# Development of a Cylinder Valve Restrictive Flow Orifice

May 1, 2022

## Introduction

Restrictive Flow Orifices (RFO) have become a key safety tool for users of highly toxic, toxic or pyrophoric compressed gases. The RFO is an effective, inexpensive safety device that limits gas discharge rate during an accidental release. It is a passive device and does not modulate flow like a mass flow controller. As the pressure decays in the cylinder the flow will also decay. In theory an RFO was expected to cause immediate ignition of a silane release, however this has not been the case. For pyrophoric gases, like silane, the orifice reduces gas flow so that the gas burns controllably at the release site; for toxic gases, the release rate is limited so that dilution alone can mitigate the hazardous condition.[1]

The RFO must be installed as close to the cylinder valve as practical. The most effective is having the RFO threaded into the outlet connection of the cylinder valve as shown in Figure 1. Here the orifice can provide protection in all likely accidental leak events including inadvertent opening of the cylinder valve while not connected to the system or a failure of the downstream piping system. The most likely leak is the cylinder valve outlet connection.

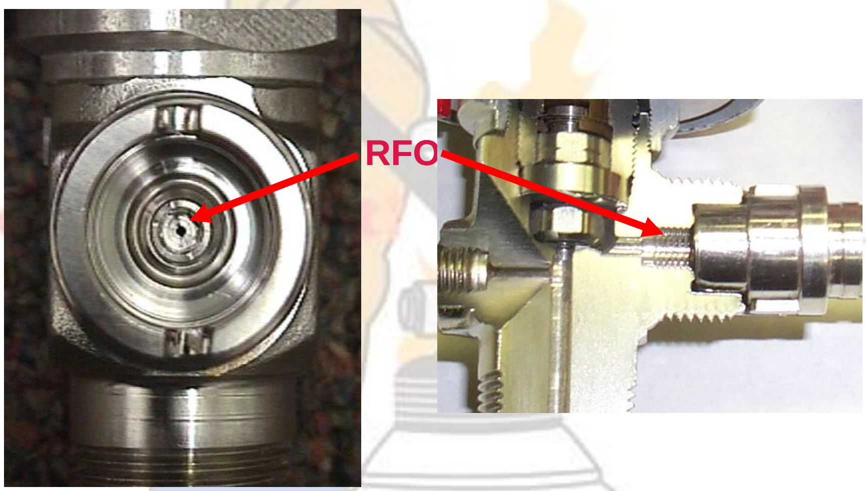



**Fig. 1: Restrictive Flow Orifice Installed in Cylinder Valve**

A cylinder cannot be filled while there is an RFO in the cylinder valve. It is installed by the gas supplier after the cylinder has been filled and just prior to shipment only if requested by the customer

The gas industry now has over 40 years of experience with RFO's. The primary users are semiconductor companies in the US, but use is expanding in other countries This article is a comprehensive review of development of the RFO.

## Background

The development of RFOs first began in the early 1980's, a time when the semiconductor industry was experiencing tremendous growth. During this period there were numerous compressed gas incidents (explosions, fires or acute exposures). There was significant concern with uncontrolled leaks, especially with silane which was the cause of some major incidents.

In the late 1970's and early 1980's many US users of silane experienced numerous fires and explosions. IBM and AT&T launched research projects and created task forces to examine this issue. Key research project from Hazards Research Corp., Battelle, Union Carbide, Intel.[2]

Britton summarized a major incident as follows:[3]

> In December 1977 a powerful explosion wrecked a piggyback trailer 11 miles east of Mojave. Eyewitnesses described a large fireball. A 5K cylinder (initially at 700 psi) was found to have zero pressure gauge. This cylinder had evidently leaked in the trailer. Ignition of silane which had accumulated at an ambient temperature of around 21 C caused the explosion. The flooring of the trailer was buckled and all sheet metal was sheared from the sides and top. Of 20 silane cylinders, 17 fell off of the train, which was travelling at 70 mph. Twenty four drums of antifreeze remained out of twenty-eight also on the trailer, and these had a concave head. Damage was consistent with a very rapid deflagration

Duct and gas cabinet vent explosions frequently occurred.

The Japanese Semiconductor Industry was also experiencing issues[4]

> Japan's industry in the past has experienced several safety related accidents involving fire, property damage and injury similar to the experiences of the United States industry In the United States the subject of Silane gas safety has been discussed at seminars under the sponsorship of SEMI (Semiconductor Equipment and Materials Institute) and SSA (Semiconductor Safety Association). Additionally, in Japan also discussions on toxic gas safety are becoming more frequent.

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

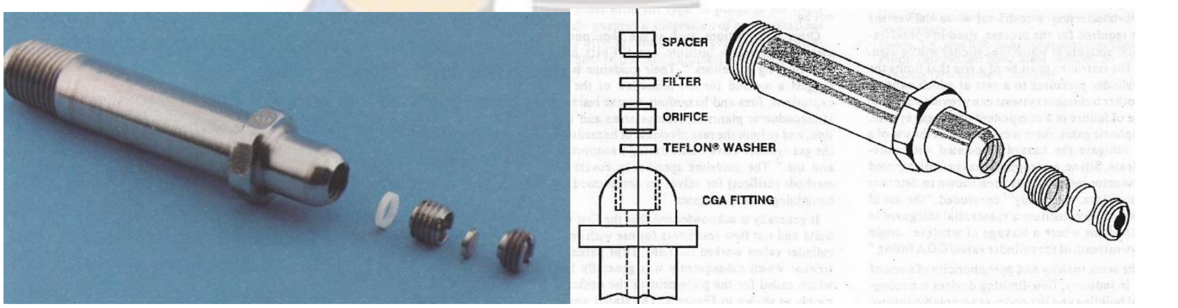



**Fig. 2: IBM Pigtail RFO[1]**

This was 0.006" dia with a 2 um filter. Bernie Meyerson, IBM was issued U.S. Patent 4,526,593 for this RFO design in 1985. [5,6]

During this time period, there was considerable interest in designing the next generation valve ("State of the Art") to improve Electronic Gas safety and quality. 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 (Compressed Gas Association).

