



Electronic Specialty Materials US Fire Standards and Regulations May 2022

Code

A code is a document containing only mandatory provisions, using the word “Shall” to indicate requirements. Examples of codes are the American Society of Mechanical Engineers (ASME), Boiler and Pressure Vessel Code and the National Electrical Code (NEC).

Standard

A standard is a document containing both mandatory provisions (“shall” rules) and advisory provisions (“should” recommendations). It is referred to as “that which is established by authority, custom or general consent as a model or example; criterion; test.” An example of a standard is the ISO publication “Gas cylinders — Cylinder valves — Specification and type testing ISO 10297:2014

Regulation

A regulation is a law created or administered by a national, regional (e.g., a U.S. state), or local governmental entity. Prime examples are the regulations of the U.S. Department of Transportation (DOT).

Major US Regulations That Affect Electronic Specialty Materials

- US Dept of Transportation (DOT), 49 CFR
- OSHA Worker Safety and PSM, 29 CFR
- EPA Toxic Air Emissions and RMP, 40 CFR
- DHS Security (CFATS)
- International Fire Code (IFC)
- International Building Code (IBC)

US Standards That Address Electronic Specialty Materials

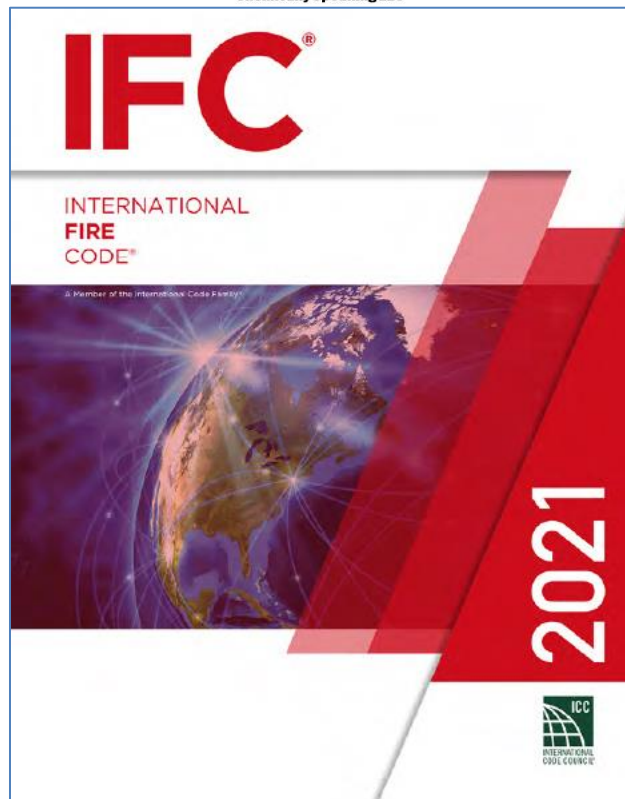
- National Fire Protection Association (NFPA)
- International Standards Organization (ISO)
- International Harmonization Council (IHC)
- Compressed Gas Association (CGA)
- Semiconductor Equipment Manufacturers (SEMI)
- Insurance (FM Global, UL)

Hazardous materials code requirements are based on both the International Fire Code (IFC) and the International Building Code (IBC).

IBC requirements are driven by the Group H occupancy classification assigned to a building.

IFC requirements are driven by the specific materials stored or used.

The majority of the Fire Codes throughout the US are based on the International Fire Code (IFC). This is a model code that is developed and maintained by the International Code Council. It is adopted as a regulation by state agencies or local jurisdictions. The code is updated every three years. Most jurisdictions lag behind by a few years in adopting the most recent edition. These can be edited to delete or insert local requirements



The IBC occupancy classifications are based on the stored materials being physical or health hazards (or both – which is very common).

When quantities of hazardous materials presenting a physical hazard exceed the MAQ, the occupancy containing such materials is classified as a Group H-1, H-2 or H-3.

H-1 Occupancies with materials that present a detonation hazard

H-2 Occupancies with materials that present a deflagration hazard or a hazard from accelerated burning.

H-3 Occupancies with materials that readily support combustion or present a high physical hazard.

