CRITERION
CATALYSTS & TECHNOLOGIES

TECHNICAL BULLETIN:

CRITERION* Hydrotreating Catalyst Reactor Loading Guidelines

Introduction

The performance of high activity hydroprocessing catalysts is heavily dependent on proper loading technique. Careful attention, during the loading process, will ensure the maximum reactor activity possible and increase the catalysts' run length. The guidelines presented in this bulletin are provided to maximize the performance of all CRITERION Catalysts.

This bulletin will provide guidelines on Planning The Reactor Load, Inspection of Reactor Internals and Cleanliness, Loading of the Reactor Outlet Support, Main Catalyst Bed Loading Techniques, Top Bed Grading, Final Inspection Of The Distributor, and Maintaining The Proper Environment After Loading.

Planning The Reactor Load

Careful planning, beginning up to 12 months prior to the actual turnaround of the reactor, is required to ensure that the catalyst loading will be performed efficiently and correctly. Below is a Check List of items and suggested lead times required to plan an efficient T/A. This is not intended to be an exhaustive list.

Table 1 - Reactor Loading Checklist

	Suggested Lead time before T/A	Check Off
1. Submit request for catalyst recommendation from supplier or support personnel.	8-12 mo.[1]	
2. Review and Accept recommended catalyst load.	4 mo.	
3. Determine if catalyst will be pre-sulfided in situ or prior to delivery.	3 mo.	
4. Finalize reactor loading diagram and order catalyst[2, 3]	3 mo.	
5. Order support material for reactor.	2 mo.	
6. Contact loading contractor or personnel involved in catalyst loading.	2 mo	
7. Order sulphiding agent if pre-sulphiding in situ.	2 mo.	
8. Order flowbins or containers for catalyst disposal.	1 mo.	
9. Contact catalyst regeneration or disposal contractor.	1 mo.	
10. Finalize shutdown, unloading, loading and startup procedures.	1 mo.	
11. Order materials for T/A(i.e. gaskets dry ice, proper labels for containers, Ceramic fiber, etc.)	1 mo.	
12. Receive catalyst and verify all catalyst has arrived.	1 wk.	
13. Receive catalyst and verify all catalyst has arrived.	3 days	
14. Have meeting with unloading and loading contractor or personnel. Communicate requirements and job responsibilities.	day of T/A	

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[1] 8-12 months will allow time for pilot unit testing, if required.

[2] Catalyst can be supplied in DOT barrels, SuperSacks (35 ft3), or in Flo-Bins (87 ft3). Significant loading time savings and transportation costs can be achieved by delivery in Flo-Bins. Work with your CRITERION representative.

[3] When determining the amount of catalyst required for the reactor load, it is best to provide the catalyst supplier with a reactor mechanical drawing in order to accurately determine the volume of each catalyst required to fill the reactor along with the anticipated loading method (sock or dense). It is recommended that an additional contingency amount of catalyst of 5% is purchased in order to prevent short loading of the reactor.

Inspection Of Reactor Internals and Cleanliness Prior To Loading
Reactor Cleanliness:
Prior to loading of catalyst or support, care should be taken to make sure any water, dust, or old catalyst is removed from the reactor. Loading contractors can usually provide video inspection in the case that the reactor is kept under an inert environment.

1. Special attention should be given to the outlet screens of the reactor and inter-reactor beds. Chips of catalyst should be removed from outlet screens. The outlet screens should be 100% clean. This is easily accomplished with a wire brush. Replace screen covers, if necesaary.

2. Coke or clumps of catalyst should be removed from all vessel walls and internals.

3. Quench section nozzles should be verified to not be plugged.

Reactor Internal Inspection

Special attention is required during the re-assembly of reactor internals to ensure that proper liquid distribution occurs and to prevent catalyst migration.

