

# Catalysts by Evonik

## Handling Procedures for Precious Metal Catalysts (PMC)

### Reactor Charging - Filtration - Deactivation

#### General recommendation

Whenever possible we recommend to use PMC in water-wet form. If water does interfere with the hydrogenation reaction and does cause any undesired side-reactions one would use a dry catalyst.

There are three options how to charge a commercial scale reactor:

1. Adding catalyst via pre-mixing vessel, where the catalyst is suspended in the solvent and fed into the reactor via a piping system. This also allows for a semi-continuous addition of catalyst suspension into the reactor under inert conditions (Figure 1).

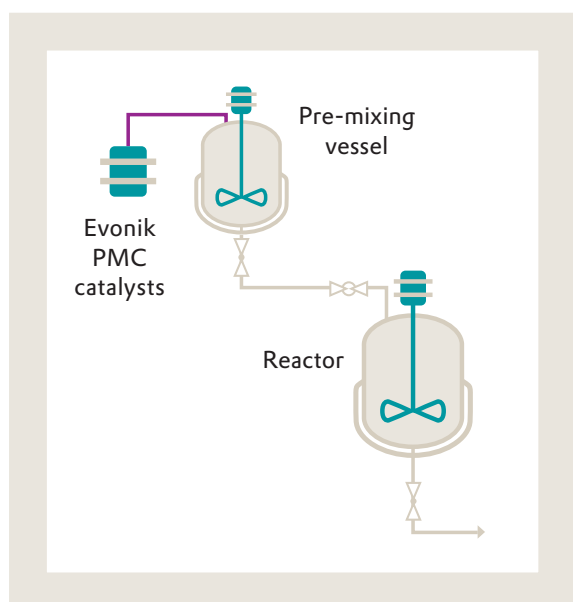


Figure 1: reactor charging with pre-mixing vessel

2. Adding catalyst via hopper or container continuously or semi-continuously under an inert atmosphere directly into the reactor (solid charging system).

3. Adding catalyst directly into the reactor (Figure 2):
- Purge reactor with nitrogen
  - Add catalyst to the air-free slurry vessel
  - Add organic solvent
  - Purge with nitrogen again, exchange nitrogen by hydrogen and start the reaction

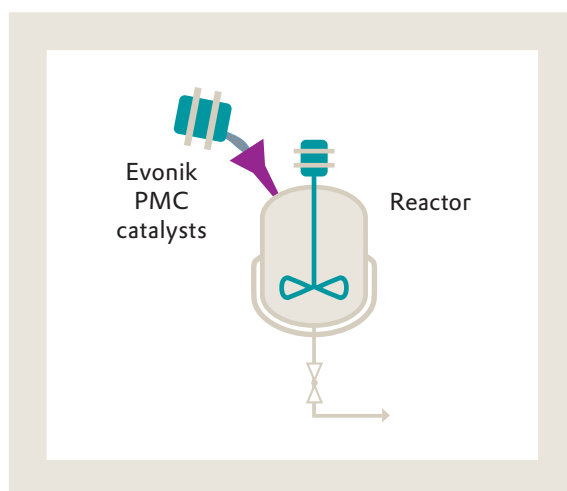


Figure 2: direct charging of the reactor

**!** Use caution when working with any solvents or liquid organic chemicals, including those that do not ignite as readily. It is therefore recommended that small-scale charging trials be carried out before starting commercial scale production.

We recommend using option 1 or 2 when wetting a PMC with a flammable solvent (Figure 1).

The wetting of the catalyst with the solvent should be carried out in a pre-mixing vessel under an inert gas atmosphere (e.g. argon or nitrogen) prior to charging the catalyst slurry into the air-free reactor. The preferred addition sequence is to add the solvent to the dry catalyst instead of the catalyst to the solvent. Cooling of both solvent and catalyst before mixing further reduces the possibility of ignition. Reduced PMC is more reactive towards flammable organic vapours or liquids in the presence of oxygen than non-reduced PMC.

Care must be taken when choosing the desired solvent as PMC can react exothermically with certain solvents at room temperature. The use of highly volatile and highly flammable solvents should be avoided with both dry and water-wet PMC. Lower alcohols (e.g. methanol, ethanol and isopropanol) and compounds such as cyclohexene that are readily dehydrogenated are especially prone to ignition.

