

# **PRECAUTIONS AND SAFE PRACTICES**

**for**

## **Handling Zeolite Molecular Sieve Adsorbents in Process Units**



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## SECTION I

# THE NATURE OF ADSORBENTS AND THEIR OPERATION

### UNDERSTANDING ADSORBENTS

In order to handle, start up, and operate adsorbents in a safe and appropriate way, it is important to have a basic understanding of their nature and their properties.

The adsorbents discussed in this document are typically zeolite molecular sieves combined with other materials, both inert and active. Zeolite molecular sieves are aluminosilicates with a well-defined crystalline structure. This crystalline structure has channels and cavities which offer an enormous internal surface area (hundreds of square meters/gram). Entry into its rigid cavities, where molecules are adsorbed, is through the precisely dimensioned openings, or pores. Many types of adsorbent crystals can be manufactured with a number of different pore sizes.

Due to the nature of this internal surface area, the molecular sieve adsorbent will attract and collect molecules from the gas or liquid to which it is exposed. This is referred to as **adsorption**. Not all molecules are attracted by the surface to the same degree. For those that are the right size to enter, the attractive forces diminish with diminishing polarity of the adsorbed molecule. This results in a **selectivity** of some molecules over others. Water and ammonia are the most polar molecules and will be preferentially adsorbed over molecules such as CO<sub>2</sub>, H<sub>2</sub>S, paraffins, olefins, etc.

A more polar molecule can actually **desorb** or **displace** a less polar molecule which had been previously adsorbed. When a more polar molecule is adsorbed, heat is released causing the temperature to rise and to further desorb adsorbed species. This crucial characteristic significantly impacts the safe handling of adsorbents.

When unloading or handling unloaded adsorbents, there is potential for adsorbed materials to be displaced or desorbed. This can happen when adsorbents are exposed to moisture either intentionally (being purposely wetted to abate dust or to deactivate the adsorbent) or unintentionally (from ambient air, rain, or snow). Any molecules which have been adsorbed during the cooling and/or conditioning step can be released to the atmosphere as they

are displaced by water. Even very small amounts of certain materials can accumulate to dangerous concentrations in enclosed environments. A particular example of this hazard is in “sour” applications where trace levels of H<sub>2</sub>S in the feed stream can accumulate to dangerous levels in the molecular sieve. *H<sub>2</sub>S can be deadly even when present in very low concentrations.*

### THEORY OF OPERATION

Adsorbents are used to dry, purify and separate a wide variety of gases and liquids. They do so by:

- ▶ Strongly attracting certain types of molecules, and
- ▶ Screening out molecules larger than their pores.

Polar molecules such as water and methanol are *strongly* attracted by adsorbents. In contrast, nonpolar molecules, such as methane and ethane, are much more *weakly* attracted. When a mixture of water (polar) and methane (nonpolar) is passed over adsorbents, the water is adsorbed, even though both are small enough to pass easily through the pores.

Water can also be removed from a strongly attracted fluid such as methanol by using adsorbents with pore openings smaller than the methanol molecule, but larger than the water molecule.

When molecules are adsorbed, heat is released. In most systems, the temperature of the process stream rises only a few degrees. However, when a high concentration (Greater than 0.5 volume %) of a strongly attracted molecule is adsorbed, the temperature can rise more than 100°F.

At low temperatures (less than 150°F), adsorbents have a relatively high capacity for adsorbed species. At high temperatures (300-650°F), the capacity is much lower. When adsorbents become saturated, the adsorbed molecules can be driven off by heating the adsorbents to 300-650°F with a gas stream. This operation is called **regeneration**. After regeneration, the adsorbent is cooled down to the normal process temperature with a low

temperature stream before returning to service. In some applications, adsorbents are regenerated by reducing pressure, rather than heating. Continuous operation is maintained by cycling two or more vessels between adsorption and regeneration.

Follow the precautions in **Section II** whenever loading fresh adsorbent into a vessel. During the start-up and operation of an adsorption system, follow the precautions covered in **Section III**. In process units, adsorbents are exposed to liquids or gases from which they pick up various materials, some of which can be hazardous (i.e. H<sub>2</sub>S, NH<sub>3</sub>, CO<sub>2</sub>, various hydrocarbon and sulfur-containing compounds). Before opening a used adsorbent vessel to the atmosphere, install appropriate blind flanges, and remove any potentially hazardous materials, as detailed in **Section IV**.

For general technical questions, contact your UOP representative.