A joint meeting during the SSA annual conference was held on April 6, 1983 which defined the features desired for the valve.[7]

Better leaktight outlet connection (minimum of $10^{-9}$ cc/sec)
Right Hand Thread
Pneumatic Operation
RFO (In Valve)
316 Electropolished Stainless Steel

Many equipment, valve and gas companies created projects to develop this cylinder valve. Key individuals and companies that took part in these efforts included:[8]

- Bill Korzenowski, Gary Johnson, Pat Taylor – Joint Project Linde (Now Praxair) & Veriflo
- Bill Koch - Nupro Valve
- Dick Martin - Martin Valve
- Bill Kalaskie - Superior Valve Corp
- Ceoduex (Rotarex)
- BOC (Airco)
- Jim Proctor & Jerry Sameth – Matheson Gas Products
- Ted Bielli – Motorola
- Philip Schull - Texas Instruments

These valves are better described in the article "Development of an Electronic Specialty Gas Cylinder Valve" which will follow this article.

Bill Kalaskie, Superior Valve Corp led the SEMI task force effort. Jerry Sameth and Jim Proctor of Matheson Gas Products tested the Superior Valve prototype in late 1984. This was the Superior Valve Corp brass springloaded metal diaphragm CGA 350 valve which was the most commonly used valve for arsine, phosphine and silane service at that time.

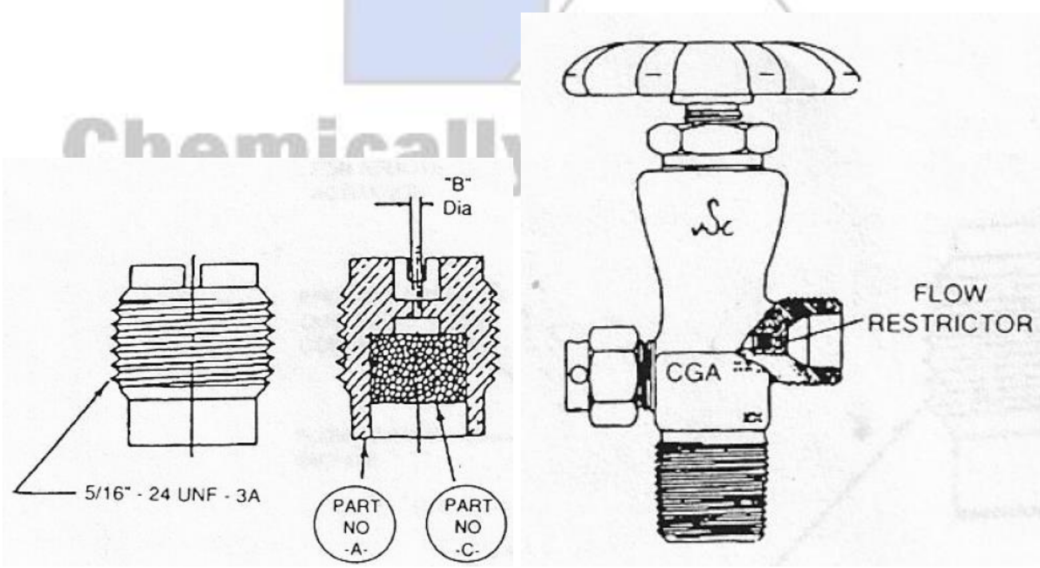



**Fig. 2: Superior Brass Valve With RFO[7]**

There were 6 versions of the RFO tested.[9]

| **Orifice Size, inch dia** | **Filter** Pore Size, um |
|---|---|
| 0.006 | 0.5 |
| 0.006 | 2.0 |
| 0.010 | 2.0 |
| 0.010 | 5.0 |
| 0.014 | 5.0 |
| 0.014 | 10.0 |

**Table 1: RFO's Tested**

They were all tested for flow using 2,000 psig nitrogen and purging of the deadspace in the valve created by the RFO.

The basis for the 0.010" diameter RFO more commonly used today for pure silane was probably 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" diameter orifice while they did occur with 0.040" diameter. A study by Southwest Research Institute #06-7725, November 1983 stated that no explosions occurred with a 15% silane mixture in nitrogen with a 0.040" RFO.[10,11]

Meyerson reported that testing determined that regardless of pressure (150-750 psig) through an 0.006" orifice or the release condition, open air, dynamic gas cabinet or static, the silane always ignited. The flame was not sufficient enough to cause any serious concern regarding the integrity of the gas cabinet or cylinder hardware.[2]

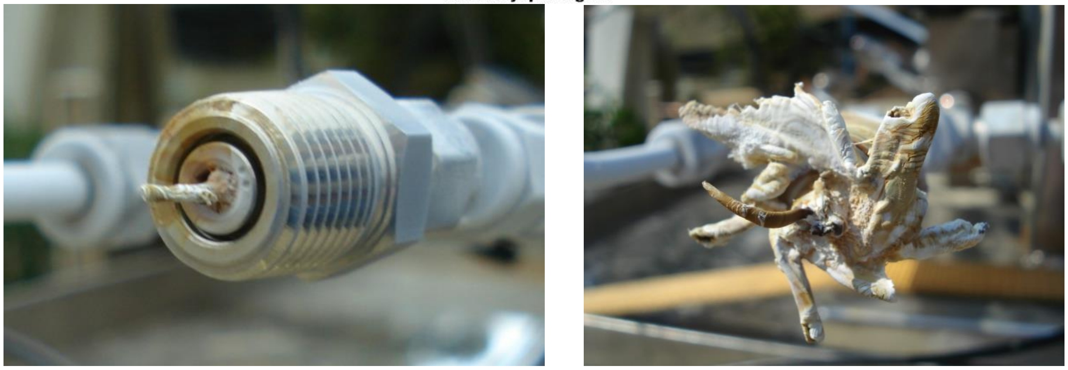
Larry Fluer goes further to state

> "It is current belief that if flow control (RFO) is provided at the source, fire not explosion is the likely event, however I have not seen data to substantiate the belief. I have questioned S. Tunkel at Hazards Research Inc. along this line and he indicated that there was no guarantee of ignition simply due to the presence of an RFO".[12]

This was confirmed by the testing Jerry Sameth conducted using a cylinder of silane filled to 150 psig. In the first test series, he used the 0.006" diameter with the 0.5 um filter and vented it directly to atmosphere. He was shocked to see no flame and when he closed the valve there was a pop and a small jet flame. He did this 2 more times with the same result. He then tested the 0.006" dia with a 2 um filter and the 0.014" diameter with a 10 um filter. These gave him the same result 3 more times.[9]

Testing by Prof Chen in 2009 further confirmed this. In 24 tests using RFO diameters from 0.006 – 0.060" only 2 had prompt ignition. The remainder did not ignite they always popped and had a jet flame when turned off.[13] He concluded:

> "The results from the dynamic release tests clearly indicate that the ignition behavior of pure silane released into air has no direct connection with the release pressure, whether the release is coming from a tube or from an RFO. It is, thus, concluded that the dominant factor controlling the prompt ignition of silane is a sufficiently low flow velocity."