H-4 Occupancies having materials that are health hazards

H-5 Semiconductor fabrication facilities and comparable research and development areas in which hazardous production materials (HPM) are used and the aggregate quantity of materials is in excess of those listed in IBC Tables 307.1(1) and 307.1(2)

To apply code requirements, hazardous materials must be identified, evaluated and classified to fit into the classification framework used by model codes

Situations

Storage

Use (open or closed)

Locations

Interior

Exterior

Physical Hazard

Aerosols

Compressed gases and cryogenic fluids

Explosives, fireworks and model rocketry

Flammable and combustible liquids



- Flammable solids
- LPG and LNG
- Organic peroxides
- Oxidizers
- Pyrophoric materials
- Unstable (reactive) materials
- Water-reactive materials

Chemical Hazards

- Highly toxic
- Toxic
- Corrosives

Hazardous Material quantity is used as the primary basis of regulation, and to address the other risk factors, the code varies maximum allowable quantities (MAQs) in non-hazardous uses based on:

- Different hazard categories.
- Different states of material (solid, liquid or gas).
- Different situations (storage, use-closed or use-open).
- Different protection features.

MAQs are the maximum quantities of hazardous materials that may be stored or used in an area before the area must be designated as a hazardous occupancy.

Control Area

Control areas containing hazardous materials which are separated by fire-resistive occupancy separation to isolate adjacent hazards. A building can have multiple areas. Each can have hazardous materials up to the MAQ

Semiconductor Fabrication Facility

A building or a portion of a building in which electrical circuits or devices are created on solid crystalline substances having electrical conductivity greater than insulators but less than conductors. These circuits or devices are commonly known as semiconductors.

Fabrication Area

Is an area within a semiconductor fabrication facility and related research and development areas in which there are processes using hazardous production materials. Such areas are allowed to include ancillary rooms or areas such as dressing rooms and offices that are directly related to the fabrication area processes.

Work Station

Is a defined space or an independent principal piece of equipment using HPM within a fabrication area where a specific function, laboratory procedure or research activity occurs. Approved or listed hazardous materials storage cabinets, flammable liquid storage cabinets or gas cabinets serving a workstation are included as part of the workstation. A workstation is allowed to contain ventilation equipment, fire protection devices, detection devices, electrical devices and other processing and scientific equipment.

IFC Chapter 50 Hazardous Materials

General provisions for hazardous materials which include compressed gases and cryogenic fluids
Contains Maximum Allowable Quantities (MAQ) for each hazard classification in a control area

Requirements for

- Gas Detection
- Emergency Power
- Exhaust ventilation



Fire Permit is required for

**TABLE 105.6.8
PERMIT AMOUNTS FOR COMPRESSED GASES**

TYPE OF GAS	AMOUNT (cubic feet at NTP)
Carbon dioxide used in carbon dioxide enrichment systems	875 (100 lbs.)
Carbon dioxide used in insulated liquid carbon dioxide beverage dispensing applications	875 (100 lbs.)
Corrosive	200
Flammable (except cryogenic fluids and liquefied petroleum gases)	200
Highly toxic	Any Amount
Inert and simple asphyxiant	6,000
Oxidizing (including oxygen)	504
Pyrophoric	Any Amount
Toxic	Any Amount

Alternate Means and Methods

The IFC is flexible in that it allows for the use of alternative and innovative materials and performance-based methods in achieving code compliance

The code enforcement official must find that the proposed design, use or operation satisfactorily complies with the intent of the fire code and that the method of work performed or operation is, for the purpose intended, at least equivalent to that prescribed in the fire code in quality, strength, effectiveness, fire resistance, durability and safety

NFPA

NFPA (National Fire Protection Association) has more balanced group representing all aspects of the product/system. The typical technical committee will have representation by

- Users
- Manufacture
- Fire Agency
- Other government agencies
- Consultants
- Insurance Company
- Industrial Associations

NFPA standards are more comprehensive and many are incorporated into the IFC by reference

- Material Specific
- Use Specific
- Extract policy
- Revised every 4 years

NFPA 55 "Compressed Gases and Cryogenic Fluids Code" which covers all compressed gases.

