

Operating Procedure for replacing FerroSorp[®] S in filter tanks for desulfurization

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1 Introduction

The flushing of the gas filter with nitrogen and the bordering system components is always required when any work being performed on the filter tank or bordering components can endanger the health of the employees, or if there is any possibility of igniting the gas/air mixture. One such possibility is during the replacement of the desulfurization pellets in the filter vessel.

The system area to be flushed has to extend to the outside of the work area. All crossing points to gas-bearing parts of the system have to be sealed.

Part of the work is performed within the Ex-Zone of the gas filter. Therefore the appropriate measures to prevent the ignition of an explosive atmosphere must be taken. Examples are the prohibition of smoking, open light sources and open flames.

Necessary work involving ignition sources have to be carried out in the period in which the formation of an explosive atmosphere is impossible.

If there is a risk of unintentional ignition sources during flushing the tank, this operation must not be performed. This is particularly the case during a thunderstorm.

Under all circumstances and at all times it is forbidden to work alone during the whole process of the change-out! At least one competent person has to be present or at least within visual range and hearing range.

2 Equipment

Listed here is the equipment that is required for proper flushing and monitoring the explosion hazard. Equipment and tools that are required for further work and after preventing an explosion hazard are not listed.

- Firefighting equipment
- Fire Blanket
- Cooling water, sufficiently long water hose and spray nozzle
- Explosion warning device
- H₂S detector
- Nitrogen cylinder with pressure reducer
- Compressed air hose with proper connection to the fan and the filter tank, at least 10 m long
- Wrench
- Explosion proof hand lamp
- Flange covers

3 Work preparation

To determine the system components to be flushed, the employee in charge of the waste water treatment plant / biogas plant is to be consulted. Together the gas pipe system is examined and appropriate crossing points to non-vented system parts are determined.

Flanged joints where gaskets can be inserted are preferred as crossing points to non-vented system parts.

Attention!

Within the plant area to be flushed there should be no manifolds with pipe branches that cannot be completely vented or securely closed off.

Particularly critical are connections to supply lines of the gas network, as gas can be pressed unnoticed into the work area.

If it is impossible to completely flush the pipe system that has to be secured or to separate it reliably from all gas-carrying parts of the system, no work at the filter tank shall be carried out.

The work area has to be secured against unauthorized access. If internal traffic routes pass through the work area, they are to be shut off.

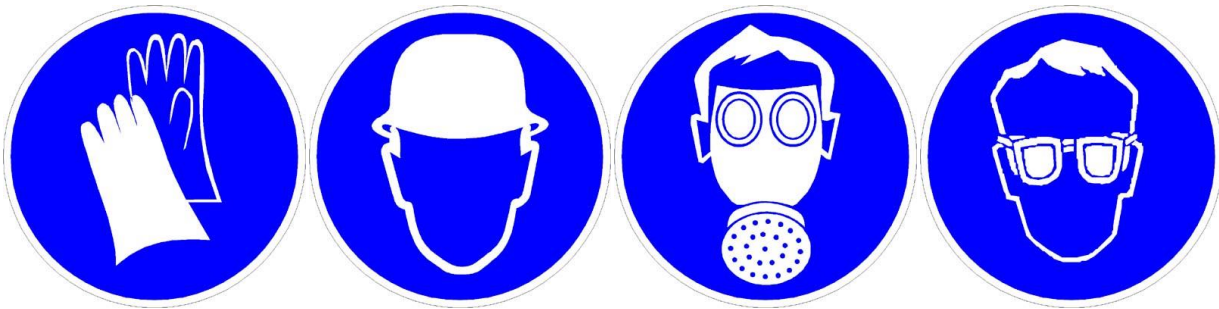
If no other, separately controllable gas filters exist, the generated gas must be stored, burned through an emergency flare or vented into the atmosphere. If the gas can't be stored, the use of the emergency flare is preferred to venting into the atmosphere. Additional measures necessary for these operations are dependent on the specific circumstances and cannot be given in general.

The following steps have to be completed in the order shown:

- Turn off gas consumers if they cannot be operated via an additional gas filter tank, which is separate and gas-proof from the area to be flushed. Close valves in the gas line from the gas filter tank to all consumers, e.g. CHP. If several valves are installed in a gas line system, all valves have to be closed.
- Open shut-off valves in the gas line between the gas source and emergency flare, or vent and prevent automatic closure. Close shut-off valve in the gas pipe leading to the gas tank.

- Close shut-off valve in the gas inlet pipe to the gas tank. If several valves are installed in the gas line, all valves have to be closed.
- Shut-off valves in the gas line from the gas source (digester) to the gas tank are to be secured by warning signs against unauthorized opening.

During the change-out process protective clothing must be worn.



4 Flushing the filter tank with nitrogen

Attention! If possible, always stand on the windward side in front of the filter tank.

Pressurized gas still remaining in the filter tank is then released by opening an exhaust valve.