**Figs. 3 & 4 $SiO_2$ Formation When Released Unignited Then Pop and Immediate Ignition[13]**

Superior Valve was able to fabricate a 303 stainless steel diaphragm valve in the for Electronic Gas Service

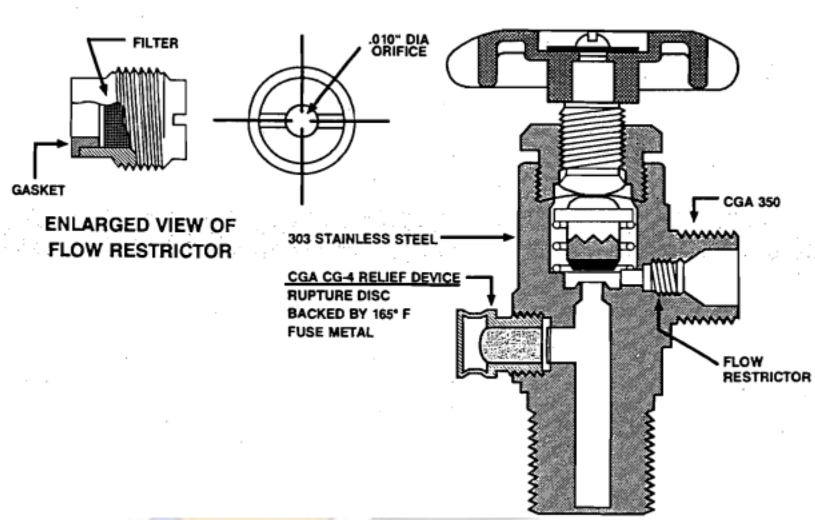



**Fig. 5: Stainless Steel Superior Valve with RFO[7, 10]**

**Fig. 4: Brass and Stainless Steel Superior Valve RFOs**

**Early RFO Design**

Other valve companies as they developed a new cylinder valve also designed RFO's for their respective valves


**Figs. 5 and 6: Nupro Valve RFO and Martin Valve RFO in Unloader**[11, 14]

British Oxygen Corporation (BOC) also developed a brass Flow Restrictor Valve (FRV) in 1982. They switched to stainless steel and added a 2 um filter in 1986.[15]

Due to concerns with users tampering with the RFO's, Superior Valve initially had a raised slot which required a special tool to insert or remove it

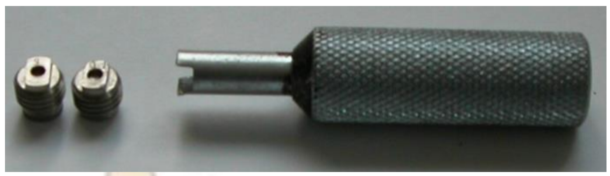


**Fig. 7 Slot Screwdriver Tool**

A later version had a screwdriver slot flat blade that was prone to stripping and damage. Some damaged RFO's had to be drilled out in order to empty the cylinder. This was a challenging and delicate task. One manufacturer briefly even offered a vandal proof screwdriver slot.

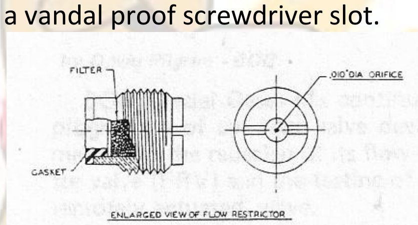



**Fig. 8: Screwdriver Flat Slot**

The RFO's now use a hex wrench which provides better torquing capability.

**Fig. 9: Raised slot, Flat Slot and Hex**

The 2 major US suppliers of RFO's now use a 4 mm (5/32") (Rotarex) or (GasFlo) 1/8" hex.[16, 17]

**RFO Identification**

The RFO is such a small device that it limited how they could be marked to indicate the orifice size. One early proposal by SEMI was to use the following marking for 3 orifice sizes.[18]

| Orifice Size | Air Flow @100 psig, 1pm | Marking |
| --- | --- | --- |
| A | 1 ± 0.5 | / |
| B | 3 + 0.5 | X |
| C | 6 + 0.5 | * |

**Table 2: SEMI RFO Marking**

As this did not provide enough sizes this was never implemented.

Initially the RFO were stamped with the size as shown in Figure 10.


**Fig.10: Superior Valve RFOs Stamped with Orifice Size**

These were a challenge to stamp. The current system uses either a series of dots or notches around the face. [16, 19]

**Filter**

Due to concerns with particles plugging the RFO, there was a sintered metal filter in the initial RFO designs. As expected, this filter reduced the flow through the RFO. Nupro™ cylinder valve testing showed that for air at 2,000 psig the flow rate through a 0.010" orifice with a 5 micron filter was reduced by 38% and with a 2 micron filter by 75%. The test report also highlighted the potential plugging and purging problems that a filter poses. A 5 micron filter on a 0.01 inch orifice will collect, and become plugged by particles only 2% of the orifice diameter. The filter will be plugged much more easily than the orifice it is designed to protect.[8, 17] With a filter present, purging also needs to be increased and insufficient purging can create particulates that will plug the filter.

The sintered metal filter has a large surface area where moisture can accumulate and discharge into the gas stream over time. The sintered metal filter is press fit into the RFO body. This creates particles that will be released into the gas stream.

Filters were a severe problem when used for corrosive gases like hydrogen chloride. The moisture on the filter reacted to form corrosive hydrochloric acid which quickly corroded and plugged the filter.

Without the filter, flow through the orifice in normal operation and under a release scenario is in the critical or choked flow range, since the inlet pressure to the orifice from the cylinder is two or more times greater than the outlet pressure. In this critical range, the gas velocity is at the speed of sound. Although gas velocity through the orifice remains the same, the flow will decrease linearly with entrance side pressure because of a decrease in the gas density in and through the orifice.[21]

**Gasket**
The brass RFO that Matheson tested in 1984 did not have a gasket which would seal the RFO and prevent any gas from bypassing it. They reported that some variation in the flow rates measured were likely due to leakage. The stainless steel RFO had a nylon gasket which disintegrated when it was tested by Solkatronic Chemicals with HCl in 1991. It was replaced by Kel F™ (PCTFE) which can absorb considerable moisture.

