Development of a DISS Cylinder Outlet Connection

In the US. Compressed Gas Association (CGA) Outlet connections were developed for industrial and specialty gases to prevent incompatible gases or of extremely high pressure from being connected together. They are designed to provide a “bubble-tight” connection, typically in the range of 1 x 10^{-3} to 1 x 10^{-5} cc of helium per second.

Types:

1. Industrial
2. Medical
3. Specialty
4. Semiconductor

Over 54 connections have been established which cannot interconnect with each other to form a gas tight seal

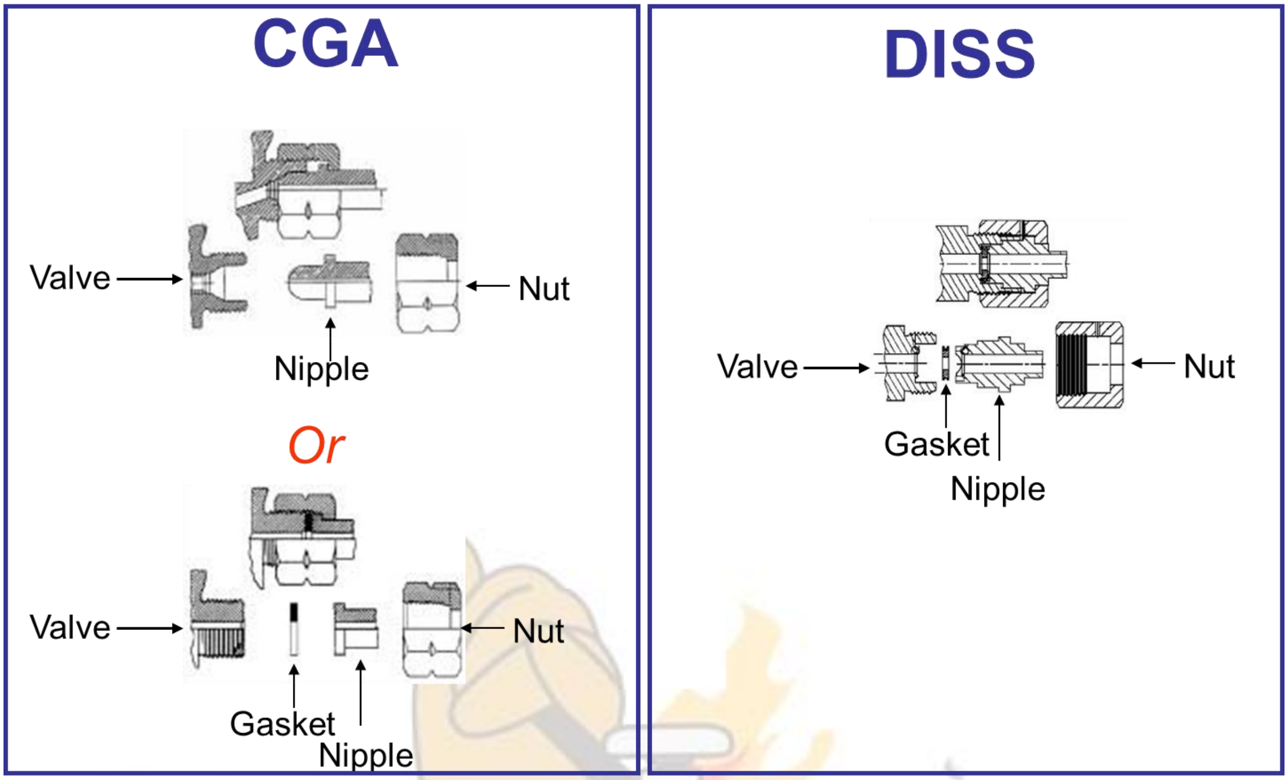
These unique connections are formed by using various combinations of:

1. Male/Female Threads
2. Different Diameters
3. Nipple Shapes
4. Right/Left-Handed Threads

Sealing is from:

1. Nipple Deformation
2. Flat Gasket
3. Tapered Thread

Ultra-High Purity (UHP) Semiconductor gases however require a mechanical connection that can achieve a leak tightness of 10^{-8} cc/sec or higher.[1]. The Diameter Index Safety System (DISS) outlet connection was designed for this purpose. The system relies on two different diameters in line to create a unique connection, thirteen are offered. Gases are grouped based on chemical and physical properties for the outlet assignment. These have been used since the late 1980’s with much success.