## **Filtration**

**a. Preparation:** After performing the desired reaction, it is necessary to separate the catalyst from the reaction solution to purify the product, recycle the catalyst for additional reactions or to remove the deactivated catalyst. The catalyst filtration is an important step for the overall cost efficiency of the process and is a potential bottleneck in many production processes. An ineffective filtration method could result in lost time, lower product yields or less catalyst recovery. The choice of the proper catalyst for the desired reaction and the equipment used to manufacture the product can help to improve catalyst filtration.

**b. Safety:** Whereas fresh, dry PMC is generally not pyrophoric at room temperature and safe in the absence of flammable organic vapours or liquids, it may spontaneously ignite after use in a hydrogenation reaction due to the highly reactive adsorbed hydrogen. When the catalyst is re-used, purging of the filter bags with the appropriate solvent under an inert gas atmosphere is the best way to get the catalyst directly back into the reactor.

**c. Filtration process:** For maximum economy in the use of PMC, the spent catalyst must be recovered with the minimum loss of material. Various types of filter (e.g. leaf, cartridge or centrifuge filters) are suitable for this operation. Any filter pre-coat used should preferably be of a combustible material (e.g. cellulose) to simplify the recovery and refining of the precious metals in the residues. The selection of filter media is critical in order to allow free flow of filtrate while avoiding the pass through of solids. At the beginning of the filtration process the fine particles pass through the filter medium. After a short while a filter cake starts to develop and the passage of fines becomes more and more limited. A practical way of filtration is to recycle the filtrate as long as no fines can be detected in the mother liquor. Sometimes filter aids can be added to help filtration either directly into the slurry or as a pre-coat on the filter medium. Filter aids include diatomaceous earth, perlite, and clay which are all inorganic. Organic filter aids include cellulosic fibres and activated carbon. If the catalyst needs to be recycled back into the process those filter aids should not be used.

**d. Deactivation and preparation for transport:**

To remove spent catalyst from the process cycle, the catalyst must be filtered off under an inert gas atmosphere (e.g. argon or nitrogen) and treated with steam. The latter step removes as much as possible of the organic residues, which could cause problems in the precious metal recovery process. Furthermore, the highly reactive adsorbed hydrogen is also deactivated. Care must be taken that the spent catalyst does not dry out during recovery as it may spontaneously ignite on reaction with residual organic compounds from the hydrogenation process. The spent catalyst should be stored in drums.

## **Spills and Deactivation**

Spills of wet and dry catalysts should be cleaned up with wet towels. To avoid dust formation, it is recommended that dry catalyst spills are moistened with water. The spilled material and towels must then be stored in appropriate waste containers and kept water-wet.

**Europe**

Evonik Resource Efficiency GmbH  
Business Line Catalysts  
Rodenbacher Chaussee 4  
63457 Hanau-Wolfgang  
Germany  
Phone +49 6181 59-13399  
Fax +49 6181 59-2699

**North America**

Evonik Corporation  
Business Line Catalysts  
299 Jefferson Road  
Parsippany, NJ-07054  
USA  
Phone +1 800 422-8773  
Fax +1 973 929-8504

**South America**

Evonik Brazil Ltda.  
Business Line Catalysts  
Rua Arquiteto Olavo  
Redig de Campos,  
105 Torre A – 13º e 14º andar  
04711-904 - São Paulo - SP  
Brazil  
Phone +55 11 3146-4100

**Japan**

Evonik Japan Co., Ltd.  
Business Line Catalysts  
Shinjuku Monolith 12<sup>th</sup> Floor,  
2-3-1, Nishi-Shinjuku,  
Shinjuku-ku  
163-0938 Tokyo  
Japan  
Phone +81 3 5323-7363  
Fax +81 3 5323-8789

**China**

Evonik Specialty  
Chemicals (Shanghai) Co., Ltd.  
Business Line Catalysts  
55 Chungdong Road,  
Xinzhuang Industry Park  
201108 Shanghai  
China  
Phone +86 21 6119-1461  
Fax +86 21 6119-1142

**India**

Evonik Catalysts India Pvt Ltd.  
F - 1/2, MIDC Phase 1  
Dombivli (East) – 421213  
District Thane  
India  
Phone +91 25 124 71716  
Fax +91 25 124 70269

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Business Line Catalysts

[catalysts@evonik.com](mailto:catalysts@evonik.com)

[www.evonik.com/catalysts](http://www.evonik.com/catalysts)

[www.evonik.com](http://www.evonik.com)

