Technical Guidelines for
**Durasorb™ HG**
Mercury Removal from Natural Gas
Adsorbent Solutions
New Jersey, USA, 10/31/2018
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Introduction

Durasorb™ HG for Mercury removal from Natural Gas

BASF Durasorb™ HG is a high performance, non-regenerable mercury removal adsorbent, produced as a mixed metal oxide containing a dispersed and promoted copper sulfide phase on a highly porous alumina carrier.

Process and Operating Data

General Information

Durasorb™ HG being a sulfided product has limited stability when exposed to air and/or moisture. On exposure, the copper sulfide in the adsorbent would slowly oxidize and generate heat. Therefore, exposure of the adsorbent to air and moisture must be eliminated during storage and transportation and minimized during the loading and unloading activities. Additionally, the adsorbent should not be exposed to mineral acids, flame or an ignition source, or temperatures above 150°C. These conditions may cause toxic vapors such as sulfur oxides and hydrogen sulfide to form. Additional product safety information on Durasorb™ HG is available in BASF’s MSDS.

Adsorbent Data

Material Properties

<table>
<thead>
<tr>
<th>Form</th>
<th>Composition</th>
<th>Design Capacity</th>
<th>LOI</th>
<th>Bulk Density</th>
<th>Crush Strength</th>
<th>N₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spheres, nominal 1/8” dia.</td>
<td>10 wt% copper, as metal</td>
<td>&gt;6 wt% Hg capacity at saturation</td>
<td>6-8 wt%</td>
<td>750-800 kg/m³ (sulfided)</td>
<td>&gt;15 N</td>
<td>&gt;200 m²/g (BET) 0.35 ml/g (PV)</td>
</tr>
</tbody>
</table>

Operating Procedures

Storage and handling

Durasorb™ HG is supplied in sealed, airtight steel drums. The net weight is about 130 kg of adsorbent per drum. This packing is for safe overseas shipping and safe storage for several years, provided a dry and non-corrosive environment. The drums should be stored in a dry and clean warehouse, away from sunlight. Drum seals should be maintained until use and drums should be handled in a manner that does not puncture or dent the containers. The vent screw on the lid should only be loosened immediately prior to loading the adsorbent into the vessel. The drums should be kept closed and opened only shortly before filling the adsorbent into the reactor. Minimize exposure of the adsorbent to moisture by loading in dry weather and not leaving open containers exposed to...
the atmosphere. It is recommended that the drum lid be opened at a platform close to the loading hopper to reduce the exposure to atmosphere.

Loading of the Durasorb™ HG needs to be performed under inert atmosphere. Therefore, appropriate personal protective equipment must be worn when handling and loading the adsorbent. 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, as dust might be generated. Working around the loading area may require use of Self containing Breathing Apparatus to prevent asphyxiation from the purge Nitrogen. When exposed to water, the heat of adsorption can cause adsorbent temperature to rise. Care should be taken to avoid contact with moist skin, mucous membranes and eyes.

Loading

Safety precautions: When using this product, the information and advice given in the Safety Data Sheet should be observed. Due attention should also be given to the precautions necessary for handling chemicals. The local safety regulations must be strictly observed when handling BASF Adsorbent materials. In situations where entry to the vessel is required, confined space safe work practices must be followed. Many countries have specific regulations for confined space work.

If flammable vapors such as hydrocarbons are present in the loading area, grounding is important to avoid discharge of static electricity that could cause an explosion or fire.

The loading crew should be knowledgeable about the hazards of nitrogen as an asphyxiant. Only trained personnel wearing the appropriate PPE should enter the vessel during the purging and loading activities. An important aspect of PPE is the Self containing Breathing Apparatus. Ensure industry applicable HSE requirements on working with SCBA are followed at all times.