Figs 1 & 2: Standard CGA Connections and DISS Connection

History

The SEMI (Semiconductor Equipment Manufacturing Industry Association) Gases Safety Subcommittee Valve Task Force created a project to work with SSA (Semiconductor Safety Association, now SESHA) and CGA on this effort. A joint meeting was held on April 6, 1983, during the SSA annual conference where they defined the following features for the valve[2]

1. Better leak tight outlet connection, 10^{-9} cc/sec,
2. Replaceable Metal Gasket, recessed to avoid damaging the seal and designed to be removed with the nipple
3. Right Hand Thread
4. Pneumatic Operation
5. Restrictive Flow Orifice (In Valve)
6. 316 Electropolished Stainless Steel

Bill Kalaskie, Chief Engineer, Superior Valve Co. proposed using the newly developed Silane Outlet Connection in 1984.[3]

1.030-14NGO-RH-EXT (Tipped Nipple)

LIMITED STANDARD CYLINDER VALVE OUTLET CONNECTION FOR SILANE
(Electronic Industry Only)

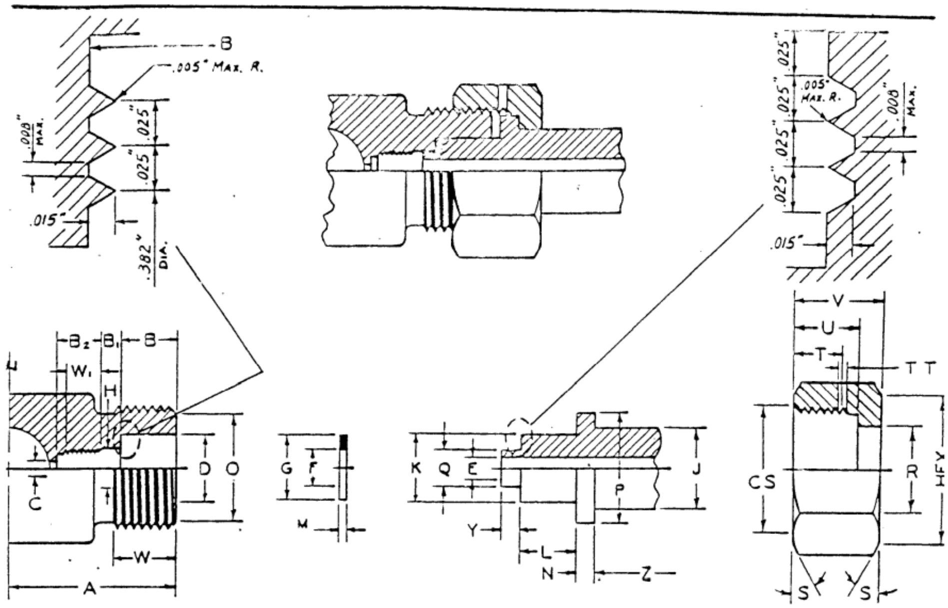


Fig. 3: Proposed Silane Connection

This however did not meet all of the committee requirements

Nupro developed a VCR type connection with keys that rotated on the face of the outlet to create a unique connection

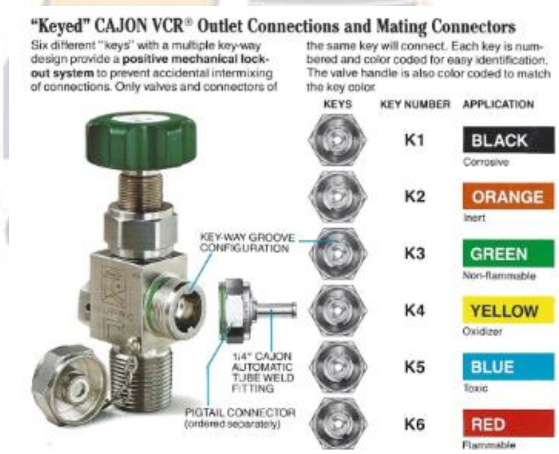


Fig. 4: Nupro Valve

Initially it was offered with 2 keys which provided 6 outlet connections. To make more connections the number of outlet keys was expanded to 4.