**For 24-hour Product Safety Emergency Assistance contact:**

<b>In USA:</b>	<b>UOP</b>	<b>847.391.2123</b>
	<b>CHEMTREC</b>	<b>800.424.9300</b>
<b>In Canada:</b>	<b>CANUTEC</b>	<b>613.996.6666</b>
<b>In Europe:</b>	<b>BIG</b>	<b>32 14 58.45.45</b>
<b>In other countries:</b>	<b>CHEMTREC</b>	<b>202.483.7616</b>

## SECTION II

# LOADING FRESH ADSORBENT

*Read and understand this section thoroughly before loading fresh adsorbent. Observe all shipping container precaution labels. Make sure that the product Material Safety Data Sheet is available at the work site and reviewed prior to handling the product.*

### PREPARATION

Adsorbents are typically supplied as pellets, beads or granules. In their fresh, unused state, adsorbents present minimal risk when appropriate protective equipment is used. Appropriate protective equipment includes gloves, long sleeve shirts and pants or coveralls to protect against skin exposure; safety glasses, goggles or face shield to protect the eyes; and dust masks or respirators if it is expected that dust will be generated. When exposed to water, the heat of adsorption can cause adsorbents to get quite hot. *Care should be taken to avoid contact with moist skin, mucous membranes and eyes.*

*Note: Some special adsorbents may contain toxic or flammable materials. These adsorbents will have special precautions on the labels of the shipping containers and in the Material Safety Data Sheets. In such cases, always follow the special precautions provided by UOP.*

If flammable vapors such as hydrocarbons are present in the loading area, grounding is especially important to avoid discharge of static electricity that could cause an explosion or fire. Even conductive footwear and conductive walking surfaces may not prevent electrostatic buildup on the human body. For example, removal of outer garments in low humidity conditions can generate sparks.

### CONFINED SPACES

If entry to the vessel is required, confined space safe work practices must be followed. Many countries have specific regulations on confined space work. Personnel should review these regulations and ensure that they are followed. Among the precautions and practices that are called for are:

- ▶ Removing hazardous materials from the vessel prior to entry
- ▶ Isolating the confined space from sources of hazardous materials or energy by installing blind flanges to inlet and outlet nozzles and decoupling pumps and instrumentation
- ▶ Providing adequate ventilation to prevent accumulation of flammable materials, combustible dusts, toxic contaminants or an environment that is oxygen deficient or excessive
- ▶ Testing for oxygen, flammable gas, and suspected toxic materials prior to entering the vessel
- ▶ Using safety attendants outside the vessel to monitor
- ▶ Communicating with personnel in the confined space
- ▶ Having notification and response procedures in place for emergency situations such as injury or loss of consciousness to personnel within the confined space
- ▶ Equipping personnel entering the vessel with the appropriate safety equipment, this may require use of safety harnesses and/or self-contained breathing apparatus (SCBA)
- ▶ Training personnel authorized to work as safety attendants, rescue personnel or persons entering the confined space
- ▶ Conducting pre-job discussions with personnel involved with the work about the potential hazards in the confined space

### LOADING

Adsorbents are normally shipped in 55-gallon sealed drums or 35-50 cubic-foot bags. A vacuum may exist inside the removable-top drums. First, loosen the vent screw on the lid and break the vacuum. Then remove the top.

Take care when moving adsorbent containers. Proper lifting techniques and equipment should be used. Full drums weigh more than 300 pounds; full bags more than 2,100

pounds. During loading, the containers must normally be lifted to the top of the vessel. Sometimes, several drums are dumped into a large hopper and this is lifted. This activity requires that standard elevated work practices be used, such as isolating the area under the lifting path and using ropes or posting signs and keeping personnel clear of the area.

Occasionally, pneumatic trucks are used to transfer adsorbent into the vessel through a hose. Keep the hose outlet pointed away from personnel. Static electricity can build up during transfer operations. Therefore make sure that the transfer and receiving system are electrically grounded to help dissipate any static charge.

## SECTION III

### START-UP AND OPERATION OF ADSORPTION SYSTEMS

*Read and understand your equipment manufacturer's operating instructions, your employer's safety practices and this section before starting up and operating an adsorption system.*

#### MAJOR OPERATING HAZARDS AND PRECAUTIONS

During start-up and operation of any adsorption system, it is critical to stay within the pressure and temperature limits supplied by the equipment manufacturers. Excessive temperatures can cause equipment failure and result in a life-threatening fire or explosion.

Three things can cause excessive temperatures in an adsorbent system.

1. Introducing a flammable or reactive fluid into a vessel containing air.
2. Introducing a high concentration of a reactive, strongly adsorbed material into fresh or regenerated adsorbent.

