



Plant Manager+ Series 1-50

As featured in **nitrogen
+syngas**

Technical discussions on urea plant issues
and operating problems, sharing experiences,
providing advice and solutions.

BCInsight

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UreaKnowHow.com
Where the Urea industry meets

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This is the first in a series of discussions from a selection of round table topics discussed on the UreaKnowHow website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers. The first subject to be discussed is the problem of foaming in the purification and concentration sections of a urea plant.

Problem No. 1 Foaming in purification and concentration section

The heart of any urea plant is the high pressure synthesis section where the two feeds, ammonia and carbon dioxide, are converted to urea with water as a byproduct. As this conversion reaction is an equilibrium reaction, a purification/recirculation section is needed to separate urea (and water) from the more volatile non converted feeds. Downstream of the recirculation section, urea and water are separated in a concentration/evaporation section (see picture). Under vacuum conditions water is evaporated and the urea melt is concentrated to about 96 or 99.6 wt-% depending on the type of finishing section present.



Mr Muhammad Ahsan Sarfraz of Fatima Fertilizer Limited in Pakistan posts a serious operational problem, which he experienced in the Pak-American Fertilizer Limited urea plant in Mianwali, Pakistan, where he previously worked:

We have been experiencing a strange problem in our urea plant. It is now the third time that all the sight glasses of the high and low pressure decomposer, concentrators and flash tank start have indicated a sort of soap foam. When this phenomenon happens we experience higher free NH_3 in the product but all the operating parameters of the synthesis and concentration section remain the same.

No abnormality in lab results are observed indicating slippage of NH_3 to the concentration section. Moreover the operating vacuum pressure in the concentrators remained the same i.e. 190 mm Hg and 45 mm Hg respectively.

The high free NH_3 in the product doesn't affect the vacuum and the operating conditions of the section also remain the same.

Is there anybody who can help me out?

Mr Majid Mohammadian of PIDEMCO in Iran indicates a likely cause:

It may be due to oil penetration into the urea plant by ammonia or CO_2 .

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands asks for some more specific information:

Majid, do you know if foaming can be caused by any kind of oil or are certain types of oil more of a problem than others?

Mr Ahmed Selim of Alexandria Fertilizers Company in Egypt confirms the cause indicated by Mr Mohammadian, asks for further clarification and provides a valuable additional consequence:

After this phenomenon, are you facing high recycle pressure in the low pressure carbamate condenser and flash tank condenser?

As Mr Majid mentioned, it may be oil leakage from the CO₂ or syngas compressors in the ammonia plant, so I think this may also greatly affect stripping efficiency causing severe fouling in stripper tubes.

Mr Sarfraz replies to the responses he received from his original post and confirms that oil can be a cause, but that he is looking for another possible cause like, for example, alcohols or formaldehyde:

The effect of oil contamination has been monitored very carefully on a regular basis as we have witnessed this problem last year when we got oil contamination from our NH₃ feed pumps. Since then, we have paid special attention to oil carry over and regular oil samples in the NH₃ and CO₂ and all the streams of the synthesis section are being carried out.

Moreover it is very evident that this oil disturbs the falling film in the HP stripper resulting in a low stripper efficiency. This would lead to high NH₃ slippage towards the recovery/purification section. But we don't face such a problem.

All the results from the stripper outlet streams are well within the desired range. The only thing we have whenever this foaming occurs is that we start to have high free NH₃ in product but the system vacuum pressure remains the same.

Is there anyone who could guide me as to the possibility of this phenomena caused by alcohols or formaldehyde?

Mr Abdul Raouf of Fauji Fertilizer Company in Pakistan confirms that foaming can also be caused by organic matter. He provides two valuable remedies and confirms that the indications are similar to the ones as experienced by Mr. Sarfraz:

We also faced this problem at our Snamprogetti urea plant due to carry over of organic matter with CO₂ from the aMDEA CO₂ removal section. We have installed a CO₂ washing system at the CO₂ compressor and also dose Amerel* in the process condensate treatment section to counter foaming.

This foaming does not affect the synthesis section or any other product quality related problem but causes operational problems. Carry-over of PCT section distillation tower is most common of all and also erratic level indications of all holders.

*Amerel® is a registered trademark used for defoamer and anti-foamer, in particular, for alkanolamines gas scrubbing systems.

Mr Brouwer of UreaKnowHow.com suggests another cause for foaming:

There is a rumour that N/C ratio is also a parameter in foaming: A high N/C ratio may cause foaming.

Mr. Nasir Hussain from Pak Arab Fertilizer Company in Pakistan makes a suggestion why the free ammonia content is increased due to this foaming problem:

This may be because of matter which is less volatile than ammonia. This material, strips in the recovery sections, leaving unrecovered NH₃, which ends up in the prilling section. ■

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Problem No. 2 High biuret level at low plant loads



Bitter harvest: biuret in urea fertilizers has been seen to damage citrus foliage.

Biuret is formed from urea according to the following overall reaction: $2 \text{ urea} \rightleftharpoons \text{biuret} + \text{ammonia}$. In fact this reaction proceeds in three steps: When heating a concentrated urea solution in water or a urea melt, urea is present in two forms, a keto and an enol form. The enol

form of urea primarily decomposes into NH_3 and HNCO (cyanic acid). Cyanic acid reacts with urea to form biuret.

All these reactions are equilibrium reactions. Biuret formation is favoured by higher residence times, higher temperatures and lower ammonia partial pressures.

Biuret is considered an impurity in urea when

used as a fertilizer as it has been identified as injurious to citrus foliage and to certain germinating seeds, but the level at which biuret is damaging to plants is disputable. The earliest urea plant designs produced urea with up to 5 wt-% biuret. From 1955 to present, many urea plants were designed to produce low-biuret product with not more than 0.3 wt-% biuret by means of a crystallisation section. The lower biuret concentration was found to be safe for these special applications which amount to about two percent of total urea fertilizer consumption.

Modern plant designs are successful in producing urea prills and granules with 0.8 wt-% biuret. These plants can handle 98% of the urea market with simpler and more cost-effective urea finishing designs that produce higher quality products with better storage and handling characteristics.



Mr Mohammed Al-Jeshi of Safco

Mr Mohammed Al-Jeshi of Safco in Saudi Arabia posts an interesting operational question:

What is the relationship between plant load and biuret content in the final product? Are there any charts readily available?

Mr. B.P. Suresh of Nagarjuna Fertilizer and Chemicals Limited in Kakinada, India replies:

Biuret formation depends only on temperature and residence time. There is a relationship between plant load and biuret formation with respect to residence time. In case of increase in load, the residence time will decrease and biuret formation will also decrease.

Mr. Mark Brouwer of UreaKnowHow.com in the Netherlands asks for some more specific information:

Does anybody have a graph available derived from practical experience: biuret in product versus plant load? Or what is your biuret level at 60% plant load? I would also like to know of any "tricks" to reduce the biuret formation at low plant loads.

Mr. Waqqar Ahmed of Fauji Fertilizer Corporation in Pakistan replies with several valuable suggestions:

At reduced load, biuret can be controlled by the following measures:

1. Keep urea solution levels in holders as low as possible i.e. MP/LP/vacuum section.
2. Keep NH_3/CO_2 ratio slightly on higher side.
3. Maintain urea stripper temperature ~ 198-200°C for Snamprogetti technology.
4. For plants using prill towers, urea melt delivery line to the prill tower is normally jacketed with low pressure steam, here urea melt gets both

residence time and temperature, reduce jacketing steam in this line in case of low load operation.

5. If dust solution recovery is ongoing reduce its flow rate.

Mr. Ahmed continues ...

The maximum allowable limit of biuret in the final product is set at 1.0 wt-% at our plant.

In normal operating conditions biuret is maintained at < 0.9 wt-%, however, at low load operation it sometimes exceeds 0.9 wt-% but remains less than the set limit.

If the biuret content exceeds the allowable limit, shipment of product is stopped and urea is stored in bulk storage, from where it is recovered again in the form of solution.

Mr. Mohammad Rastegaryan of PIDEMCO, Iran asks for some clarification:

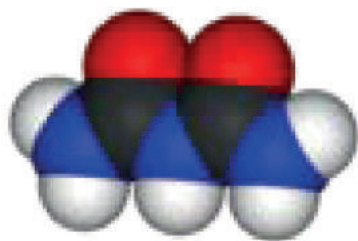
Is it possible to add on-spec urea in order to reduce the biuret content in urea storage.

Mr. Ahmed replies:

Off-spec product due to moisture, biuret, prill shape, fines etc is diverted in bulk store (separate from on-spec stored urea). A urea solution of ~ 70-80 wt-% concentration is prepared from the off-spec piles to recycle back to the vacuum section.

Mr. Rastegaryan suggests another solution to handle off-spec product:

You are right. but sometimes you can pour fresh, on-spec urea (directly from the plant) via a tripper over the off-spec product in bulk storage to achieve an acceptable range. In addition, when it is transferred to the bagging system it will be better mixed.



Biuret molecule:
the IUPAC name is
2-Imidodicarbonic
diamide

Mr. Muhammad Adnan Hanif of Fauji Fertilizer Corporation in Pakistan adds his experiences to the discussion:

I agree with you that the off-spec urea may be mixed with on-spec urea in such a proportion that the mixed product remains in the specified limits.

Recycling of off-spec urea by dissolving in water and then repassing it through processing units such as the vacuum and prilling section is not a good solution in term of product quality (as biuret will again increase) and energy utilisation in terms of steam in the vacuum section will also increase.

Then Mr. Al-Jeshi who started the discussions originally comes back and gives the background of his initial question. He wants to influence the biuret level in the final urea product as he likes to operate the urea plant at a lower load due to a shutdown of one of the ammonia plants.

I understand that biuret can be controlled in a Snamprogetti process by increasing the NH_3/CO_2 ratio. What modifications can be considered in a Stamicarbon process (CO_2 stripping) to control the product quality? We have one ammonia plant S/D and want to utilise all excess CO_2 from another ammonia plant to maximise our urea production. Unfortunately, the product quality is expected to be off spec with respect to biuret when our urea plant is operated at 70% load.

Mr. Majid Mohammadian of PIDEMCO in Iran replies with several suggestions and a question:

As you know in a urea plant biuret formation is caused by two factors: residence time and temperature. So, I have following suggestions:

1. Confirm the level measurement in the urea solution tank, evaporators, rectifier, HP stripper, level tank of rectifier outlet and suction of the melt pump and maintain these levels as low as possible.
2. Minimum recommended temperature in areas with higher than 50 wt-% urea concentration.
3. Higher N/C ratio if possible.
4. Higher capacity if possible.

At 70% plant capacity how much is your biuret content?

Mr. José Azócar of Fertinitro, Venezuela introduces another problem related to biuret levels:

At present, we are experiencing levels of more than 0.9 wt-% biuret content in the final product in one of our units, even when the plant load is 100%; we have tried to reduce this content doing all the

recommended procedures (lowering the level of the decomposers holders, lowering the temperature of the high pressure stripper etc.). We have considered taking samples at several points in both units to find out any differences between them. My question is: Is there any other procedure we should consider in order to reduce the biuret content?

Mr. Prabhat Srivastava of TATA Chemicals Limited in India shares his valuable experience:

Almost all possible causes of formation of biuret have been covered. No matter which urea technology is used, biuret increases with increase of urea concentration, high temperature and residence time. It is clear that temperature has a significant impact on the biuret formation. Other than this, if we run the plant load at low capacity and maintain the same operational parameters which we maintain at high plant load, then the residence time increases because of low flow velocities in comparison to a high plant load.

Also, if urea melt (which already has a high biuret concentration) is recycled it will have an impact on the biuret levels in the final product. To avoid this, if plant load is low than optimise the process temperatures in HP/MP and LP and maintain low levels wherever possible. Likewise in the vacuum and pre-vacuum section, where the rate of biuret formation is very high due to concentration, level can be kept low. An optimised rate of recovery can be lined up at low plant load.

Mr. Sam Q. Raman of Fertco in India introduces another interesting cause for biuret formation and provides also valuable suggestions to reduce biuret levels at lower plant loads:

Check the urea melt pump efficiency: If you have lower efficiency you will have a larger temperature difference between the evaporator and discharge temperature at the prill tower top. You could consider having two parallel melt lines to the prill tower. A reduced size line can be used for low load to increase the velocity.

