ultrasonic examination (UE) described in DOT Special permit 12022 or by a retest facility approved by OHMSPA for ultrasonic examination of these type cylinders provided each cylinder meets all of the following:

- (i) The cylinder is used exclusively for argon, helium, krypton, neon, nitrogen, oxygen, xenon, permitted mixtures of these gases in accordance with \$ 173.301a(a)(1) that contain less than 13 percent carbon monoxide, a dew point of below minus  $52^{\circ}F$  at 1 atmosphere.
- (ii) The cylinder is hammer tested in accordance with CGA pamphlet C-6.
- (iii) The cylinder is not used for underwater breathing.
- (iv) Each cylinder is stamped with five-point star as described in § 180.209(b)(vi) immediately following the UE.
- c. OPERATIONAL CONTROLS Filling limits specified in § 173.302a (b) are not authorized. These cylinders must not be filled to a pressure exceeding the marked service pressure at  $70^{\circ}F$ .

#### 8. SPECIAL PROVISIONS:

#### a. Reports:

- (1) Prior to the initial shipment of cylinders made to any specific design, or from a repeat heat treated lot, a report of test results specified in §§ 178.37(d)(5) and/or 178.37(n) must be submitted to OHMSPA.
- (2) The maker of the cylinder under this specification must retain the test reports required by this specification indefinitely as long as these cylinders are authorized.
- b. A copy of the Inspector's report for each of the first three lots produced must be submitted to the OHMSPA prior to shipment.
- c. 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.

- d. Each packaging manufactured under the authority of this special permit must be marked with a <u>registration symbol</u> designated by the Office of Hazardous Materials Special <u>Permits and Approvals Program</u> for a specific manufacturing facility.
- e. A current copy of this special permit, in its current status, must be maintained at each manufacturing facility at which this packaging is manufactured and must be made available to a DOT representative upon request.
- f. In accordance with the provisions of Paragraph (b) of § 173.22a, persons may use the packaging authorized by this special permit for the transportation of the hazardous materials specified in paragraph 6, only in conformance with the terms of this special permit.
- g. A current copy of this special permit must be maintained at each facility where the package is offered or reoffered for transportation.
- h. Transportation of Division 2.1 materials (flammable gases) and Division 2.3 materials (gases which are poisonous by inhalation) are not authorized aboard cargo vessel or aircraft unless specifically authorized in the Hazardous Materials Table (§ 172.101).
- i. The transportation of oxygen by aircraft is only authorized when in accordance with § 175.501.
- j. No new cylinders may be manufactured under the terms of this special permit after February 26, 2010.
- 9. MODES OF TRANSPORTATION AUTHORIZED: Motor vehicle, rail freight, cargo vessel, cargo aircraft only (see restrictions in paragraphs 8.h. and 8.i.).
- 10. MODAL REQUIREMENTS: A current copy of this special permit must be carried aboard each cargo vessel or aircraft used to transport packages covered by this special permit. The shipper shall furnish a current copy of this special permit to the air carrier before or at the time the shipment is tendered.

- 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. Section 5101 et seq:
  - o All terms and conditions prescribed in this special permit and the Hazardous Materials Regulations, 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 49 CFR 107.601 et seq., when applicable.

Each "Hazmat employee", as defined in 49 CFR 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 49 CFR 172.700 through 172.704.

No person may use or apply this special permit, including display of its number, when the 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. REPORTING REQUIREMENTS: 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

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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.:

for Dr. Magdy El-Sibaie

Ward By

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-30, 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 <a href="http://hazmat.dot.gov/sp\_app/special\_permits/spec\_perm\_index.htm">http://hazmat.dot.gov/sp\_app/special\_permits/spec\_perm\_index.htm</a>
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PO: MT/dl