NFPA 400 "Hazardous Materials Code" which merged all of the hazardous materials standards for pyrophoric liquids, solids, corrosives, reactives into one standard

Use specific codes will extract sections directly from the materials specific codes. For example section on compressed or cryogenic gases will be from NFPA 55 for NFPA318 "Standard for the Protection of Semiconductor Fabrication Facilities"

IFC and NFPA are divided into use and material specific chapters



The most current IFC is the 2018 edition. The most relevant Chapter is Chapter 27 Semiconductor Fabrication Facilities.

Other applicable IFC chapters include

Chapter 50 Hazardous Materials

Chapter 53 Compressed Gases

Chapter 58 Flammable Gases and Flammable Cryogenic Fluids

Chapter 60 Highly Toxic and Toxic Materials

Chapter 64 Pyrophoric Materials

Chapter 27 lists the MAQ for Semiconductor Fabrication Facilities

Table 5003.8.2 Summarizes when detached room is required for highly hazardous materials

Silane

15 kg cylinder contains 396 ft³. 6 cylinders will exceed the threshold quantity of 2,000 ft³ therefore mandating construction of a detached room.

ANSI/CGA G-13

7.3.2.2 Barricade construction

7.3.2.2.1 Unattended operations

Rooms or areas used to contain silane sources used in unattended operations shall be constructed to meet the requirements for barricade construction designed to address the potential for a detonation of released material.

Ordinary construction methods and the use of explosion venting or relief systems are not allowed as a means to offset the effects of a detonation.

“Semiconductor Industry Association (SIA) Fire and Building Standards Committee - Code Proposal to Modify Detached Building Requirements for Pyrophorics”, (attached) that amended Table 5003.8.2 Detached Building Required, by adding footnote d to read:

d. Detached buildings are not required, for gases in gas rooms that support H5 fabrication facilities, and are separated from other areas by a fire barrier with a fire resistance rating of not less than 2 hours, when the gas is located in internally sprinklered gas cabinets, equipped with continuous leak detection and automatic shutdown, the supply is from cylinders that do not exceed 125 lb (57 kg) water capacity per 49 CFR 173.192 for Hazard Zone A toxic gases, and gas cabinets are not manifolded upstream of pressure control devices.

No longer require detached building for pyrophoric gases above 2,000 ft³.

NFPA 55 Proposal to Modify Detached Building Requirements for Pyrophoric Gases (attached)

Amend Table 6.5 (Table 6.6 after A2019 First Draft) Detached Buildings Required Where Quantity of Material Exceeds Amount Shown, by adding footnote a to Gas Hazard Pyrophoric Gas to read:

a. Detached buildings are not required, for gases in High Hazard Gas Rooms that support Protection Level 5 fabrication facilities separated from other areas by a fire barrier with a fire resistance rating of not less than 2 hours, when the gas is located in internally sprinklered gas cabinets, equipped with continuous leak detection and automatic shutdown, the supply is from cylinders that do not exceed 125 lb (57 kg) water capacity per 49 CFR 173.192 for Hazard Zone A toxic gases, and gas cabinets are not manifolded upstream of pressure control devices.

E. Ngai Response to NFPA 55 Technical Committee Comments On Intel Proposal, Rev 2, Nov. 16, 2017 in support (attached)

These are in conflict with the ANSI/CGA G-13 requirements for Barricade construction (H-1). Barricade construction requires a detached room.



ANSI/CGA G-13 Storage and Handling of Silane and Silane Mixtures

Work began on a CGA Safety Standard in 1994 for the storage and use of silane. A silane release test study was conducted in 1996 to develop setback distances for ISO Tube Modules. This was adopted as CGA P-32 in 2000. For recognition by NFPA and the International Fire Code it had to be an approved ANSI document. This approval process took 4 years and it was adopted as ANSI/CGA G-13 2nd edition in 2004. In the United States the International Fire Code (IFC) and the National Fire Protection Association (NFPA) all require that the standard be followed for silane. It is also a globally harmonized standard. It has been adopted by the European Industrial Gas Association (EIGA), Japanese Industrial and Medical Gas Association (JIMGA) and Asia Industrial Gas Association (AIGA). The attachment that was labelled EN-14770-2 is actually AIGA 052/16 which is an exact duplicate of ANSI/CGA G-13 3rd edition. It wasn't even changed to reflect national regulations such as transportation, PRD, etc. This was done when EIGA adopted the 2nd and 3rd editions.

