



Properly Purge and Inert Storage Vessels

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Inert gases are used to prevent fires and explosions in the vapor spaces of equipment. Correct selection of the oxygen concentration and the inert gas-flow rate are critical to ensuring safety.

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Purging and inerting (or blanketing) process vessels and equipment are two common, yet distinctive, practices to control the concentration of oxygen, thereby reducing fire and explosion hazards. Purging usually refers to the short-term addition of an inert gas (*e.g.*, nitrogen or carbon dioxide) to a tank, process vessel, or other piece of process equipment that contains flammable vapors or gases to render the space nonignitable for a specific time period (say, during a maintenance outage). In contrast, inerting (or blanketing) is the long-term maintenance of an inert atmosphere in the vapor space of a container or vessel during operation.

Safe oxygen levels

The goal of these methods is to reduce a vessel's oxygen concentration below the limiting oxygen concentration (LOC). The LOC is the concentration of oxidant below which a deflagration cannot occur in a specified mixture. The LOCs for common flammable gases and vapors, using a nitrogen or carbon dioxide diluent, are listed in Table 1. A safety margin must be maintained between the LOC and the normal working concentration in the system. Conservative control typically uses 2–4 percentage points below the LOC. That is, if the LOC of ethanol using nitrogen as a diluent is 10.5%, the control point would be 6.5–8.5%.

Although nitrogen or carbon dioxide are the most common inerting gases, steam is sometimes used. If so, it must be supplied at a rate sufficient to maintain the vessel temperature at 160°F or higher and care must be taken so that condensation by cooling does not draw in atmospheric air or collapse the vessel by implosion (*I*).

This temperature is experience-based and is considered sufficient to prevent condensation of the steam, which would hinder the protection

Safety

to the vessel from the steam purge.

Possible sources of purge or inerting gases include commercially available gases supplied from high-pressure cryogenic tanks or standard cylinders, or on-site air separation plants that remove oxygen from the air and recover nitrogen by liquefaction followed by absorption, chemical reaction, or membrane permeation. Cross-connections between the source of inerting gas and any other system should not be allowed. The gases from an enclosure or vessel being purged must be vented at a safe location. The purging or inerting gas should be introduced and exhausted so that effective mixing is ensured and the desired reduction in oxidant concentration is maintained throughout the system being protected. Multiple inlets and outlets are desirable to promote diluent distribution.

Several methods may be used to form and maintain a noncombustible atmosphere in an enclosure. These include batch modes for one-time use, such as purging equipment during a shutdown, and continuous inerting to assure safe conditions during normal operations. Each method will be described and illustrative sample design calculations will be made.

Syphon and vacuum purging

The common batch purging methods are syphon, vacuum, pressure, and sweep-through.

Syphon purging involves filling the vessel to be purged with liquid (*i.e.*, product or water) followed by introducing the purge gas, typically nitrogen, into the vapor space as the liquid is drained. The required purge-gas volume equals the volume of the vessel. The rate of application corresponds to the volumetric rate of liquid discharge. Syphon purging may not be appropriate if the liquid is above its flashpoint due to evaporation into the space.

Vacuum purging is one of the most common vessel inerting procedures provided that the vessel is designed for the maximum vacuum that can be developed by the source (*e.g.*, a vacuum

Gas or Vapor	LOC for N ₂ /Air O ₂ , vol. %	LOC for CO ₂ /Air O ₂ , vol. %
Cyclopropane	11.5	14
Natural gas (Pittsburgh)	12	14.5
<i>n</i> -butyl chloride	14	—
Acetone	11.5	14
Carbon disulfide	5	7.5
Ethanol	10.5	13
Hydrogen	5	5.2
Methyl ether	10.5	13
Methyl ethyl ketone	11	13.5

Source: Adapted from Ref. 3.

pump). The steps in a vacuum purge are: (1) drawing a vacuum on the vessel until the desired level is achieved; (2) relieving the vacuum with an inert gas such as nitrogen to atmospheric pressure; and (3) repeating Steps 1 and 2 until the desired oxygen concentration is reached. The amount of purge gas required depends upon the number of evacuations needed to develop the desired oxygen concentration.