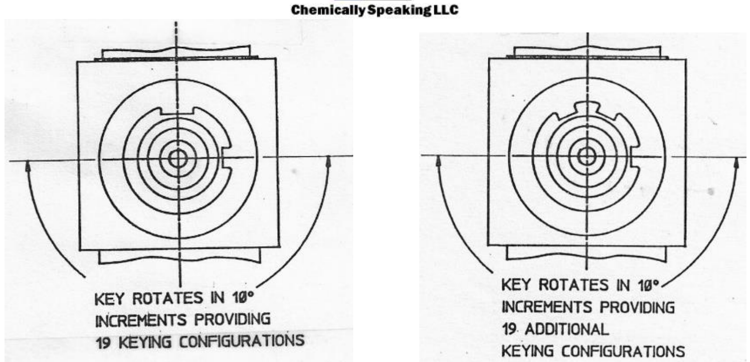


Fig. 5: Additional Keys

This increased the number of connections to 57

One gas supplier tested the valve using HCl by

1. Inserting a new valve into a cylinder and filling it with high purity HCl
2. With the vapor tight outlet cap on the valve is opened allowing the HCl to fill the valve deadspace for 8 hrs
3. Shut the valve and remove the vapor tight outlet cap and allowing it to be exposed to air overnight
4. Repeat step 2 and 3 every 3 days over a period of 30 days
5. After 30 days allow the cylinder to sit without the vapor tight outlet cap on for 60 days
6. After this period connect the cylinder to a pigtail and see if the connection seals and the valve is operable

The Ceodeux and Nupro valves pass the test. The Superior and Martin valves did not

The gas supplier had over 500 cylinders in service for a major US Fab. The problem was that a new valve had to be installed each time due to cleanliness concerns. There was no cleaning procedure or rebuild kits.

The Nupro valve however had 2 significant issues which were highlighted by the CGA Valve Connection Committee.

1. Proprietary connection
2. Did not follow CGA Outlet guidance

The valve also offered only limited PRD options which did not meet the CGA requirements. Due to these issues, no gas supplier would use the valve.

When the prototype DISS connections were tested, it sometimes created rotational friction as the Nut was tightened, this twisted the gland against the gasket causing damage to the toroidal bead. To prevent this, anti-rotation keys were machined into the gland and slots in the outlet connection

CGA outlet connections are designed to provide a “bubble-tight” connection, typically in the range of 1 x 10^{-3} to 1 x 10^{-5} cc of helium per second.

However, with the DISS connections the maximum outboard leak rate shall not exceed a value of 1 X 10^{-7} cc (He)/sec at 2000 psig Helium when the connection is tightened to the recommended torque level (e.g. 35 ft-lb using a Nickel washer).

When this outboard leak rate is achieved it is reasonable to expect that an inboard leak rate (one normal atmosphere) no greater than 1 X 10^{-9} cc (He)/sec will result.

The standard CGA 350 connection used for hydrogen or silane is not capable of routinely providing such a high leak integrity with repeated use. This bullet connection relies on a nut mechanically compressing a nipple into the valve outlet connection. The gas seal is formed by deformation of the nipple and/or outlet connection. The surfaces over time will become scratched and may not be capable of providing a high leak integrity. Fine metal particles can also be generated due to friction at the sealing point. In addition, stainless steel surfaces will work harden each time it is deformed, making it more difficult to seal. Leak rates for the connection after repeated use is typically 10^{-5} cc/sec.