For Silane, IFC and NFPA Reference ANSI/CGA G-13

Bulk silane release testing was conducted in 1996 to develop the setback distances for storage and use of cylinders and bulk supply.

Assumes a nest of silane cylinders containing 10,000 ft³. 50% of the cylinders will be discharging from the PRD at any time. It will take 10 minutes to empty the cylinders

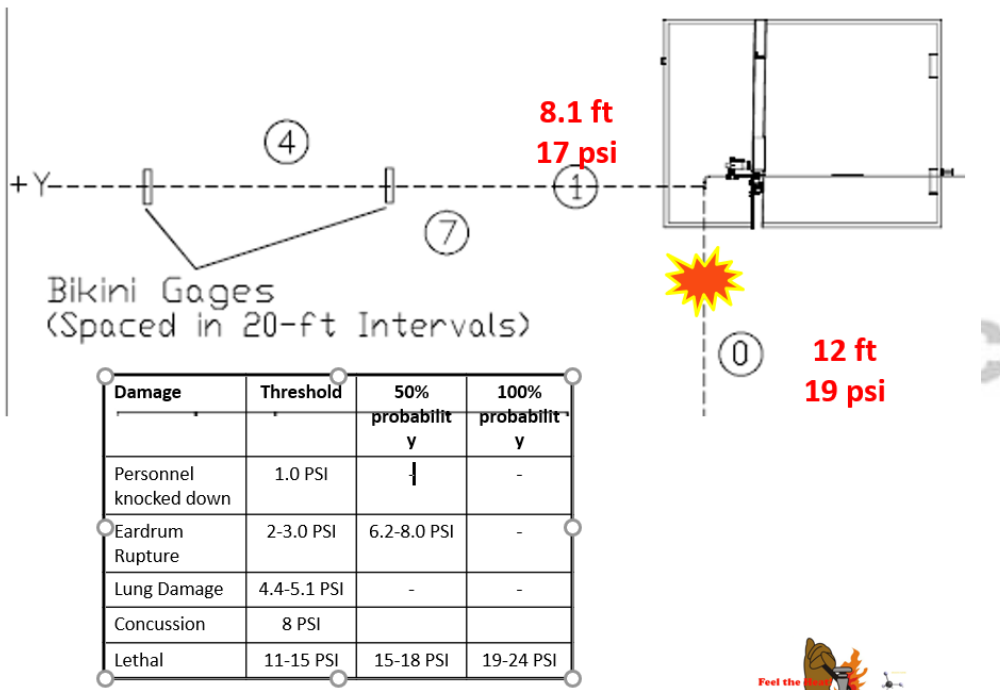
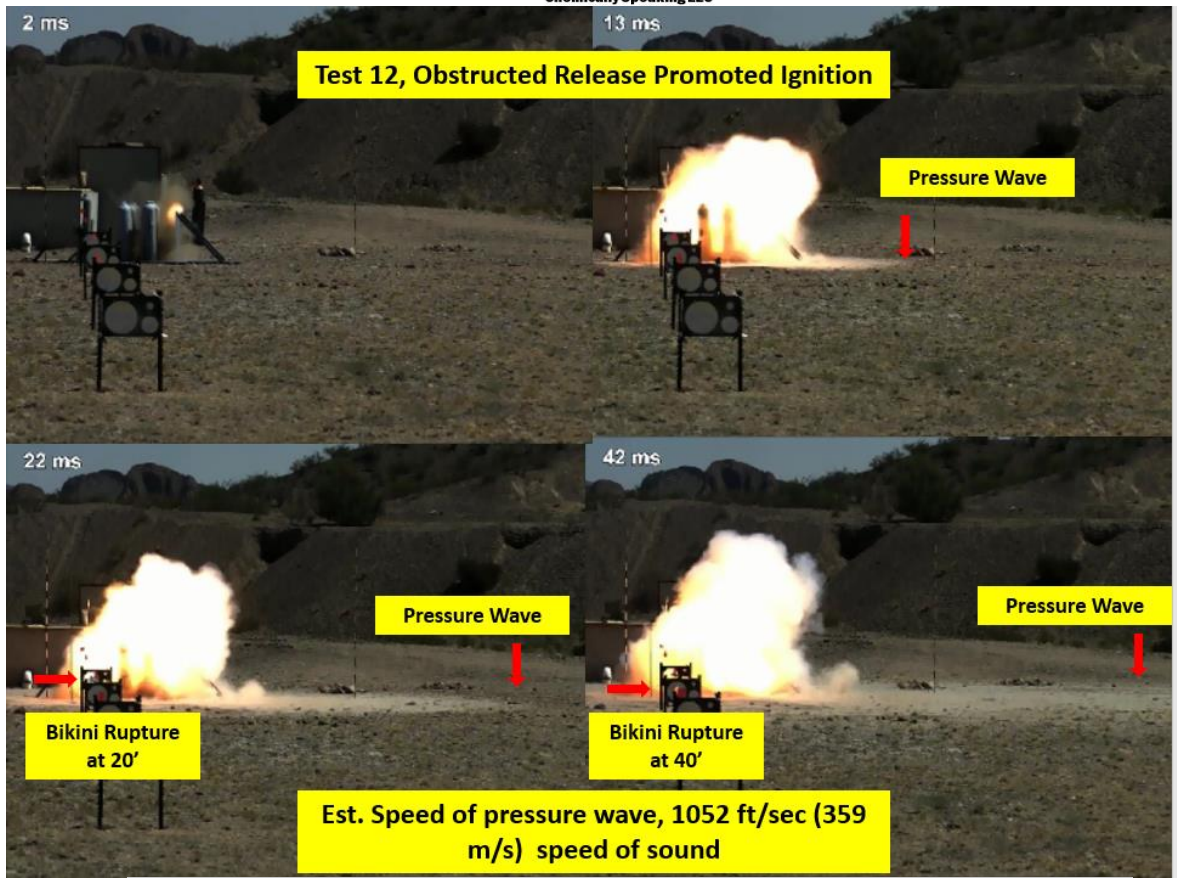
Revision of ANSI/CGA G-13, Started in 2008.