The oxygen concentration x after k purge cycles (vacuum and relief) is described by Eq. 1, assuming that the pressure limits P_H and P_L are identical for each cycle (2):

$$x_k = x_o \left(\frac{P_L}{P_H} \right)^k \quad (1)$$

The quantity of inert gas required may be calculated by Eq. 2 for k cycles, based on the ideal gas law (2):

$$V_2 = k \frac{(P_H - P_L)V_1}{P_H} \quad (2)$$

Example 1: Determine the number of purges required and the total consumption of nitrogen to reduce the oxygen concentration in a 5,000 gal tank to 2% before introducing acetone. The temperature is 75°F and the vessel is initially charged with air at atmospheric pressure. A steam ejector is used that reaches 25 mm Hg absolute and the vacuum of each cycle is relieved with pure nitrogen until the pressure equals 1 atm.

Solution:

$$x_o = 0.21 \text{ lb-mole O}_2/\text{total moles}$$

The number of purge cycles k is determined by rewriting Eq. 1 to solve for k (2):

$$k = \frac{\ln \left(\frac{x_k}{x_o} \right)}{\ln \left(\frac{P_L}{P_H} \right)} \quad (3)$$

Substituting in (2):

$$k = \frac{\ln \left(\frac{0.02}{0.21} \right)}{\ln \left(\frac{25}{760} \right)} = 0.69 \text{ or } 1 \text{ cycle} \quad (4)$$

The total nitrogen used is determined from Eq. 2 (1):

$$V_2 = \left(\frac{P_2 - P_1}{P_2} \right) V_1 \quad (5)$$

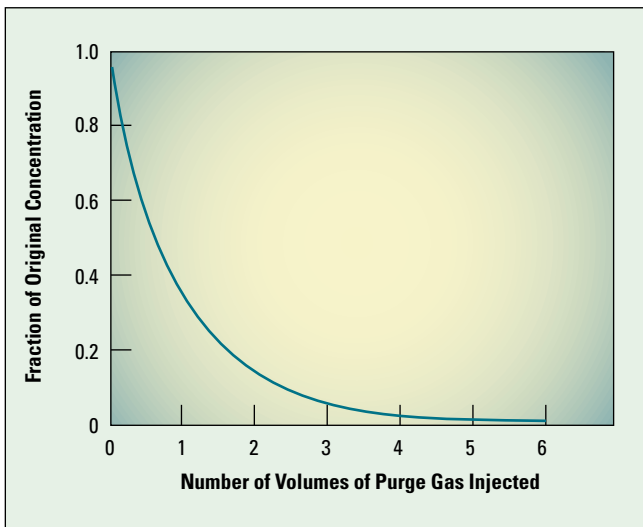
$$V_2 = \left(\frac{760 - 25}{760} \right) 5,000 \text{ gal} \times$$

$$1 \text{ ft}^3/7.48 \text{ gal} = 646.5 \text{ ft}^3 \quad (6)$$

or 1.66 lb-mol.

Pressure purging

Vessels may also be purged by adding inert gas under pressure and, after the gas has sufficiently diffused, venting to atmosphere. As with vacuum purging, more than one pressure



■ Figure 1. Dilution ratio for purging at atmospheric pressure, assuming complete mixing.

cycle may be necessary to reduce the concentration of oxygen to the desired level. The mathematical relationship that describes the pressure purging process is identical to Eq. 1, except that the initial concentration of oxygen in the vessel x_o is computed after the vessel is initially pressurized.

Example 2: Determine the number of nitrogen pressure purges required and the total consumption of nitrogen needed to reduce the oxygen concentration in the 5,000 gal tank described in Example 1 to 1% oxygen. The nitrogen is supplied to the vessel at 100 psig and 75°F.