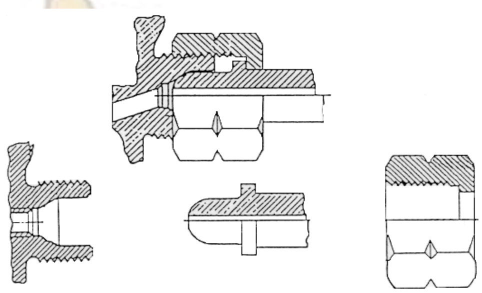
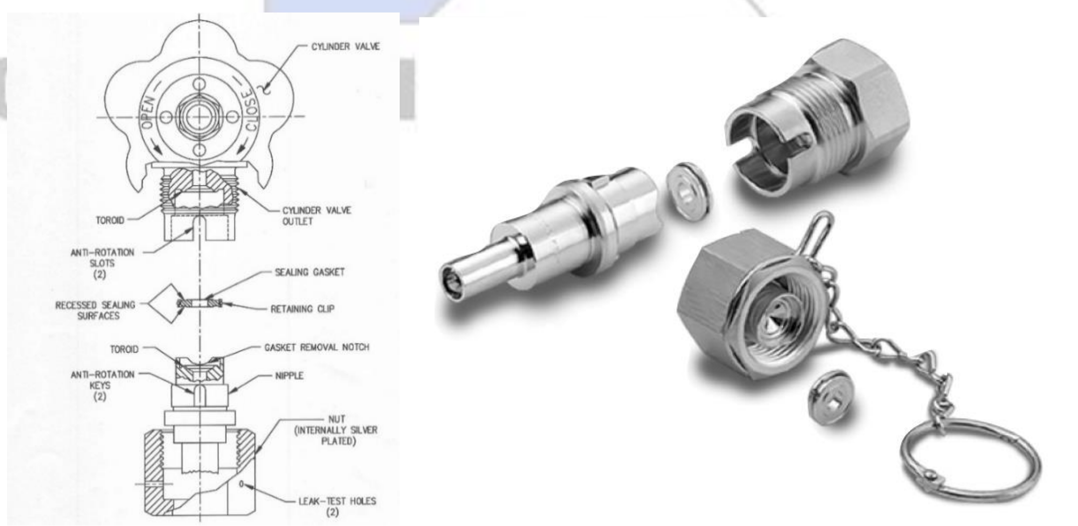


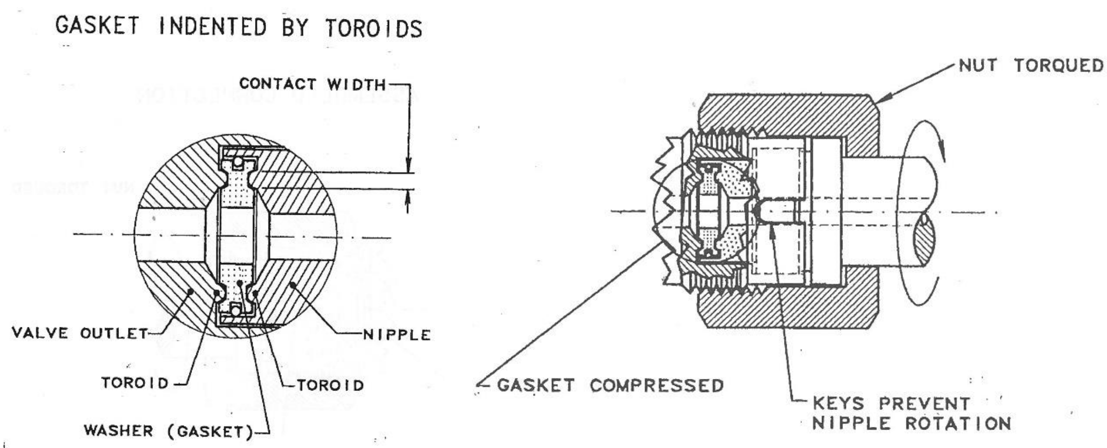
Figure 6: CGA 350 Outlet Connection, Bullet

A properly designed DISS connection can meet all the requirements. An additional benefit of the DISS connection design is the reduction of dead space in the outlet connection. Dead space can accumulate contaminants such as moisture when the outlet is exposed to air. These will be released over time into the gas stream as a virtual leak.



