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## Question 1: What is your best practice for safe and quick decontamination of solid media beds?

### ABIGAIL SLATER (HollyFrontier)

Safe and quick decontamination of solid media beds requires a good working knowledge of the unit, the bed material, and the nature of the contamination. Media beds include feed filter-coalescer elements, reactors, guard bed reactors, chloride guard beds, sulfur guard beds, etc. The nature of the contamination may involve any number of biological, organic or inorganic materials which present hazards to handling the media after service.

Generally, the decontamination of solid media involves bringing Lower Explosive Limits (LEL), Hydrogen Sulfide (H<sub>2</sub>S), and benzene into acceptable ranges. Decontamination practices are also used to passivate pyrophoric or self-heating catalysts, which present safety hazards when exposed to oxygen.

One of the most common contamination issues is that of adsorbed benzene – a well-recognized toxic material. Benzene can sometimes absorb on the walls of the vessel or the media. A great deal of time can be lost in decontaminating solid media and the unit as a whole. For that reason, many solid media beds are often dumped or vacuumed out under an inert atmosphere. A hot hydrogen purge, followed by a hot nitrogen purge, is typically successful in liquid-freeing the solid media bed but not always successful in removing LELs and benzene. Hot hydrogen, or even fuel gas, can be used when nitrogen is not readily or easily available, this serves as a cost saver but must be followed by a hot nitrogen purge.

Another method to benzene free a vessel is steam out. However, many operators have found this approach to be inefficient, often requiring multiple steam out attempts, and hence, extra time to bring residual benzene levels down to within acceptable exposure limits. Steam out can also cause undesired results. For example, steaming out a chloride guard bed can potentially increase the corrosivity or even react violently with the media inside the vessel. Therefore, many have turned to using nitrogen and chemical cleaning to more quickly benzene free solid media beds and the unit in general.

An increasingly popular method to decontaminate LELs, H<sub>2</sub>S, and benzene has been to chemical clean. Some companies have produced proprietary organic chemicals which have proven to be successful at freeing a unit of LEL, H<sub>2</sub>S, and benzene. The chemical cleaning technique is to inject the chemical during the cool down step while circulating the chemical for a designated time. The H<sub>2</sub>S is first scavenged, and then the hydrocarbon is displaced to flare. Once the chemical cleaning is complete, the unit or vessel is swept with nitrogen. An added benefit of chemical cleaning is that it generally reduces decontamination time by several hours, or even days, by reducing the time to hold a hot nitrogen purge. This is a large economic driver. It can also reduce the safety hazards of working in an inert atmosphere.

Overall, the decontamination of a solid media bed will greatly depend on the application of the vessel. Hot nitrogen purge and steam out is a tried and true method to reduce LEL, H<sub>2</sub>S, and benzene levels, but it takes a large amount of time and the vessels are still held under an inert atmosphere. Chemical cleaning is becoming more popular and is proving to be successful and reduces decontamination time

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by several hours or days.

### **THOMAS PORRITT (Chevron U.S.A.) (2)**

My company has had limited success reaching acceptable benzene and lower explosive limit levels when cleaning media beds with hot nitrogen. For that reason, whenever possible, we choose solid media that is tolerant of steam during the cleaning phase. This allows us to clean and dump the beds more efficiently. For steam cleaning to be effective we have found that flow is a key factor. The flow should be enough to avoid forming condensate in the vessel.

### **DAVINDER MITTAL (HPCL-Mittal Energy)**

The methods and processes for decontamination of solid media beds largely depend upon the final objective to be achieved such as replacement of full charge of solid media in reactor system, replacement of one or more beds or only skimming of catalyst bed, reactor inspection only after unloading catalyst or bottling up of the unit due to a longer shutdown.

Different methods are available specific to the unit depending upon whether it is a hydro-processing unit or some other unit.

For hydro-processing units, most people still prefer conventional proven methods like hot hydrogen stripping with a minimum required hydrogen sulfide content in recycle gas to avoid catalyst reduction followed by cold hydrogen stripping, depressurization and nitrogen purging for skimming, partial replacement of catalyst, bottling up of unit due to longer shutdown or only Reactor inspection.

The full charge replacement is relatively less cumbersome as it requires plain hot hydrogen strip without hassles of maintaining a minimum hydrogen sulfide followed by cold hydrogen stripping, depressurization and nitrogen purging before catalyst unloading.

