

JM ProcessWise webinar Fixed bed absorbents - troubleshooting

July 2020

Questions and answers

- Q1. I regularly analyse and monitor the mercury concentration, but I am having difficulty because the analysis results are not stable. Is it preferable to continuously purge during analysis, or is it preferable to purge immediately before sampling? (Please teach us if you have any knowledge about an appropriate sampling method).
- A1. Mercury can readily adhere to pipework which can make it challenging achieving a representative sample for analysis. Ideally sample lines would be continuously purged during operation to ensure that an equilibrium is reached and the subsequent sample taken is representative. If you require further guidance then please contact us.
- Q2. After loading ZnO catalyst and start up, it caused a lot of dust downstream, this blocked many downstream valves and required shut down. How can we handle this in coming times?
- A2. It is typical for a fixed bed reactor to have a dust filter downstream to protect from dust generated during the catalyst loading. During reactor commissioning (after the catalyst has been loaded) it is possible to do a de-dusting procedure by passing an inert gas over the catalyst bed to remove any dust to a safe downstream location. Dust is not expected to be generated during normal operation.
- Q3. Am I right in thinking that a lead-lag system would help prevent early breakthrough?
- A3. Correct, a lead-lag system would ensure that once the lead bed has started the breakthrough, the lag bed is brought online to treat the stream. It also serves the purpose of ensuring the maximum capacity of the lead bed is achieved.
- Q4. Apart from elemental Hg, can Hg guard bed removes other forms of Hg compounds?
- A4. **PURASPEC** mercury guards only remove elemental mercury.
- Q5. At start up of our MRU, we noticed much higher pressure drop than design that has persisted. We have not done a bed pressure survey to diagnose yet, but have you seen this at greenfield start-up before? Maybe due to scales?
- A5. It is possible for scale or debris to accumulate in an inlet distributor when a unit is first brought online.
- Q6. Beside loading, any other cause for beads crushing that can cause excessive dust? E.g. pressure fluctuations? Any other?
- A6. **PURASPEC** absorbents have a high strength to withstand many scenarios. However, there are factors that can cause breakage of absorbent or ceramic particles which include: high velocities, damaged distributor, reverse flow and fluidisation. Installing a layer of 100 mm ceramics on top of the absorbent is good practice to protect the absorbent.
- Q7. Do you typically recommend online mercury monitoring systems or manual periodic type testing?
- A7. Both online analysis or taking field samples for analysis in a laboratory are viable methods of monitoring the performance of a MRU. Online analysis will provide better visibility of the performance. In either case the sampling system needs to be designed to ensure a representative and accurate reading is achieved.

- Q8. Can a Saturated bed with H₂S removal be used for Hg removal?
A8. Co-removal of Hg and H₂S can be achieved but product selection needs to account for both contaminants.
- Q9. Can adsorbents be used for sulphur (in particular dimethylsulphide) removal from NGL streams with reasonably high sulphur (250-300ppmw). Would the quantity required be practical?
A9. It is not possible to directly remove dimethylsulfide at ambient temperatures from an NGL stream using an adsorbent. The removal is typically achieved by first breaking them down into H₂S using catalytic hydrodesulfurisation followed subsequent reaction with an adsorbent. The practicality of the solution will depend on the amount of sulfur that needs to be removed which depends on the flowrate of the stream.
- Q10. Can H₂S and Hg removal can be performed in a water saturated feed?
A10. **PURASPEC** adsorbents can remove both H₂S and Hg from water saturated feeds. Note that water saturation is not essential for the reaction, and **PURASPEC** can remove contaminants just as effectively in dry and wet streams.
- Q11. Can H₂S and light mercaptans be removed simultaneously in sulfur guard bed?
A11. Yes, although a split bed using different products is expected to be required. However it may be possible to install both inside the same vessel.
- Q12. Can you provide the main reactions of Hg and Catalyst?
A12. $2MS + 2Hg \rightarrow M_2S + Hg_2S$ (M = metal, S = sulfur, Hg = mercury).
- Q13. What is the difference between an adsorbent and absorbent?
A13. An absorbent reacts with the contaminant such that it is chemically bound. The reaction takes place throughout the absorbent. An adsorbent reacts physically with the contaminant (generally on the surface) such that the reaction can be reversed using means such as temperature or pressure.
- Q14. Could you please give an overview of the side reactions generating impurities during loading? Is H₂S formed as well from exposure of metal sulfides to atmosphere?
A14. No side reactions should take place during loading as metal sulfide adsorbents are required to be loaded under an inert atmosphere.
- Q15. Could you provide explanations on cautions while loading the Hg removal catalyst?
A15. Mercury removal adsorbents react exothermically with oxygen and are therefore classed as self-heating (not pyrophoric). Therefore they need to be loaded and unloaded under an inert atmosphere to prevent contact with air.
- Q16. Define what level of liquid tolerance for adsorbent?
A16. **PURASPEC** adsorbents can recover from isolated incidences of liquid carryover. However continuous exposure to free liquids can permanently affect the performance and/or integrity of any adsorbent material.
- Q17. Design of adsorbent vessels typically by EPCIC contractor. How to ensure proposer flow distribution?
A17. JM work with EPCIC contractors during the design stage and offer recommendations on the design of the distributor and vessel to ensure good flow distribution.