Import urea solution from other plants (if you have second train of urea plant).

For a temporary load reduction the best option is still to store it as off spec product and mix it with on spec product in proper proportions.

Mr. Brouwer of UreaKnowHow.com in the Netherlands adds to it:

Also, check the temperature of the steam heating the melt lines. The melt temperature at the prilling bucket should be at a minimum to have minimum biuret. Biuret also increases crushing strength!

Has anyone experienced a difference in crushing strength between 0.8 wt-% biuret prills and 1 wt-% biuret prills?

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.

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Problem No. 3 High vibration in high pressure pipelines

In the high pressure synthesis section of a urea plant there are several high pressure pipelines prone to higher vibration. For example, the discharge lines of reciprocating high pressure ammonia and carbamate pumps or CO₂ compressors and pipelines with a large pressure drop e.g. pipelines from the high pressure synthesis section to the significantly lower pressure recirculation stages.

Special precautions are needed to reduce and control the vibrations in these lines and to ensure safe and reliable operation. It is not always an easy and straightforward exercise as shown by the round table discussions below.



FFC fertilizer complex
at Rahimyar Khan,
Pakistan



Mr. Muhammad Adnan Hanif of Fauji Fertilizer Corporation (FCC) in Pakistan posts an important topic, namely higher vibrations of high pressure pipelines:

This thread is to discuss vibration issues in urea plants especially:

- at high pressure section pipelines;
- CO₂ compression section, especially at final discharge line;
- high pressure NH₃ feed pump discharge line.

At our plant, we are facing the last two problems. Any suggestions to reduce them, especially at higher than design plant loads?

Mr. Bimal Bhayani of Gujarat State Fertilizers and Chemicals in India replies with several valuable practical suggestions:

As regards high pressure ammonia pump discharge piping problems:

- The pump must have suction and discharge dampeners to reduce the root cause of vibrations. If the booster pump to the high pressure pump is a centrifugal type the problem becomes easier to solve.
- Check the sizing of the discharge piping. Under-designed piping at higher load can increase vibrations.
- Discharge piping should be of long radius type and without sudden 90° bends.
- Use of wooden supports as supporting clamps in discharge piping before the discharge valve will reduce vibrations drastically as wood is a very good absorber of vibrations. We have successfully implemented this recently.

Mr. Kashif Naseem of Pak American Fertilizers in Pakistan has some additional suggestions:

Check that lines are properly designed as per desired load design. At high load under-designed pipelines show this kind of behaviour. If these lines are at the carbamate feed pump discharge, ensure that the solution specific gravity is as per design, because at low density this may happen.

Mr. Gamal El-Washahy of EFC in Egypt contributes to the discussions with his own experiences:

For ammonia lines check the temperature of the inlet ammonia as it should be between 15°C and 22°C. At lower temperatures

vibrations can occur. Also, if you have steam on dampeners you have to close it during normal operation.

Mr. Muhammad Adnan Hanif replies:

Yes our pump suction temperatures are higher than 22°C, but steam remains open in its suction dampeners. Why do you suggest closing the steam during normal running conditions, is it normal practice at your plant?

Mr. Anees Ahmad of Dawood Hercules Chemicals Limited in Pakistan contributes to the discussions with some practical solutions:

Vibration in pipelines can be due to many reasons. However, for dampening you can use rubber pads like engine mountings with a steel plate at the bottom.

Mr. Ahsan Muhammad Sarfraz of Fatima Fertilizer Ltd in Pakistan provides practical information about his experience and solutions:

We have also witnessed the same problem at Pak American Fertilizer Limited. For the ammonia pump discharge line we found that:

- Vibrations started at over 113% load (load kept same nothing could be done).
- We witnessed high oil accumulation in the dampeners from the pump. The oil was drained and the purge frequency increased to curb the oil accumulation in dampener.
- It was also observed that the major vibration is after the ammonia is preheated (from 30 to 60°C). We decided to reduce the ammonia temperature by 10°C and across same conditions there was no vibration later on. High temperature is indeed a factor contributing to higher vibrations.
- The supports of the line were strengthened.

Mr. Muhammad Adnan Hanif replies to Mr. Sarfraz as he now recognises the cause of the vibration problem in his plant and asks for some more clarification:

Thanks a lot. I am also of the same opinion with regard to the steam jacket in the suction dampener. Did you get the complete effect from just reducing the steam/temperature of the suction ammonia? However, when we cut off steam to the dampners in an 8 hour trial no impact was observed. In our case high vibration is more significant due to higher than nameplate

capacity plant load. Who is the vendor of your reciprocating ammonia pump i.e. Peroni, Ebara or Uraca?

Mr. Ahsan Muhammad Sarfraz provides some extra information:

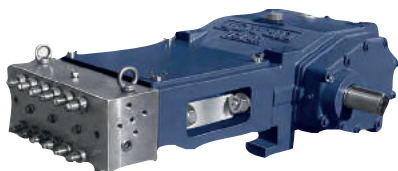
Our ammonia preheater is located downstream of the dampeners. It uses the hot condensates from the stripper steam saturation drum as heating media. In our case we came to the conclusion that the issue was more prominent at higher load together with high ammonia temperature. We therefore keep the temperature on the lower side while operating at higher ammonia load. Subsequently, after strengthening the line supports the issue has been resolved. So you can say that in our case with old supports it was due to high ammonia temperature. The pumps are manufactured by Peroni.

Mr. Joe Geronimo of Chemac Inc. in the USA, a key supplier of high-pressure equipment to the urea industry clarifies several issues with valuable background information and summarises the discussions with several solutions:

Regarding NH₃ reciprocating pump and pipe vibrations, 99% of the time excessive vibrations are caused by poor suction conditions. Here are some things to look at:

- NH₃ temperature and pressure. Liquid must be maintained in the suction line at all times. At 22°C you need minimum 9-10 bar otherwise the ammonia will flash. Check your pump data sheet regarding conditions of service.
- The suction dampener should be steam traced on the upper one-third section only, a gas cushion will then be maintained. You may want to install a level indicator to see where the separation point is in the bottle.
- Make sure the suction pipe is sized correctly otherwise liquid velocity will be too high and could cause cavitations.
- Make sure the pump is in good working order. Suction and discharge valves must seal 100%. If a valve fails the pump will vibrate excessively.

The discharge dampener will only help the piping, not the pump. Triplex reciprocating pumps have a normal flow variation of 7% above and 17% below discharge pressure. A correctly sized discharge dampener should reduce this to around 2-3% peak to peak on your discharge piping.



Triplex reciprocating pump.

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Problem No. 4 Gaseous ammonia and dust emissions in urea plants

The urea industry continuously strives to reduce ammonia and urea dust emission from urea plants. The European Fertilizer Manufacturers' Association has described the Best Available Technologies for emission reduction; please refer to "Best Available Techniques for Pollution Prevention and Control in the European Fertilizer Industry, Booklet No. 5

of 8: Production of Urea and Urea Ammonium Nitrate, 2000" which one can be downloaded from their website (<http://www.efma.org>).

The aim of this round table discussion is to collect the different solutions actually applied in urea plants to reduce gaseous ammonia and urea dust emissions.



Mr Mark Brouwer of UreaKnowHow.com in The Netherlands posts a topic on gaseous ammonia and dust emission points in urea plants, an increasingly important issue for urea plants worldwide:

Who has implemented a process to reduce gaseous ammonia and dust emissions from a urea plant? Can you provide a

general description (without confidential information)? Who is looking for a solution to reduce gaseous ammonia emissions?

Mr Waqqar Ahmed of Fauji Fertilizer Corporation in Pakistan replies with a valuable suggestion to reduce ammonia emissions from the medium pressure section:

In Snamprogetti's plant, inerts from the synthesis section are vented through the medium pressure section, which also carries ammonia with it. Inerts and ammonia vapours pass through the inerts washing column, where ammonia is absorbed/scrubbed with cold condensate. The vented mixture of inerts contains about 4.5-6% ammonia. To reduce ammonia losses we have recently modified the vapour distribution system in the column locally, which helped to reduce losses < 1%.

Mr Muhammad Adnan Hanif of Fauji Fertilizer Corporation in Pakistan adds another interesting suggestion to reduce ammonia emissions from the low pressure section:

In Snamprogetti urea plants, the most concentrated ammonia emission is from the low pressure section vent valve. In order to reduce the ammonia emissions, we provided a small by-pass line upstream of the final pressure control valve to the waste water section over-head condenser. This modification has reduced ammonia emissions significantly as the vent valve remains mostly in the closed position.

M. Majid Mohammadian of PIDEMCO in Iran proposes a new possible solution to reduce the ammonia emission from another source:

I have a proposal: Injection (spraying) of the treated process condensate outlet from the desorption section of the plant to the main stack of the urea plant in order to reduce the amount of waste ammonia from the stack. What do others think about this proposal?

Mr Mark Brouwer widens the discussion and asks:

What about ammonia emissions from prill towers and granulation scrubbers? Who has experience of reducing these?

Mr B.P. Suresh of Nagarjuna Fertilizer and Chemicals Limited in Kakinada, India describes their solutions to reduce urea dust emissions:

We implemented a dry-dedusting system two years ago for pickup of urea dust in the conveyor belt areas, which has reduced emissions fairly well. The process sequence is as follows: pickup points (chutes) → cyclone separator with blower → collected urea → urea dissolving tank. We are now planning to apply the same modification in our second plant. We have considered dedusting collection with hot water on top of the prill tower top and prepared a design for it.

Mr Mark Brouwer asks for some more details:

Who is the designer/vendor of the dry-dedusting system? With regard to the prill tower dust washer, do you operate a forced or natural draft prill tower? Which vendor are you considering? Do you have emission restrictions from the government?

Mr B.P. Suresh replies:

The dry-dedusting system is an in-house scheme, designed and implemented by us. The cyclone separator was manufactured in our workshop and only the blower was purchased from a vendor. This system collects urea dust from the belt conveyors at different pick up points.

For prill tower dust, a separate dedusting system with water circulation is available above the bucket floor. The collected dust is sent to the urea lumps dissolving tank and recovery to the system. We operate a natural draft prill tower with a free falling height of 75 m.

We have state pollution board regulations on prill tower dust emissions. The limit for urea dust in the atmosphere is 50 micrograms/m³ and we achieve around 13-26 micrograms/m³ as a maximum level.

Mr Muhammad Ahsan Sarfraz of Fatima Fertilizer Limited in Pakistan provides information about their dust scrubber on the prill tower:

In Toyo's ACES process they have a specially designed prilling tower. This includes an air scrubbing and washing system at the top of the bucket assembly room before the induced fans. It includes two packed beds, one circulating pump, one water sump and one spraying system to keep flushing demisters. The concentration in the sump is maintained at

nitrogen + syngas

The urea industry continuously strives to reduce ammonia and urea dust emissions...

30~40 wt-% urea circulated via the circulation pump onto the packed bed. This dedusting system significantly reduces urea dust/ammonia from the prilling tower. The dust emission is normally 20~25 mg/m³ of air. Ammonia in air would also be well in range as our ammonia consumption factor is 0.568 t ammonia per t urea after thirteen years of plant operation.

Mr Mark Brouwer asks for some more background:

It would be interesting to also check the ammonia content as the experience with other scrubbers is that with water only the ammonia emission is hardly reduced by the water. How do you measure dust? Is it done continuously?

Mr Muhammad Ahsan Sarfraz replies:

The circulating water is a basically a urea solution of 30~40 wt% concentration. We have a device made for the purpose. It sucks air at the outlet of the induced fans. The amount of urea dust deposits and volume of air sucked could be measured easily. The result would be in mg of urea/m³ of air. Toyo's prilling tower is the best I have seen so far in terms of emissions.

Mr Juan Jose Pestana of Soluciones Quimica P El Campo La Indu in Mexico provides another valuable solution to reduce ammonia emissions from the urea melt plant:

In our 1970 Snamprogetti plant, ammonia with inerts from the synthesis section are vented through the medium pressure section, along with off gas from the very low pressure system and sent to an ammonium nitrate neutraliser.