1. Distributors trays should be verified to be level**. The tray should be leveled within .1-.3% of the reactor diameter or 6 mm(1/4 in), whichever is smaller. This is only a general guideline. Check with your distributor manufacturer for specific recommendations.**

2. Inspect all reactor outlet screens for bent or distorted wires. Make appropriate repairs as required.

3. If the reactor has an outlet screen (sometimes referred to as an "elephant stool"), make sure that it fits snugly in the outlet nozzle of the reactor. Make sure the gap that exists where the outlet screen fits into the outlet nozzle of the reactor is sealed or is at least 3 times smaller than the support material that will be used in the bottom of the reactor.

4. Ensure that all gaskets and ceramic fiber have been installed so that the distributor tray is completely sealed. Flow distribution problems can result from a tray that has not been properly sealed. Special attention should be given to the areas where thermowells and dump tubes penetrate the distribution tray. Also inspect the outer diameter of the distribution tray and seal with ceramic fiber if gaps exist.

5. Inspect the same areas as mentioned in 4 on interbed outlet screens to prevent catalyst migration. Catalyst migration will cause significant damage to the distribution tray below.

6. Quench sections should be inspected for levelness. The level should be within normal fabrication practice.

Loading Of The Reactor Outlet Support

Figure 1 and 2 show sample loading diagrams for loading support in the bottom of a reactor with an outlet screen and an inter-bed outlet screen.

Figure 1

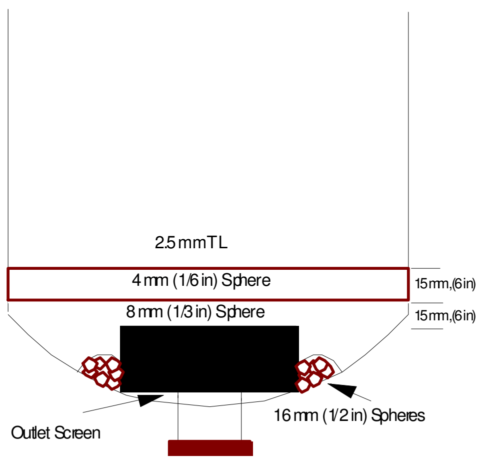
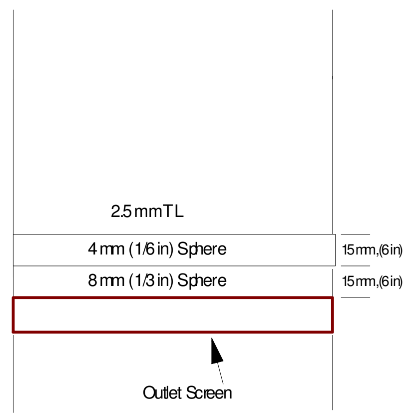


Figure 2



In Figure 1, the outlet screen ("elephant stool") is typically designed with a sleeve that fits into the outlet nozzle of the reactor. It is very important that the outlet screen fits "tight"(ie, there is not a large gap between the screen sleeve and the outlet nozzle). A quick test for this is to try to move the outlet screen while it is in place. Any movement signifies that the outlet screen sleeve needs to be properly sealed. Attention to the bottom edge of the outlet screen where it meets the bottom head of the reactor needs to be part of the reactor internal inspection. Large gaps between the base of the outlet screen and reactor head **(>3mm,1/8in)** should be sealed with ceramic fibre. This area should be sealed with **>16 mm (1/2 in)** ceramic spheres to prevent catalyst migration under the outlet screen.

In Figure 1 and 2, we recommend that support media (Active or Inert) is used as the attrition and crush strength of these materials resist breakage and fines generation which can plug the outlet screen. In processing services where maximum activity is required, CRITERION recommends **8 mm (1/3 in) active support spheres** are loaded to **150 mm(6 in)** above the top of the outlet screens. This recommendation holds unless the slot spacing of the outlet screen is greater than **2.5 mm (1/10 in.)**. If the slot space is greater than **2.5 mm**, we recommend spheres that are **3 times** larger than the slot space of the outlet screens. This rule of thumb prevents broken pieces from the large support from being able to pass through the screen. For Figure 1 and 2, we recommend that the next layer should be less than **3.5 times** smaller in order to prevent migration of the smaller material above**.** **4 mm (1/6 in.) active support spheres** are available, however in cases where maximum activity is the goal, the next size material can be 2.5 mm (1/10 in.) catalysts. Refer to tech. bulletin **"Loading Your Hydrotreating Reactor For Maximum Activity".** This layer should be at least 6 inches. The material used as support material should possess **an Attrition Index* greater than 95 % and a bulk crush strength greater than the maximum expected pressure drop + the weight of catalyst above.**

* % wt retained on 20 mesh screen after tumbling for an hour.