Redid FEMA analysis in 2008-2009 and gather gas industry PRD incidents.

CGA Silane Release Test, June 2011 & 2012 Las Cruces, New Mexico



Chemically Speaking LLC



Aug 2019

Semiconductor Facility US
Codes and Standards

One obstructed release with promoted ignition had an overpressure of 19 psig at a distance of 12 ft which is the threshold for 100% lethal



Key ANSI/CGA G-13 Revisions

Replaced Table 4 overpressure with thermal radiation with and without PRDs

Added new table to cover Bulk Storage and Bulk Use

Overpressure can be estimated by calculations in Appendix D

Added chapter for gas fillers

Canopy allowed over bulk

Removed 150 fpm must analyze ventilation for deadspots where silane could accumulate

Revised setback based on thermal model and with or without PRD

Table 3—Distances to exposure for outdoor cylinders in storage or use up to 50 L

Type of exposure	Minimum distance to exposures ^{1), 2), 3), 4), 5), 6)}			
	Without PRD		With PRD	
	Cylinders in use ⁷⁾ ft (m)	Cylinder in storage less than 200 000 ft ³ (5660 m ³ or 7620 kg) ⁸⁾ ft (m)	Cylinders in use ⁷⁾ ft (m)	Cylinder in storage less than 10 000 ft ³ (283 m ³ or 381 kg) ft (m)
Off-site places of public assembly ⁹⁾	25 (8)	25 (8)	29 (9)	50 (14)
Property lines ⁹⁾	25 (8)	25 (8)	23 (7)	45 (14)
Buildings on-site, noncombustible nonrated construction ⁹⁾	20 (6)	20 (6)	23 (7)	26 (8)
Buildings with 2 hr fire rating and no openings within 25 ft (8 m)	5 (1.5)	5 (1.5)	5 (1.5)	5 (1.5)
Buildings with 4 hr fire rating and no openings within 25 ft (8 m)	0 (0)	0 (0)	0 (0)	0 (0)
Compatible materials or other silane nests ¹⁰⁾	20 (6)	20 (6)	20 (6)	20 (6)
Incompatible materials ¹⁰⁾	20 (6)	20 (6)	23 (7)	26 (8)

NOTE—Distances to exposures may vary due to local regulatory requirements. Any reduction to the distances given shall be subject to an engineering analysis and/or a risk assessment.