- Q18. Do you have any recommendations for the maximum level of oxygen in the feed to the MRU and for the SRU?
- A18. Oxygen levels must be specified to the supplier at the design stage so that they can be taken into consideration with product selection. The maximum permissible oxygen content can vary from case to case but a typical maximum is 0.1 mol% (1,000 ppmv). A detailed assessment at the design stage will consider aspects such as the partial pressure of oxygen as well as the source (to assess whether the concentration could increase during operation).
- Q19. What is the maximum level of hydrogen in the feed to an MRU or SRU?
- A19. The presence of hydrogen requires consideration of concentration, partial pressure, operating pressure and absorbent volume to determine the acceptable level. This information is used to calculate the expected exotherm from the reaction of hydrogen with the absorbent. A detailed assessment at the design stage will consider aspects such as the partial pressure of hydrogen as well as the source (to assess whether the concentration could increase during operation).
- Q20. Does JM know any company that can take spent MRU catalyst?
- A20. Yes, JM have strong relationships with organisations that can ensure the safe reprocessing of spent mercury absorbent that avoid landfill. Where possible, JM may also be able to provide a service to assist in managing the reprocessing of spent MRU waste. Please contact us for more details.
- Q21. Does the current online analysers technology can accurately detect mercury at ppb levels?
- A21. Analysers can detect to very low levels. The challenge though is getting a representative sample to measure at such low levels accurately and reliably.
- Q22. Does the SRU can remove elemental sulphur from LPG streams?
- A22. No, elemental sulfur is not removed.
- Q23. Does the SRU removes organic sulfur and COS, or only H₂S.
- A23. **PURASPEC** catalysts will hydrolyse COS to form H₂S and our absorbents can subsequently remove the H₂S. It is possible remove lighter organic sulfur compounds using an absorbent but this will depend on the species and process conditions.
- Q24. Where is the best location for an MRU in the flowsheet?
- A24. It is advisable to locate an MRU as upstream as possible after the initial separation. This protects all downstream equipment from mercury contaminant. **PURASPEC** is water tolerant and thus can be located in upstream locations where the streams are water saturated as long as the beds are operated in single phase.
- Q25. For the lead lag, will the pressure drop be higher because you have two beds in series?
- A25. Operating two beds in series will have a higher pressure drop than a single bed. But the lead-lag solution can be designed to meet pressure drop requirements of the project. This can include consideration for radial flow vessels to reduce the system pressure drop.
- Q26. For these temperature rise, please make clear is it for zinc oxide or carbon bed?
- A26. The exothermic reaction of a metal sulfide absorbent with oxygen applies to zinc sulfide absorbents and not zinc oxides. JM do not offer activated carbon materials and therefore cannot comment on their reactivity with air.