The reactor unloading nozzles should be filled with inert material to minimize coking and to prevent potential reactive areas with no flow. A layer of ceramic fibre should be placed at the bottom of each unloading nozzle above the stopping plate to ease the removal of the plate. Refer to Figure 3.

The support material is generally sock or bucket loaded by a loading technician who is stationed inside the reactor. Care should be taken to ensure that support does not free fall more than **60 cm (2 ft)** to prevent breakage. Ensure that dust and chips in the bottom of the original containers are not loaded into the reactor when transferring support material to the reactor.

For reactors with multiple beds, which use "dump tubes" to allow dumping of all beds without entry, fine aggregate is typically loaded into the tubes. This material is installed to prevent liquid bypassing of the interbed distributor, but allows the catalyst to free flow through the tube when the reactor catalyst is unloaded.

Figure 3

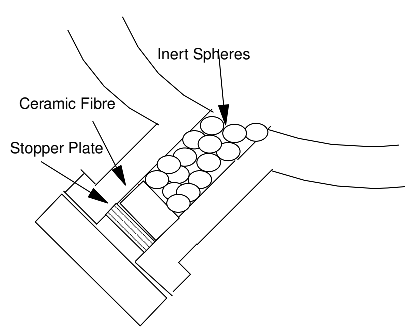
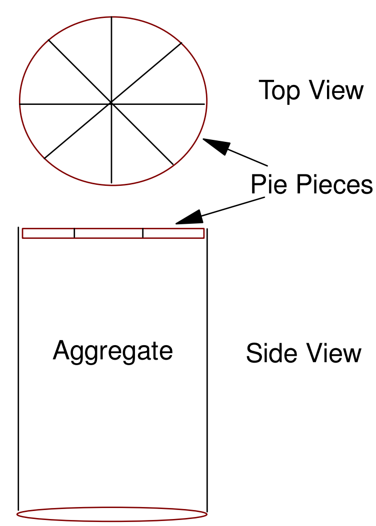


Figure 4



There have been problems with "dump tubes" coking and not allowing the catalyst to free flow during unloading. This is caused by liquid that is trapped in the "dump tubes" during normal operation with low H_2 partial pressure present. There are several ways to improve the reliability of "dump tubes". The first is to provide a chain that is attached at the inside top of the reactor and passes through the dump tubes and is anchored at the bottom of the reactor. If there is a problem with coking in a "dump tube", the chains can be moved around to break up the coke in the "dump tube". Another method is to place metal "pie pieces" which fit at the top of the "dump tube" filled with aggregate as shown in Figure 4. These metal "pie pieces" will minimize the amount of liquid that enters the dump tube, which should minimize the amount of coke formed. These "pie pieces will pass through the dump tubes during the unloading process.

Main Catalyst Bed Loading and Techniques

Now that the support has been loaded into the reactor, catalyst loading by "sock" or "dense" loading methods can commence. Generally**, sock loading should be performed until the catalyst is out of the bottom head of the reactor**. Catalyst should be graded in decreasing sizes using a ratio of 2-3 until the main bed catalyst size is reached.

In general, dense loading is the preferred method for reactors. Dense loading of the reactor can lead to improved flow distribution, increased activity from incremental pounds of catalyst loaded, and reduced possibility of channelling. However, SOR pressure drop will be 50-80% higher with dense-loaded catalyst and the unit must be carefully assessed to ensure that the unit will not become hydraulically limited by the dense loaded bed. Dense loading is more expensive as 10-15% more catalyst is loaded, loading is slower than sock loading and royalties must be paid for using certain dense loading methods/devices, such as Densicat. Table 2 is a comparison of the two methods of catalyst loading.