Solution: Equation 1 is used to determine the number of purge cycles. The initial oxygen concentration in the vessel is calculated after the first pressurization using a simple pressure ratio where P_L is the starting or atmospheric pressure, whichever is appropriate.

$$x_o = 0.21 \left(\frac{P_L}{P_H} \right) \quad (7)$$

$$\begin{aligned} x_o &= 0.21 \left(\frac{14.7 \text{ psia}}{100 \text{ psia} + 14.7 \text{ psia}} \right) \\ &= 0.03 \text{ lb-mole} \end{aligned} \quad (8)$$

Applying Eq. 1, the number of cycles is:

$$k = \frac{\ln\left(\frac{0.01}{0.03}\right)}{\ln\left(\frac{14.7}{114.7}\right)} = 0.54 \quad (9)$$

Thus, the number of purge cycles = 1, and the nitrogen consumption is calculated as follows:

$$\begin{aligned} V_2 &= \left[\frac{(100 - 14.7) - 14.7}{(100 + 14.7)} \right] \times \\ & \quad 5,000 \text{ gal} = 582.8 \text{ ft}^3 \\ & \quad \text{or } 11.65 \text{ lb-mole} \end{aligned} \quad (10)$$

Note the increased consumption compared to vacuum purging (Example 1), because the vessel must be pressurized to 100 psig during each cycle.

Sweep-through purging

This method introduces a purge gas into a vessel at one opening and withdraws the mixed gas at another opening and vents it to the atmosphere (or an air-pollution control-device), thus, sweeping out residual flammable vapor. The quantity of purge gas required depends upon the physical arrangement. Sweep-through purging is commonly used when the vessel is not rated for pressure or vacuum. The purge gas is introduced and withdrawn at atmospheric pressure.

The relationship between the number of volumes of oxygen-free purge gas (e.g., nitrogen) that enters the vessel and the reduction in the vessel's oxygen concentration, assuming complete mixing, is shown in Figure 1 (3).

An oxygen material balance around the vessel is (2):

$$V \frac{dc}{dt} = C_o Q - C Q \quad (11)$$

Equation 11 can be rearranged and integrated, assuming $C_o = 0$ (2):

$$Q = \int_0^t dt = V \int_{C_1}^{C_2} \frac{dc}{c} \quad (12)$$

Integrating Eq. 12 yields Eq. 13, the volumetric flow rate required to reduce the vessel's oxygen concentration from C_1 (initial condition) to C_2 (2):

$$Qt = V \ln \left(\frac{C_1}{C_2} \right) \quad (13)$$

Equation 13 assumes perfect mixing. Since this is not normally the case in actual practice, a correction factor K is used. Table 2 lists values of K for certain conditions. Since little data exist on defining the degree of mixing, conservatism recommends a value of K no greater than 0.25. Equation 13 then becomes (3):

$$Qt = \frac{V}{K} \ln \left(\frac{C_1}{C_2} \right) \quad (14)$$

Example 3: A 20,000 gal storage vessel that contains 100% air must be inerted with nitrogen until the oxygen concentration is 2% by volume. How much nitrogen must be swept through? Assume $K = 0.25$.

$$\begin{aligned} Qt &= \frac{20,000 \text{ gal}}{0.25} \ln \left(\frac{0.21}{0.02} \right) \times \frac{1 \text{ ft}^3}{7.48 \text{ gal}} \\ &= 25,148 \text{ ft}^3 \text{ N}_2 \text{ or } 9.41 \text{ air changes} \end{aligned} \quad (15)$$

Table 2. Mixing efficiencies for selected ventilation arrangements.

Source of Supply	Efficiency, <i>K</i>	
	Single Exhaust Opening	Multiple Exhaust Openings
Infiltration through cracks, open doors, or windows	0.2	0.3–0.4
Powered air supply through:		
• Grills and registers	0.3	0.5
• Diffusers	0.5	0.7
• Perforated ceilings	0.8	0.9
• Pipes and ducts	0.25*	0.25*

* Conservative recommendation based on NFPA 69 (3).