- Q27. How is the performance of trilobe adsorbents and how to troubleshoot for crushed adsorbent bed other than changeout?
- A27. JM do not offer trilobe shaped adsorbents for H₂S or Hg removal. We have chosen spherical granules for our **PURASPEC** products based on decades of manufacturing experience that have optimised our product shape and form. There are a number of ways to troubleshoot for adsorbent crushing including doing a pressure survey; checking bed velocities; and CFD simulations. See **Error! Reference source not found.**
- Q28. How would you determine the location of the issue if pressure drop is a problem?
- A28. A pressure survey would help locate the source of the pressure drop within the vessel or whether it is caused by upstream or downstream equipment.
- Q29. If cycle life end how I can treat this residual, am I going out my country for this case? Or can you have technology for treat in situ?
- A29. Spent adsorbents can be sent for metal recovery via audited environmentally friendly routes. Depending on your location the spent adsorbent may need to go outside of your country for this process. It is not possible to treat H₂S or Hg adsorbents in situ as they are non-regenerable.
- Q30. If liquid carry over will this cause adsorbent to fuse together, then how we discharge, we need to break and how break?
- A30. **PURASPEC** adsorbents are resistant to isolated incidences or low levels of liquid carryover and performance can recover. It is only excessive, continuous carryover that can lead to unlikely incidences of fusing or agglomeration. In these cases then mechanical means to break the fused adsorbent would be required.
- Q31. If the flow through the unit is fluctuating for a period of time. Could this lead to a damage/crushing of the adsorbent or ceramic media?
- A31. Only if the flow rate exceeds the design of the feed distributor and/or the velocities inside the vessel result in fluidisation of the media. JM can help to assess the impact of fluctuating flow on the adsorbent.
- Q32. If you have liquid carryover, will pressure drop problems only be at the top or is it possible that some of the liquid could travel down the bed?
- A32. It is possible for liquid carryover to migrate through the bed and therefore affect more than just the top layer of adsorbent.
- Q33. If you increase the amount of ceramics or sacrificial material at the top of the bed for pressure drop issues, will this not increase the pressure drop?
- A33. The ceramic materials that we recommend for protection tend to have a higher voidage than **PURASPEC** so the pressure drop would in fact be lower at start of run compared with if that volume was occupied by more adsorbent.
- Q34. Is it common for fixed bed adsorbents to have temperature indicators?
- A34. It is not common to measure the temperature of a MRU or SRU and would only be recommended if, during the design phase, it was deemed to be necessary because of the feed contaminants.
- Q35. Is dust filter normally is in same vessel as adsorbent or separate unit upstream?
- A35. Dust filters are always separate unit operations and can be located both upstream and downstream of a fixed bed adsorbent system depending on what equipment requires protection. We do not recommend installing foam filters inside the adsorbent bed as this locks any pressure drop contribution from dust inside the vessel until the adsorbent is unloaded. Any dust generated during loading usually leaves the vessel shortly after commissioning and **PURASPEC** does not generate dust in normal operation.

- Q36. Is it common to have a DP gauges, inlet/outlet TIs and several intermediate bed sample points on a SRUs?
- A36. Differential pressure drop and intermediate bed sampling points are more common than temperature indicators.
- Q37. Is skimming the bed for pressure drop problems a standard practice? And should we do this if we think the bottom hasn't been saturated yet?
- A37. It would be recommended only if it is possible to confirm that the problem is caused by the top layer of material.
- Q38. Is the fixed bed (same bed) is same for both H₂S and Hg?
- A38. It is possible to remove both H₂S and Hg in the same vessel, we would generally refer to these as co-removal vessels. The vessel designs are similar for single duty and co-removal.
- Q39. Is the spent absorbent considered as hazardous waste and how it is disposed normally?
- A39. Spent **PURASPEC** is considered to be hazardous waste and our recommended disposal route would be to send the material reprocessing. This route prevents the material from going to landfill and converts the metals into a feedstock. JM offer this as part of our **PURACARE** service.
- Q40. Please could you explain again what is the issue of having oxygen in the feed and also is there normally a limit?
- A40. Oxygen reacts with a metal sulfide absorbent exothermically which can heat the absorbent rapidly as well as deactivate performance. See Q18 for information on recommended limits.
- Q41. Sorry I didn't understand, please explain again how carry over causes maldistribution?
- A41. Excessive liquid carryover does not generally impact the bed uniformly and can lead pockets of absorbent that have been affected. This can lead to channels forming causing maldistribution.
- Q42. Sulphided 32-5 should be assumed to be potentially combustible because of adsorbed H₂/hydrocarbons and finely divided carbon that can be present?
- A42. See A26.
- Q43. There are many potential root causes of each symptom. However, do you have rule of thumb on how to identify potential root causes prior to execution i.e. if we observe pressure drop, can we observe some process parameter to identify that root cause is from liquid carry over rather than solid particle?
- A43. Regular performance monitoring is key in root cause analysis. It is recommended to evaluate trends in operating data such as flow, temperature, pressure, pressure drop across the bed, and pressure drop and performance of upstream and downstream filters. The collection of such data along with the knowledge of design parameters can significantly help a root cause analysis investigation. Also, analysing discharge samples is of great importance if the absorbent did not perform as expected. In summary, a comprehensive root cause analysis is required to clearly identify potential causes and solutions.
- Q44. Upstream of our gas bed, there is coalescer filter. What will you suggest the configuration of this CF vessel, horizontal? vertical?
- A44. Vertical configuration with cartridge filters is recommended. This will enable better liquid drainage due to gravity and thus improving performance.

