ADVANCED PRESSURE TECHNOLOGY

Product Note, PN 408, Revision 1
Source Regulator Outlet Pressure Rise in NF_3 Service

May 4, 2016

Introduction

AP Tech customers are requesting products for higher NF_3 flow rates in source systems and some customers are installing NF_3 bulk specialty gas systems to meet the rising demand. Increasing flow rates and consumption are leading to problems such as system high delivery pressure alarms and regulator failures.

A short review of NF3 properties

NF_3 is supplied in cylinders at ~1500 psig (103 bar) at ambient temperature. Many facilities use the cylinder until the pressure drops to 150 psig (10 bar). NF_3 is a strong oxidizer that readily supports combustion. At high temperatures (>300ºC), NF_3 will disassociate into reactive fluorine species. Rapid pressurization of high pressure lines in NF_3 service can result in high temperatures due to adiabatic compression. These high temperatures could cause NF_3 to disassociate or ignition of plastics used for seats. For high flow bulk gas systems, heat is sometimes used to increase the gas enthalpy to counter the effects of Joule-Thompson cooling.

So what's the problem?

In some cases, the delivery pressure rise is simply due to pressure regulator **supply pressure effect** as the source cylinder empties. Supply pressure effect is the rise in outlet pressure caused by a drop in source pressure. Liquefied gases (such as hydrogen chloride) maintain a constant source pressure as the cylinder empties, but NF_3 source pressure drops by ~1300 psig as the cylinder empties. Regulator models that can deliver higher flow rates also have higher supply pressure effect values. Depending on the regulator model, the delivery pressure rise due to supply pressure effect can be almost 50 psig. Table 1 summarizes the delivery pressure rise due to supply pressure effect in several regulator models. If the manifold in service has a high delivery alarm, switching over to a standby manifold might cause the standby manifold to alarm depending on the equipment design. Customers may want to consider a two stage regulation system on high flow systems to reduce supply pressure effect.

Table 1. Supply Pressure Effect Summary

Regulator Model	Max Recommended NF3 Flow Rate	Supply Pressure Effect, delivery rise per 100 psig source pressure drop	Delivery Rise as NF3 Cylinder Empties from 1500 to 150 psig
AP1510	5	0.25 psig	3.4 psig
AP1410T	150	1.6 psig	21.6 psig
AP1210	600	3.5 psig	47.2 psig

In other cases, returned regulators have been inspected and significant **contamination** has been found on the PCTFE seat that prevented the poppet from making a leak tight seal. The advantage of the tied-diaphragm design is the sealing load increases as the outlet pressure rises above set pressure. The increased load can embed contamination in the seat, in effect moving the obstruction out of the way. In many cases the regulator will then seal but the outlet pressure rise can be more than 10 psig above the set pressure. In these cases, customers may want to consider the use of a filter to protect the regulator but consideration should be given to the strong oxidizing properties of NF_3 and the possibility of ignition due to adiabatic compression or particle impact.

In a few cases, returned regulators have been inspected and it appears that the PCTFE seat has been **eroded or chemically attacked** by the NF_3. A visual inspection has not been able to determine if the

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damage to the PCTFE seat was due to adiabatic compression, gas impurities, higher flow rates, reactive fluorine species, or some other factor. If a customer has this problem, they should consult with their gas supplier for recommendations. A comparison of different seat material properties is shown in Table 2. This information was collected from various published sources such as product data sheets and technical standards. The table shows that for chemical compatibility PTFE and PFA are the best materials. But, due to the cold flow and low tensile strength characteristics of these fully fluorinated plastics, AP Tech does not provide these materials in high pressure (high load) applications such as NF_3. In general, PI (Vespel®) does not have good compatibility with acids and bases, and therefore testing would be recommended before use. Traditionally, PCTFE (e.g. Kel-F®) has been used as the seat material in pressure regulators and valves in NF_3 service with no major problems. One AP Tech customer, who had regulator creep problems in NF_3 service due to erosion, installed several AP1410T regulators with a PVDF (Kynar®) seat. The regulators have been in service for over one year with no problems, compared to bi-monthly replacement.

AP Tech recommends that customers use PCTFE for regulator seats in NF_3 systems because PCTFE is rated better than Kynar in terms of oxygen index and auto-ignition temperature, and there is a long successful history of usage confirming that chemical compatibility is good.

Table 2. Properties of Seat Materials

Seat Material	Tensile Strength at 70F, psi	Tensile Strength at 160F, psi	Limiting Oxygen Index	Auto Ignition Temperature	General Chemical Resistance to Oxidizers	Chemical Compatibility with NF3
PCTFE (Kel-F)	5,700	2000	100%	388ºC	Good	Satisfactory
PVDF (Kynar)	7,000	4,000	44-60%	268ºC	Good	Satisfactory
PI (Vespel)	12,500	11,000	53%	300ºC	Fair-Poor	Unknown
PTFE (Teflon® PTFE)	4,000	No data	Did not propogate	434ºC	Excellent	Satisfactory
PFA (Teflon PFA)	3,600	3,600	100%	424ºC	Excellent	Satisfactory

So, how to prevent high delivery pressures?

If **supply pressure effect** is the problem, then there are several options: (1) use a lower flow regulator that has a lower supply pressure effect, (2) use a two stage regulation system, or (3) monitor the cylinder pressure and periodically rotate the adjustment wheel 1/4 turn counterclockwise as the cylinder empties.

If **contamination** is the problem, then using a particle filter and a tied diaphragm regulator will help increase the lifetime of the regulator, but eliminating sources of contamination is the real solution to this problem.

If **erosion or chemical attack** of the PCTFE seat material is the problem, then customers should consult with their gas supplier and might consider using PVDF, which may have better chemical compatibility with NF_3 but may increase the risk of seat ignition.

Please consult the factory or your local distributor for further information or assistance.

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