The pre-load checklist includes:

1. Check the vessel to ensure that it is clean and free of equipment and debris.
2. Verify that all dump nozzles and inspection manholes not required for loading have been cleaned and sealed, and the gaskets of the proper size and material are in place.
3. Verify the adsorbent support screen and/or grating are correctly installed and secure.
4. Prepare a loading diagram depicting weights, outages, and material loading sequence.
5. Prepare an equipment check list to ensure that no equipment is inadvertently left in the vessel.
6. Ensure all material is on-site and nearby.
7. For the sulfided adsorbent, a high purity continuous nitrogen (minimum 99.9%) purge for the vessel is required. A provision for this purge should be done from the bottom of the vessel.
8. As the loading is done under inert atmosphere conditions, the area around the loading should be barricaded and appropriate safe work procedures should be employed for working in hazardous conditions. This exists even at the top of the vessel where the purge nitrogen will be escaping.
9. No loading should be done during inclement weather, rain, high humidity and heat.

Required loading devices: Stationary hopper fitted on the reactor top with a slide valve. Several canvas sleeves (adequate length, 150 mm diameter). One to three mobile hoppers containing approx. 1 ton of adsorbent. Adequate lifting devices to move the ground hopper to the hopper at the reactor top. This could be either a crane or a winch system. At ground level, a platform should be available for proper work with the equipment described above, and to empty the adsorbent drums.
into the shuttle hopper. A vacuum cleaner must be available to clean the reactor and to collect dust and adsorbent fines inside the reactor. Proper lighting and personal protective equipment is required.

Take care when moving adsorbent containers. Proper lifting techniques and equipment must be used, since containers must normally be lifted to the top of the vessel using hoists or cranes. Proper lifting techniques must be performed in accordance with government regulations and/or other precautions and practices.

Procedure:

- Confirm adsorbent material is on site. Check that the reactor is isolated from the rest of the system. Check the reactor drawings and make sure that the reactor is fully assembled as shown in the drawings. Ensure all safety, personal protection equipment (gloves, dust masks, safety clothes, respirators), and communication devices are available.
- After the manhole is open, check if the reactor is suitable for entry, and take respiratory precautions or verify the oxygen levels, as the reactor might be oxygen depleted.
- Make sure that the reactor is clean and that no water is present in the reactor or in the tubes going to the reactor. Especially check in elbows, where water might have condensed.
- Ensure that the bed supports are properly installed and that there are no gaps in the screen and the vessel wall which may lead to containment issues.
- Before adsorbent loading, the drums should be examined for any possible damage suffered during transit. The adsorbent may be charged either directly from drums or by using a chute or a filling pipe. It is advised to restrict the free-fall height to about 0.5 meter. Boards should be placed on the adsorbent for protection if it is necessary to walk on it.
- The inspection activities and the inert media loading activities should be done under normal vessel entry conditions i.e. in presence of oxygen.
- Before loading the adsorbent, the vessel should be purged with high quality Nitrogen. Check the nitrogen content at the top of the vessel to ensure it is free of air. Continuous monitoring of the Oxygen content is required during the loading process. If during the loading process it is noted that the oxygen content is increasing, the operators should increase the purge volume to keep air out of the loading vessel.
- Open the adsorbent drums only when they are ready to be loaded in the vessel. Do not leave opened drums exposed to atmosphere. In case of shift break, ensure that the vessel has been sealed with high purity nitrogen and there are no opened drums left outside.
- Adsorbent loading should be done continuously, without interruption. Once the adsorbent is loaded into the vessel the reactor should be closed and the vessel sealed by high purity nitrogen or any other inert gas. The quickest and most proven procedure of filling a vessel evenly with a adsorbent is to use a canvas sock fitted to a hopper which is supported outside the manhole. The sock is always kept full of adsorbent and slowly raised to allow the adsorbent to flow into the vessel in a controlled way. The sock must be guided to avoid adsorbent discharge at the same location. By this procedure uneven packing of adsorbent is avoided and even flow distribution across the adsorbent bed is ensured, resulting in a highly effective adsorbent bed. Periodically, the sock must be shortened to prevent kinking during the filling procedure.
- In case operators must enter the vessel during or after charging the adsorbent, planks should be used so that they have not to step directly on the adsorbent.
- At the time when operator has to enter the vessel for levelling the adsorbent, the loading activities should be stopped. Also the operator should be fully trained in working in Oxygen deficient environment and must have appropriate PPE including a Self containing Breathing Apparatus.
Ensure that the bed adsorbent layers are level and not stacked in the centre. All adsorbers should be loaded in similar way to ensure equal flow distribution during normal operations.