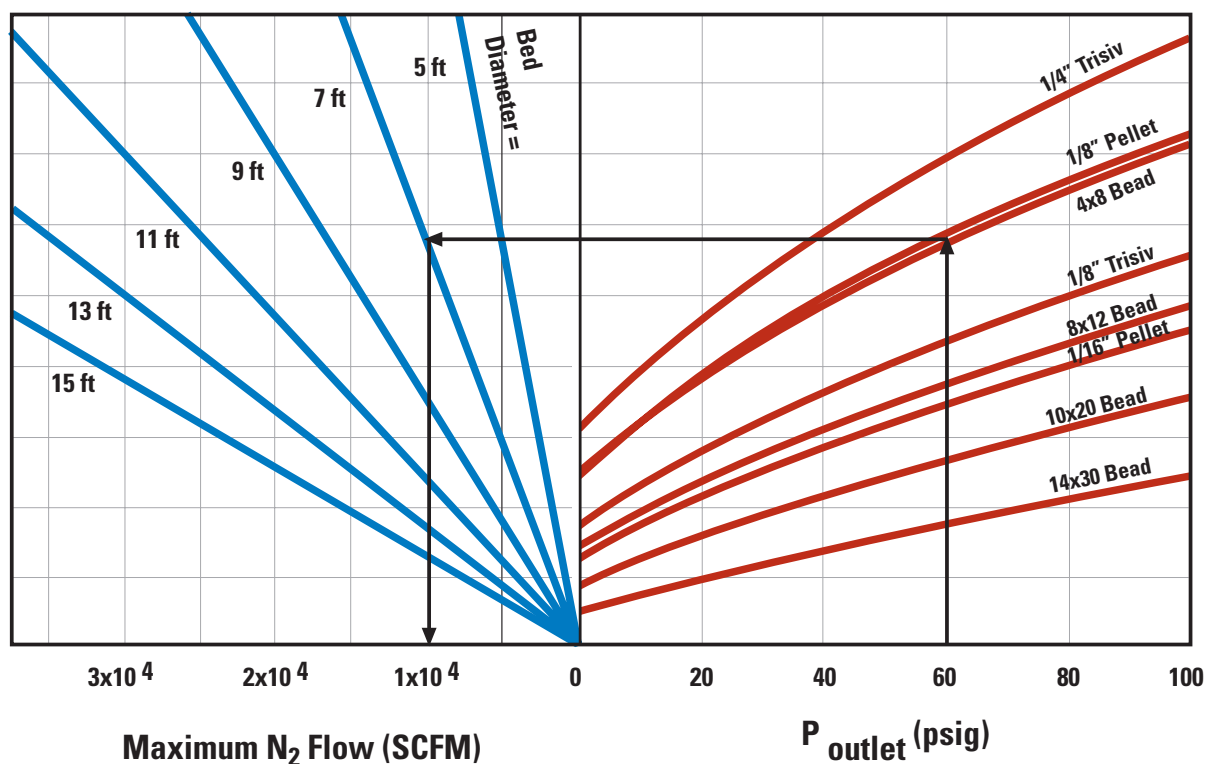
3. Using a highly reactive fluid to heat or cool the bed. (Examples of reactive fluids would include ethylene, propylene and other olefins.)

#### INITIAL EXPOSURE TO A PROCESS STREAM

Guidelines to avoid temperature excursions:

1. After loading the air must be removed using a nitrogen purge before introducing the process stream into an adsorbent vessel. The amount of available nitrogen is typically inadequate to provide the gas velocities needed for effective flow distribution within the adsorbent bed. The repetitive pressurization and depressurization of the bed with nitrogen is an alternative way to purge the bed void volume. The nitrogen purge gas should flow down (top to bottom) through the vessel to preclude lifting the bed and support material due to high velocities. If downward flow is not possible, see **Figure III-1** for guidance in setting the flow rate or contact your

**FIGURE III-1**



UOP representative for specific recommendations for your unit. Continue purging until the oxygen content of the outlet mixture is less than 0.5 volume %.

2. Do not introduce reactive, strongly adsorbed fluids (such as ethylene, propylene and other olefins) into a fresh or regenerated bed without proper preparation. The heat of adsorption can increase the temperature sufficiently to start a self-sustaining, heat-releasing reaction. If unchecked, the temperatures can increase enough to rupture the vessel and create a life-threatening situation. For these systems, always use the special procedures provided by the adsorbent supplier in the process design. These procedures involve exposing a fresh or regenerated bed to an inert stream such as nitrogen to which a low concentration of the reactive fluid is added. The concentration of the reactive fluid is slowly increased until the outlet temperature drops back down and approaches the inlet temperature. If the process stream is **ethylene**, refer to the UOP Bulletin, entitled, *Safe Practices for use of Molecular Sieves in Ethylene Purification*.
3. Do not use extremely reactive fluids to heat or cool a bed. The elevated temperatures during regeneration could start a hazardous heat-releasing reaction.
4. Never change the regeneration gas or feed stream specified in the process design to another fluid without first consulting with your UOP representative. Such a change could create the hazardous conditions discussed in items 2 and 3 above.

Complete the introduction of the reactive fluid and bring the vessel up to normal operating pressure. Establish normal flow and closely monitor any intermediate and outlet temperature indicators until each bed has completed one adsorption step. If a readily adsorbed component in the process stream is present at more than 0.5 volume %, both temperatures will rise. However, if either temperature rises more than 150°F above the inlet temperature, **stop the process immediately**. This condition indicates that a heat-releasing reaction may have started.

## HEAT RELEASING REACTION

In the event of a heat releasing reaction, stop it by taking the following steps:

1. Close the valves on the lines leading into and out of the vessels to stop the flow of fluid into the adsorbent.
2. Open the valve on the flare line to vent the bulk of the reactants from the vessel to the flare for combustion.
3. Purge with nitrogen to the flare to remove the remaining reactants and cool the bed.
4. Contact UOP for assistance.