Testing has shown that it takes a long time to dry the system. Even with the filter removed from the testing has shown that drying by flowing a non-water soluble gas takes over 5 hours to reach a level of 1 ppm in the system.[22]

**Purging and Cleaning**
The RFO makes gas system purging more difficult by creating a small deadspace (1-2 cc) in the cylinder valve behind the small hole. Studies have shown that purge cycles have to be substantially increased to reach the same level of purity as previous and times increased.[1, 10] The maximum vacuum that a gas cabinet vacuum venturi can pull is -12.5 psig while the use of a mechanical vacuum pump that can achieve -14.7 psig is more effective.[22]

Table I: Effect of RFO on Cycle-Purging of a Gas Cabinet Connection

| Cylinder Valve RFO Size | Pigtail RFO Size | 21 Cycles (ppb $SF_6$) | 40 Cycles (ppb $SF_6$) | 60 Cycles (ppb $SF_6$) |
|---|---|---|---|---|
| none | none | 67 | 15 | 6 |
| 0.06” | none | 156 | 25 | 15 |
| 0.01” | none | 165 | 51 | 28 |

Insignificant differences were observed between the valves fitted with either the smallest (0.006” diameter) and the largest (0.16”) RFO’s. Since the calculated conductances for these orifices vary by almost three orders of magnitude, it must be concluded that the orifice itself is not the limitation to the dry-down of the valve under the conditions of the test.

Air Products study of impurities after 60 purge and evacuation cycles. Sulfur hexafluoride at 300 psig was used as the tracer (process) gas and UHP $N_2$ at 90 psig was used as the purge gas. Vacuum to -12.5 psig was provided by a nitrogen-driven venturi generator.[22]

Figure 11 is the effect with the evacuation times extended from 15 seconds to 40.

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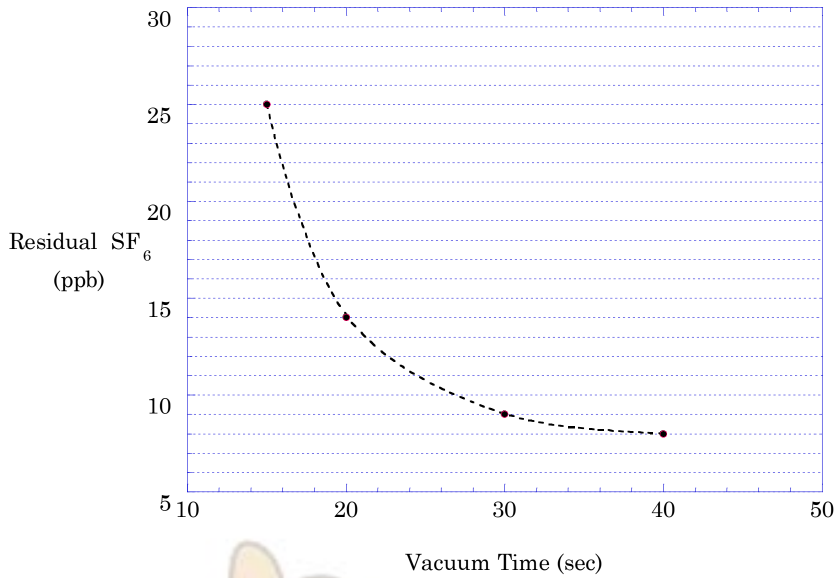



**Fig. 11: $SF_6$ Impurity After Varying Evacuation Times**

**Hazardous Consequences with and without RFOs**

The use of an RFO does not provide significant risk reduction during cylinder transportation, onsite handling or storage. This is because cylinders containing highly hazardous gases are required to have the valve handwheel secured shut and a vapor-tight outlet seal installed except during cylinder connection and usage. Pneumatic cylinder valves are normally closed and have a manual lock. However, an RFO installed in a valve outlet does provide additional risk mitigation when the vapor-tight outlet seal is removed for valve inspection, cylinder connection and product usage. During this time, personnel and equipment may be exposed to an accidental release of product via equipment malfunction and/or operator error. The RFO will significantly limit the sonic flowrate achievable from the container versus the wide-open withdrawal valve flow coefficient (Cv).

All product hazardous effects during container connection and usage are reduced via a properly positioned RFO. These include jet fire, vapor cloud explosion and downwind dispersion distances for outdoor release. Therefore, safety systems associated with the gas delivery and distribution may be reduced accordingly, such as room or enclosure ventilation rates to capture releases and treatment systems sized for emergency releases.

**Figs. 14 & 15: Silane Flames With and Without a 0.010" (0.25 mm) RFO (1,100 psig)[23]**

**Regulations and Standards**

With the exception of silane, worldwide there are no regulations mandating the use of RFO. The Northern California Fire Chiefs Hazardous Materials Code Committee 1990 report by R. Hanselka Pyrophoric and Self Detonating Gas Fact Finding mandated the use of a 0.006” diameter RFO and the cylinder must be stenciled. This was adopted by communities in Silicon Valley.
ANSI/CGA G-13 Storage and handling of Silane and Silane Mixtures 10.2.4 requires a RFO of 0.010” diameter or less installed in silane cylinder valves. This is a globally harmonized standard that has been adopted by:

1. European Industrial Gas Association (EIGA)
2. Asia Industrial Gas Association (AIGA)
3. Japan Industrial and Medical Gas Association (JIMGA)

In the US, ANSI/CGA G-13 is referenced in the International Fire Code (IFC) and the National Fire Protection Association (NFPA) making it a regulatory requirement for silane.

One of the initial drivers for RFO use in the US for gases other than silane was the 1987 edition of the Uniform Fire Code Article 80, Hazardous Materials. For highly toxic and toxic gases, an abatement system had to be sized to reduce the worst-case flow from the largest container/cylinder to ½ IDLH at the point of exhaust ventilation discharge to the atmosphere (8003.3.1.3.5.3 Performance) For a non liquefied gas a vent duration of 5 minutes and liquefied gases a vent duration of 30 minutes was to be used (8003.3.1.3.5.6) to size the abatement system. While it did not require the use of RFO’s it allowed the user to take credit for a reduced accidental gas flow to size the abatement system if an RFO was installed in the cylinder valve since it is a passive device.