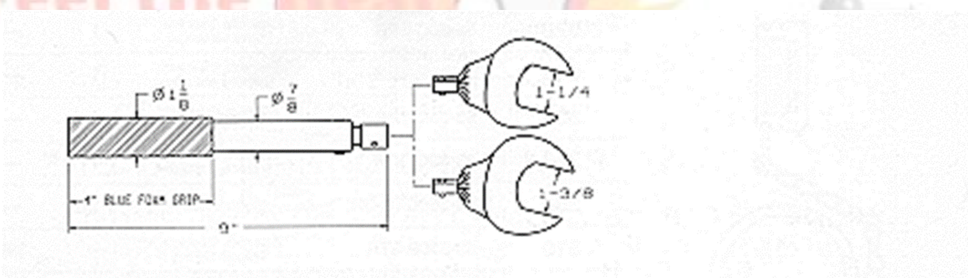
Figs. 7 & 8: DISS Connection, Outlet and Gland

The sealing points on the nipple and valve outlet are highly polished toroidal beads. When the nut is screwed to the valve threads, it pulls the nipple into the valve outlet compressing the gasket between the two beads. The polished beads are mechanically compressed by the nut into the gasket forming a gas tight seal.[4]



Figs. 9 & 10: DISS Gasket Sealing

To tighten the connection a torque wrench must be used. If not torqued properly it will leak



EWAL Number	Factory Set Torque	For Use With	CGA 632-642 Hex Flat	CGA 712-728 Hex Flat
77-DISS-TW	35 ft-lbs	Nickel Gasket	1-1/4	1-3/8
77-DISS-TW-K	12 ft-lbs	Kel-F® Gasket		

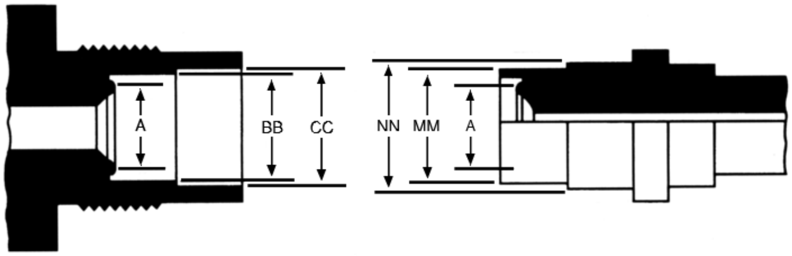
Fig. 11: Torque Wrench

Each DISS nut has a leak check hole where the connection can be tested using an electronic leak detector.

It has been in use successfully for almost 30 years.

There are 13 DISS connections. It is the only cylinder connection that is recognized and used worldwide.[5]

ISO 10692-1 Gas cylinders — Gas cylinder valve connections for use in the micro-electronics industry — Part 1: Outlet connection. Reaffirmed 2017