Table 2 -Comparison of Sock and Dense Loading

FEATURE	SOCK LOADING	DENSE LOADING
Personnel in reactor during loading	Yes	No
Special contractors & tools required	No	Yes
Speed, catalyst loaded m^3/hr (ft^3/hr)	4-9 (140-300)	2.2-14 (80-500)*
SOR Pressure drop	Base	+ 50-80%
Slumping after start-up	Potential	No
Loading costs	Low	Higher
Amount catalyst loaded per ft^3 (m^3)	Base	+10–20%
Risk of maldistribution	Higher	Low

* Rate is heavily dependent on which method of dense loading is used. Consult your loading contactor.

General Guidelines for both "sock" and "dense" loading methods.

1. Prior to loading any catalyst, perform a simulation of expected loading density as described in the "Loading Density Simulation" section. This will provide an initial target for loading density and will help to identify problems during the loading process.

2. Due to the hydroscopic nature of catalyst in the oxide state, it is not recommended to load in damp conditions. Damp conditions include rain, mist, or very high humidity. The catalyst must be kept protected from the rain. In conditions of mist or high humidity, it would be considered too damp if the inside wall of the reactor is "sweating". Catalyst bags should be kept sealed until actual catalyst loading is occurring. The issue with moisture is that vaporization of water during initial startup could result in catalyst damage. Also, excessive moisture may interfere with the sulfiding process during startup.

3. Care should be taken when standing on the catalyst bed. Snow shoes or boards are typically used to distribute the weight of the loading technician.

4. A vacuum system should be used, when loading catalyst, to remove dust from the reactor. Dust and fines will impact the start of run pressure drop.

5. Catalyst bed level should be measured every 4 ft**.** **Acceptable bed level** is determined by the following equation **+-.015 x bed diameter**. This number is based on industrial experience with various dense loading methods and acceptable flow distribution after startup. If the bed does not meet these level requirements, the bed should be manually leveled. Do not use a rake unless the prongs of the rake have been sealed with duct tape. This will prevent catalyst breakage.

6. It is critical to ensure that the interface of different catalyst types and sizes meet the level criteria. It is recommended to have the loading technician visually verify the level of the bed at the interface. The outlet perimeter of the bed should be carefully inspected and leveled as needed to provide a level surface at the interface.

7. **Catalyst bed density should be measured every 4 ft**. This will help to identify problems with the loading method. The loading personnel should record the loading densities on a loading report. This information will aid in the troubleshooting process if problems are identified after startup.

8. Records of apparent dust, problems with loading equipment, etc. need to be very carefully documented.

9. Shift change of loading personnel is when problems may occur. It is important to ensure that a proper turnover occurs between each shift and that some amount of redundant coverage is provided for at least one hour during shift change.

10. Dust removal equipment should be operating when catalyst is being loaded as excess dust will reduce the void fraction in the catalyst bed and cause higher SOR pressure drop.

Specific Guidelines for Sock Loading

Sock loading is usually only recommended for vapor phase units, units with hydraulic limitations and chronic fouling, units loading spherical catalyst, and top beds using hollow cylinders or other high void shapes. However, several refiners will dense load even in these cases. The main reason being that sock loading involves having personnel in the reactor during loading. Catalyst slumping of as much as 10% may occur during operation of a sock loaded catalyst bed.

Vapor Phase Units:
In the case of vapor phase units (naphtha/kerosine), dense loading is usually not justified due to small incentives for increasing the total pounds of catalyst in the reactor. In addition, maldistribution is less of a concern in a vapor phase operation. The net result of sock loading a vapor phase reactor is usually a faster loading time, more pressure drop tolerance, due to the higher void fraction, and a lower start of run pressure drop.

Spherical Catalyst:
There is usually no activity benefit from dense loading spherical catalyst. There have been cases where dense loading has been performed on spherical catalyst with percieved benefits of increased loading density. This may be explained by non uniform spherical shape and variations in actual density verses expected density. However, it is generally an accepted practice to sock load spherical catalyst unless safety or other factors are issues.