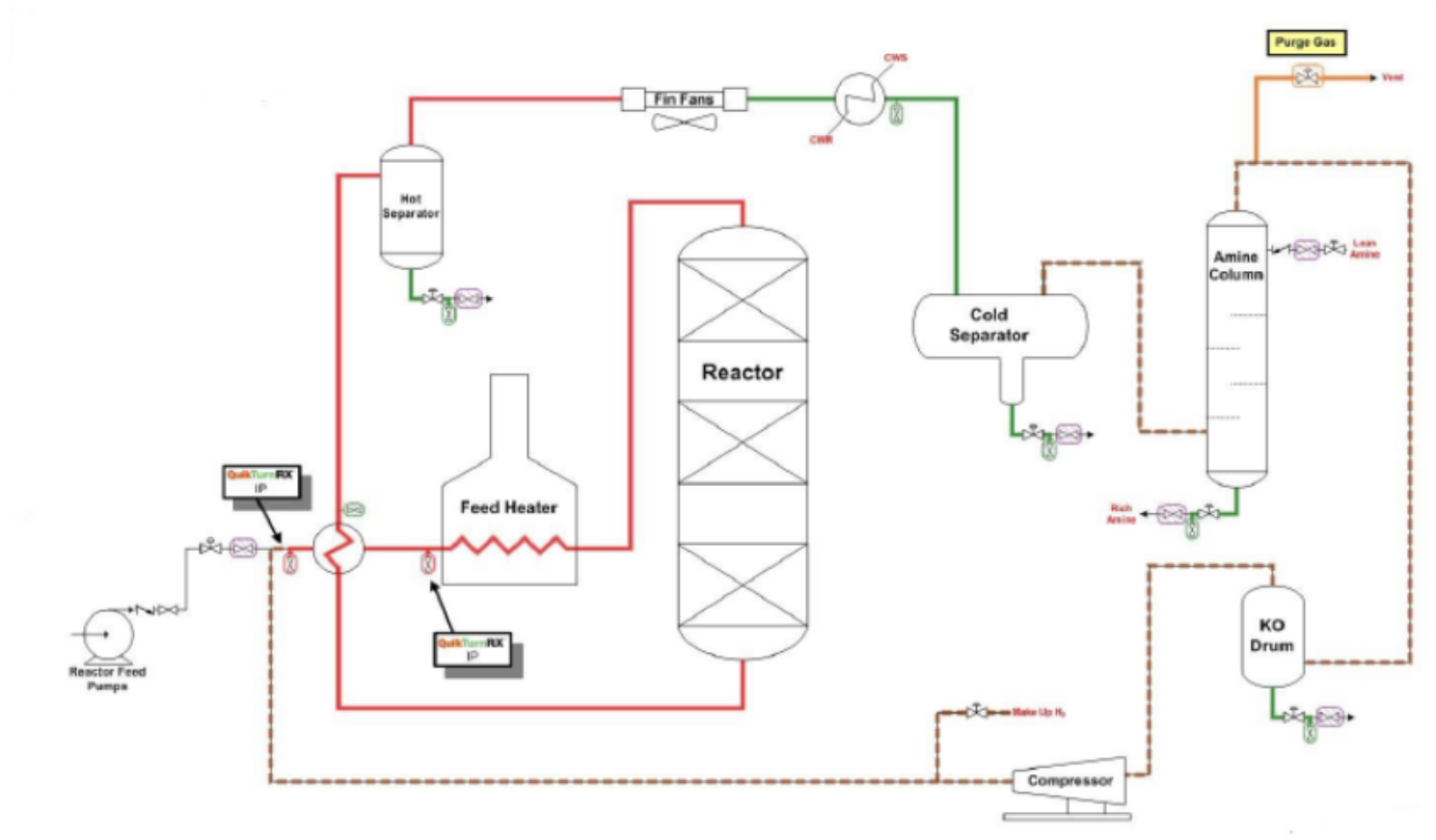
Hot nitrogen stripping or water flooding (aqueous solution of soda ash) is also practiced at times to reduce LEL before unloading of catalyst. Hot nitrogen stripping is a costlier option while reusability of catalyst is lost after water flooding.

The catalyst unloading or reactor man entry is essentially done under nitrogen in both the above cases.

A proprietary catalyst passivation technology and actual case studies are available to eliminate hot hydrogen strip during catalyst decontamination before unloading. Majority of cooling happens under liquid oil circulation thereby reducing conventional unloading time. The technique, passivates pyrophoric or self-heating catalysts by application of a catalyst coating material during special liquid circulation step. The catalyst and equipment surface are coated with an organic film which retards oxygen penetration and reaction providing significant stabilization. The passivated catalysts can be unloaded safely under air thus eliminating hazards related to inert entry. The most important feature of this technology is that it eliminates the life threatening nature of working in a nitrogen atmosphere. Safety is further enhanced by handling passivated catalyst and minimizing hazardous dust normally present with catalyst removal. Another important feature of the technology is that catalyst remains fully re-generable. The technology has successful references for more than 200 reactors till date and has been licensed for application in Europe and Middle East.

One more patented technology is available wherein a proprietary organic chemical (Distillation range:

330- 380oF, Vapor pressure: 1.4 mm Hg at 20oC) is injected during gas sweep process in the unit. The injection takes 4 to 5 hours and the displaced hydrocarbons are routed to flare and slop. The technique claims to shorten decontamination time lines, reduce or eliminate hot hydrogen strip step, no residue LEL and reduction in nitrogen requirement.



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### **Picture-1: Typical scheme showing possible injection location of organic chemical**

Also, the proprietary techniques are available for accelerated cooling of the solid media during cool out phase of decontamination which saves significant cooling and purging time for refinery process units like hydrocrackers, hydro-treaters and reformers, where catalyst handling is often a critical path activity. In this technique, typically, liquid nitrogen is injected in a controlled manner through proprietary equipment into the recycle gas stream to cool the gas before the gas enters the reactor. It is claimed that this technique requires about 1/3rd the amount of nitrogen as required in once through cool down.

For CCR and ISOM, the technology suppliers generally rely on conventional techniques for catalyst decontamination as the procedure is not as complex as in hydro-processing units.

The dry hydrogen stripping of catalyst followed by blinding and sweeping with cold dry nitrogen is followed in ISOM unit and the catalyst is unloaded under nitrogen atmosphere. For CCR, hot hydrogen stripping followed by cooling with recirculating hydrogen and nitrogen is conventional before unloading of catalyst from bottom of reactor/regenerator under nitrogen.

For chloride adsorbents in ISOM and CCR, the nitrogen stripping generally suffices for decontamination before unloading of adsorbent.

For sulfur guard in NHT for example, the drum may be flooded with clean water for decontamination, if vessel and foundation design permits and catalyst may be unloaded in wet condition.

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