This provision reduced the size of these systems considerably. To meet the Fire Code requirement an abatement system would have to reduce the exhaust concentration to 1 ½ ppm from a 50 lb arsine cylinder venting at 233 lpm. If a 0.010” RFO is used the flow rate drop to 4.5 lpm or over fifty times less. Most users specify a smaller diameter RFO, 0.006” for highly toxic gases.

This dramatically increased the use of RFOs in the US. The 2021 edition of the IFC 6004.2.2.7.5 Portable tanks and cylinders has the same provision. These same provisions have been carried over into NFPA 318 Standard for the Protection of Semiconductor Fabrication Facilities (7.7) and NFPA 55 Compressed Gases and Cryogenic Fluids Code (7.9.3.6)

The Santa Clara County, CA Toxic Gas Ordinance 17.78.400 when it was first introduced in 1992 required the use of a RFO for all highly toxic DOT Zone A gases such as arsine, diborane, phosphine as well as silane.
FM Global Standard 7-7 “Semiconductor Facilities” 2010 edition requires the use of an RFO

> 2.2.12.5 Provide an excess flow valve, or an excess flow switch connected to the emergency shutoff valve, for all cylinders of process gases. In addition to this shutoff valve, a restrictive flow orifice (RFO) should be provided in the gas cylinder valve body. For all process gases, except for silane and silane mixtures, the RFO should be sized at 0.010 in. (0.25 mm) unless a larger orifice is needed to meet process demands. For 100 percent silane and silane mixtures, RFO size should be in accordance with Section 2.2.11.3 *Silane Cabinets and Enclosures.* Cylinders containing any one of the following compressed and liquefied gases should be equipped with a restrictive flow orifice:

silane, arsine, phosphine, diborane, hydrogen, methane, disilane, germane, hydrogen selenide, hydrogen sulfide and stibine. An RFO cannot be used with low vapor pressure gases such as dichlorosilane, chlorine trifluoride, tungsten hexafluoride and boron trichloride.

An RFO cannot be used for the low pressure gases as it would not allow sufficient flow into the system.

SEMI S5 - Safety Guideline for Sizing and Identifying Flow Limiting Devices for Gases (2021 edition) requires RFO's for

| *Hazardous Gas* | *Standard Density (D), 1 atm., 0°C [kg/m³] ([lbs/ft³])* | *Cylinder Pressure (Pcyl) Typical Max. @ Room Temp.#1 [kPa_gauge] ([psig])* |
|---|---|---|
| *Arsine, $AsH_3$* | 3.45 (0.216) | 1,410 (205) |
| *Carbon Monoxide, CO* | 1.250 (0.078) | 11,385 (1,650) |
| *Diborane, $B_2H_6$* | 1.250 (0.078) | 12,410 (1,800) (1% in N2) |
| *Germane, $GeH_4$* | 3.415 (0.213) | 607 (88) |
| *Hydrogen, $H_2$* | 0.090 (0.0056) | 15,180 (2,200) |
| *Nitrogen Trifluoride, $NF_3$* | 3.200 (0.199) | 10,000 (1,450) |
| *Phosphine, $PH_3$* | 1.519 (0.095) | 4,095 (594) |
| *Silane, $SiH_4$* | 1.433 (0.090) | 8,280 (1,200) |
| *Stibine, $SbH_3$* | 1.444 (0.090) | --- |

**Table 3: SEMI S5 Gases Requiring RFO**

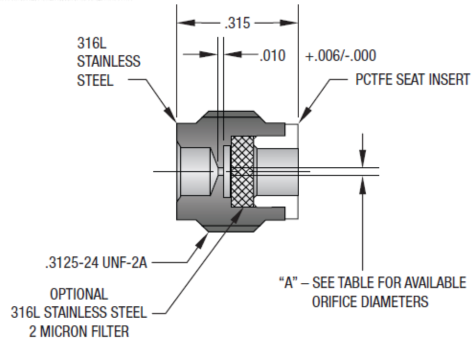
Most companies in Asia use RFO's to only comply with ANSI/CGA G-13 requirements but not for the highly toxic gases since there is no regulatory requirement to do so. US companies located in Asia however require adoption of the US requirements at their sites.

In Europe some users are requiring RFO's. As in Asia, the US owned facilities require them. The British Compressed Gas Association (BCGA) for example Code of Practice 18: The Safe Storage, Handling and Use of Special Gases in the Micro-Electronics and Other Industries requires a RFO for pyrophoric and highly toxic gases

**Current Design**

After years of development and testing, the primary Electronic Specialty Gas (ESG) valve supplier in the US is now Rotarex. The primary RFO supplier is GasFlo Products Inc. Fairfield, NJ. RFO's are also available from Rotarex

RFO's are available in sizes 0.005"-0.125" diameter, electropolished 316 stainless steel with a PCTFE seat. Rotarex has optional Nickel or Hastelloy RFOs for use with the corrosive gases. 1/8" hex wrench is used to insert or remove the GasFlo RFO or a 4 mm (5/32") hex wrench for the Rotarex. [16, 17, 19]




**Figs. 16 & 17: GasFlo RFO**

As shown in Figure 17, the RFO is about 3/8 inch in length. A Kel F (PCTFE) gasket is used a seal between the restrictor and the valve body. It has a 5/16 inch 24UNF-2A thread. The threads are truncated to allow the gasket make a seal. RFO's from Rotarex or GasFlo can be interchanged.
Drilling such small holes in an RFO has been a challenge. The holes can be made by drilling, laser or electro-discharge machining (edm). One supplier stated that laser drilling does not produce a round hole and generates slag on the outer edges of the orifice. The actual flow rate from a RFO can vary due to eggshape, ragged edge. A CNC can drill down to 0.010" diameter.

Rotarex in a 2008 e-mail stated[24]

1. Our flow restrictor are laser drilled with tolerance +/-0.02mm + electropolishing
2. Test in production: we make visual control under microscope at 100% in order to check no burrs and no wastes, cleaning in CREST machine and for diam. <0,5mm we check the flow according pressure and flow limits given by R&D; for diam. >0.5 mm we do not check the flow (diam. 0,5 + 1mm are controlled to the incoming inspection by samples with pin).
3. To determinate the flow and hole size, R&D dpt. uses an equation according to CGA V9.
4. Our flow restrictors are 100% tested to verify that they meet the flow requirement. They are tested with air or nitrogen. We are drilling the holes whenever possible since there is a larger tolerance when using a laser to create the hole. It is our opinion that due to the variances in hole size, they should be checked 100%.