- Q45. We are about to change out our bed, is the material normally free flowing or is mechanical agitation always necessary?
- A45. **PURASPEC** absorbents are tolerant to intermittent liquid carryovers and thus it is very unlikely that mechanical agitation is required. Discharge of **PURASPEC** beds is expected to be free flowing.
- Q46. We had the pressure drop problem. We measured the pressure drop across the vessel, discovered that most of the drop was on the bottom of the vessel and also we collected some ceramic residues on a filter located downstream from the vessel. So it looks like ceramics were somehow crushed inside the vessel and accumulated close to the discharge point. Under what conditions could the ceramics be crushed? they seemed to be very solid when we loaded the vessel?
- A46. Operating the beds beyond design flow limits may cause fluidization and crushing of absorbent or ceramics. During loading, minimizing free fall of ceramics and absorbent is best practice. This is easily done by using a lay flat sock and ensuring that bottom of the sock is no more than 1 meter above the layer to be loaded. Reverse flow can also bump the material and mix the bottom layers leading to migration of smaller material to the bottom collector. A comprehensive root cause analysis should be performed to identify the issue. Some questions will only be answered after the beds are discharged.
- Q47. We have encountered several occasions where the gas bed has a higher outlet reading (indicating breakthrough) whilst the top, middle and bottom bed profile are reading lower values. What typically causes this behaviour? And how can we improve it without shutting down the bed?
- A47. We recommend checking for causes of sampling contamination. Sample bombs and lines should be dedicated for each sampling positions, and should be flushed before taking any samples. We recommend, if possible, to take another sample further downstream from the bed outlet sampling point. This will help verifying false high readings at the outlet. Also, the reason could be traced back to flow maldistribution within the bed. This can be caused by either liquid/solid carry overs, bed fluidization, malfunction of inlet distributor, or operating at higher than design flow rates. Surveying the unit, including the filters and units upstream, may give further indication on what is the cause. If liquid/solids carry over is suspected, then purging the bed with an inert gas may drive off the liquids/ dust and therefore bed may recover performance. In all cases a comprehensive root cause analysis needs to be done to pin down the reason for high readings at outlet and low reading within the bed.
- Q48. What is the typical life of a fixed bed absorbent and how can the performance or life be checked?
- A48. Mercury removal units can be designed to operate for many years. However, each unit is specific to the site where it is located and thus to the feed being treated. We recommend to check what bed life was guaranteed by the supplier and to collect operating data (flow, T, P, Hg readings at inlet, outlet and interbed, if available) and feedback that information the catalyst vendor. Knowing the product in the bed and the installed volume, the catalyst vendor can then advise on the remaining bed life.
- Q49. What are the common adsorbents in your SRU?
- A49. The sulfur removal absorbents are mixed metal oxides. Mainly Copper oxide, and in some products Copper and Zinc oxides.
- Q50. What are the pros and cons to install Hg guard bed upstream of Acid gas removal unit instead of downstream of dehydration unit?
- A50. Installing Hg guards upstream will protect the downstream unit from corrosion and contamination. Installing downstream of dehydration is usually a cleaner duty, that is dry and contaminants are minimal. We would generally recommend installing the MRU upstream of acid gas removal. See A24.