¹⁾ The distances are based on permissible exposure to thermal radiation. See Appendix C for thermal radiation data.

²⁾ Cylinders referred to in this table shall be 50 L water volume or smaller. For larger cylinders, use Table 4.



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ANSI/CGA G-13 prefers “outside” (3 sides open) use areas. The industry best practice is to use open enclosures



Where this is not done, single cylinder cabinets are preferred for cylinders 49 liter or smaller.

Extensive testing in the 1990's by FM Global International determined that a single cylinder gas cabinet exhausted at 250 times the RFO flow rate could have a delayed catastrophic explosion as a result of a silane leak but it was not violent enough to rupture the gas cabinet. The over pressure would “pop” open the door with minor damage to the gas cabinet. This was increased to 300 times in the 1st edition of ANSI/CGA G-13. This was reduced back to 250 times in the 3rd edition. The silane flow rates through the RFO increased with increasing fill pressures, therefore limiting the fill pressure will reduce the exhaust flow requirements.

Sprinkler

A water sprinkler has proven to be very effective with silane or other pyrophoric gas leaks and fires. They are meant to cool the cylinder, not put out the fire.

The following incident occurred as a result of, the user using a PTFE gasket rather than a nickel gasket in the DISS cylinder connection. This cylinder had been installed a day earlier and was the standby cylinder on the right side (B). The process was being fed from the left side (A) cylinder. At gas pressures above 500 psig, PTFE can “cold flow” causing the connection to leak after a period of time. Since it was a small leak it immediately ignited, the fire increased the leak rate until it caused the water sprinkler to activate and the system to shutdown. Since the cylinder did not have a pneumatic cylinder valve the automatic shutoff valve was on the pigtail. Since it was a valve connection leak this did not stop the leak. This fire burned for over 9 hours until the gas supplier Emergency Response Team arrived on site and manually shut the valve.



Since both cylinders of silane were made of aluminum there was concern of thermal embrittlement which can cause the cylinders to rupture. The sprinkler water cooled the cylinders preventing this from happening. The reason there is fire only on the right side is because of the metal separation plate required between aluminum cylinders of silane for the same reason.

Metal Separation Plates

Aluminum cylinders exposed to radiant heat from fires can embrittle and rupture

Worst case incident, Silane Fire, Aug. 23, 1997. 44 partially full Sulfur Hexafluoride (SF_6) Aluminum Cylinders adjacent to an outdoor Silane nest of >200 cylinders ruptured from radiant heat exposure
None of the silane steel cylinders failed

Individual cylinders containing silane shall be secured in open steel racks and separated from other silane cylinders to prevent flame impingement from a silane release to an adjacent cylinder or valve area. Separation shall be by $\frac{1}{4}$ in (6 mm) thick steel plates with the plate extended a minimum of 18 in (460 mm) below the centerline of a cylinder valve and a minimum of 6 in (150 mm) above the centerline of the cylinder valve or other means providing equivalent protection

Dedicated high pressure purge

The Gollob Analytical (1988) and Osaka University (1991) incidents were all believed to have been caused by backflow of Nitrous Oxide through the common system purge line into the silane cylinder creating an explosive gas mixture. The 2 explosions killed 5 people and caused significant damage.

ANSI/CGA G-13 requires a dedicated high pressure purge cylinder for silane

One incident occurred because the user conducted the DISS connection leak check using a low pressure of 100 psig rather than the full silane cylinder pressure of 1,450 psig. The industry best practice is to leak check at the silane cylinder pressure.

UVIR flame detection

Approved UVIR detection systems are critical to detection of a leak and fire. It will automatically shutdown the system and alarm. To prevent false alarms a time delay of 10-30 secs is used. The most common leak during use is from the cylinder connection. Without a UVIR detector a leak and fire can burn for a long period of time without detection. One user used a heat detector for fire detection but since it was located outside of the exhaust ventilation flow it did not activate. It was not until an operator saw flames flickering through the access window that they realized there was a leak. They believe that this leaked for over a day. When silane is burning there is no gas detection.