The two primary RFO manufacturers have different systems of identifying the orifice size, GasFlo (Parker) use a series of dots around the outside rim while Ceoduex uses a series of notchs

**Figs 18: Rotarex, Notches and GasFlo, Dots**

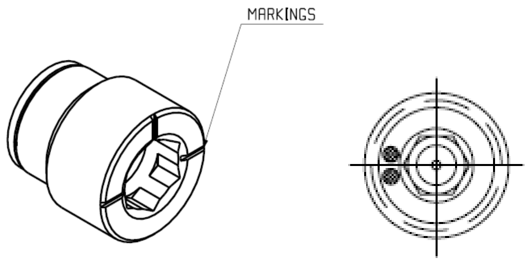



**Fig. 19: Rotarex, Notches and GasFlo, Dots[16, 17, 19]**

| Size, in | | 0.005 | 0.006 | 0.007 | 0.008 | 0.010 | 0.012 | 0.014 | 0.015 | 0.016 |
|---|---|---|---|---|---|---|---|---|---|---|
| Size, mm | | 0.12 | 0.15 | 0.17 | 0.20 | 0.25 | 0.30 | 0.35 | 0.40 | 0.40 |
| GasFlo ID | | | | | | | | | | |
| Rotarex ID | | 3 notch | 2 notch | | 4 notch | 1 notch | none | 9 notch | | 7 notch |

| Size, in | 0.020 | 0.030 | 0.035 | 0.040 | 0.050 | 0.060 | 0.070 | 0.080 | 0.090 | 0.125 |
|---|---|---|---|---|---|---|---|---|---|---|
| Size, mm | 0.50 | 0.75 | 0.87 | 1.00 | 1.25 | 1.50 | 1.75 | 2.00 | 2.30 | 3.00 |
| GasFlo ID | | | | | | | | | | |
| Rotarex ID | 5 notch | 10 notch | | 6 notch | 11 notch | | | 12 notch | | |

**Table 4: Rotarex and GasFlo ID for Orifice Sizes**

Cylinders with an RFO installed must be identified with a label or marking indicating the presence of an RFO and the orifice diameter.

Many users want to verify the presence of the RFO and determine if the requested size has been installed. It is not safe to visually inspect the RFO by peering directly into the valve outlet. Use a mirror to view the cylinder valve outlet shown in Figures 20 and 21.

**Figs. 20 & 21: Mirror Inspection of RFO**

This may be difficult to see as the cylinder will be in a gas cabinet where lighting is poor and visual access is not that good. Some companies use a flexible fiberoptic boroscope which has a LED light to illuminate the outlet. Many also have a magnifier to allow closer inspection of the RFO. Some companies will also use it to take a photograph to use as evidence of compliance with the Fire Code.

**Fig. 22: Fiberoptic Scope Inspection**

**Gas Flow**

Numerous methods have been used to estimate gas flows. FM Global originally used the ISA Equation. It greatly over estimated flow.[25]

| Silane Flow Rate [slpm] (@70°F Source Temperature, 0 psig Downstream Pressure) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| RFO Dia. [in.] | Source Pressure [psig] | | | | | | | | |
| | 1500 | 1200 | 1000 | 800 | 600 | 400 | 200 | 100 | 50 |
| 0.020 | 355 | 277 | 214 | 157 | 108.5 | 67.8 | 32.7 | 16.6 | 8.74 |
| 0.014 | 174 | 136 | 104.9 | 76.9 | 53.2 | 33.2 | 16.0 | 8.13 | 4.29 |
| 0.010 | 88.8 | 69.2 | 53.6 | 39.3 | 27.1 | 16.9 | 8.18 | 4.15 | 2.19 |
| NOTE: The flows through the 0.014 and 0.010 in. RFOs are equal to 49 and 25% of the flow through the 0.020 in. diameter RFO. | | | | | | | | | |

**Table 5: FM Global Estimates of Flow Rates Using ISA Equation**

This was replaced using a FM Global developed model.[26]

| Silane Flow Rate [slpm] (Source Temperature: 77°F; Downstream Pressure: 0 psig; Discharge Coefficient: 0.8) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| RFO Diameter (inch) | Source Pressure [psig] | | | | | | | | |
| | 1500 | 1200 | 1000 | 800 | 600 | 400 | 200 | 100 | 50 |
| 0.020 | 284 | 223 | 171 | 123 | 85.5 | 54.4 | 26.4 | 14.1 | 7.86 |
| 0.014 | 139 | 104 | 83.8 | 60.2 | 41.9 | 26.6 | 13.2 | 6.89 | 3.85 |
| 0.010 | 70.9 | 55.8 | 42.8 | 30.7 | 21.4 | 13.6 | 6.72 | 3.52 | 1.97 |
| Note: The flows through the 0.014 and 0.010 inch RFOs are equal to 49% and 25% of the flow through the 0.020 inch diameter RFO. | | | | | | | | | |

**Table 6: Revised Flow Estimates Using FM Global Developed Model**

It is critical that the silane flow esimates are accurate as possible. The research by Dr. Tamanini, FM Global for Sematech determined that silane releases can cause significant over pressures. He concluded that in order to keep the gas cabinet intact if an explosion were to occur, overpressure had to be limited to a maximum overpressure of 1 psi. A gas cabinet exhaust ventilation flow 250 times the RFO flow would achieve this under all likely conditions. Overpredicting silane flow rates would be costly in energy usage.[27]

When ANSI/CGA G-13 was developed, Air Products was using the Ultraflo capillary model which also over estimated the flow. They switched to the ASME Section VIII Orifice equation for compressible (non-ideal) gases flowing at sonic velocity that is based on a thin wall model. This closely matched the FM Global value (Table 6) s and was used by AP to develop the ANSI/CGA G-13silane flow tables.