Mr Bimal Bhayani of Gujarat State Fertilizers and Chemicals in India finally provides another efficient and effective solution to drastically reduce ammonia emissions:

We have implemented an emission control scheme to recover ammonia from our two urea plants plus our two melamine plants. We recover ammonia from the off gases of these plants by scrubbing with 98% sulphuric acid in a column. We recover this ammonia as 35% ammonium sulphate solution which is finally recovered as ammonium sulphate by evaporation and then used as a fertilizer. We recover nearly 2.5 t/d ammonia in this system. Our final vent gases to the atmosphere are totally free from ammonia.

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.



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Problem No. 5 H_2SO_4 injection in urea melt

The urea industry continuously strives to reduce ammonia and urea dust emissions from urea plants. The previous reported Round Table discussion (No.4) collected different solutions actually being applied in urea plants to reduce gaseous ammonia and urea dust emissions. This time a new, interesting and challenging possible solution, injecting sulphuric acid (H_2SO_4) in the urea melt, will be

discussed. This solution was tested at Phu My Fertilizer plant in Vietnam and immediately prompted a lively discussion in the UreaKnowHow.com Round Tables. The mixing of urea with sulphuric acid was already the subject of research programmes in the early 1980s and the information available in the public domain was helpful in gaining a better understanding of the phenomena observed.



Mr Le Ngoc Ban from Operations at Phu My Fertilizer plant in Vietnam posts an interesting experiment in the Round Tables:

Today I injected sulphuric acid (H_2SO_4 98 wt-%) into the urea melt solution at the top of the prill tower. The injection point is at the suction of the urea melt pump. The problem is that the current and discharge pressure of the pump are gradually

decreasing when the acid flow rate is gradually increasing. What happens with the melt pump? Does it cavitate? Does anyone have experience of this?

Mr Muhammad Farooq of Pak-American Fertilizer Limited in Pakistan asks for some clarifications as this experiment is quite unique in the industry:

Can you tell us why 98 wt% H_2SO_4 has been injected into the urea melt? What are the expected benefits?

Please provide more details of your experiment.

Mr Mark Brouwer of UreaKnowHow.com in The Netherlands also requests additional information and provides some initial suggestions:

An interesting experiment!

How much sulphuric acid did you inject?

Do you want to reduce the ammonia emission from the prill tower or do you want to produce urea/AS product?

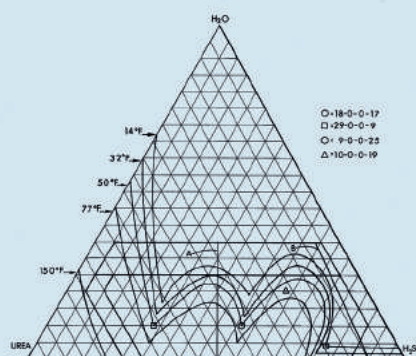
I would expect the formation of ammonium sulphate, which is able to dissolve in a urea melt up to a certain percentage.

I guess the density increases when injecting H_2SO_4 which should lead to a higher current at the same flow rate, but maybe gas formation occurs. Did the flow decrease when you injected H_2SO_4 ? Did the temperatures change?

Mrs Saima Abdul Rasheed of Pak-American Fertilizer Limited in Pakistan replies with valuable information:

The reaction between sulphuric acid and urea is highly exothermic (US Patent No. 4445925 and 4310343). Some vapour formation might have occurred.

Solubility in the system urea – sulphuric acid – water



Mr Le Ngoc Ban replies to the questions raised earlier:

To Muhammad Farooq: The purpose of this injection is to reduce the ammonia content in the prilling top as per environmental requirements.

To Mark Brouwer: The H_2SO_4 injection flow rate is 0.11 m³/h (design), but when I did this test, I increased the flow rate in three steps: 0.04 m³/h; 0.08 m³/h; 0.11 m³/h.

- Yes, I want to reduce the ammonia emission from the prill tower exhaust.

- Yes, the urea melt temperature increased by about 2.7 - 3°C resulting in a biuret increase!

When I increase the acid flow rate, the current of the melt pump decreases so I think the density of the urea melt decreases. You said that gas formation occurs, but what is the gas? When I stop the acid injection the current and discharge pressure of the melt pump increases again.

Steps: 0.04; 0.08; 0.11; 0.00 m³/h

Current: 117; 115; 113; 119 A

Discharge pressure: 13.4; 13; 12.8; 14.5 bar

Biuret content: 0.97; 1.07; 1.09; 0.95 wt-%

The plant load did not change.

Mr Muhammad Farooq suggests an alternative to bypass the problems:

I would like to suggest that you inject H_2SO_4 in the discharge line of the urea melt pump near the prilling bucket by using an acid dosing pump. Do you have any washing/scrubbing system at the top of your prill tower?

Mr Muhammad Naeem of the Technical Services Department of Pak-American Fertilizer Limited in Pakistan contributes to the discussion with a valid remark:

I think the proper way to inject any kind of fluid irrespective of whether it is UF-85 or H_2SO_4 is to inject it at the suction of the urea melt pump to achieve a better mixing of both fluids.

Mrs Saima Abdul Rasheed replies to Mr Le Ngoc Ban's question:

The vapours formed are water and the acid.

Mr Mark Brouwer elaborates further:

Yes and urea can also decompose into NH_3 and $HNCO$ gases: this reaction might also be promoted when H_2SO_4 reacts with NH_3 . Plus, when the temperature increases, more vapours form.

Mr Le Ngoc Ban raises some more questions:

Do you have any documents about H_2SO_4 injection into urea melt? What kind of side reaction happens in this solution?

How do you explain the increase in biuret content?

And why does the urea melt pump cavitate?

Mr Mark Brouwer replies:

Please study carefully the patents indicated by Saima Abdul Rasheed. These patents mention exothermic reactions and even explosion risks. As by-products CO_2 , NH_3 and toxic components like ammonium sulphamate and sulphamic acid are mentioned. Be careful, when the ammonia concentration reduces due to reactions with sulphuric acid, the biuret formation reaction: $2 \text{ urea} \rightleftharpoons \text{biuret} + NH_3$ goes to the right side. This leads to more biuret. More gases cause cavitation of the pump.

Mr David Herrero from Operations at Fertiberia, S.A. in Spain gets involved in the discussion and provides valuable new information:

One can find several patents on the web regarding sulphuric acid injection into urea melt. They claim that the ammonia emission from the prilling tower could drop to 5 mg/Nm^3 without further air treatment. The biuret content will increase slightly and the final content of the ammonium sulphate would be around 0.5%. The water content would not be affected if 98% sulphuric acid is used. The point of injection is at the pump suction line to assure correct mixing and reaction with the free ammonia. If enough gases are present due to the reaction, cavitation, of course, will take place.

The ammonium sulphate content makes this urea only valid for agriculture (not industrial grade). As far as I know, there was limited real experience (I guess this would have been more widely practiced if it was very successful).

Did you run the trial for a long enough time to enable the ammonia emission to be measured? If so, what were the values you got?

Mr Ahsan Muhammad Sarfraz of Fatima Fertilizer Ltd in Pakistan asks a few more questions:

Really interesting experiment, but may I ask what the present level of ammonia emission at your prilling tower is that you are trying to control? In addition, what was the impact on the ammonia emission of the tower during the time you had dosed the acid (did you measure the impact?).

What emission level are you aiming for – TEC and Stamicarbon prill towers usually operate at around 25 mg/Nm^3 . We are operating at 14 mg/Nm^3 .

Mr Le Ngoc Ban replies:

The test lasted for 7 hours, from 9 am to 4 pm.

Steps: 0.04; 0.08; 0.11; $0.00 \text{ m}^3/\text{h}$

NH_3 : 78; 49; 89; 94 mg/m^3

Actually, the ammonia level at the prill tower depends on where you take the sample. In our case, the highest level is 94 mg/m^3 and the lowest level is below 60 mg/m^3 . My target is to reduce the level of ammonia to below 60 mg/m^3 .

By the way, do you think the prill hardness could improve after H_2SO_4 injection?

Mr Basheer Al-Awami of SAFCO in Saudi Arabia contributes some new and interesting aspects to the discussion:

It is an interesting subject. Injection of H_2SO_4 or ammonium sulphate is going to improve the mechanical strength of the urea prills and also reduces the ammonia emission. However, one should be worried about the corrosion that may occur in the prilling tower structure. Also the colour of the final product may appear yellowish, which may be not accepted by farmers.

Mr Vedantam Srinivas of SAFCO in Saudi Arabia contributes to the discussions with an interesting alternative to reduce the ammonia emission:

You were doing a good experiment.

Do you have a dedusting arrangement in the prill tower top?

What is the height of the tower and flow of air through the tower?

Based on the above, you may consider using water or H_2SO_4 as the scrubbing media. Ammonium sulphate can also be produced as a byproduct.

Mr Le Ngoc Ban replies:

We do not have a dedusting system in the top of the prill tower.

Mr Gh. Serpoush of R&D of Shiraz Petrochemical Company in Iran confirms the biuret increase and raises another question:

OK, the urea melt temperature increases about $2.7\text{--}3^\circ\text{C}$, which increases the biuret content.

What is the ratio (mole H_2SO_4 /mole urea) in your test or how many tonnes of urea were prilled during the test?

Mr Le Ngoc Ban replies:

The plant load when I did the test was about 99 t/h , you can calculate the H_2SO_4 /urea mole ratio.

Now we are awaiting the result of the prill hardness test. We hope that the hardness will be improved.

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Problem No. 6 Choking of urea fluid bed granulator nozzles

The nozzles used in urea fluid bed granulation form the most essential part of a fluid bed granulation plant. Each licensor has developed its own specific nozzles and its features have a significant impact on the performance and efficiency of the granulation plant. Common to all nozzles is the mixing of urea melt with air although in which way the mixing is taking place differs widely between the different technologies. In Uhde Fertilizer Technology's fluid bed granulation, the nozzle produces tiny droplets which solidify and join the seed material by an accretion process leading to a very hard granule. Stamicarbon's fluid bed nozzle produces a layer of urea melt, which solidifies on the seed material forming an "onion" kind of granule leading to a very spherical shape. In Toyo Engineering

Corporation's fluid bed granulator each nozzle forms a spouted bed, where vigorous mixing occurs, leading to round, uniform granules. Urea Casale applies a nozzle, which generates very fine droplets due to the fact that in the nozzle an emulsion of urea melt and air is formed, which is sprayed out of the nozzle. The very fine urea melt droplets make it possible to apply a urea melt feed with a higher water content to the granulator as the water is able to evaporate from the fine droplets.

This round table discussion discusses the problem of nozzle choking: What are the causes and solutions? From several rounds of questions and answers the problem area and a most likely root cause has been identified, which could solve the problem for urea producers.



Mr Saadat Motamedi of Operations at Pardis Petrochemical Company in Iran introduces to the round tables his problem with choking fluid bed urea granulation nozzles (see photo) and asks:

What do you think is the cause of the choking as shown in the picture? Is it from the urea melt or atomisation air stream and why?

Mr Prabhat Srivastava of TATA Chemical Limited in India

replies: Seeing your pictures it is my assumption that it may be possible that a particular nozzle gets partially choked and the urea melt/air mixture could not exit with the desired velocity.

Mr Ramchandra Nesari, Freelance Consultant in India adds the following valuable information:

The choking of the nozzle can be due to the following reasons: Trapping of foreign material in the spray nozzle internals may result in a change in the atomization profile of the urea melt. Once this starts, the choking of the nozzle starts building up and finally the nozzle gets choked completely. Also, sometimes, the internals of the nozzle get damaged due to foreign material which also changes the atomization profile of the urea melt.

That is why the atomization profile of nozzles in a row is normally observed through an inspection window and any malfunctioning nozzles are noted down. Choked nozzles are inspected during a short shutdown of the granulator and choked nozzles/internals are replaced with spare nozzles. What were your observations of choked nozzles/its internals?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands posts some more questions:

A very nice topic and good discussion. I would like to ask a few more questions:

Is this problem at certain plant capacities only?

Did this problem occur in the start up period of the plant or only after a certain period?

How long can you keep the granulator running before this problem becomes too big. Is this problem determining the operating time between stops?

Mr Majid Mohammadian of PIDMCO in Iran also adds some

questions: In addition to Mr. Brouwer's questions please let me know the following: After what period of time were you faced with the problem?

How many choked nozzles did you find in each bed?

As you know the type of choking in nozzles varies, so which is most typical?