Smoke detection

Small silane leaks may not be detected by the UVIR. As a backup smoke detection is required. A proposal for NFPA 318 to allow a HSSD to replace UVIR



Pneumatic Cylinder Valve

As a best practice, many users are requiring a pneumatic cylinder valve to be able to shutoff the silane at the cylinder. In addition the plastic pneumatic line provides a secondary activation of the valve when it melts in a release and fire. Pneumatic valves rely on springs to close the valve. With use the valve seat can accumulate SiO₂ particles that can cause some seat leakage. The Ceodux D-388 valve is a knife edge design that eliminates this problem.

Semiconductor Cylinder Valve Outlet Connections (DISS)

To provide high integrity connection via use of replaceable metal gasket between two polished beaded surfaces, 10⁻⁹

Cleaner connection by eliminating virtual leaks and degassing from elastomer gaskets

To prevent contamination via use of two different diameters

For safety all are right handed threads

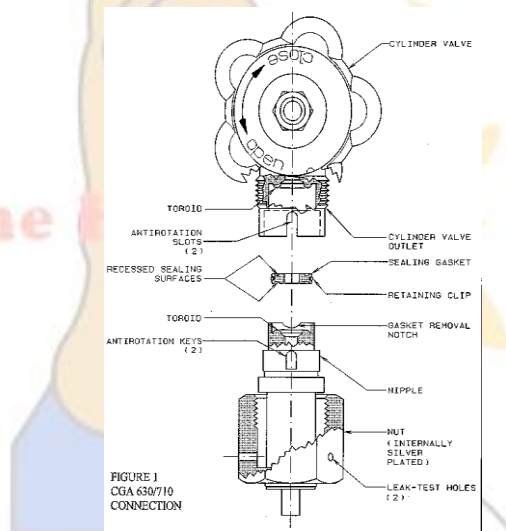
Diameter Index Safety System (DISS) with 16 unique connections CGA 600 & 700 series

Rotational damage of beaded surfaces. Antirotation fingers added to prevent

Torque wrench is required. Under and Overtorque can cause leak

Will not fit under standard cylinder cap

Adopted by ISO 10692 as International Standard



For proper seal of DISS 632

New uncoated nickel 200 gasket must be used

This must be fully annealed after machining

Maximum hardness of 100 Brinnel and a surface finish of 10 Ra (microinches)

Restrictive Flow Orifice (RFO)

Due to the many silane incidents in the early 1980's IBM funded research on silane behavior and developed the first Restrictive Flow Orifice (RFO) for silane. Bernie Meyerson, IBM was issued a US patent on this device in 1985.⁵

IBM was one of the first users to develop and routinely use an RFO that could be inserted into the CGA Connection on the system pigtail.



IBM Pigtail RFO

A CGA Task Force in the 1980's worked closely with cylinder valve manufacturers to develop a removable RFO that would fit in a cylinder valve outlet.

Restrictive Flow Orifices (RFO) have become a key safety requirement for the use of highly toxic, toxic or pyrophoric gases. The RFO is an effective, inexpensive safety device that limits gas flow to a predetermined discharge rate during an accidental release. The RFO is a passive device and does not modulate flow like a flow controller. For pyrophoric gases, like silane, the orifice reduces gas flow so that the gas burns controllably; for toxic gases, the release rate is limited so that dilution can mitigate the hazardous condition.⁶

Originally thought that silane would always ignite when released through an RFO directly into air. This proved to be incorrect when Matheson first tested RFOs with silane.



Silane Release without and with a 0.010" RFO, 1000 psig silane cylinder

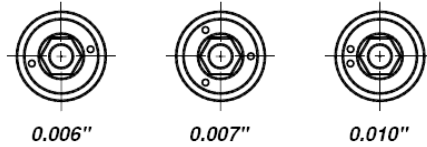
The basis for the 0.010" dia RFO more commonly used today for Silane in cylinders is due to the results of the testing Hazards Research Inc conducted for IBM in 1982. Report #5038, May 11, 1982 states that explosions did not occur with a 0.015" orifice while they did occur with 0.040" and larger. A study by Southwest Research Institute Report #06-7725, November 1983 stated that no explosions occurred with a 15% mixture in Nitrogen when a 0.040" RFO was used.