GAS NAME	CONN.#	DIAMETERS IN mm				
		*A	*BB	*CC	*MM	*NN
Arsine, Diborane, Disilane, Germane, Hydrogen, Selenide, Phosphine, Silane, Trimethyl Silane	632	.418-.422	.649-.653	.796-.800	.646-.642	.793-.789
Hydrogen Chloride, Boron Trichloride, Hydrogen Bromide	634	.418-.422	.663-.667	.782-.786	.660-.656	.779-.775
Dichlorosilane, Trichlorosilane, Silicon Tetrachloride	636	.418-.422	.667-.681	.768-.772	.674-.670	.765-.761
Tungsten Hexafluoride, Hydrogen Fluoride	638	.418-.422	.691-.695	.754-.758	.688-.684	.751-.747
Nitrogen Trifluoride	640	.418-.422	.705-.709	.740-.744	.702-.698	.737-.733
Boron Trifluoride, Silicon Tetrafluoride, Phosphorus Pentafluoride, Arsenic Pentafluoride, Germanium Tetrafluoride	642	.418-.422	.719-.723	.726-.730	.716-.712	.723-.719
Nitrous Oxide	712	.418-.422	.649-.653	.883-.887	.646-.642	.880-.876
Oxygen	714	.418-.422	.663-.667	.869-.873	.660-.656	.866-.862
Carbon Dioxide, Sulfur Hexafluoride, Halocarbons 11, 12, 13, 14, 23, 115, 116, Perfluoropropane, Pentafluoroethane, Octafluorocyclobutane and perfluoro-2-Butene	716	.418-.422	.677-.681	.855-.859	.674-.670	.852-.848
Nitrogen, Neon, Helium, Argon, Xenon, Krypton	718	.418-.422	.691-.695	.841-.845	.688-.684	.838-.834
Ammonia	720	.418-.422	.705-.709	.827-.831	.702-.698	.824-.820
Hydrogen Sulfide	722	.418-.422	.719-.723	.813-.817	.716-.712	.816-.806
Hydrogen, Carbon Monoxide, Deuterium, Ethane, Ethylene, Methane, Methyl Fluoride, H32	724	.418-.422	.733-.737	.799-.803	.730-.726	.796-.792
Diethylzinc, Dimethylzinc, Triethylaluminum, Diethyltelluride	726	.418-.422	.747-.751	.785-.789	.744-.740	.782-.778
Chlorine, Fluorine, Nitric Oxide, Chlorine Trifluoride	728	.418-.422	.761-.765	.761-.765	.758-.754	.758-.754

Fig. 12: DISS Assignment

The DISS connection makes the valve too large to fit under the standard 3 ¼" diameter cylinder cap. A much larger 5 ¼" diameter cap must be used. A collar adapter is attached to the standard cylinder collar to accommodate this larger cylinder cap. It is held in place by a hex screw.

Fig. 13: Cylinder Collar Adapters

Fig 14: Larger Cylinder Cap

Causes of DISS Connection Leakage

While DISS and VCR connections have high leak integrity, they are mechanically weak. Physical impact, vibration or twisting can break the seal causing a leak. A CGA 350 connection is mechanically stronger.

Some causes of DISS leakage in silane service

Expansion cooling

Expansion cooling of silane by the pressure reduction from 1450 psig to 50 psig or less (Joules Thomson Expansion) can create a large amount of cold liquid silane. At flow rates of 50 slpm or greater this is a significant problem. This has caused leakage in bulk systems at the DISS connection due to differences in thermal contraction of the stainless steel and the nickel gasket.