Mr Saadat Motamedi replies: The problem started after only two days in service. There were four choked nozzles in each bed. After opening the atomisation air stream line, we realised that we had a special type of congestion (choking) that could not be solved with steam condensate and has the characteristics of plaster.

Mr Majid Mohammadian asks further: Did you find the choking problem in the main header of the atomisation air or in the inlet of choked nozzles?

Mr Saadat Motamedi replies: Choking was not in the main header but it was at the inlet of the choked nozzles.

Mr Majid Mohammadian adds: In case you found the reason I think you can solve the problem because you should look for poor distribution or the reason for insufficient air on those nozzles.

The problem of nozzle choking

**Mr Ali Azhar of Technical Services of FFBL in Pakistan**

Referring to your comment “we found a special congestion (choking) that could not be solved with steam condensate and has a characteristic like plaster” I have the following question: Is this some sort of plastic or jelly-type material normally used for sealing purposes? RTV?

Mr Mark Brouwer adds some suggestions : It is also possible that there is a mechanical problem. For example, in the event that urea melt leaks into the injection air, it will cause the injection air to slowly clog. Proper attention needs to be paid to the Teflon closing rings. For example, always use new ones when installing a nozzle and use sufficient Teflon tape when installing the nozzles in order to ensure proper tightening. Another possibility is that debris is present in the atomisation air, but this typically happens only in the first weeks of operation. The things to do is clean the atomisation air pipes by hydro jet to remove the biuret and check the individual sprayers for urea leaks. If the problem occurs on only one nozzle and you find a leak it is merely an incident. If you have several problem nozzles, you should check all nozzles and do a leak test procedure.

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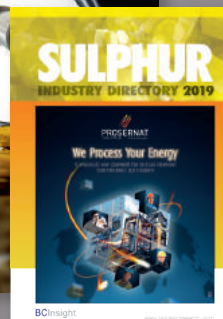
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Problem No. 7 Urea melt pump damage

The urea melt pump is a challenging application of a centrifugal type of pump: On the discharge side it has a relatively high pressure as the urea melt needs to be pumped up to the top of the prilling tower, which is typically some 100 metres high; while on the suction side vacuum pressure exists. Furthermore, the urea

melt needs to be pumped with a minimum residence time to minimise biuret formation, so the residence time at the suction is limited. Finally the urea melt can easily crystallise transforming into biuret, triuret or further poly-urea products, which can cause cavitation and damage the pump.



Mr Muhammad Usman of the Process Engineering Department of Fauji Fertilizer Company in Sadiqabad, Pakistan introduces his problem of damage to the urea melt pump:

We have a centrifugal pump for urea melt at the bottom of the second vacuum concentrator, which pumps around 99.7 wt-% urea melt to the prilling tower.

Urea polymer lumps frequently falling in the melt pump suction line are causing cavitation of the pump and in a recent incident even damaged the pump shaft. Using a suction strainer to avoid lumps in the pump suction proved to be unsuccessful, due to its choking. Frequent flushing of the system is carried out; however, the issue is ongoing. Do you have any suggestions to improve the problem?

Mr Juan Jose Pestana of Operation Department of Soluciones Químicas para el Campo y la Industria in Mexico has also experienced this type of problem in his plant:

After a lot of problems of this kind we had to open the second stage vacuum separator and make a manhole to take a look and clean up all the polymer urea. This was a lot of work for us and I think there is a better solution. If you cannot wait for a longer shutdown, try the following:

With a plant shutdown close all steam to ejectors and decrease the cooling water pressure (we took off one cooling water pump). Completely fill the vacuum separator with hot water and when water comes up to the vacuum condenser, feed steam to the evaporator heater to boil (5 to 10 minutes). Maintain a small flow of water with this operation. After this, drain all equipment. Operate your urea solution pump until all water remains clean.

Mr Nour Dw. of Delta Co in Egypt provides some recommendations to avoid damage of the urea melt pumps:

Adjust the temperature of the urea melt and the vacuum pressure in the vacuum separator to avoid these kinds of problems according to the % H₂O and % biuret analysis of product. Typical conditions in the second evaporation stage are 0.03-0.04 kg/cm² and 138-140 °C. High vacuum pressure may damage the urea melt pump, especially in cases of low load of the evaporation unit.

Mr Yousaf Mehboob of Production Department of Fauji Fertilizer Company in Pakistan offers another possible solution:

If you have frequent problems of lumps falling in the urea melt pump suction, you should install a grid plate inside the second stage vacuum separator holder neck to prevent lumps from travelling to the pump suction side. The lumps will dissolve on the grid without disturbing the process and equipment.

Mr Muhammad Farooq of Pak American Fertilizer Ltd in Pakistan introduces several valuable suggestions:

You need to identify your site conditions. The following points are important:

1. Proper level of urea melt in the final concentrator holder. If the holder contains bubbles, the level set point in the holder needs to be increased to avoid pump cavitation. Increasing the level set point will result in less damage.
2. Ensure that the pump never runs without urea melt if polymer urea lumps fall – then the pump needs to be stopped immediately or put in standby in service within the shortest possible time.
3. Ensure the urea melt liquid has the right density (to avoid pump cavitation).
4. Avoid quick changes in vacuum system during vacuum pulling/breaking.
5. Check the diameter of the shaft and consult a mechanical expert to review the diameter, sleeve, impeller nut etc.
6. Check that the free ammonia content in the urea melt is as per design.

Mr Girish Prakash of Tata Chemicals Ltd in Brabala, India introduces another solution which has given them excellent results:

The easiest and safest solution we have found to the problem is to have periodic urea melt dome flushing (preferably fortnightly). The line for flushing is drawn from the melt pump discharge. The results obtained were excellent. In addition to this you can install a conical perforated suction strainer (like the one installed in lube oil pumps) in the melt pump suction line.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands asks a further question:

Are prilling plants (two stage evaporator steps) more prone to this problem than granulation plants with a one stage evaporation step?

Mr Nour Dw shares his positive experiences with urea melt pumps:

In 18 years we have never faced this problem in our prilling plant. We have no strainer in the suction line and we always monitor the level indicator in the suction line, located nearly one metre above the pump. We control the level as low as possible and monitor vacuum and temperature (one point at the suction side and another before the bucket with alarms and achieve good specifications for biuret and water content. We usually open the discharge valve located before the bucket 100% and maintain a normal flow rate of urea melt to the evaporation section.

Mr Le Ngoc Ban of Operation Department of Phu My Fertilizer plant in Vietnam asks about lump formation in the first stage evaporator:

I agree with Girish 100%, we had the same problem and solved it by weekly urea melt dome flushing. But we find it is difficult to clean the first vacuum separator dome because we do not have a urea melt line to the first vacuum separator. Do you have experience of first stage vacuum washing?

Mr Girish Prakash replies to the previous questions:

You can take a branch line from the line for second stage flushing with proper flushing connections and interconnecting valves and then do flushing of both the stages in any sequence you want. As for Nour's observation, the polymer formation is more severe in plants with pre-vacuum concentrators.

Mr Le Ngoc Ban asks further:

Did you modify your vacuum washing system?

Mr Girish Prakash replies again:

Yes, you need to modify the separator dome flushing system.

Mr Muhammad Umair Ali of Production Department of Fatima Fertilizer Company in Pakistan adds his experience:

The best way to tackle this problem is to avoid the formation of polymer urea lumps and I am in complete agreement with Girish that urea melt flushing once a week will give you excellent results. At Fauji Fertilizer Bin Qasim we employed urea melt flushing, which is recommended by Stamicarbon, and we are satisfied with the results.

Mr Muhammad Usman asks a further question:

We perform flushing of the first and second stages fortnightly with hot condensate. The operating conditions of the concentrators are 137-142°C and 0.5 and 0.06 kg/cm² (abs). We are also operating with a pre-concentrator. This adds to the formation of polymers in the vacuum stages. One option i.e. the use of melt urea from pump discharge for flushing could be useful as mentioned earlier.

However, would installation of a grid in the second stage help? How can we avoid choking of the grid in the case of frequent falling lumps. Would it add to the flow starvation of the pump?

Mr Rajeev Gupta of Urea Operations at GPIC in Bahrain contributes to the discussion:

As Girish has said, wash the top domes at regular intervals. Urea melt is better as it has been observed that it dissolves the urea polymers better at the higher temperature. In case a polymer lump has fallen down, watch the urea melt pump discharge pressure. If the temperature is increased, it will dissolve faster. In cases where there is no provision of urea melt to wash the domes, condensate can be used until urea melt can be made available in next stoppage. It must be noted that a special sprayer is required that must be obtained from a reputable licensor.

It is also worthwhile to investigate if the melt pump rotates in reverse direction in case the pump loses suction level. Normally a level control valve can be closed temporarily to close the discharge control valve. Do it as early as possible to ensure avoidance of reverse rotation. This can be realised by interlocking with the discharge pressure transmitter and current drawn in series.

Mr Majid Mohammadian of the Engineering Department of Pidmco in Iran provides some more valuable suggestions:

Muhammad Usman, I agree with you regarding the reason for this problem. As you know, due to vacuum in the evaporator separators we are faced with physical entrainment phenomena and thus we should reduce this entrainment to control the lump formation in the top part of separators.

Normally there is a Chinese hat (impingement plate) in the separator to control the urea melt entrainment. However, if the diameter of the Chinese hat is insufficient, some urea melt droplets can escape, eventually resulting in the formation of lumps. One solution therefore is to check the diameter of this Chinese hat and if required increase the diameter to reduce the entrainment.

The heating coil in the separators should also be checked. Urea melt circulation is a good solution if you can control the biuret increase accordingly.

Mr Muhammad Kashif Naseem of Process Engineering at Safco in Saudi Arabia gives his suggestions:

The most efficient and valuable solution for the removal of urea polymers is to install a urea melt flushing system sprayer. It will not add any additional components to the urea melt and waste water system and the plant will run smoothly.

If time is available before start-up of the unit, e.g. after a shut down, water flushing should be carried out.

Mr Waheed Ahmad of Urea Operations of Fauji Fertilizer Company in Mirpur Mathelo in Pakistan adds:

We faced this problem after a plant up-rate. Our new pumps were of high capacity and we were operating below the normal flow. Our flow has now normalised.

Check the level in the suction of the pump. Increase it and check your suction level transmitter.

The grating should be inside the second stage vessel to avoid lumps in the pump suction, as in the latest designs.

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Problem No. 8 Urea prilling buckets

Prilling buckets are the oldest technology to form solid urea in a urea plant. In the top of a concrete prilling tower a bucket with numerous holes rotates releasing urea melt droplets, which cool and solidify against the rising air. At the bottom of the prilling tower solid urea prills are collected and sent

to storage. It is estimated that about 80% of all urea plants worldwide produce prills. Various designs of prilling buckets are available in the market. What are the design criteria for prilling buckets and what are the experiences with the various designs?



Mr Girish Prakash of Tata Chemicals Ltd in Babrala, India introduces an interesting topic to the Round Table: What are the differences in technical features and performance of prilling buckets designed for Saipem and Stamicarbon plants? What are the design considerations for the prilling bucket?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands replies:

When designing a prilling bucket the following criteria are important:

- size distribution of the prills should be as even as possible
- maximum usage of the prill tower cross section to maximise
- heat exchange between prills and air.

Typically, prills are designed to have an average prill size of 1.7 mm. I would like to suggest the consideration of a larger diameter (for example 1.8 or 1.9 mm) as the design figure. This will only influence the prilling tower height and will give much more flexibility to increase the capacity of the prilling tower later and reduce caking problems. Prills from CIS countries are typically 1.9 mm average and are suitable for export. I have never noticed much difference between the performance of prilling buckets from different licensors.

Mr Muhammad Adnan Hanif of Fauji Fertilizer Corporation in Pakistan shares his experiences: I totally agree with Mark in view of taking a higher average prill size as a design basis. We have practical experience of this with our two plants having different prilling tower sizes. The bigger tower (designed for a lower load) is performing better at higher production rate at the same inlet conditions. The product quality is also relatively good for the bigger tower.

The prilling tower diameter is also important for even heat distribution with air.

Mr Girish Prakash specifies more in detail his interests:

The basic design criteria for prilling buckets and towers are correctly mentioned and remain independent of the technology licensor. But what I am interested in is the other criteria specific to prilling buckets like:

1. L/D ratio of the bucket and its impact on the prill quality
2. Type and size of holes on the bucket surface and its impact on prill granulometry.