When one adsorption step is completed on each bed without excess temperature rise, the system is ready for normal processing.

## REGENERATION

Once the adsorbent bed is saturated, it is no longer effective and must be regenerated. This is normally done by passing a gas stream over the adsorbent at 300-650°F. The actual temperature is set by the design of the unit.

Because of heat losses, the bed outlet temperature should always be lower than the temperature of the inlet during heating. If the outlet temperature starts to increase above the inlet temperature, **stop the heating step immediately**. This condition indicates that a heat-releasing reaction may be occurring. Close the inlet and outlet valves to the vessel being heated, depressurize it to the flare, and purge it with nitrogen. Contact UOP for assistance.

During regeneration, the adsorbent bed may be as hot as 650°F. Always check the cooling outlet temperature after regeneration to ensure the bed is cool before exposing it to the feed stream. Failure to cool the bed before returning it to service could result in **downstream equipment damage** or possibly initiate a **heat-releasing reaction** in the case of reactive feed streams. Both consequences can create a safety hazard. The adsorbent has high heat capacity and low thermal conductivity. It can stay hot for weeks if a shutdown occurs before the bed has been completely cooled. For systems with automatic switching, always check following a shutdown to ensure that a hot bed is not switched from cooling prematurely.

## SECTION IV

# DEACTIVATION OF SPENT ADSORBENT FOR UNLOADING

*Read and understand this section thoroughly before opening a used adsorbent system to the atmosphere.*

Adsorbents exposed to a gas or liquid stream will adsorb various compounds from the process stream. Trace compounds can be concentrated on the adsorbent during its use. They may be toxic, flammable, or explosive.

Exposure of spent adsorbent to ambient air may generate heat and desorb toxic materials creating a hazardous environment. While the adsorbent is regenerated prior to unloading, there are various reasons why there may be residual adsorbed material remaining on the adsorbent. Some potential causes would include:

- ▶ Bed channeling
- ▶ Adsorbent agglomeration
- ▶ Coke formation due to reactive species
- ▶ Liquid carryover
- ▶ Inadvertent contamination after regeneration
- ▶ Incomplete regeneration

Adsorbed species can be displaced by atmospheric moisture or liquid water resulting in heat release. If reactive species such as olefins or other hydrocarbons are present, an uncontrolled reaction could occur resulting in fire and/or release of toxic materials. Therefore, it is important to remove any potentially hazardous adsorbed materials and render the adsorbent incapable of picking up more hazardous material. This procedure is referred to as deactivation. Several **deactivation** methods are described in this brochure

Deactivation should be done before the vessel, piping or valve bodies are opened to the atmosphere, and before any attempt is made to remove adsorbent or other materials from the vessel.

### INERT GAS (NITROGEN) PURGING

Nitrogen will not effectively displace materials concentrated within the internal cavities of the adsorbent, except oxygen. Nitrogen purging is done to remove the residual process gas from the void volume of used adsorbent beds.

1. Regenerate the bed: heat and cool it with a gas. If the gas normally used to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas. Your UOP molecular sieve representative will help select an alternate gas and determine the proper flow rate, temperature and pressure.
2. Completely cool the bed with gas, then isolate the vessel and depressurize it. Install blind flanges on all inlet and outlet lines except the flare line. Connect a nitrogen source at the opposite end of the vessel.
3. The amount of available nitrogen is typically inadequate to provide the gas velocities needed for effective flow distribution within the adsorbent bed. The repetitive pressurization and depressurization of the bed with nitrogen is an alternative way to purge the bed void volume. The nitrogen purge gas should be downflow (top to bottom) through the vessel to preclude lifting the bed and support material due to high velocities. If downward flow is not possible, see Figure III-1 for guidance in setting the flow rate or contact your UOP representative for specific recommendations for your unit.
4. Continue to pressurize and depressurize the vessel to the flare with nitrogen purge gas until the outlet mixture contains less than 10% of the lower explosive limit (LEL) for any flammables. The lower explosive limit in air of several common fluids is shown in Table 1. Purge until free of toxic materials. Then, proceed to one of the alternative deactivation methods described below.

**TABLE 1**  
**LOWER EXPLOSIVE LIMIT\* OF SOME COMMON FLUIDS**

Fluid	Volume % in Air	Fluid	Volume % in Air
Methane	5.0	N-Butane	1.6
Ethane	2.9	Hydrogen	4.0
Propane	2.1	Benzene	1.4

\*Lowest concentration of vapor in air that is capable of propagating a flame through a mixture.

## ADSORBENT DEACTIVATION

There are several options for deactivating the spent adsorbent. All involve the saturation of the adsorbent with a displacement fluid.

### WATER FLOOD

Water is the preferred material for deactivating the adsorbent. Unlike nitrogen, water is more strongly attracted to the adsorbent than nearly any of the potentially hazardous materials which may be present. The simplest method is to fill the bed from the bottom up with liquid water, after preparing the adsorbent bed.