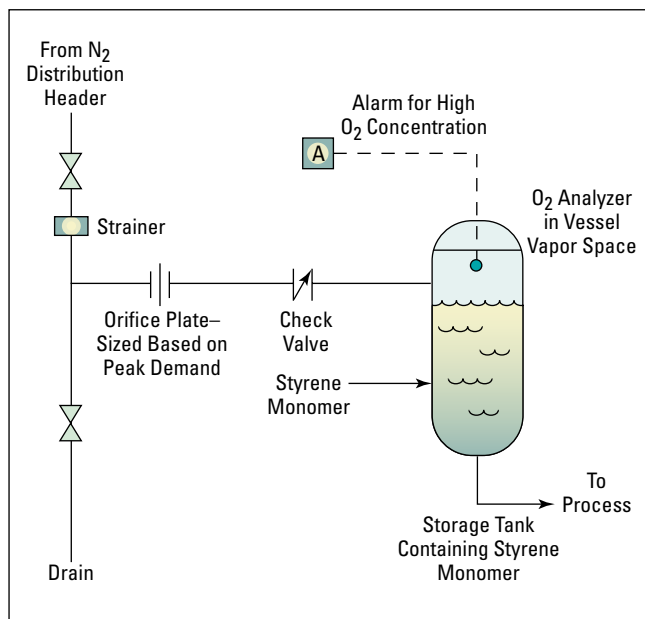
Inerting

Continuous inerting methods are fixed- and variable-rate (or demand). *Fixed-rate application* involves continuous feeding of inert gas into an enclosure (e.g., vessel) at a constant rate and the corresponding release of a mixture of inert gas and flammable vapor that has been picked up in the vessel's headspace. To ensure that the vessel is completely protected, the rate must be sufficiently to satisfy peak-demand requirements.

The peak demand for continuous inerting is typically controlled by the maximum liquid withdrawal rate coupled with potential temperature changes. For a vessel containing a flammable liquid, the inert gas demand based on liquid withdrawal is the capacity of the largest pump used to withdraw liquid or the maximum possible gravity outflow rate, whichever is greater.

The maximum demand from a temperature change will occur in outdoor tanks operating at or near atmospheric pressure as a result of the sudden cooling from a summer thunderstorm. The rate of inert gas necessary to prevent the vessel's pressure from falling significantly below atmospheric pressure is determined as follows: (1) for tanks over 800,000 gallons capacity, 2 ft³/h of inert gas per square foot of tank surface (shell and roof); and (2) for smaller tanks, 1 ft³/h of gas per 40 gal of tank capacity. Optionally, an inert gas rate corre-

Figure 2. One method of flow control for a fixed-rate application.



sponding to the mean rate of change of the vapor-space temperature of 100°F/h may be used. The rates corresponding to a temperature change and liquid withdrawal must be added together to determine the peak rate. These numbers are experience-based and are found in Refs. 1 and 3.

Variable rate or demand inerting involves feeding inert gas into the vessel at a rate that is a function of demand. Demand is based on maintaining a pressure within the vessel that is slightly above that of the surrounding atmosphere (e.g., ~1 in. H₂O).

Variable application offers an obvious advantage over continuous in that inert gas is supplied only when it is actually needed, thereby reducing: (1) the total quantity of inert gas required; (2) product loss; and (3) disposal problems. A disadvantage is the dependence upon flow control devices actuated by very low pressure-differentials that are sometimes difficult to maintain.

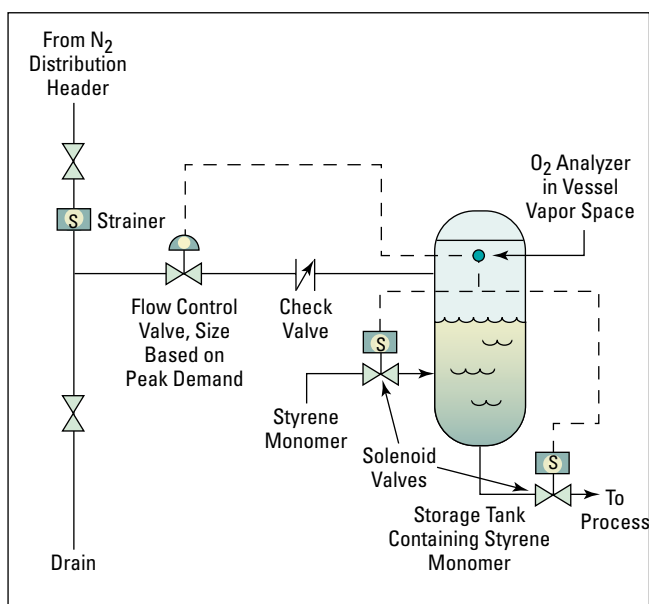
For variable-rate application, an inerting system is required to maintain such an atmosphere in the vapor space above the liquid. Ideally, this system should include an automatic inert-gas addition valve or backpres-

sure regulator to control the oxygen concentration below the LOC. The control system should feature an analyzer to continuously monitor the oxygen concentration and allow inert gas to enter the space to maintain the oxygen concentration at safe levels with a reasonable margin of safety. An increase in the concentration above the set point should initiate an alarm and shut down the operation. Where the oxygen concentration cannot be continuously monitored, it should be designed to operate at no more than 60% of the LOC and checked on a regularly scheduled basis.