As far as I know, and correct me if I am wrong, there are no tangential holes on the prilling buckets supplied for Stamicarbon plants in contrast to Saipem plants where the bucket surface has both tangential and straight holes.

Mr Mark Brouwer replies: I have seen Tuttle buckets and Stamicarbon buckets. I think with Saipem buckets you mean Tuttle? The L/D of Stamicarbon buckets is somewhat larger than that of Tuttle buckets.

I have never seen any differences in prill quality because of this. Other parameters are more important like correct design, operating with optimum speed, urea melt concentration and prill temperature at bottom of prilling tower. I believe neither buckets have tangential holes, all are straight drilled.

Mr Nauman Talat of Pakarab Fertilizer Limited in Pakistan

introduces vibro prilling: Please let me know if any one of you has worked on a vibro priller. Please share your comments regarding this prilling technique:

- What is the impact of the vibro priller on average prill size?
- What is the impact of vibro priller on product temperature?
- Which vendors provide vibro prillers?
- Description of vibro priller unit.

Mr Henry King of PCS in the United States shares his

experiences with prilling buckets: We have replaced the Tuttle and Stamicarbon buckets with the vibration-assisted bucket designed and manufactured in Ukraine. The improvement in prill size uniformity and approach to the air temperature with this technology is quite significant.

Mr Bhupendra P. Mehta of Indian Farmers Fertiliser Co-operative Limited (IFFCO) in Kalol, India asks some more questions:

As you have operating experience with vibration assisted prill buckets, can you please tell us the following:

- Prill temperature at tower bottom before and after vibration assisted prilling bucket
- Exhaust air temperature before and after vibration assisted prilling bucket

Mr Muhammad Kashif Naseem of Process Engineering at Safco in Saudi Arabia introduces another question:

With a small diameter prilling tower and having induced fan technology, can we realize a more even prill size distribution (less under size <1mm) by increasing the air flow (increase of induced fan speed)?

Mr Gamal El-Washahy of EFC in Egypt shares his view:

I think a bigger prill size is better even for lower capacities as mentioned by others. A larger prill size will also help to reduce dust formation (an environmental issue), reduce product loss (financial issue) and affect product quality.

Mr Henry King provides more information about his experiences with vibration-assisted prilling buckets: In our plant the prill temperature leaving the tower at 100% load was about 8-10°C lower with the vibration assisted prilling bucket than with the Tuttle bucket, at the same ambient air temperature. The Uniformity Index of the prills increased from 55-60% (Tuttle) to 75-80%.

It is clear that the improvement is completely due to the effect of the vibration, because the small motor that induces the vibration onto the bucket can be switched off at any time without interrupting production. If this is done, the vibration-assisted prilling bucket is similar to the Tuttle bucket. The temperature of the air leaving the prill tower is not routinely monitored in this plant.

Mr Mark Brouwer asks further: When you replaced the old prilling bucket by a vibration-assisted one: How old was the original bucket, did it have internal vanes and was it designed for the current plant capacity?

Mr Henry King replies: The old prilling buckets had internal vanes and were 2-3 years old. They had been "tweaked" by Tuttle for higher rates. The vibration-assisted prilling buckets were provided by Technochim of Sumy, Ukraine.

Mr Muhammad Ahsan Sarfraz of Fatima Fertilizer Limited in Pakistan raises an interesting question:

We have asked Tuttle to design a prilling bucket for a mean size of 2 mm but the strength was so low that about half of the product turned into fines on the vibrating sieves. Now we are running it at lower average size. A Tuttle prilling bucket does have tangential holes in the bottom part and straight holes at the top. What is the maximum strength value for a prill size of 1.8~1.9mm?

Mr Mahmood Ahmad of FFC Goth Machhi in Pakistan gives his view on vibro prilling: If you use a Vibro Priller you will get a prill size of 2 mm but the product temperature will be high due to a low rpm of vibro priller (66-90 rpm).

Mr S.K.Gupta of Indian Farmers Fertiliser Co-operative Limited (IFFCO) in India asks another interesting question:

The prill temperature depends on the air flow through the tower, melt temperature, size of the prills etc. Will someone elaborate on how a Vibropill Bucket, producing same size of prills in a particular prilling tower produces prills at a lower temperature than a normal prilling bucket.

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.

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Problem No. 9 Unblocking pipelines after a urea plant shutdown

In several sections of a urea plant, pipelines can become easily blocked when temperatures become too low and crystallisation can take place. In the evaporation section, urea melt is present which crystallises at about 133°C when in the pure form.

Furthermore, urea can also polymerise to biuret, triuret and poly-urea with high crystallisation temperatures when urea is kept for a longer period at high temperatures. In addition to pipelines becoming blocked, the internals of several equipment items can also suffer from these problems

as shown in the picture (right).

In the recirculation section ammonium carbamate is present which crystallises at about 153°C when in pure form. And in the high pressure synthesis section, urea and carbamate mixtures are present. During normal operation the risk of blocked lines is small, however, during a shutdown of the plant, blocking can occur if the right measures are not taken. This Round Table discussion discusses which measures to take and what to do when a line is blocked.



Mr Manzoor Ahmad Taraqdar of Bangladesh Chemical Industries Corporation in Bangladesh initiates the discussion:

When restarting our urea plant after a shutdown, on several occasions we have experienced a line blockage. We are not sure whether the white material is ammonium carbonate or ammonium

carbamate. Normally this blockage can be removed by hot water. In cases where a water wash is not possible without dismantling the pipeline, we have tried flame heating of the pipe from the outside by removing the insulation. However, most of the time it did not work and we have had to dismantle the pipeline.

I would like to know the following:

- What is the white material?
- How do we avoid its formation after a shutdown?
- What is the standard method for unblocking lines?

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia asks:

Please define the area where you have found this material.

Mr Nasir Hussain of Pak Arab Fertilizers in Multan, Pakistan provides the first answers:

This white material could be urea or carbamate, both of which solidify upon cooling or when there is sufficient time for their solidification. A possible reason for this phenomenon could be the improper flushing of the sections during shutdown. Therefore, after shutdown flush all sections thoroughly with hot condensate.

Mr Manoj Chaturvedi of Star Aria Ammonia Company in Iran contributes to the discussion with his own experience and provides further recommendations:

This will either be urea or ammonium carbamate. The only solution to unblock the lines is by heating either directly by injecting condensate or steam into the pipeline or by indirect heating through flame or steam outside the pipeline. If the point of the blockage can be located it can be targeted. If the location of the blockage is unknown, some of the flanges in between the line can be opened and direct heating can be applied. I experienced the same thing

mostly in urea melt lines where the solidification is quick after shutdown if the drain line is also found to be blocked.

To avoid blockages, it is always recommended that in each shift the drains of all the critical lines which are subject to blocking are checked.

Mr Siddharth of Tata Chemicals Ltd in India contributes to the discussions with a valuable warning:

Be careful while unblocking the pipelines in the urea plant by flame heating because overheating or localised heating may cause thinning and damage to the pipeline. We experienced this when unblocking our reactor drain line.

Mr Zeeshan Shoaib of Fauji Fertilizer Company in Pakistan shares his experiences and recommendations:

We experienced a similar problem at our plant, but fortunately we were able to rectify the problem after a long struggle. It occurred in the discharge line of P-106 (a pump taking the suction from the pre-concentrator holder and sending the solution into the vacuum unit) when we applied the feed to the reactor after the shutdown. This blockage was caused by carbamate that had solidified due to poor flushing. We had to cut off the feed because the level was increasing and the solution reached the condensate tank. We have three recommendations:

- Apply flushing after heating the line.
- Ensure that the drain/sample point of that line is flushed.
- Finally, if the line has tracing it should be in service and ensure that the steam trap isolations are removed and that they are in service.

Mr Malik Sohail of Pak-American Fertilizer Limited in Pakistan asks a question related to blocking of internals of equipment items:

If carbamate solidifies in equipment like the carbamate condenser or in the high pressure loop equipment, can it be removed by hot water or should some other method be used to remove this solid?

Mr Ajkumar Kulkarni of RCF LTD in India summarises the recommendations and introduces an interesting solution to minimise the problem of blocking.

It is preferable to avoid the blockage problem by following a standard operating procedure.

- After shutdown, drain the solution.
- Circulate hot condensate and drain through all the drain points provided to confirm that there is no solution available / remaining in the system which can cause blockage.
- Ensure that the tracing lines are in line and steam traps are in line and in working condition so no condensate hold up happens.
- Dilute the solution (in case the solution is being held) with condensate.

I have a suggestion to identify the area where the temperature has dropped due to any problem: Provide skin temperature measurements for the most probable and difficult to access points. This is a low cost solution which can immediately detect any problem and helps to isolate/identify the problem area (blockage) to enable the problem to be dealt with quickly.

Mr Tarun Batra of National Fertilizers Limited in India confirms the importance of having a proper flushing procedure and asks for a solution for a blocked heat exchanger in the urea melt section:

We all know that pipelines in a urea plant are very prone to blocking due to solidification of ammonium carbamate/urea during unplanned stoppages of the plant when adequate flushing with hot condensate/steam of the pipelines has not been carried out.

Thus it is always preferred to have a foolproof arrangement of condensate/steam to adequately flush out the system after plant stoppages.

Another critical area is high concentrated (99%) urea heat exchangers in the vacuum stage. Concentrated urea solution, if allowed to remain stagnant at higher temperature in the heat exchanger, will result in pyrolysis of the urea forming biuret/triuret. This problem can arise say if 99% urea pumps lose suction due to any reason and steam to the heat exchanger is open and solution is stagnant in the heat exchangers for some time. The removal of this type of blockage is not possible by steaming.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands confirms the risk of overheating urea grade materials:

Please realise that heating up austenitic stainless steels like 316L UG or 25-22-2 and duplex steels can deteriorate the corrosion resistance of these materials.

For example heating 316L UG above 500°C will increase the corrosion rate. ■

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Problem No. 10 High CO₂ content in liquid outlet of a high pressure CO₂ stripper in urea synthesis

It is well known that the high pressure CO₂ stripper efficiency in a urea plant is defined by the ammonia content in the liquid bottom outlet. The ratio NH₃/CO₂ in the bottom outlet is determined by equilibrium equations. Problems have been experienced with a relatively high CO₂ content.

Several possible causes are explored. One cause could be that CO₂ slips through with the liquid bottom outlet due to a defective level measurement in the stripper bottom. Measur-

ing the liquid level is not an easy task due to the corrosive nature and the risk of crystallisation. Sometimes a delta-P level transmitter is applied with flushes in its legs, but this is not a reliable option. Radioactive level measurements are often used but still safety risks, complicated calibrating procedures, limited lifetimes and maintenance costs are significant concerns. The radar level measurement developed by Stamicarbon offers a new and reliable solution.

Mr Joko Raharjo of PT Petrokimia Gresik in Indonesia introduces an interesting topic to the round table:

In March 2010 we carried out some maintenance on our high pressure CO₂ stripper due to high NH₃ content in the liquid bottom outlet. After start-up, the NH₃ outlet improved (down from 15.8 wt-% to 14 wt-%, while the design figure is 12.8 wt-%). However, CO₂ is still high (before shutdown 16.4 wt-%, now 16.2 wt-%, design is 13.38 wt-%). Now our recovery section is getting hotter. In my opinion, the higher the decomposition rate of carbamate in the stripper, the lower the NH₃ content in the bottom of the stripper, as with CO₂. However in my case, the NH₃ content in the outlet is lower but the CO₂ is still high... I welcome comment on what's going on with this high CO₂ problem.

Mr Majid Mohammadian of Pidemco in Iran comes up with a possible cause:

Check for gaseous CO₂ slip from the bottom of the stripper if N/C ratio in the synthesis section is normal.

Mr Cristiano Azevedo of Qatar Fertilizer Company (Qafco) in Qatar shares his experiences:

We had the same problem some years ago. I suggest you check the stripper level indication, which can be wrong due to bad calibration or vortex formation. Incorrect level indication will permit CO₂ bypassing, which you cannot easily figure out. Try to close the stripper outlet valve more than usual (by 5%) and wait, the high stripper level alarm comes on, you will see a change in the stripper bottom temperature and in the recirculation pressure.