- Q51. What are the recommended temperature instrument types for the vessel? We cannot use thermowell type because of solids and frequent absorbent change-out?
- A51. Thermowells may be used and a mesh can be installed to protect it. Another option is installing the temperature instrumentation in piping rather than on the vessel itself.
- Q52. What causes Puraspec 2070 media to turn white over time?
- A52. **PURASPEC** could appear white if covered by dust, for example from an upstream molecular sieve. We have never seen **PURASPEC** 2070 turn white all of the way through the granules.
- Q53. What could cause blockage to only the bottom collector area?
- A53. It could be caused by blockage to the mesh surrounding the collector. If there is no mesh or it is damaged, this may cause the open slots in the collector to be blocked by ceramics. If the bed has been fluidized, then excessive crushing or dust formation may lead to blocking the collector. The collector itself may also be damaged which can contribute to blockages.
- Q54. What could foul the absorbents?
- A54. The absorbent beds should be operated in single phase. Therefore, any carry over to the bed by not being in the same phase as the feed may be considered a foulant. Examples are free liquids such as water, glycols, amines, methanol, hydrocarbon liquids dropping out as a separate phase. It is important to share the most representative feed composition at design stage to account for all possible contaminants.
- Q55. What instrumentation is recommended for monitoring the performance of the absorbent vessel?
- A55. It is typical to measure differential pressure over the absorbent bed and the contaminant concentration at the exit of the vessel as a minimum. We would generally also recommend measuring the contaminant concentration at the inlet, as well as having sampling points within the vessel at 25%, 50% and 75% intervals to monitor performance. These can also be used to carry out a pressure drop survey if required.
- Q56. What is the maximum concentration of H₂S or Mercury is acceptable for fixed bed absorbent use?
- A56. There is not necessarily a maximum concentration for these contaminants. It is usually down to economic feasibility. For Hg, this is not commonly an issue as the amount of Hg is typical low and does not reach percentage levels. For H₂S, fixed bed absorbents are typically an economic choice for levels of sulfur below 500 kg/day.
- Q57. What is the recommended oxygen content in Nitrogen for bed purging activities to prepare vessel for unloading?
- A57. Nitrogen purity should be at least 99.9%. If lower purity nitrogen is the only option, JM should be consulted ahead of **PURASPEC** unloading and sulfided **PURASPEC** loading.
- Q58. What is the sacrificial material which can take care of liquid carryover?
- A58. It is a ceramic type material (alumina/silica) which has a high void fraction and high surface area to capture liquid carryovers and protect the bed. The high void space make it is possible to maintain low and stable pressure drops.
- Q59. What is the typical increase in flow rate acceptable to use the same bed to achieve the same product quality in case of capacity augmentation of existing plant?
- A59. Numerous factors can impact whether a bed can be operated above the design flow rate. A review by the catalyst vendor is required to evaluate if the bed can perform at higher flow rates and what is the impact. In many cases, a different product can be used or addition of volume will be required to meet a higher flow rate.

- Q60. What is the value of Delta P which I see I have a problem?
A60. The pressure drop of **PURASPEC** beds should not increase during operation. Therefore, if normalized pressure drop is increasing, surveying the vessel and upstream filter is essential to determine the root cause. The normalised pressure drop takes into account the impact of flow on the measured pressure drop and normalises the value against the design flow rate.
- Q61. What's the recommended operating temperature range and pressure range? How does temperature & pressure impact H₂S and COS removal?
A61. Typically, the operating temperature range is 0-100 °C and pressures from atmospheric to 200 barg. However, operating beyond these ranges is possible and can be studied at the design stage. For H₂S and COS removal, the higher the temperature the better the kinetics. Product selection will take into consideration temperature for desulfurisation duties.
- Q62. Why is the breakthrough observed before complete bed saturation?
A62. It is because of the mass transfer zone (MTZ) required for the reaction. The MTZ is the length of bed required to go from the inlet concentration of contaminant down to non-detectable levels. **PURASPEC** products have sharp kinetics and thus a small MTZ. As the reaction progress and the bed become progressively saturated, breakthrough will happen when the MTZ reaches the bottom of the bed. With a lead-lag configuration, that MTZ can be captured by the lag bed, allowing the lead bed to run until it reached its maximum capacity.
- Q63. You discuss the O₂ impact on mercury removal. How does O₂ impact sulfur removal?
A63. As H₂S is removed, the oxidic material converts to a sulfide and therefore reaction of O₂ with the sulfided material creates sulfate and this reaction is exothermic leading to temperature rise. See A18.
- Q64. You mentioned putting some extra ceramics at the top of the bed for extra protection, is this for dust or for liquid?
A64. A ceramic sacrificial layer at the top of the vessel is mainly to protect the bed from liquid carryover. For dust, the best protection is a dust filter upstream of the bed.

Further Information

Please contact your local Johnson Matthey representative for further information or send your enquiry to polly.murray@matthey.com.

All information comprised in or relating to the contents of this report shall be treated by the parties as confidential and shall not be disclosed to third parties by any party without the prior consent of the other.

The information, data and results contained in this report together with any suggestions, advice and/or recommendations contained therein (individually and collectively "the Information") are given in good faith but Johnson Matthey does not warrant or guarantee the correctness, adequacy or suitability of the Information and it shall be the sole responsibility of the customer to assess the Information and whether or not to accept, act on or implement the same. If the customer does elect to do so, then Johnson Matthey shall not have any for any loss or damage suffered as a result, whether in contract, tort (including negligence) or otherwise (other than that arising from death or personal injury caused by Johnson Matthey's negligence).