If the sock loading method is performed, the following additional guidelines are provided.

1. Use at least a **15 cm (6 in) diameter** sock in order to provide an acceptable loading rate. Typical loading rates will range from **4-9 m3/hr (140-300 ft3/hr)**. The loading rate is dependent on distance from catalyst hopper to the bed, with the lower beds requiring more time.

2. A loading technician should operate the sock from inside the reactor. Proper personnel protection equipment should be used for the person inside the reactor.

3. The technician should move the sock in a circular pattern as the catalyst is unloaded from the sock. When this method is used, raking and leveling of the catalyst bed is not required.

4. The sock should be kept within **30 cm (1 ft)** of the catalyst surface to minimize dust and freefall of the catalyst.The sock should be cut at regular intervals as the catalyst bed level rises. This will be dependent on the diameter of the reactor.

Specific Guidelines for Dense Bed Loading

In the absence of hydraulic limitations, it is recommended to apply the dense bed loading for trickle phase operations. Well known methods are COP from Arco, proprietary methods from Cat Tech (CRI) and Densicat applied by Petroval. Consult with these companies for additional information.

In dense bed loading, the catalyst is sprayed radial into the reactor which orientates the catalyst particles in a mostly horizontal position. This is accomplished by a distribution device located at the top of the reactor bed. The net effect of dense bed loading is a higher loaded density and a lower void fraction. Table 3 shows a comparison of typical loading density and void fraction for CRITERION catalysts using sock and dense bed loading methods.

If the dense bed loading method is applied, the following additional guidelines are provided.

1. Each licensor will apply their own procedures. In general the equipment must be level and centered at the top of the reactor (and at each distributor tray in multibed reactors).

2. Catalyst must not impinge on the reactor walls as this could result in catalyst breakage and fines generation. In COP (Arco) and the Cat Tech (CRI) method, one technique is to initially turn up the air pressure until you can hear the catalyst impinging on the wall, then slowly back off on the air pressure until you can no longer hear the catalyst hitting the wall.

3. Prior to beginning loading with a dense loader, take a measurement of the length of the dense loader. As the bed approaches within 1m (3 ft) of the bottom of the dense loader, dense loading should be ceased and sock loading should be used to complete the reactor loading.

Table 3
Typical Loading Density and Void Fraction for Criterion Catalysts

Criterion Catalyst	Diameter mm, (in)	Sock Loaded Density g/cc, (lb/ft^3)	Dense Loaded Density g/cc, (lb/ft^3)	Void Percent Sock Loaded	Void Percent Dense Loaded
DN-200 NiMo TL	2.5	0.64 (40)	0.75 (47)	49	41
DC-185 CoMo TL	1.3	0.56 (35)	0.66 (41)	49	40

Top Bed Grading

A graded top bed in a reactor is usually recommended in order to improve flow distribution and improve fouling tolerance (especially in the lead reactor). The use of high void active and inert support is a common industry practice when it comes to top bed grading. The shape of commercially available top bed catalysts and supports (Hollow Cylinders, Spheres, etc.) provide for improved flow distribution by improving radial flow throughout the catalyst bed. When these large void catalysts and support are properly installed, significant increases in fouling tolerance will result due to the ability to accumulate a larger volume of particulates between catalyst particles. See Table 4 for a comparison of void of different shapes. Contact your CRITERION sales representative for recommendations on top bed grading.

Table 4 - Void Volume of Shaped Materials

Top Bed Catalyst Shape	Void Percent – Sock Loaded
Medallion	65 - 70 %
Hollow Cylinder 8 mm, 6.4 mm, 4.8mm	50 – 60 %
TriLobe Shape 2.5 mm	40 – 45%
Sphere 8 mm, 4mm	30 – 35%

Guidelines for application of top bed grading are presented below.

1. Contact your CRITERION Sales Representative for specific recommendations on top bed grading. Top beds can be customized to provide the optimum fouling tolerance for specific fouling problems.