During the charging process adsorbent dust can be produced, therefore, respiratory protection must be provided for anyone who may have to enter the vessel during this time. It is recommended to write a loading protocol showing the number of drums and the adsorbent weight loaded into the reactor, including the adsorbent lot numbers. Additionally, the bed height, the adsorbent volume and the loaded density of the charged adsorbent should be reported, as well as the time needed for adsorbent charging.

Measurement of each layer of adsorbent should be done and confirm that the height of the adsorbent is consistent with the loading diagram.

After filling, the vessel is sealed and leak tested. Keep the high purity Nitrogen blanket inside the vessel at above atmospheric pressure. Regularly monitor this pressure to ensure that there are no leaks and atmospheric air does not ingress into the vessel.

Start-up
Prior to startup, the vessel should be under a positive high purity Nitrogen blanket to keep it free of any moisture and air.

For gas phase systems, increase pressure down flow slowly with feed (less than 50 psi/minute) to operating pressure by cracking open the inlet (top) valve or small pressure change bypass valve. When the vessel is at feed pressure, open the inlet valve fully and crack open the outlet (bottom) valve to slowly increase pressure downstream. Once the downstream is pressurized, establish flow through the vessel by fully opening the outlet valve. Some temperature rise is expected due to adsorption of the carrier stream.

Operation
Durasorb™ HG has been designed to operate at temperatures between 15 °C and 65 °C. The adsorbent is operated at pressures between 20 bar and 80 bar.

Shut-down
Hydrocarbon feed stream is stopped. The vessel is slowly depressurized and is purged with high purity nitrogen.

Unloading
A high purity (minimum 99.9%) nitrogen purge must be used during unloading. The spent adsorbent will contain a mixture of copper sulfide, mercury sulfides, hydrocarbons, elemental mercury (physiosorbed but did not react), and alumina. These compounds are generally stable under ambient conditions if they are not exposed to oxygen. In addition to these components, other trace contaminants from the process stream may be adsorbed by or deposited on the adsorbent. These trace compounds can become concentrated on the adsorbent during its use and may cause the spent adsorbent to exhibit toxic, flammable, or unstable characteristics. Among the contaminants of greatest concern are hydrogen sulfides, iron sulfides (from metal scale in the vessel), and residual hydrocarbons.

To help minimize the presence and reaction of these other trace contaminants, it is essential that certain precautionary practices be employed prior to and during unloading the adsorbent from the vessel. Importantly, the bed must be completely purged with high purity nitrogen prior to unloading to remove trace contaminants and create an inert atmosphere free of oxygen and water. The vent purge gas should be sent to flare. Continue the purge step with nitrogen until the outlet mixture meets the HSE requirements for the Lower Explosive Limit. An inert atmosphere must also be maintained.
during unloading operations to ensure that any remaining trace contaminants do not become displaced or react with oxygen or moisture contained in ambient air to create a hazardous atmosphere.

In addition, always assume a hazardous atmosphere exists within the vessel. Do not enter the vessel unless confined space entry procedures that include provisions for safe entry under inert/hazardous atmosphere are followed.

Personnel involved with the unloading operation that are working outside the hazardous atmosphere perimeter 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 if adsorbent dust is generated.