: Constant Enthalpy Expansion, Single Stage Pressure Reduction

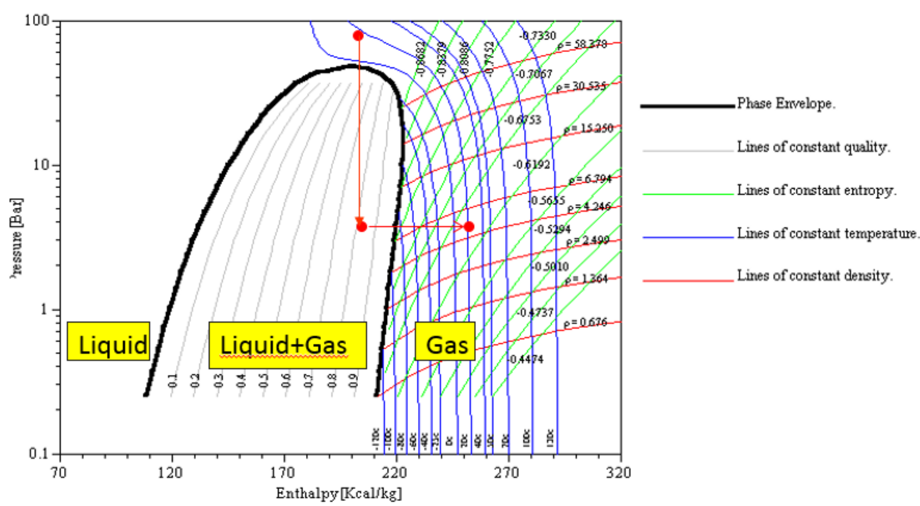


Fig. 15: Constant Enthalpy Expansion, Single Stage Pressure Reduction

As noted in the thermodynamic chart, a single stage expansion of silane at constant enthalpy will enter the liquid region. Silane expansion cooling at high flow rates is estimated

1200 psig to 0 psig, -124°F (-87°C), 19 wt% liquid
1200 psig to 200 psig, -55°F (-48°C), 26 wt% liquid

In one case the expansion cooling occurred at the tube valve when it was opened to feed the system. The cold liquid thermally contracted the ISO Module outlet DISS connection within 10 secs causing a leak and fire as shown below. This was because of the difference in contraction of nickel versus stainless steel.

Figure 16: DISS Connection leak caused by cold liquid silane

The 1994 SEMATECH Silane Safety Improvement Report S71 noted that Kel F and Silver Coated Stainless Steel Gaskets are not affected by expansion cooling when silane expands from 1600 psig to 0. There have been leaks however with nickel and stainless steel gaskets.

This can not only occur during high use rates but also during system startup when the silane must fill a large dead volume. In another incident, a bulk system passed an inboard leak test as well as an outboard leak test. The pigtail was pressurized with Silane at 1450 psig for almost 7 minutes without leakage. It was not until the system valve was opened to bleed silane into the long fab line that the DISS connection at the Y container leaked. The cooling occurred for almost 30 sec, with the pigtail pressure slowly increasing until the operators heard a “pop” and flames of 50 cm from the DISS connection.

Mechanical Force

The nickel gasket can be damaged if there is impact or excessive torque on the pigtail. This can deform the gasket causing a leak.
Vibration of the system loosens the connection.

Improperly supported pigtail that applies stress on the DISS connection damaging the gasket.

Back purging through the connection has loosened the gasket

A properly made connection with a nickel gasket at 35 ft-lbs only requires 10 ft-lbs to loosen.

Fig. 17: Silane ISO DISS Leak

Initially silane ISO Modules and Cylinder packs used a DISS 632

Gasket

A new uncoated nickel 200 gasket must be used each time the connection is made. This must be fully annealed after machining. Maximum hardness of 100 Brinell and a surface finish of 10 Ra (microinches). In one case the user reused the nickel gasket. This leaked as soon as the valve was opened. A nickel gasket will continue to work harden with use. It will increase from 75 Brinell to 125-135 Brinell and if used a second time and increase to 200 Brinell. This will be too hard to seal at the specified torque.

Metal gaskets are easily damaged and if they are opened and dropped on the ground they must not be used. If a tool or other heavy object is laid on the gasket, it can be damaged and cause a leak

Returned DISS Connections must be rigorously cleaned and inspected before they can be used again. Magnifying glass is used to visually inspect the toroidal beads.

DISS outlet connections can be damaged during use:

1. Pulling on the gland when the DISS is connected
2. Not parking anti rotation slots when tightening Nut
3. Worn anti rotation keys which allow over torque

Special repair procedures are required to return these to use.

Figs.16 & 17: Damaged DISS Connections

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References

1. CGA/DISS Valve Outlet Comparison, Air Products and Chemical Summary for CGA, November 2005
2. Kalaskie, W., A Superior Automatically Operated Cylinder Valve for the Semiconductor Industry, SSA Newsletter Vol 8, No. 1, 12-14, Jan 1986
3. News Update, Semiconductor Safety Association, Nov 1984
4. Aldridge, L. L., Sealing The CGA 630/710 Series of Connections, Presented to CGA, EWAL, Nov 1993
5. Matheson Tri-Gas Gases and Equipment Catalog Pg xxii, 2007