We have installed a box inside our stripper. This device is used to avoid vortex formation and allow us to obtain the correct level indication. Your ammonia content seems to be very high, which kind of urea process are you talking about?

Mr Mohammad Rastegariyan of Pidemco in Iran introduces new process parameters to double check and correct:

It seems the stripper efficiency is low. I suggest you investigate the saturator drum pressure and its level, N/C ratio and adjust reactor temperatures.

Mr Mohammad Sarfraz of Fatima Fertilizer Ltd In Pakistan confirms that unreliable level measurement could be a possible cause:

I am of the same opinion that there is every possibility of CO₂ slippage to the recovery section. Another explanation could be

that urea hydrolyses into NH₃ and CO₂ but in your case as the NH₃ contents are the same this can be ruled out. From your design conditions it seems that you have a TEC ACES process. If this is correct then the only thing could be the problematic flushing water pumps to the stripper level transmitter.

Address the issue and hopefully you will get a positive result.

Mr Mark Brouwer of UreaKnowHow.com provides and asks for some more information:

Gresik operates a TEC ACES urea plant. The reactor pressure is higher than in a Stamicarbon plant and the stripper efficiency is lower. A medium pressure recirculation section is therefore part of an ACES plant. What are your design and actual reactor pressure and design and actual stripper efficiency?

Mr Raharjo provides some answers:

Yes, we have the ACES process. Before posting this topic, we had already taken some action.

We regulated the stripper bottom level. Normal operation is 40%, we increased it to 50% and the CO₂ content is still the same. We also decreased it to 25% and had no effect.

We regulated the saturation drum level. Normal level is 61%, steam pressure 17.4 kg/cm².g. We decrease it to 50%, the steam pressure was also run down (to 16.4 kg/cm².g), bottom stripper temperature was increased to 170°C. The CO₂ content is still high.

Our conditions for the reactor and stripper are: reactor design pressure 175 kg/cm².g, actual is 170 kg/cm².g; N/C ratio design is 4, we maintain it in the range 3.96-4.04; design stripper efficiency is 72.8%, actual is 67.2%.

Mr Sarfraz of Fatima Fertilizer Ltd. In Pakistan shares his experiences:

We have the same conditions at our plant but have never had this high CO₂ content problem. Can you tell me what the results at the reactor outlet and stripper outlet? Can you tell me whether CO₂ in the reactor remains OK? I still believe that it is CO₂ slippage from the stripper. Does your ammonia feed to the reactor have the same rate as earlier at the same CO₂ load?

As the stripper bottom temperature you are operating at is not favorable for hydrolysis I would rule this cause out.

Try to further increase the stripper level. When we had similar problems we increased it up to 100%. There are two temperature indicators at the stripper bottom which can give you an idea about

level position. Moreover the CO₂ compressor discharge pressure will start increasing when the level approaches the sparger area.

Mr Nassar Hussain of Pak Arab Fertilizers in Multan, Pakistan suggests another way to determine the cause:

There may be a problem in the level transmitter of the stripper, low level of stripper causes passing of the gases to the lower pressure recovery section. It can be judged by the temperature of the stripper off gases, if it is lower than normal than the stripper efficiency is will be lower with a low level in the stripper bottom.

Mr Muhammad Adnan Hanif of Fauji Fertilizer Company Ltd in Pakistan provides some suggestions:

In my opinion, you should post more details such as the current and design operating parameters; pressure and temperature of the stripper, reactor inlet and outlet compositions along with conversion, steam to stripper pressure, and stripper bottom and top temperatures. These are necessary to investigate the higher CO₂ at the stripper outlet.

With the available information, I can only suspect the N/C ratio is on the lower side than that reported. How do you measure the N/C ratio i.e. through laboratory or some instrument? In the stripper there are two types of stripping in progress; chemical stripping and mechanical stripping. So, if NH₃ is stripped off there is no reason that CO₂ does not strip-off, based on the same N/C ratio.

Mr Muhammad Khashif Naseem of SABIC in Saudi Arabia contributes to the discussion:

Many assumptions can be made for this case but I suggest the following:

- first check that the reactor conditions are normal, in particular the N/C ratio;
- check the calibration of the stripper bottom level transmitter;
- operate your stripper at higher level set point than normal. This problem is probably CO₂ slippage caused by wrong indication of the level transmitter.

Mr Raharjo provides some more answers:

The N/C ratio is measured by laboratory analysis. We have a normal N/C ratio as stated earlier. Last week we increase the saturation drum pressure from 16 K to 17 K. The result is quite good, the bottom stripper temperature increase to 174°C, the efficiency of the stripper increased to 68.4% from 67.2%, the synthesis loop pressure also increased from 170 K to 171.4 K. Now the NH₃ outlet stripper is 14.1% while CO₂ is 15.4%.

Mr Mohammadian asks for some more information and makes some suggestions:

What is the condition of your recovery system condition after increasing the steam pressure of the stripper shell? Your design N/C ratio for the stripper outlet stream is 2.5 and at present this ratio is 2.36, so I think that first of all, you should try to reach the design N/C ratio.

I think it is better to decrease the amount of CO₂ outlet to the recovery section with throttling of level control valve in the stripper outlet to reach the higher ratio.

Mr Mark Brouwer introduces another cause:

Another cause could be that the relatively high CO₂ content is caused by a bad sampling method. Taking a good sample from the

liquid bottom outlet is not easy due to the high pressure, high temperature, crystallization of the media, formation of a gas phase due to the pressure drop and the hazardous ammonia. BHDT in Austria has developed a no dead volume high pressure sampling valve which makes taking a sample much safer and easier.

Mr Muhammad Farooq of Pak-American Fertilizers Ltd in Pakistan contributes to the discussion:

I have seen this plant and am familiar with Toyo ACES loop characteristics, hence I would like to add that the problem you are facing is due to little or too little CO₂ conversion in your reactor which can be improved to obtain optimum results.

The steps required are:

- increase steam saturation drum pressure (UFA-101 in your case) gradually 0.2 to 0.5 kg/c only 17.1 - 17.8 kg/cm²), so that the stripper bottom remains in your desired range. Keep synthesis pressure around 172 kg/cm². Try to maintain N/C ratio of 4.0 in the urea reactor.
- Counter the rest of the pressure hike by R/C.

An increase in synthesis pressure will gradually reduce your CO₂ content at the stripper outlet and recovery will dictate these condition.

Mr Muhammad Khashif Naseem adds the following suggestions:

I suggest you check the following:

- loop pressure is normal;
- condenser bottom temperature is normal;
- calculate stripper efficiency based on ammonia looking OK but on basis of CO₂ is less;
- check that the stripper bottom level transmitter is working normally. I think the level at the bottom of the stripper is less and CO₂ slipping.

Mr Waqas Habib of Engro Fertilizers Ltd contributes to the discussion:

If the level of the stripper is the reason, the CO₂ usually increases from the bottom only if the level is high and is near the CO₂ sparger i.e. somewhat above the bottom holdup. This is because the direction of CO₂ sparger holes in the TEC stripper is downwards and not upwards. I think the following two possibilities also need consideration.

In the TEC stripper, the bottom CO₂ sparger has pipes with holes of variable size. It is quite possible that these sparger pipes are not reinstalled as per drawings and there is a mal-distribution of CO₂ at the bottom so check this at the next available opportunity.

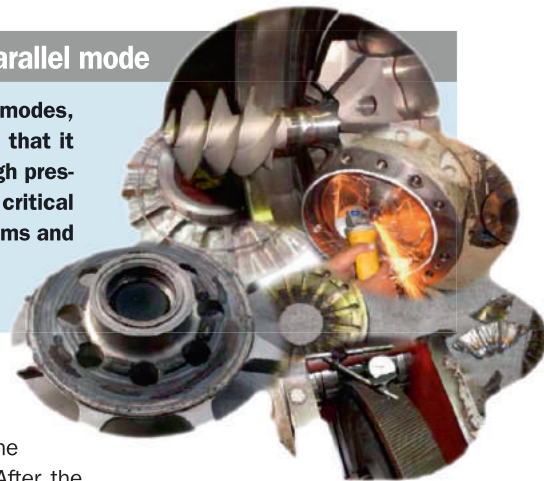
There may be some level built-up in the bottom of the stripper shell due to higher steam flow or some restriction in piping. This causes the stripper bottom temperature to decrease more than expected and thus increases the solubility of CO₂ in the stream leaving the stripper bottom. This can be verified by comparing the change in delta-T with change in stripper efficiency. ■

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Problem No. 11 How to run high pressure centrifugal pumps in parallel mode

When operators have a bad experience when operating critical equipment in certain modes, how do you convince them that operating in a certain mode should be possible and that it will bring benefits? This round table discussion discusses the parallel operation of high pressure centrifugal carbamate pumps. High pressure centrifugal carbamate pumps are critical equipment items and several process requirements need to be fulfilled to avoid problems and damage. Sharing experiences can improve the knowledge of how to operate these critical equipment items and help operators to overcome their bad experiences.



Mr João Amilton Raharjo Silva Sampaio of Petrobras in Brasil introduces a very interesting practical problem:

I have some questions about centrifugal high pressure carbamate pumps. We have two Ebara pumps driven by a steam turbine. The pumps are used to pump the carbamate solution from the medium pressure recirculation section to the high pressure reactor.

Operating conditions are: suction pressure 23 bar, discharge pressure 240 bar, temperature 100°C, flow 50 m³/h.

About 20 years ago, an operator tried to put the pumps in parallel arrangement to avoid the shutdown of the CO₂ compressors. When the pumps were put in parallel mode they suffered serious damage caused by shaft locking. Today operators are of the opinion that the pumps can never operate in parallel mode. Who is familiar with the Ebara high pressure centrifugal pump? Is the opinion of the operators correct? What causes shaft locking in a high pressure pump when the equipment is in parallel arrangement?

Mr Nawal Gupta of Kribhco in India replies with a possible cause and recommendation:

In parallel operation, the flow through each pump might have reduced to below the minimum required flow through the pump. This can result in seizure of the pump shaft. If run in parallel, flow should be first increased by recycling.

Mr Muhammad Farooq of Pak-American Fertilizer Limited in Pakistan confirms the cause and elaborates on the previous reply:

Ebara high pressure centrifugal pumps can be operated in parallel provided that you maintain all the requirements such as suction flow rate, temperature, pressure and recycle line flow rate. If there is no flow rate through recycle, it will lead to choking and can affect suction flow rate. Operating the pump below the minimum suction flow rate can lead to major damage.

I couldn't understand the relationship between shutdown of the CO₂ compressor, please elaborate further.

Mr João Amilton answers:

With regard to the relationship between Ebara and the CO₂ compressor shutdown, it is a logic of the DCS to protect the medium pressure recirculation section, especially high level in the absorber and ammonia recovery tower. In a steady-state, if the carbamate

recycle pump stops, the system stops the CO₂ compressors. After the CO₂ compressors, the system stops the ammonia pumps. So, the load from the reactor doesn't go to the medium decomposer tower and the level of the medium absorber doesn't increase very quickly. During start-up, the logic is by-passed during a certain time to put in operation the pumps and compressors. Such logic was implemented by the operators recommendation.

Mr Krisnan Hanit of Qafco in Qatar shares his experiences and provides a procedure to switch from one pump to the other:

Running Ebara or any other pump in parallel mode is difficult or dangerous but you can change over the pump without a CO₂ compressor trip if you follow the procedure described below.

The discharge pressures of both pumps will be different and any high pressure carbamate pumps to run in parallel require that the discharge pressure of both lines is almost similar. To avoid a CO₂ compressor trip, you can start one pump on recycle and slowly close the discharge valve of the other running pump to less than 50%. Then stop the pump and quickly close the discharge valve. Immediately line up the running recycle pump to the system by opening the discharge valve and you can avoid a CO₂ trip without MP absorber level trouble (there will be some level rise).

Mr Riyadi Fikri of P.T. Pupuk Sriwidjaja in Indonesia gives his experiences:

We are using Ebara high pressure carbamate pumps and have been since 1977. We were unable to operate the Ebara centrifugal pumps in parallel mode for a long time, because we had difficulties in regulating the flow, pressure and temperature in normal conditions. Operation of the pump outside its specified conditions will cause fatal damage. We run parallel pumps only for switching with the standby pump for the purpose of maintenance.