The Gas Constant (C) equation is:

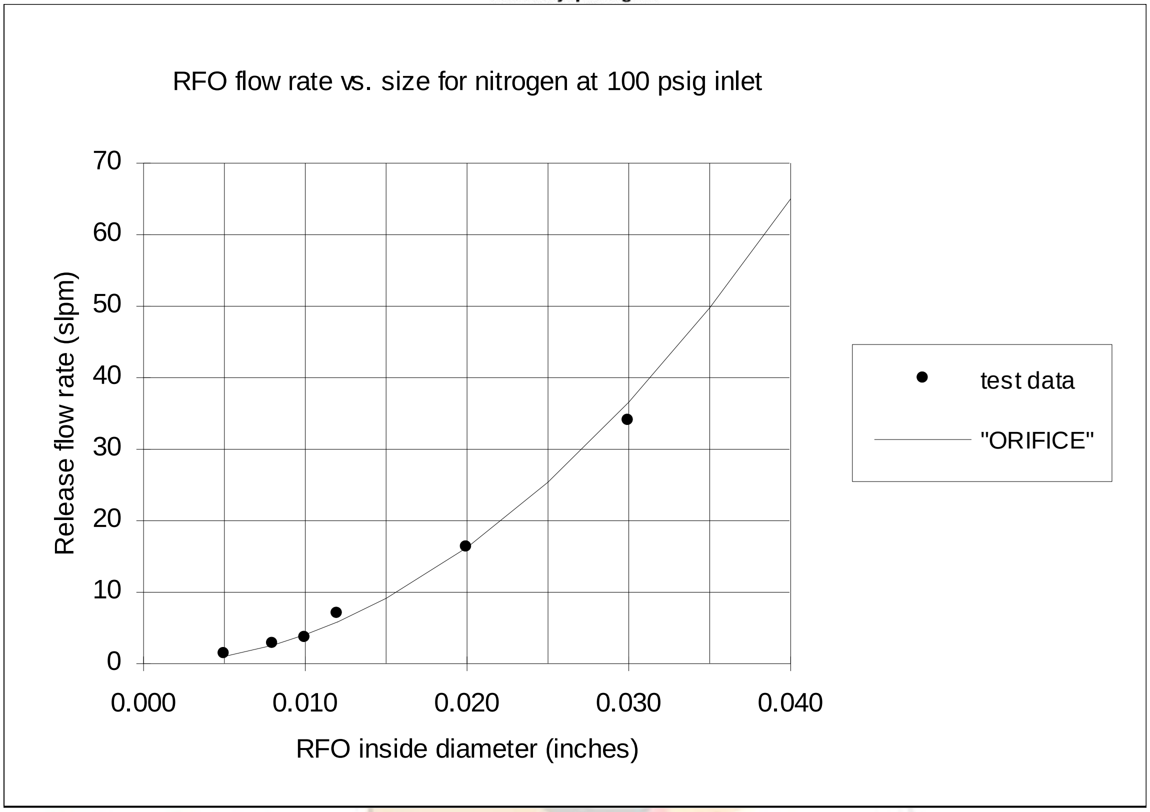
$$C = \sqrt{\left[\frac{k}{k+1}\right]^{\left(\frac{k+1}{k-1}\right)}} \quad \text{where } k = Cp/Cv$$

K is calculated for Cp and Cv specific heats at STP.
So C = 342.1 as a constant for silane.

$A = 7.85 \times 10^{-5}$ $in^2$
K = 0.8
P1 = 1515
MW = 32.1
Z = 0.424
T = 530

Result is 2.47 cfm

The Air Products Orifice program as shown in the following graph accurately predicts $N_2$ flow at various orifice sizes




**Graph 1: Air Products Orifice Software Estimate of $N_2$ Flow and Actual**

Other flow estimates NFPA 55 7.9.3.6.2

$$CFM = (767 \times A \times P)\frac{(28.96/MW)^{1/2}}{60}$$

where:

$CFM$ = standard cubic feet per minute of gas of concern under flow conditions

$A$ = area of orifice in square inches *(See Table A.7.9.3.6 for areas of typical restricted flow orifices.)*

$P$ = supply pressure of gas at NTP in pounds per square inch absolute

$MW$ = molecular weight

Most use $N_2$ flow rates and the gas specific gravity to estimate flow using the following equation

$$\text{Flow} = \text{The Flow Rate of } N_2 \text{ at the Same Pressure} \sqrt{\frac{1}{\text{Specific Gravity}}}$$

| Gas | Specific Gravity |
|---|---|
| Arsine | 2.70 |
| Diborane | 0.95 |
| Disilane | 2.20 |
| Germane | 5.20 |
| Hydrogen selenide | 2.80 |
| Methyl silane | 1.60 |
| Nitric oxide | 1.00 |
| Phosphine | 1.20 |
| Silane | 1.10 |

**Table 8: Gas Specific Gravity**

For a 1500 psig silane cylinder with a 0.010” RFO there is a wide variance of estimated silane flow rates

| Estimate Method | Flow, cfm |
|---|---|
| NFPA 55 | 1.44 |
| Sandia | 1.61 |
| AP Ultraflo | 2.17 |
| ASME | 2.47 |
| FM Global | 2.50 |
| ISA | 3.17 |

**Table 9: Flow Estimates**

Of these the FM Global and ASME (AP Orifice) have proven to be the most accurate

**Procedure for Reuse**

Cylinder gas suppliers can reuse RFOs after following a strict cleaning and testing procedure. Typically, RFOs from used cylinders are removed and segregated based on the product to ensure that there will be no cross contamination. The RFOs are placed in an ultrasonic cleaning bath for three (3) 20 minute cycles, each with fresh deionized water. RFOs are blown dry with a nitrogen gun and placed in a bake out oven at 220$^{o}$F for 8 hours. The RFOs are then visually inspected and those showing thread or gasket damage are discarded. A calibrated gauge is used to gauge the size of the orifice to determine if the hole is the specified size. RFOs that pass the gauge test are bagged and labeled with the product serviced, orifice size and inspector’s name.

**Other RFO’s**

For gases other than Highly Toxic or Pyrophoric, a cylinder valve RFO is normally not available. For users that require RFO’s in these gas systems, GasFlo offers CGA DISS Nipples that are tapped for an RFO.