After the gas has been released by the internal pressure of the filter tank, the exhaust adaptor should be opened, and a gas cylinder filled with nitrogen should be connected to the inlet valve. Provided that the compressed air hose or the pipes to the filter tank require no other restrictions, the pressure reducer on the gas cylinder should be regulated to not more than 2.5 bar.

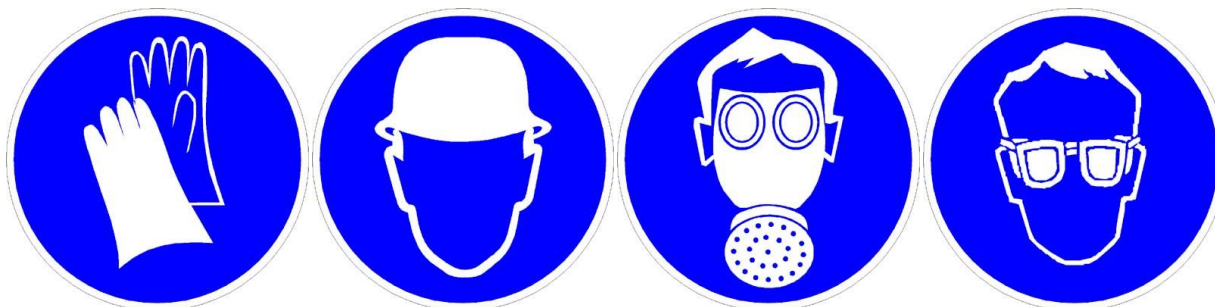
The amount of nitrogen used for flushing must be at least twice the volume of the empty filter tank - including dead volume. Then the success of inerting must be checked. The methane concentration should be below 1% by volume (20% LEL) and the concentration of hydrogen sulfide below 7.1 mg / m³ (5 ppm). Otherwise flushing with nitrogen must be continued until methane and hydrogen sulfide concentrations are met.

5 Removing the desulfurization pellets

Note: Please follow the removal instruction of the provider for the currently used desulfurization material if it is not FerroSorp® S.

After all pipes and the filter tank have been sufficiently flushed the desulfurization pellets FerroSorp® S can be removed. For this purpose any flanges or other caps on the outlet have to be removed.

Protective clothing must be worn.



Attention!



In contact with oxygen spontaneous heat build-up of FerroSorp® S is possible. It is important to ensure that the temperatures in the gas and at the vessel wall remain under 60 °C (140 °F). Stricter regulations on the part of the filter tank prevail. **Attention! The temperature usually increases after a few minutes, but very rapidly!** The risk of significant heat build-up can be minimized by maintaining a continuous concentration of oxygen during the operation of the gas filter that reaches at least double the concentration of hydrogen sulfide. However spontaneous heating cannot be ruled out entirely.

It must be ensured that sufficient space for the unloading of the material is available. If possible, the material has to be filled into a suitable heat-resistant container or hutch immediately. If not available the material should be released onto the floor. To avoid excessive heating, the desulfurization pellets FerroSorp® S should be spread out immediately after emptying in an open area. Larger amounts have to be emptied and spread out in batches. In case of spontaneous heating FerroSorp® S should be cooled with water immediately. In order to prevent inhaling the potentially toxic fumes the water should be sprayed with a hose and nozzle from a sufficient distance, and sprayed from the windward side.

If the material does not completely empty from the filter tank, carefully assist using a sweeper or shovel, without causing damage to the tank. **If possible, nobody should enter the filter.** However if necessary, it has to be the responsible person of the plant or an instructed, competent subordinate who enters the filter. This also applies to all other work in confined space.

After emptying the filter tank it should be cleaned to the greatest extent possible with a broom or something similar. All sieves and gas routes must be cleared of remaining particles. Subsequently the gas filter outlet can be closed.

6 Storage and disposal of FerroSorp[®] S

For intermediate storage, FerroSorp[®] S has to be kept in a dry and wind protected place. If a heat build-up was observed during chapter 5, the media has to be watched in the first days after change-out. If necessary, it has to be cooled.

In Germany the desulfurization material FerroSorp[®] S is classified as nonhazardous waste and under certain conditions it can be used in agriculture. Following a recommendation of the Working Group of the German Federal States on fertilizer traffic control, with elemental sulfur loaded FerroSorp[®] S is legally classified as "processing aids using elemental sulfur of industrial origin" after usage for gas treatment in biogas plants. Thus this product is a kind of fertilizer according to DüMV (Fertilizers Regulation) Appendix 2, Table 8.1, line 8.1.4 "precipitant" in conjunction with Section 1, Table 1.5, "Guidelines for secondary raw material fertilizer", line 1.5.9 "Elemental Sulfur," that may be added to the fermentation residue of the biogas plant before the application in agriculture.

To prevent a possible reformation of hydrogen sulfide from the sulfur contained in the loaded material, FerroSorp[®] S should only be added shortly before application or agricultural use of the digestate into the digestate storage tank. This applies especially to covered digestate storage tanks, whose gas chamber is connected to the gas tanks of the biogas plant.