2. The top layer of support should exhibit a loading density greater than .72 g/cc (45 lb/ft^3) and be at least 10-16 cm (4-6 in) deep when a distributor or impingement plate is present to prevent movement of the bed. If a distributor or impingement plate is not present, inert support with 1.4-1.7 g/cc (90-110 lb/ft^3) is recommended.

3. Top bed catalysts (hollow cylinders, TL) should have a flat plate crush strength of at least 1.5 lb/mm and an Attrition Index greater than 95% in order to prevent catalyst breakage in fouling services.

4. Graded top beds should only be loaded by sock loading methods to prevent significant breakage and to provide the maximum void.

5. The top layer of support should be loaded within 15-30 cm (6-12 in) of the distributor. It is usually not recommended to load right up to the bottom of the distributor as flow maldistribution may occur.

6. Each layer of top bed catalyst and support should be carefully leveled prior to placing the next layer on top.

7. When beginning to load a new layer in the top bed, the catalyst should be discharged very slowly from the sock in order to prevent significant disturbance to the level of the bed. Do this until at least a 8 cm (3 in) layer of material completely covers the previous layer.

Final Inspection of the Distributor

After the reactor has been completely loaded, careful installation of the distributor tray should be carried out.

Use the following checklist during the final inspection.

1. All debris and catalyst has been removed from the tray deck.	
2. The outer diameter of the distribution tray is completely sealed by gasket material.	
3. All tray sections are sealed by gasket material.	
4. Any gaps around the thermowells or dump tubes have been completely sealed.	
5. The tray leve still meets guidelines provided in the “inspection of reactor internals”.	
6. All tray sections are in tight (e.g. they don’t rattle when you hit them with a hammer).	
7. The inlet pipe has been completely cleaned of debris to prevent depositing the debris on the tray dec when the inlet pipe is installed.	

Maintaining A Proper Environment Prior To Startup

After the reactor has been loaded, it is recommended to install the inlet pipe spool as soon as possible. If this is not possible, the inlet manway needs to be sealed to prevent any additional moisture from entering the reactor. After the inlet pipe spool is installed, the reactor should be purged with nitrogen in order to remove as much moisture and oxygen from the reactor. After this is complete, maintain a nitrogen environment over the catalyst.

This simple procedure allows you to predict sock and dense loaded densities.

Dense Load Simulation

1. Take an initial weight of an empty 1000 ml graduated cylinder.

2. Slowly tap catalyst sample into the graduated cylinder. This should be done slow enough that the catalyst lays flat in the graduated cylinder.

3. Fill graduated cylinder to 500 ml with catalyst.

4. Weigh graduated cylinder and subtract initial empty weight.

5. Calculate density by dividing the net weight by 500 ml.

6. Convert the density into units that will be used during loading (lb/ft^3, kg/m^3,etc.)

7. This is the approximate dense loaded density.

Sock Load Simulation

1. Perform the dense load procedure described above.

2. Place a cloth over the end of the graduated cylinder and turn the cylinder over 3 times.

3. Measure the new volume and calculate the density.

4. This is the approximate sock loaded density.

HEALTH, SAFETY AND ENVIRONMENTAL PRECAUTIONS

CRITERION has evaluated the hazards of CRITERION products as required by OSHA Hazard Communication Standard 29 CFR 1910.1200. Full attention to these hazards and to appropriate health, safety, and precautionary information is essential. Before handling, testing, or using this catalyst, a Material Safety Data Sheet must be obtained by contacting a CRITERION Catalyst Sales Representative.

ADDITIONAL INFORMATION

All catalyst information supplied by CRITERION is considered accurate but is furnished with the express understanding that the customer receiving such information shall make its own assessments to determine suitability of such information for customer's particular purpose. All purchases of catalyst from CRITERION are subject to CRITERION's standard terms and conditions of sale (including CRITERION's product warranties) set forth in a sales proposal, sales contract, order acknowledgement, and/or bill of lading.

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