



Research Catalysts, Inc.

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Reduction Guidelines for Copper Catalysts

For O₂ removal from gases and liquids, copper-based catalysts such as our GetterMax® products must be activated by reduction of the copper oxide to metallic copper. The reduction is accomplished using a gas mixture having a minor amount of reducing agent in an inert carrier or diluent gas. H₂ is the reducing agent commonly used - although CO can also be used; and the carrier/diluent is generally N₂ - although other inert gases such as helium or argon (or CO₂) can be used as well.

The keys to a successful reduction are (1) maintaining catalyst temperature between about 175°C and 250°C; (2) sufficient dilution of the reducing agent (H₂) to avoid excessively high temperatures; and (3) feeding a sufficient total quantity of hydrogen to achieve a complete reduction of the catalyst. For small beds (10 kg or less) RCI recommends a 1.5% H₂ / 98.5% N₂ reducing gas; while for larger beds a 2% H₂ / 98% N₂ mixture can be used provided the bed is equipped with catalyst temperature measurement and the reactor will be monitored and cooled with H₂-free gas as necessary if/when any zone in the catalyst bed exceeds 225°C. PLEASE NOTE: Most copper types can be operated at temperatures as high as 250°C with minimal loss of lifetime or capacity, and a few short-term excursions (spikes) to 275-300°C can often be tolerated with minimal damage to the catalyst. Thus, the “max. 225°C” guideline provides a good “margin of safety” for the reduction procedure. Once the bed has been reduced as far as possible with the 1.5-2.0 % reducing gas, the H₂ (or CO) level can be increased (optional completion step) e.g. to 5 or 10% or even 100%, to ensure that a complete reduction is obtained.

A detailed, step-by-step procedure is provided below. This procedure assumes that the gases being used are H₂ and N₂. In principle, the reducing gas may either be purchased premixed to the desired dilution ratio (1.5 – 2.0% H₂); or the two gases can be separately metered and flow-controlled, and then mixed (by sufficient length of tubing/piping and/or an inline static mixer) prior to contacting the catalyst. If a diluent gas other than N₂ is used, the dilution ratio should be adjusted to compensate for the difference in the heat capacity of the chosen diluent gas versus that of N₂. For example, CO₂ has a higher heat capacity than N₂, thus with CO₂ the H₂ concentration can be slightly higher, whereas with He or Ar it should be lower. The reduction can be performed at pressures from slightly above atmospheric to several bars. However, lower pressure is preferred, especially for smaller systems, as it results in higher superficial gas velocity which promotes good fluid dynamics and uniform flow distribution.

- (1) Preheat the catalyst bed to 175-180°C with a H₂-free inert gas (e.g. N₂) that is preferably heated to this temperature range before contacting the catalyst. The reactor should be insulated to prevent heat loss. Wrapping smaller vessels with heating tape or a heating mantle with variable power input and using tube-skin temperature measurement to control power input to the surface heating element on the reactor, also works well to help maintain catalyst at reaction temperature during the reduction. A gas flow of at least 5 SLPM per kg of catalyst is recommended for efficient heating of the catalyst.
- (2) Once the catalyst has been heated for at least two hours as described in step (1), the reduction can commence. Switch from H₂-free gas to reducing gas (1.5% or 2% H₂) and adjust the flow rate between about 5 SLPM and 25 SLPM of total gas per kg of catalyst, depending on the gas delivery capacity of the system. Maintain the inlet temperature of the gas at 175-180°C. With 5 SLPM of a 2%-H₂ gas mixture per kg of catalyst, it takes ~24 hours to feed the total amount of H₂ required to fully reduce a virgin (fully oxidized) catalyst with about 40% CuO; with 15 SLPM per kg, the same reduction is completed in about 5 hours; for catalysts with lower copper content, it takes proportionally less time and H₂.
- (3) If the reactor is equipped with thermocouples for catalyst temperature measurement, a “temperature wave” can be seen moving from the top to the bottom of the reactor if in down-flow (or bottom to top if up-flow), as a “hot-spot” (reaction front) generally in the range of 200-220°C travels through the catalyst bed. Should the temperature exceed 225°C anywhere in the bed, switch to H₂-free gas until the hot zone cools back down to 200-210°C, then resume feeding the reducing gas (1.5% - 2% H₂) mixture. Repeat as necessary till all bed temperatures have peaked, preferably without exceeding 225°C.
- (4) As an optional completion step, once all bed temperatures have dropped and the bed is no longer consuming any H₂, increase the H₂ in the gas to 5% or 10% for 1 hour.
- (5) The end-point of the reduction can be detected in several ways:
 - a. If catalyst temperature measurement is available, the hot-spot will have traveled fully through the bed; no further heat release will be evident; and all catalyst bed temperatures will be stable or subsiding.
 - b. If measurement of H₂ concentration in the gas is available, the H₂ concentration in the effluent will have reached the same concentration as in the feed.
 - c. If the water of reduction is being condensed and measured, the formation of water will be seen to have stopped.

- d. A moisture analyzer on the effluent gas can be used. When the reduction has been completed, the moisture level in the gas will drop off to a low level (if there is any O₂ in the reducing gas, there will remain a corresponding amount of moisture in the effluent).
- (6) For small units having no catalyst temperature measurement, no hydrogen analysis and no measurement on water, basically one must feed a known amount of reducing gas (with some excess) at the right temperature to be sure of completely reducing the catalyst.
- (7) Once the reduction has been completed, feeding additional reducing gas (even pure H₂) has no effect on the catalyst: no further heat of reaction is released and no further water formation will occur.
- (8) Once the reduction has been completed, the reactor may be put immediately into service. However, if processing a feed stream with significant amounts of unsaturates such as ethylene or propylene, the H₂ must be purged out of the reactor before introducing feed, otherwise a potentially large exotherm can occur, as reduced copper-type catalysts can catalyze the hydrogenation of unsaturated compounds such as acetylenes, dienes and olefins.
- (9) If the reactor is not to be used immediately, purge with N₂ until the bed is fairly free of H₂ and moisture, and keep it blanketed under N₂ or other inert gas until needed.
- (10) The activated catalyst is highly reactive towards oxygen, and pyrophoric in air. With the catalyst in the reduced state it is imperative that no air be allowed to enter the reactor, and that the catalyst not be removed into air without being first oxidized or wetted with water – otherwise it can get hot enough to permanently incapacitate itself and/or cause ignition of any flammable materials it may contact.

Following the above guidelines for the reduction and regeneration of the copper based catalysts will ensure safe operation, optimum performance and long lifetime with these highly efficient O₂-getter catalysts. In case of any questions about this procedure or the application of the catalysts, please call 281-935-8123 (my cell) or email us at sales@researchcats.com.

August 29, 2012



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