If any agricultural use of the loaded FerroSorp[®] S is not possible we recommend disposal of the loaded desulfurization pellets FerroSorp[®] S using waste code ASN EWC 060603 - "wastes containing sulphides other than those mentioned in 060602" according to the European Waste Catalogue (EWC).

In Germany many municipal waste landfills and construction landfills accept the loaded desulfurization material FerroSorp[®] S, which is a nonhazardous waste, after submission of a declaration analysis.

7 Re-filling the filter tank

It must be ensured that all the openings of the filter tank, except for the filling opening, are closed and gas-proof. For Big Bags we recommend the use of a filling adapter and hose extension (see photo). This ensures a gentle filling of the material into the tank. Attention should be paid to keep the hose end close to the material surface. During the filling process, the hose should be lifted consistently, so that it always remains above the surface of the material and does not get jammed in the material. This is even more important for large drop heights!

If possible, nobody should enter the filter. However if necessary, it has to be the responsible person of the plant or an instructed, competent subordinate who enters the filter. This also applies to all other work in confined space.



8 Startup of the filter tank with FerroSorp[®] S

After filling the tank, the filling port has to be resealed gas proof. Leak tightness of the system has to be examined, by creating an overpressure with nitrogen, which is higher than the standard operating conditions. An appropriate value has to be set by the plant operator. After successfully testing the system for leaks, the nitrogen can be discharged via the exhaust valve.

Then the exhaust valve stays open and the vessel has to be further flushed with nitrogen again (see above). The quantity should be at least twice the volume of the filter tank.

Before the next steps of the start-up can be taken, possibilities to cool the filter wall and/or the media have to be taken into account and, if applicable, have to be prepared for the case of unexpected complications. A good method is to have nitrogen available and still connected, which could replace the biogas or dilute it. Other suitable options might be a water hose at hand or make water nozzles in a filter ready for use.

Now flushing with biogas can be initiated. The procedure is different for FerroSorp[®] S, FerroSorp[®] Sd and FerroSorp[®] Sk. The following steps are for FerroSorp[®] S as well as FerroSorp[®] Sd and must be performed in the order shown:

- All valves in the gas inlet pipe leading from the fermentation tank to the filter tank, which are open in standard operation mode, have to be opened.
- If an emergency flare is present, open its valves and close the exhaust valve. Otherwise keep the exhaust valve opened. Then open the shut-off valve in the gas line to the gas source in order to flare the gas for the first 30 minutes.
- At the contact of sewage or biogas with the desulfurization pellets, the exothermic reaction of CO₂ with the Ca(OH)₂ to CaCO₃ and H₂O takes place and thus excessive heating occurs. It is important to ensure that the temperatures of the gas and on the vessel wall remain below 60 °C (140 °F). Stricter regulations by the manufacturer of the filter tank have priority. To control the reaction, the gas stream is to be reduced accordingly or diluted with nitrogen at the beginning (about 30 minutes). **Attention! The temperature usually increases just after a few minutes, but then very rapidly!** If the temperature rises above 60 °C (140 °F) the gas flow must be stopped immediately until the temperatures are significantly lower again.
- **A more safe way is to dilute the gas with nitrogen** until the reaction is finished. To achieve that, the dilution of the gas should decrease over time.
- As soon as the filter contains usable gas and the gas temperature as well as the temperature at the filter wall is within normal range, all valves in the gas lines to the consumers (e.g. CHP, boiler, etc.) can be opened. If vents are available, the pipes can be de-aerated. After closing all valves and vents, the consumer can be activated.
- Accumulated water behind the gas filter tank should be drained by opening the condensate trap.
- Existing dust filters upstream to the consumer must be replaced with new, clean filters.

The FerroSorp[®] Sk has been conditioned at the factory, thus has been charged with CO₂, so that a large part of the described chemical reaction has already taken place. For technical reasons, however, it cannot be guaranteed that the reaction has been completed. Accordingly, the temperature profile should also be observed. In a modification of the above points, the start-up with FerroSorp[®] Sk can proceed as follows:

- All valves in the gas inlet pipe to the filter tank and all closed valves in the pipe to the gas source have to be opened. The exhaust valve has to be closed.
- Now the gas filter tank can be filled completely with sewage gas/biogas. After filling the filter tank, the gas concentration is, depending on the dead volume of the filter tank, about 80 - 90%. Whether this mixture can be utilized by the consumer, e.g. CHP, depends on their technical specification and must be assessed by the operator of the biogas/sewage plant. If the gas cannot be used it must be either burned by means of the emergency flare, or discharged through an outlet valve into the atmosphere. Depending on the structure of the pipeline system it may be necessary to burn multiple tank volumes of freshly generated gas by means of the emergency flare.
- As soon as the filter contains usable gas, all valves in the gas lines to the consumers (e.g. CHP, boiler, etc.) can be opened. If vents are available, the pipes can be de-aerated. After closing all valves and vents the consumer can be activated.
- Accumulated water behind the gas filter tank should be drained by opening the condensate trap.
- Existing dust filters upstream to the consumer must be replaced with new, clean filters.

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