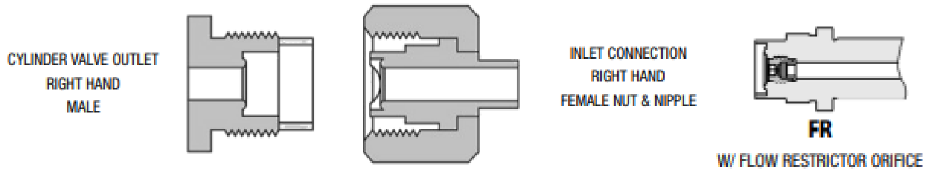



**Fig. 24: GasFlo CGA DISS Nipple Threaded for RFO**

It uses the same RFO as those made for cylinder valves.

Sandia designed and tested a Swagelok pipe adapter that has an orifice[30, 31]

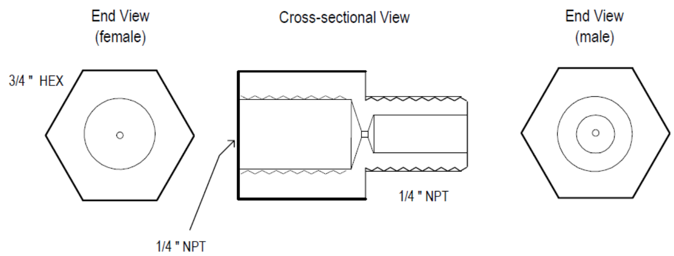
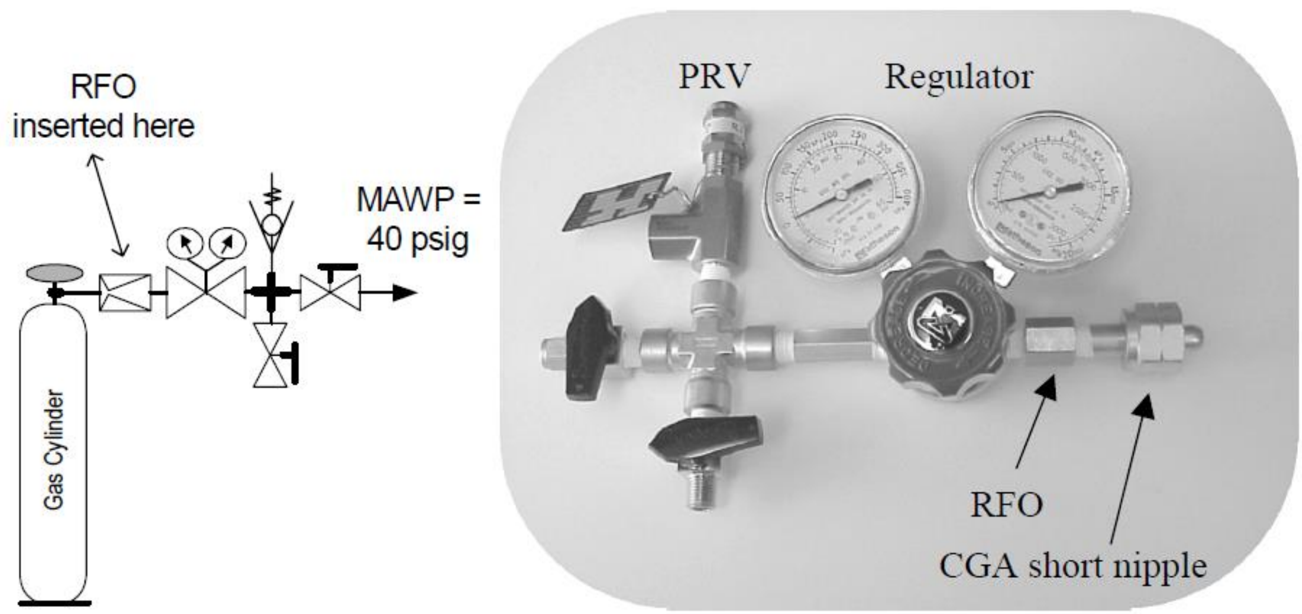



**Fig. 25: Sandia/Swagelok Pipe Adapter RFO**

This design also attempts to minimize the effects of manufacturing tolerances on the gas flow through the RFO. The length of the RFO is greater than the orifice diameter to allow a consistency of orifice diameter and symmetry. Approach velocity can have considerable effect on flow through an orifice: approach velocity variations are minimized due to the large inside diameter of the fitting (from either side) compared to the small orifice diameter size. This design limits the flow in either direction to the same rate.




**Figs 26 & 27: Use of Pipe Adapter RFO in a System**

**Miscellaneous Issues**

As expected there continues to be issues with the use of RFOs. These are the most common:

1. Plugging of orifice. This has been a problem periodically; removal of the filter reduced this problem. Proper purging of the cylinder valve before opening the valve minimizes this. When this plugging occurs, the cylinder must be sent back to the gas supplier. The RFO is removed and visually inspected to determine why it was plugged. If it cannot be easily removed, it must be drilled out.
   Ed Van Schoick a 50 year veteran in the gas and waste disposal industry has had to deal with many plugged or damaged RFO's that were primarily silane related. He stated that

   > "The clearance between the RFO and the valve seat wasn't much. A standard drill bit would barely penetrate the RFO before it touched the valve seat. My concern was that damage to the seat could create an uncontrollable leak. I tried using bottom mills, but they were difficult to control. I also tried left handed bits, hoping that some of the RFOs would unscrew during the process. Nothing met the criteria of being safe and reliable. We, therefore, abandoned the project and continued our practice of drilling the sidewalls."
2. Corrosive gases such as chlorine or hydrogen chloride remain an issue due to corrosion. Aluminum Silicon Bronze RFO's have not worked well.
3. Expansion cooling. The RFO acts as an expansion valve during high flow rates. During system startup of a Silane Y container feed system, the RFO acted as an expansion nozzle. This was initially opened to a long piping system that connected the exterior pad to the Fab building. It immediately cooled the silane down to <-50°C creating liquid silane. This thermally contracting the DISS connection and caused a leak at the connection. Procedures need to be developed to recognize this problem to ensure that it does not happen.
4. Larger RFO's are being supplied for use on low vapor pressure corrosive gases such as boron trichloride or tungsten hexafluoride to get adequate flow rates.

**Conclusion:**

RFO's have been in regular use for 40 years with silane and the highly toxic gases. Initial problems such as plugging and leakage have been resolved with changes in design and/or procedure. They are now an inexpensive and simple device that adds a passive layer of safety in the use of pyrophoric and highly toxic gases.

Eugene Ngai



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