Figure 2 shows one simple method of flow control that can be used with a continuous introduction of purge gas. Figure 3 depicts a method that can be used with variable rate application.

Practical considerations

Operation of a system with an oxygen concentration low enough to prevent a deflagration does not necessarily mean that incipient fires are absolutely prevented. Smoldering can occur in fibrous materials at low oxidant concentrations, later resulting in a fire or explosion when ex-



■ Figure 3. Typical setup for flow control in a variable-rate application.

posed to a higher concentration. The physical and chemical properties of the flammable or combustible materials involved will govern the type and required purity of the purge gas needed.

The following factors should be considered in the design of any system to reduce the oxidant concentrations to safe levels:

- Required reduction in oxidant concentration;
- Variations in the process, process temperature and pressure, and materials being processed;
- Purge gas supply source and equipment installation;
- Compatibility of the purge gas with the process;

- Operating controls;
- Maintenance, inspection, and testing;
- Leakage of purge gas to surrounding areas; and
- Need for breathing apparatus by personnel.

The minimum achievable oxygen concentration will depend upon the purity of the inert gas, the vessel's gas-tight integrity, and the presence of back diffusion from the exhaust line. To maintain a given oxygen concentration, it may be necessary to continue to supply inert gas even after attainment of the desired oxygen level.

Flowing inert gas will remove heat from the vessel (*e.g.*, a heated reactor or oven). Care should be taken that this heat removal will not

upset a reaction or cause exhaust system damage. The flowing gas will also carry volatile materials (solvents) from the vessel. Consider what these materials will do in the exhaust system. Traps, filters, or absorbers may be necessary to treat the exhaust.

Finally, there can be no doubt that without nitrogen (or other inert gas) purging and inerting, many more people would be injured or killed by fire and explosion. Nevertheless, we have paid a heavy price for the benefits of nitrogen. Many employees have been asphyxiated by it. During the period from 1960 to 1978, one group of companies reported that 13 employees were killed by fire or explosion, 13 by toxic or corrosive chemicals, and 7 by nitrogen.

If a person enters an atmosphere of nitrogen, he or she can lose consciousness without any warning symptoms in as little as 20 s. Death can follow in 3–4 min. A person falls as if struck by a blow on the head. In one incident in which the author was involved, a viewing hatch in a vessel containing vegetable oil was removed, but the nitrogen was kept flowing to protect the material from oxidation. A supervisor did not ask for an entry permit as he intended only to peek in. Fortunately, someone noticed that he was no longer moving and he was rescued in time. Whenever nitrogen (or other asphyxiating gas) is used for inerting or purging, employee training, strict adherence to a confined space entry procedure and permit system, and vessel labeling are a must.

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Nomenclature

C	= concentration of oxygen in the vessel
C_o	= inlet oxygen concentration (0, when inerting with nitrogen)
k	= number of purge cycles
K	= correction factor in Eq. 14 for degree of mixing
P_H, P_2	= initial high or atmospheric pressure
P_L, P_1	= initial vacuum pressure
V	= vessel volume
Q	= volumetric flow rate of inerting gas
t	= time
V_1	= total volume of tank or enclosure
V_2	= volume of inert gas required, measured at P_H
x_k	= final oxygen concentration after k purge cycles
x_o	= initial oxygen concentration under vacuum

Literature Cited

1. "Inerting and Purging of Tanks, Process Vessels, and Equipment," Factory Mutual Loss Prevention Data Sheet 7-59, Factory Mutual Engineering Corp., Norwood, MA (1977).
2. Crowl, D. A., and J. F. Louvar, "Chemical Process Safety — Fundamentals With Applications," Prentice Hall, Upper Saddle River, NJ, pp. 195–200 (1990).
3. "Standard on Explosion Prevention Systems," NFPA 69, National Fire Prevention Association, Quincy, MA (1997 edition).

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