*Note: Water flood should not be done without doing Step 1 (regeneration) below.*

*Important: This procedure renders the adsorbent unfit for re-use. Furthermore, water flooding of an internally lined vessel may cause damage to the liner. Once wet, the liner is difficult to dry out.*

1. Regenerate the bed: heat and cool it with a gas. If the gas normally used to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas. Your UOP molecular sieve representative will help select an alternate gas and determine the proper flow rate, temperature and pressure.
2. Completely cool the bed with gas, then isolate the vessel and depressurize it. Install blind flanges on all inlet and outlet lines except for the flare line at the top of the vessel. Attach a water line to the bottom of the vessel. Be sure that the water is obtained from a safe and appropriate source and is compatible with the equipment's materials of construction.
3. As the vessel fills with water, displaced gases are vented to the flare. To avoid overfilling the vessel and flooding the flare line, monitor the water filling rate and the liquid level. This can be easily done with a properly calibrated DP indicator if one is available. Alternatively, calibrated pressure gauges can be temporarily mounted at the top and bottom of the vessel. (Note: 14.7 psi is approximately equal to 34 ft of water.)

4. After the vessel is completely filled, shut off the line to flare and open a vent port or vent valve to break the vacuum on the vessel during draining. Then, drain the water to an approved disposal location.
5. Pressurize the vessel with nitrogen and establish sufficient flow to allow gas monitoring to ensure the outlet of the vessel is below 10% of the LEL and is free of toxic materials. If the nitrogen leaving the vessel is not less than 10% LEL or contains toxic materials, repeat the water flood procedure and gas monitoring until these results are achieved.

### WATER VAPOR DEACTIVATION

There are several drawbacks to the water flood procedure. Internal vessel liners (if present) can be damaged, the adsorbent cannot be reused, the waste water must be expelled, residual liquid water must be completely removed in water-sensitive applications (i.e. cryogenic or turbo-expander facilities).

The following procedure avoids most of the problems associated with the use of liquid water. However, it requires the use of nitrogen as a carrier fluid for vaporized and entrained liquid water.

1. Regenerate the bed: heat and cool it with a gas. If the gas normally used to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas. Your UOP molecular sieve representative will help select an alternate gas and determine the proper flow rate, temperature and pressure.
2. Completely cool the bed with gas, then isolate the vessel and depressurize it. Install blind flanges on all inlet and outlet lines except the flare line. Connect a nitrogen source at the opposite end of the vessel.
3. Start a flow of nitrogen at the highest rate possible. Introduce water into the nitrogen purge (carrier) gas by either a) the injection of steam through a restricting orifice, or b) the injection of liquid water with a metering pump and atomizer. Nitrogen saturated with water vapor will generally not contain enough water to allow for complete deactivation of the adsorbent bed within an acceptable time frame. Most

of the required water will be entrained as small droplets within the nitrogen purge gas. For purposes of estimating the amount of water, nitrogen and time required to accomplish this water vapor deactivation, the following nomographs, Figures IV-1a and b, may be helpful. Please contact your UOP representative with any questions regarding this procedure.

4. After sufficient water has been introduced into the bed to accomplish deactivation, monitor for LEL and toxicity, as described above in Inert Gas (Nitrogen) Purging Step 4. Repeat the process as necessary until this standard (less 10% LEL and no toxicity) has been met.