Mr João Amilton asks some more questions:

What kind of power source do you have for the Ebara pump – steam turbine or electric motor? Could you describe the procedures to put your pumps in parallel mode to switch the pumps?

Mr Riyadi Fikri replies:

The carbamate recycle solution feed pump Ebara pump specification is as follows:

- Source power: steam turbine
- Pressure: 42 kg/cm²
- Temperature: 390°C
- Liquid pumped: urea carbamate solution
- Inlet temperature carbamate: 110°C (max), min : 80°C (min)
- Capacity: 85 m³/h
- Discharge pressure max: 260 kg/cm²g, normal operation: 250 kg/cm²g
- Suction pressure: 24 kg/cm²g
- Pump speed: 6500 rpm
- Minimum flow: 38 m³/h.

The procedure to switch Pump A to Pump B when operating in parallel mode is described below.

Pump A: normal operating conditions

Pump B: increase vacuum, increase heat, start with curve performance from vendor until pressure, flow, and temperature are in the same condition as the other pump (pressure: 250 kg/cm², flow: 85 m³/h, temperature: 100°C) → operation rate 100%.

Condition Pump B: Discharge fully closed, bypass throttle and

then...Pump B → fully open discharge. Crack open bypass Pump A, close bypass Pump B, close discharge Pump A.

If Pump B is in good condition, then stop Pump A according to the procedures supplied by the vendor. (Warning: if below the minimum flow (<38 m³/h) pump can be damaged).

Mr Rajkumar Kulkarni of RCF LTD in India shares his valuable experiences:

Ebara centrifugal pumps can be operated in parallel. As some of us have already mentioned, it is important to have the proper/sufficient suction flow to the pumps. Pump flow is controlled by the recycle control valve. Under no circumstances should the pump be allowed to operate below the minimum flow. The flow meter is in the common suction line of the pumps. ■

Ebara centrifugal pumps can be operated in parallel.

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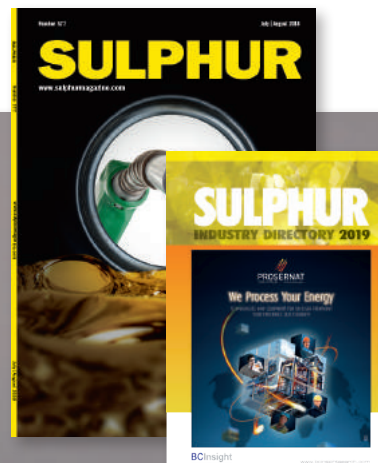
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Problem No. 12 Oil fouling of the high pressure stripper in a urea plant

The efficiency of the high pressure stripper is a very important parameter in the optimum operation of any urea plant. Fouling of the HP stripper can reduce the efficiency and oil is a typical cause of fouling. Oil can originate from, for example, oil seal systems of high pressure reciprocating ammonia and carbamate pumps and carbon dioxide compressors. Oil can easily cause fouling of the liquid distributor system in the top of the HP stripper (where small holes are present) as the oil can become thick due the stripping of the light components. What are the best methods of solving oil fouling problems and cleaning the HP stripper?

Mr Malik Sohail of Agritech Ltd in Pakistan introduces a very interesting practical problem that has a big impact on the performance of a urea plant – oil fouling in the high pressure stripper:

We have a TEC ACES plant with a HP stripper of Stamicarbon design. There has been a problem with stripper efficiency and when the ferrules were opened during a shutdown it was found that 150+ tubes (about 10%) had oil in the stripper bottom. The ferrules are OK. No blockage of holes was observed but there was evidence of pitting. The oil consumption at the NH_3 feed pump was high before the shutdown and the HP stripper bottom temperature was on the high side with high NH_3 slippage.

I would like to ask the following questions:

- How can the oil be removed?
- What are the possible causes of it?
- What procedure should be used to wash the stripper tubes?

Mr Mark Brouwer of UreaKnowHow.com replies: Oil can come from a variety of sources such as the HP NH_3 pump, compressors in ammonia plant, ammonia storage tank, etc. Oil in the HP stripper can be removed using a caustic solution. Contact Stamicarbon for the cleaning procedure.

Oil fouling can result in a lower stripper efficiency but other factors can also affect the efficiency, for example:

- non-uniform liquid distribution over the tubes. Did you check if there is a difference in liquid level between the centre and the outer area?
- poor tightening of the ferrules on the tube ends, resulting in liquid/gas bypass.

Mr Muhammad Farooq of Agritech Ltd in Pakistan adds: With regard to the liquid level at the tube sheet, the level was higher on the wall side than in the middle.

The swirls fittings were good. We carried out a leak test which indicated no leakage apart from a few tubes, so we concluded there was no liquid bypass there. But the stripper efficiency is still low, what measures should we take next?

Mr Janusz Maćkowski of ZCh"Police" in Poland provides some solutions: If you have an ongoing problem with oil fouling you could apply some of the procedures described in UreaKnowHow.com's Technical paper: 2010 09 Ren Lanhua UreaKnowHow.com Stripper Efficiency Problems.

You could also try washing the stripper using a HP water pump at every stoppage.

Muhammad Farooq: We have already tried one of the washing procedures referred to in the paper, item 4.1. We dismantled the stripper which highlighted the problems.

Mark Brouwer: What level difference did you observe between the centre and wall side? When you have droplets from some tubes in the leak test, it is a sign that some liquid is bypassing. This can also result in gas bypassing during operation. Did you observe blue oxide layers on the inside of the ferrules where these are fixed to the tubes? What is your stripper efficiency? How much lower is it compared to design? Did you do a delta-P test of the ferrules? I suggest contacting Stamicarbon to re-check the liquid distributor system based on your findings.

Janusz Maćkowski: You could also check whether the geometry of the heat exchanger part of the stripper has changed (check the tubes are still vertical and not twisted), whether the stripper tubes are fouled and whether they have the same diameter along their entire length (as any restrictions will have an adverse effect on the stripper).

Mr Patel Rasikal Dahyabhai of Notore Chemicals Ltd in Nigeria adds his recommendations: You can clean the stripper tubes and ferrules using Teapol (just like soap solution but it must be chloride free). If you have big oil problems in the stripper, it is best to convert the plunger packing lubrication system of the HP ammonia pump (forced feed lubricator) from oil to flushing water just like HP carbamate pump. The ammonia surge drum/suction vessel should also be thoroughly cleaned during the annual turnaround.

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia offers his experience and practical solutions: Our HP stripper has also had a problem due to uneven liquid level indicated on the swirls.

For oil cleaning I suggest filling the stripper with condensate from the bottom up to 6 inch above the top of the tubes and heating up the condensate to 120°C for 3-4 hours before draining it. Repeat this procedure 5 to 6 times to completely wash out the oil. But, in my opinion, your problem may be due to the internal fittings, so refit them and carry out a pressure test.

Muhammad Farooq: Although we carried out back washing during plant start up, the stripper efficiency did not improve.

After that we had a shutdown and inspected the stripper which highlighted problems. Our plant is now in operation again.

The liquid level was around 10-12 inches towards the wall side and 5-7 inches in the middle, judging from the change in metal colour.

Although the leak test was OK (zero leakage with 30 mm water level on tube sheet), around 450/1824 tubes show a blue oxide layer. The stripper efficiency (alpha) is 66.5-68.7% We haven't done a delta P test of the liquid dividers yet.

Mark Brouwer: Here is some advice for the next turnaround: The level difference between the centre and outer area seems too high, so improvements should be made to the liquid divider system; perform a delta-P test on the ferrules; check the dimensions and tolerances of the fittings of the ferrules on the tube ends.

How much does the efficiency differ compared to design? Did you remove the oil? If yes, how?

Mr Sunil Kulkarni of Mangalore Chemicals & Fertilizers Ltd in India shares his experiences: The most effective way to clean stripper tubes is EDTA cleaning. Stamicarbon can provide the procedure. Ammonia pumps plunger packing can be changed over to water flushing to eliminate oil ingress. We have done both of these at our plant with good results.

Muhammad Farooq: Please send more details of the system installed for switching to water flushing at ammonia pumps plunger packing.

Mr Vedantam Srinivas of Safco in Saudi Arabia confirms one of the suggested solutions: To eliminate the root cause, change the NH₃ pump packing sealing system from oil to cold condensate. This will bring many benefits to the plant as well as providing a clean environment. The stripper and downstream equipment will normalise gradually on their own. The cost of the modification is low. The condensate outlet from the packings can also be collected in a drain collecting tank to recover the ammonia.

Mark Brouwer: The cleaning procedure with EDTA is for removing oxide scale, not for removing oil. The seal system for the HP NH₃ pump is a water seal system not a water flush system. I believe all HP NH₃ pump vendors have a water seal system now as a standard feature, so they will also have information on how to do that. The principle is that water is sealed at about 5 bar between the low pressure packing rings and the high pressure packing rings. Any NH₃ leakage through the high pressure packing rings will be absorbed in the water and measured by the temperature difference of the water. The water should be free of solids.

A HP carbamate pump has a water flush system, which washes away carbamate crystals.

Muhammad Farooq: Which vendor should we contact to switch the sealing system of our Peroni HP ammonia pump from oil to water? The injection oil sealing pressure at plunger packing is 33-35kg/cm² with a discharge pressure of pump=185kg/cm².

Mark Brouwer: I believe every pump vendor has developed a water seal nowadays, so Uraca, Peroni etc. Please check with them.

Mr Le Ngoc Ban of Phu My Fertilizer in Vietnam shares his experiences: Our plant is in a turnaround and we are finding it difficult to remove oxide scale from inside the ferrules and on the tube side of the MP decomposer. Does anyone have any advice for cleaning with EDTA or any experiences to share?

Malik Sohail: Are there any operational problems with water sealing of HP Ammonia pumps? If there is wear of the stuffing box, water will carry with ammonia and H/C in the loop and ultimately urea conversion will be affected. Which is more damaging, oil or water carryover with ammonia?

Mark Brouwer: During normal operation, leakage of water or oil to the process side should be minimal if proper maintenance procedures are applied. In the case of an upset, oil fouling can cause bigger problems than water, at least in stripping plants. Even during an upset the amount of water from the pumps is much less than from the carbamate recycle. The liquid holes in the liquid dividers in the top of the stripper act as a kind of filter for the whole synthesis section. Oil will also be stripped in the stripper at the high temperatures and the heavy boiling part of the oil will collect around the liquid holes.

Sunil Kulkarni: The oil carryover is mainly due to oil from HP ammonia pumps. At MCF we have changed to water for plunger flushing instead of oil. The results are good. The packing life is around 3 months. The oil, if none is eliminated naturally, ends up at the stripper bottom. We have also carried out EDTA cleaning of stripper tubes back in 2000 for 316 tubes and achieved good results. Stamicarbon provided the procedure. Now we also have a Safurex stripper and no problems were observed at the first inspection.

Mark Brouwer: Oil can enter the urea synthesis from the reciprocating HP pumps and CO₂ compressor. Compared to the total amount of reactor solution, the amount of oil is small. The reactor solution and oil form an emulsion. The continuous phase is the reactor solution and the oil droplets form the discontinuous phase. In the top of the stripper (without a special fluid distribution system), the emulsion is relatively calm, i.e. no turbulence. Here, the oil has time to coalesce to larger droplets, which rise and form an oil film on the surface. As more reactor solution with oil enters the stripper, more and more oil coalesces and forms a thick film of oil. This oil film is polymerised at the surface in the gas phase. Solid particles are formed which may sink due to their larger density and clog the holes in the liquid distribution dividers.

SBN's solution for this is to prevent coalescence of the emulsion by keeping the liquid (emulsion) constantly in motion. The oil droplets will then pass with the reactor solution through the holes of the liquid distribution dividers. A special fluid distribution system can be installed above the support plates of the liquid distribution dividers. This solution has been proven in several urea plants for many years.

Contact Mr Hermann Kernberger of SBN,
H.Kernberger@christof-group.com for more information.

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Plant Manager+

Problem No. 13 3-Way valves in urea melt lines

In nearly all urea plants, urea and water is separated in an evaporation or concentration section at vacuum pressures. The urea melt can have a concentration of 99.6 wt-% when the urea melt is sent to a prilling tower or a Sandvik Process Rotoformer or a somewhat lower concentration when a granulation technology is applied.