Due to the hazardous nature of the spent material, BASF recommends that materials be immediately offloaded into containers approved for transportation of hazardous/dangerous goods per local regulations. Selection of the specific container should be established beforehand in consultation with the disposal/treatment/transportation company who will be receiving the spent material.

The unloading could be performed either by vacuuming removal or manual removal.

Vacuuming removal:

- A vacuum truck will be installed close to the vessel, and the spent adsorbent will be vacuumed from the top and filled into the steel drums at ground level.
- Personnel, operating outside the manhole, must be equipped and trained to operate in an inert/hazardous atmosphere. The product should be discharged into clean, dry, sealable nitrogen purged transport containers.
- Once the adsorbent is removed to below the bottom port, the bottom manhole/(dump) port can be opened and personnel equipped and trained to operate in an inert/hazardous atmosphere can enter under confined space procedures through the top manhole and use the vacuum hose inserted through the bottom entry to remove any remaining adsorbent.
- Vacuum out any remaining balls into separate containers until no adsorbent is observed under the floating screen or metal bed support screen. Turn off the nitrogen purge.

Manual Removal:

- Use of larger bins will speed up the unloading operation as the adsorbent will likely free-flow by gravity at a quick rate. If a bin will not fit under the dump port, dumping will have to be done into nitrogen purged drums. All containers should be maintained with a nitrogen purge and be plastic-lined.
- Position personnel equipped and trained to operate in an inert/hazardous atmosphere at the open manhole at the bottom of the vessel. These personnel should be equipped with garden-type hoes to help remove the adsorbent from the vessel. Take care not to dislodge or damage either the floating screen in an inert bed support system or the top screen in a metal bed support system. The bottom port flange can be used control or stop the flow of product out of the dump port when changing bins or drums.
- Once most of the adsorbent has been removed from the vessel, remove the inert bed support material above the floating screen or top bed support screen into separate containers being careful not to dislodge or damage the floating screen or the top metal bed support screen. Vacuuming the ceramic material from outside the vessel may be the easiest way to accomplish
this step. Check from outside the vessel to ensure that no adsorbent is under the floating screen or on the top bed support screen, and then shut off the nitrogen purge on the vessel.

- After all the adsorbent has been removed, prepare the atmosphere in the vessel for safe entry. Remove the nitrogen purge line 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 personnel. Monitor for the presence of any hazardous contaminants as well as the oxygen level in the vessel and at the outlet of the air mover to ensure that the nitrogen and hazardous contaminants have been removed. Once safe levels have been established, personnel can access the vessel under appropriate confined space entry procedures. Always leave the air mover on while personnel are in the vessel and periodically monitor the vessel atmosphere to ensure safe levels are maintained.

**Disposal**

The spent adsorbent is hazardous and should be disposed of or reclaimed in accordance with governmental requirements. It is technically feasible to reclaim the active copper component in the adsorbent and, where applicable regulations permit, the reclaimed copper may be returned to the open market.

A key consideration is the safe disposal and/or recovery of the adsorbed mercury. The mercury recovery process normally includes the removal of mercury at elevated temperatures followed by vacuum distillation at specifically licensed mercury handling facilities. Following mercury reclamation, the active copper component of the spent product can be recovered via a smelting process. You may contact BASF for assistance in contact with spent mercury guard processing facilities.

**Image Durasorb™ HG**

Durasorb™ HG spheres
About Us

BASF’s Catalysts division is the world’s leading supplier of environmental and process catalysts. The group offers exceptional expertise in the development of technologies that protect the air we breathe, produce the fuels that power our world and ensure efficient production of a wide variety of chemicals, plastics and other products, including advanced battery materials. By leveraging our industry-leading R&D platforms, passion for innovation and deep knowledge of precious and base metals, BASF’s Catalysts division develops unique, proprietary solutions that drive customer success.

BASF - We create chemistry
Annex 1: Product Data Sheet

1. Durasorb<sup>TM</sup> HG attached