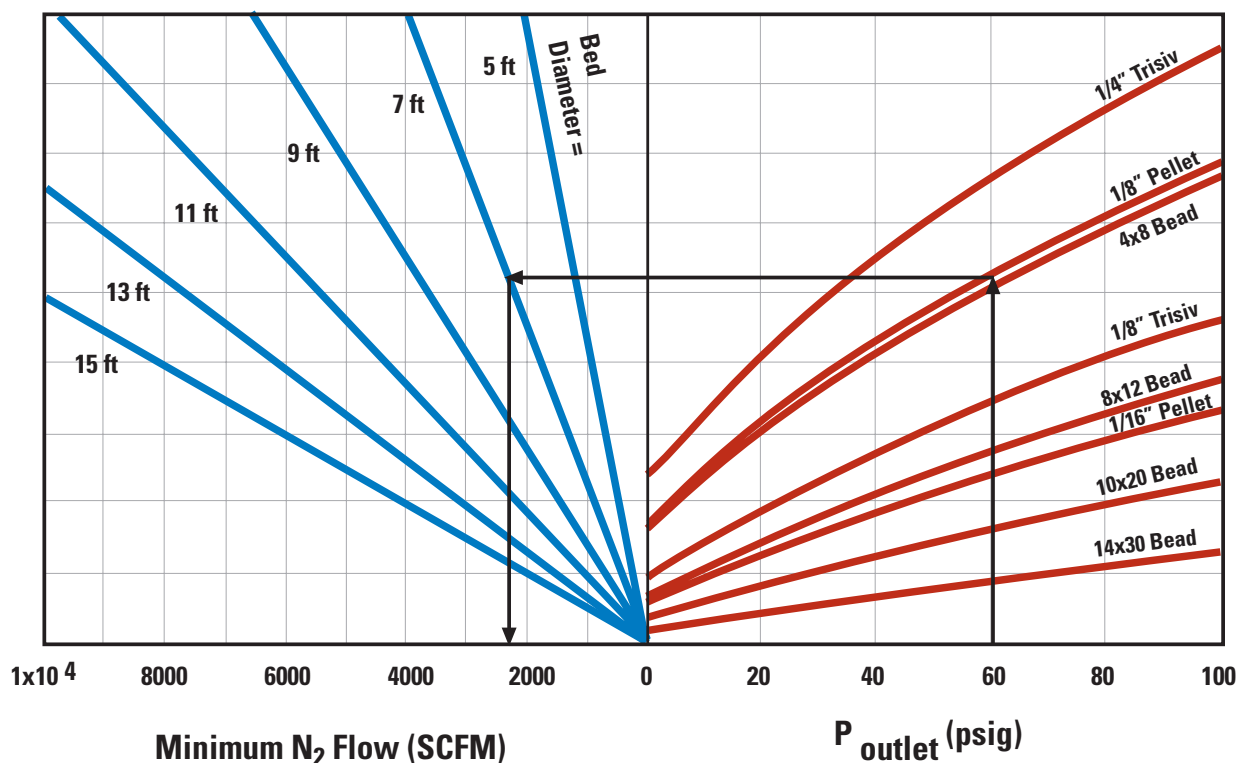
### STEAM DEACTIVATION

The use of saturated steam achieves deactivation and avoids most of the problems associated with the generation of liquid water. To prevent condensation of liquid water in the bed, it must first be preheated with regeneration gas, or hot nitrogen purge gas, to a temperature above that of the saturated steam.

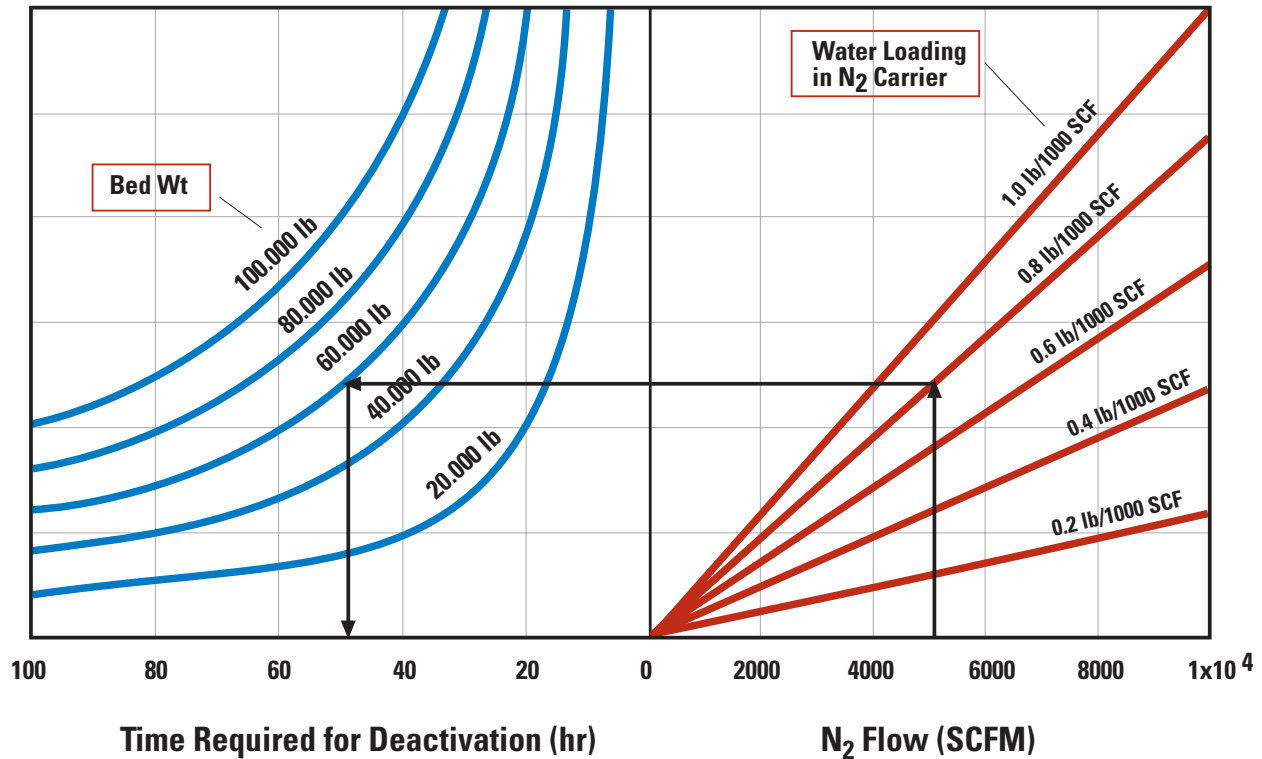
1. Regenerate the bed, but do not cool it. If the gas normally used to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas. Your UOP molecular sieve representative will help select an alternate gas and determine the proper flow rate, temperature and pressure.
2. Isolate the vessel and depressurize it. Install blind flanges on all inlet and outlet lines except the flare line. Connect a steam source at the opposite end of the vessel.
3. Steam should be directed downflow through the bed and out the bottom to the flare. Condensate can be drained from the flare knockout drum. Test for contaminants at the bottom of the bed. Maximize the steam flow within the DP limits of the adsorber.