Rather than minutes to empty a full silane cylinder, it has been estimated that will take over 10 hours to empty a 10 kg 49 liter cylinder through a 0.010" (0.25 mm) RFO.

ANSI/CGA G-13 requires cylinders used indoors in a gas cabinet to have a 0.010" RFO or smaller installed in the cylinder valve outlet



Gasflo uses a series of holes to identify the RFO size Ceodux uses notches (0.010" (0.25 mm) is one notch)



Never look directly into cylinder valve outlet for RFO. Use a dental mirror or fiberoptic borescope In gas cabinet a light might be required



DOT and TC Regulations require a valve protection device for Flammable, Toxic and Corrosive Gases. The most commonly used is cylinder cap
 Must be made of ductile metal, a fire resistant non-metal may also be used.
 Must withstand the testing described in CGA V-9, Appendix

Summary of Regulations and Best Practices for silane Cylinder

- 0.25 mm RFO (0.010")
- Pneumatic cylinder valve, Ceodux D-388
- No PRD
- DISS 632 connection
- Replace metal (nickel) gasket for each connection (quality control of annealing)
- Use torque wrench to tighten DISS connection

Gas Cabinet

- Min. exhaust ventilation of 250 times RFO flow
- Air baffles & plenum to distribute air
- Exhaust air flow monitor and alarm
- Access window with door and window automatic closers
- Fire Sprinkler
- Silane gas detector
- UVIR Flame detector approved for silane, 5-15 sec time delay
- High sensitivity smoke detection (HSSD)



- Pressure indication on pigtail
- Weigh Scale
- Leak check at full Silane cylinder pressure
- Automated purge and evacuation
- 10 sec delay on startup for Operator to move away from cabinet before cylinder valve opens
- Excess flow valve
- Use solid stainless steel pigtails not flexible lines.
- Purge nitrogen cylinder is in gas cabinet. In the event of a backflow and leak it will also be in a suitably protected area.
- Cylinder change with a buddy

The more likely silane leaks will be small, most will not be detected by the UVIR system or gas sensor.



As a backup, users are installing High Sensitivity Smoke Detection (HSSD) in the exhaust duct.

Additional Safety Items

- Autogard this is a key feature that dramatically reduces the potential for an operator to disconnect a cylinder while silane is still in the pigtail
- A video camera mounted in the gas room to provide security and the ability to visually assess the area when an alarm is activated.

Chlorine Trifluoride

IFC 6004.1.2 Gas cabinets. Gas cabinets containing highly toxic or toxic compressed gases shall comply with Section 5003.8.6 and the following requirement:

The average ventilation velocity at the face of gas cabinet access ports or windows shall be not less than 200 feet per minute (1.02 m/s) with not less than 150 feet per minute (0.76 m/s) at any point of the access port or window.

IFC 6004.2.2.3 Leaking cylinders and tanks. One or more gas cabinets or exhausted enclosures shall be provided to handle leaking cylinders, containers or tanks.

Over 30 years ago ClF_3 was mistakenly classified by Larry Fluor (Gas Industry and Fire Code expert) as being

- Oxidizing gas, liquefied
- Unstable Reactive Class 3 detonable
- Water Reactive Class 2
- Corrosive
- Highly Toxic

The classification of Unstable Reactive Class 3 detonable requires that the gas cabinets be in a detached room constructed to H-1 requirements



The author in 2005 formally requested Larry Fluer to reconsider the classification based on the actual hazards associated with ClF_3 . He responded with the letter Larry Fluer to Eugene Ngai, "Classification of chlorine trifluoride CAS 7790-91-2", June 15, 2005.

He agreed that a mistake was made and reclassified it as

- Oxidizing gas, liquefied
- Water Reactive, Class 3
- Corrosive
- Toxic

This removed the requirement for the detached room to be built to H-1 construction

Eugene Ngai