The minimum theoretical amount of steam required for deactivation may be estimated based upon the adsorbent capacity for water (a nominal value of 20 pounds of water per 100 pounds of adsorbent).

FIGURE IV-1A  
MINIMUM FLOW REQUIRED FOR GOOD DISTRIBUTION



**FIGURE IV-1B**  
**DEACTIVATION TIME VS N<sub>2</sub>/WATER FLOW**



- Each 100 pounds of adsorbent requires 20 pounds of water for deactivation.
- Each 1,000 SCF (ft<sup>3</sup>) of nitrogen carrier gas will hold one (1) pound of water.

However, the amount of steam is ultimately determined by the LEL and toxicity monitoring to be done following completion of the steam flood, as described above for the other deactivation methods.

Once low contamination exit levels are confirmed, continue to steam for at least another 2 hours. After this, purge and cool the bed with nitrogen to ~ 50°C (120°F) while continuing to direct the effluent to the flare.

### **CO<sub>2</sub> (CARBON DIOXIDE) DEACTIVATION**

A deactivation procedure which uses carbon dioxide (CO<sub>2</sub>) as a displacement fluid to effectively remove potentially hazardous materials from the adsorbent bed eliminates all the problems associated with liquid water, and readily allows for the reuse of adsorbent.

1. Regenerate the bed: heat and cool it with a gas. If the gas normally used to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas.

Your UOP molecular sieve representative will help select an alternate gas and determine the proper flow rate, temperature and pressure.

2. Completely cool the bed with gas, then isolate the vessel and depressurize it. Install blind flanges on all inlet and outlet lines except the flare line.
3. There are a number of ways by which the CO<sub>2</sub> may be introduced into the adsorbent bed.
  - a. The CO<sub>2</sub> may be blended with nitrogen purge gas, as a carrier.
  - b. Pure CO<sub>2</sub>, is generally available as liquid, and requires vaporization into the adsorbent bed or the nitrogen purge transfer line.
  - c. Alternatively, the product from an inert gas (combustion type) generator, will contain a nominal level (10%) of CO<sub>2</sub> with the balance (90%) nitrogen.

- d. In a few instances, dry ice (solid CO<sub>2</sub>) can be loaded on top of the adsorbent (under a nitrogen blanket to avoid exposing the adsorbent to the atmosphere). A flow of nitrogen purge carrier gas will then be used to disperse the vaporizing CO<sub>2</sub> over the adsorbent.
4. Approximately 20 pounds of CO<sub>2</sub> per 100 pounds of adsorbent are required to deactivate the adsorbent regardless of its means of delivery to the adsorber vessel. The determination of deactivation must be based upon the results from monitoring this purge gas from the bed, to ensure that it meets the standards of less than 10% LEL and no toxicity. If this standard is not achieved, further CO<sub>2</sub> displacement, by repetitive pressurization and depressurization, should be done. If, after repeating these steps, the LEL and toxicity standards still cannot be met, then another deactivation method should be used.

**Note:**

- ▶ *If H<sub>2</sub>S is present on the adsorbent, CO<sub>2</sub> will not desorb it. In such case, the H<sub>2</sub>S will remain in adsorbent pores until a more polar compound, like water, displaces it. If H<sub>2</sub>S is present, CO<sub>2</sub> is not recommended for deactivation.*
- ▶ *Inert gases such as CO<sub>2</sub> and nitrogen can concentrate in the pores of the adsorbent. Ambient moisture can desorb these gases allowing them to concentrate in enclosed spaces. The resulting atmosphere could be depleted in oxygen creating a personnel hazard.*
- ▶ *Do not allow personnel to enter confined spaces containing spent adsorbent without proper precautions.*

## KEY POINTS FOR DEACTIVATION OF ADSORBENT

1. Regenerate with gas. If the gas you normally use to regenerate the bed is unavailable or contains toxic components at hazardous levels, use nitrogen or an alternative nontoxic gas. Your UOP molecular sieve representative will help you select an alternate gas

and determine the proper flow rate, temperature and pressure.

2. Completely cool the bed with gas, then block-in the vessel and depressurize it to the flare. Install blind flanges on all vessel inlets and outlet lines except the flare line. Install a nitrogen tap at the opposite end of the vessel.
3. Select an appropriate deactivation fluid (in addition to nitrogen) to effectively displace potentially hazardous materials from the internal cavities of the adsorbent bed. Establish a detailed procedure for using this deactivation fluid.

**Note: Both nitrogen and CO<sub>2</sub>, described in the above procedures, presents a danger in and of themselves. Both gases are heavier than air, and both gases can lead to asphyxiation.**

4. Continue the deactivation procedure until the outlet gas from the adsorber vessel contains less than 10% of the lower explosive limit (LEL) and is free of toxic materials.
5. Particular care should be taken in applications where the process streams contain H<sub>2</sub>S, COS, mercaptans and other sulfur-based compounds – even if these compounds are only present in minute quantities. **Inhalation or exposure to these products can be fatal.**
6. Contact your UOP representative with any questions you may have regarding these procedures.

## SECTION V

# UNLOADING AND HANDLING OF SPENT ADSORBENT

*Read and understand this document thoroughly before unloading used adsorbent.*

### PREPARATION

It is important to maintain a nitrogen blanket on the vessel during unloading regardless of what technique of adsorbent deactivation and purging is used in preparing the adsorbent for unloading. *Always assume that some hazardous and toxic materials remain on the adsorbent. Do not enter the vessel while the adsorbent remains inside.* Hazardous materials may be released during unloading of spent adsorbent creating a toxic, flammable or reactive environment.

Be prepared for these situations. Limit access and contact with spent sieve, have fire-fighting equipment available and test the area around the unloaded sieve for flammable or suspected toxic materials to establish whether you have a safe environment. If monitoring or experience indicates the environment may contain toxic materials in excess of safe levels, use appropriate respiratory protection such as respirators or self-contained breathing apparatus.

*Note: UOP recommends that the spent adsorbent be deactivated prior to unloading. Spent material that has not been fully deactivated could present a safety hazard by still containing toxic gases despite having been regenerated prior to unloading. Furthermore, it could present a safety hazard by having the potential to release a significant amount of heat energy if it is wetted either with hose water, rain or snow.*

### UNLOADING

*Contact with used adsorbent should be avoided.* Workers unloading the used adsorbent should wear gloves, long sleeve shirts and pants or coveralls to protect against skin exposure; safety glasses, goggles or face shields to protect the eyes; and dust masks or respirators.

Normally, there is a stainless steel screen on top of the adsorbent bed. Inert balls lie on top of the screen. To make unloading easier, secure the screen to the top of the

vessel. This can be done without entering the vessel. Remove the loading port and secure the screen using a harpoon-type tool.

Next, open the dump port and allow the adsorbent to flow out. Once the adsorbent no longer flows out freely, remove the remaining portion using a rake, hoe, shovel or a vacuum truck. *Never enter the vessel to remove the remaining adsorbent.*

*If at any time during unloading, materials on the adsorbent start to burn or to react (as indicated by a large heat release), stop unloading.* Evacuate personnel from the area. Properly trained personnel with self-contained breathing apparatus can then return to stop the reaction or put out the fire. To stop the reaction, or put out the fire, use water from fire hoses to flood the adsorbent outside the vessel. Contact UOP for assistance.

*Note: Hazardous gases may be desorbed from the spent material as a result of the heat release or fire, or as a result of dousing the spent material with water.*

Once all the adsorbent has been removed, prepare the atmosphere in the vessel for safe entry. Remove the nitrogen purge line if used and attach an air mover so that air is expelled out of the bottom of the vessel below the bed support. The air will carry any remaining vapors out of the vessel and away from the workers. Such vapors may be released by adsorbent trapped under the bed support. Extra care should be exercised for internally insulated vessels. Hydrocarbon and toxic vapors may be slow to outgas from the insulation. Leave the air mover on at all times. *Never use a plant air supply to purge the vessel with air – a plant supply could become contaminated or be hooked up to another source by mistake.*

Once the actions have been completed to establish a safe environment for vessel entry, personnel entering the vessel must follow confined space work practices as outlined in **Section II**.

## DISPOSAL

Either dispose of spent adsorbent immediately or store it in a manner that will not impact the environment until disposal can be arranged. It is recommended that spent adsorbent be stored in containers such as drums or portable luggers. If containers are not feasible, spent adsorbent should be stored on an impermeable surface such as concrete, asphalt or plastic sheeting that is heat and chemical resistant. It is recommended that spent adsorbent be protected from rainfall to prevent the possibility of generating contaminated storm water runoff. If water flooding has been used during unloading operations, additional spill prevention and controls such as temporary dikes may be needed to contain free liquid that may be generated when the spent adsorbent is unloaded.

In its fresh unused state, adsorbent is considered non-hazardous for purposes of disposal. However the adsorbed material on the spent adsorbent may change the classification for purposes of disposal. Refer to national, state and local regulations to determine proper disposal of spent adsorbent. For any questions concerning disposal of used adsorbent, contact your local UOP representative.

In summary, adsorbent can be handled and operated safely if proper safety precautions are followed. If in doubt about the safety of your adsorbent adsorption system, please contact your local UOP representative.

## KEY POINTS

1. Prepare adsorbent using one of the recommended procedures.
2. Limit access to the area.
3. Unload from outside the vessel.
4. Should reaction occur in the adsorbent during unloading, stop unloading immediately.
5. Dispose of adsorbent immediately or store safely until such time as it is possible.

**IN CASE OF A MEDICAL OR FIRE  
EMERGENCY RELATED TO UOP  
ADSORBENTS, CALL +1-847-391-2123**



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