Ovviously it is important to control the temperature of the urea melt just above the crystallisation temperature in order to maintain it in liquid condition. On the other hand, when the temperature is too high several negative side reactions in the urea melt occur, resulting in higher biuret contents in the final product and higher ammonia and urea dust emissions from the finishing section.

Therefore, urea melt lines are typically jacketed pipes and the pressure of the steam in the jacket is controlled by the required urea melt temperature. Urea melt pumps and 3-way valves (at the suction and discharge side of the urea melt pumps and just upstream of the prilling bucket) function in a challenging environment: handling a process liquid close to its crystallisation temperature while at the same time proper sealing is vital. The picture shows the sealing challenges of a traditional urea melt pump. Fortunately, successful innovations are available that provide a reliable solution.



Mr Mark Brouwer of UreaKnowHow.com initiates a discussion about experiences with 3-way valves in urea melt lines: In several urea plants, 3-way valves in urea melt lines have experienced leakage problems.

Damage of the typical PTFE coated plug or ball valves can be caused by 1) overheating (welding the valves in the line during construction, using too high pressure steam) causing damage to the PTFE coating and/or 2) solids causing damage to the PTFE coating. Solids can originate from urea crystals/biuret when tracing/insulation is not properly applied, and also from the urea solution recycle (dissolved waste urea), which may contain, for example, sand. When this recycle is not sent to the urea solution tank but is sent directly to the evaporators, these solids reach the 3-way valves of the melt pumps. Installing proper filters in the urea recycle line is a good suggestion.

Please share your experiences with 3-way valves of urea melt pumps:

- What are your experiences with 3-way valves in urea melt service?
- Which type have provided a good experience?
- Do you have flush connections in the valve?
- Do you have coated valves?

Mr Girish Prakash of Tata Chemicals Ltd In Brabala, India replies: We have faced this situation many times, sometimes even immediately after valve replacement. However, no definitive reason could be attributed to it.

Mark asks for further clarification: Did you see the damage to the PTFE coating? Do you have any pictures? How you handle the urea recycle?

Mr S.K. Gupta of IFFCO in India contributes to the discussion and shares his valuable experiences: We have 3-way valves in the urea

melt discharge line at the top of the prilling tower supplied by Parcol of Italy. We have also faced problems with leaking of this valve. It is a ball type valve (ball made of steel) with ports. The seating surface is glass filled PTFE. Because of its compact size and hence reduced seating surface area, any scratch in the seating PTFE results in leaking of the valve. As crystallised urea at the surface of the ball may damage the seating PTFE, we ensure that steam in the control valve body jacket is always on and auto steam flushing system for the downstream lines works as per the sequence defined in interlock. On many occasions we have observed that leaking of the valve increases when the urea solution is lean i.e. when taking the vacuum in the 1st and 2nd stage evaporation at initial startup. Once the urea solution is almost 100% pure and ready for prilling the leaks stop; maybe because now the leakage path is blocked.

After dismantling the leaking valve we find scratches in the PTFE, sometimes part of the PTFE is also blown. Proper fixing of the PTFE seating ring, ensuring a urea melt free of any foreign particles and no crystallisation of urea melt at the face of the ball will certainly reduce the risk of the valve leaking.

Girish replies to Mark's questions: We do not have any pictures but we have observed twisted seats. The urea solution recovery is through filters.

Mark asks for more clarification:

- What do you mean by a twisted seat ?
- Do you mean that the seat no longer fitted correctly in the body?
- What was the cause of the twist?
- Did you see damage to the PTFE coating?
- What is the mesh of the filters you use in the urea solution recycle?

Girish comes back with more answers: It didn't fit in the body anymore. The PTFE coating was damaged.

Lately we started using micron filters (similar to the ones used in GV solution filtration system). The number of incidences before and after using these filters has not been recorded.

Mr Muhammad Farooq of Agritech in Pakistan shares his valuable experiences: We have installed a 3-way valve at the inlet of prilling basket and have never faced any problems in 13 years of operation. The valve is from Fisher, Type-YD.

Its features are:

- Economy – balanced valve plug construction in the Type YD design permits use of smaller, lower-cost actuators. A single one-piece valve accommodates both trim designs and uses Fisher easy-e bonnets, gaskets, and packing, thus cutting spare part inventory costs.
- Excellent sealing capabilities – the Enviro-Seal packing system option is available. This packing system provides excellent sealing, guiding, and loading force transmission. The Enviro-Seal packing systems features PTFE, graphite ULF, or Duplex packing with live-loading for reduced packing maintenance.

Mr Salam M. Malih of North Fertilizer Co. in Iraq poses a very practical question: We want to buy 3-way valves for the suction

and discharge of the urea melt pumps. Who is the best manufacturer of these valves and which is the best metal for this purpose?

Mr FA Khan of FFCL in Pakistan shares his experience: You can buy AZ Armaturen or Xomox valves for this service.

Mark summarises the discussions: The discussion above has resulted in the identification of several problems related to 3-way valves in urea melt applications. Solutions to the problem and several vendors were recommended to improve the reliability of 3-way valves in urea melt applications.

In order to ensure that no crystallisation will occur in the 3-way valve and at the same time guarantee easy maintenance of the valve, I further suggest applying the ControHeat bolt-on jackets developed by Controls Southeast Inc. (CSI) for a reliable solution, which requires lower investment and operating costs. CSI also has a ControTrace product as a lower cost alternative for a jacketed pipe (more info at www.csiheat.com). Further, for a reliable urea melt pump I would recommend you seriously consider the Bungartz hydrodynamic sealed melt pumps (more info at www.bungartz.de). Please note these products can also be applied for ammonium nitrate melt applications. ■

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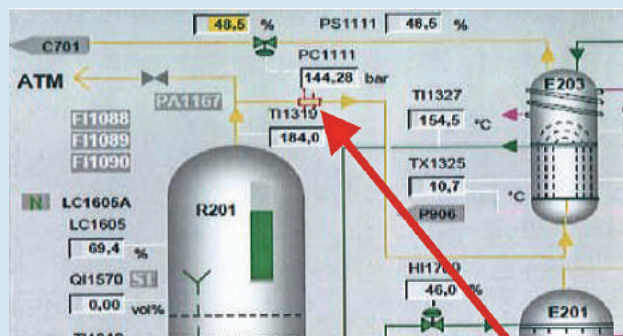


Plant Manager+

Problem No. 14 High synthesis operating pressure

Operating at the wrong synthesis operating pressure is always a tricky situation for any urea plant operator, knowing that operating at too high a pressure will lead to the pressure safety valves blowing off. Such an incident would not only cause a significant environmental impact but also a forced shut down of the plant as typically the pressure safety valves no longer close properly after blowing. Servicing these valves becomes necessary and plant down time can be a costly incident. On the other hand, operating at too low a synthesis pressure will lead to lower conversion figures, higher carbamate recycle streams and higher energy costs.

The key is to measure the synthesis pressure in a reliable way. In an ideal situation, the synthesis pressure would be measured as close as possible to the pressure safety valves, e.g. in a high pressure carbamate gas line. Until recently this was not possible because of the risks of crystallization and corrosion so the synthesis pressure has mostly been measured at locations where these risks are absent or less likely.



Synthesis pressure measurement in HP carbamate gas line

Now LESER, together with SKW Piesteritz, has developed a reliable solution to measure the synthesis pressure in the high pressure carbamate gas lines. But what should others do who don't have reliable synthesis pressure indication?

Mr Kumar Srivastava of Notore Chemical Limited in Nigeria starts up a discussion about a high synthesis operating pressure: Our urea plant is based on Stamicarbon CO₂ stripping technology. We are facing problems of high synthesis pressure when we try to go to a load of approximately 100%. My observations are as below:

- In our plant the synthesis loop pressure reference is taken from upstream of the high pressure ammonia ejector (in the ammonia feed line) and the instruction is to maintain the synthesis pressure at 150 kg/cm² based on this HP ejector upstream pressure. The ejector pressure control valve is kept fully open. However, from the ejector data sheet we can learn that at normal plant load the differential pressure across the ejector is 15.5 kg/cm².
- My concern is that carbamate may be building up, leading to a false high synthesis pressure indication and poor reactor conversion due to the low reactor pressure (seeing differential pressure), which increases the water recycle in the system, thus reducing reactor conversion even further due to the high water ratio.
- In our plant there is no gas chromatograph (or molar N/C ratio analyser).
- I am seeking help: When keeping the HP ejector pressure control valve fully open, is it possible to maintain a differential pressure across the HP ejector as per design value? This HP ejector is installed in the HP ammonia pump (7 plunger reciprocating type) discharge line. Is it possible to throttle the HP ejector valve to increase the driving pressure, which may help to create a higher differential pressure, which in turn will help to reduce the supposed build up of carbamate from the HP ejector?

Mr Salam M. Maleh from North Fertilizer Complex in Iraq replies:

You can control the differential pressure (18 bar) by controlling the HP ejector throttle valve (close ejector throttle valve for example 5%).

Kumar asks for further clarification: Does your plant have the same technology? I don't know the capacity of your plant or if you have a HP ejector in your plant. I understand the concept of the HP ejector very well, but I don't have the HP ejector performance curve.

Mr Shoaib Minhas of FFBL in Pakistan contributes to the discussions with his experience: We have a centrifugal type HP ammonia pump and we manipulate the ejector valve to control the back-pressure (ammonia pump discharge pressure). You may also wish to consider this as you have a reciprocating type pump.

Mr Muhammad Farooq of Agritech in Pakistan shares his view: In a Stamicarbon urea plant it is more important to control the N/C ratio and reactor top temperature to control the synthesis pressure.

Mr Majid Mohammadian of OCI Nitrogen in the Netherlands requests more details: Can you tell us the following?

- Plant nameplate capacity
- Capacity you are limited to
- Synthesis pressure
- Pressure upstream of the HP ejector
- Scrubber overflow line temperature and level
- Feed ratio and if possible N/C on reactor downcomer
- Top temperature of the reactor
- Gas outlet temperature of the HP scrubber
- Is it a vertical HP carbamate condenser or pool condenser type?

Kumar replies:

- Plant nameplate capacity 1,500 t/d
- Capacity limitation >90%
- Synthesis pressure about 140 kg/cm²
- Pressure upstream of the HP ejector 158 kg/cm²

We have a vertical HP scrubber. The solution outlet temperature is 165°C and the reactor vapour temperature is 168°C. This indicates to us high ammonia and we tried to increase the CO₂ a little.

Do you have level indication in your HP scrubber? I ask because in our plant there is no level indicator so we do not know if there is any level rise when there is an increase of carbamate flow. But the increased opening of the vent valve indicates that there is a level increase in the HP scrubber which is why I want to operate the HP ejector to increase differential pressure across the HP ejector.

Majid replies: Normally in our Stamicarbon plant there is a level indicator in the scrubber overflow line to control the level in the line because we need to prevent any gas passing or gas circulation in the synthesis section. Please provide the following information:

- With the synthesis pressure transmitter being located and as per your design what should the synthesis pressure be at 100% capacity?
- What is the temperature of the gas outlet from the scrubber?
- As per your PFD, what is the inlet and outlet pressure of the ejector at 100% capacity?

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia requests some further information: Please tell me the followings to help solve your problem:

- HP vent out temperature
- LP steam pressure
- Reactor top /bottom temperatures
- Stripper top /bottom temperature
- Stripper steam pressure

Kumar replies: We don't have any level indicator in the HP scrubber vessel. Please send me the details for a level transmitter in the HP Scrubber such as the vendor and where it is mounted.

We also have level transmitter issues in the 2nd stage evaporator: Presently the level indicator is mounted in the suction line of the melt pump and is not working. We need to change this level transmitter. In your plant where is this level transmitter mounted and who is the supplier?

Mr Muhammad Adnan Hanif of Fauji Fertilizer Company Limited in Pakistan shares his experiences and asks for some clarifications: What is the reactor bottom pressure [any pressure transmitter or local PI installed downstream of the HP ejector or CO₂ inlet line to urea reactor just downstream of the shut down valve]? Normally the pressure throttle valve installed on the HP ejector is utilised to regulate carbamate flow. If your plant N/C ratio is satisfactory or higher than required you can utilise this valve to increase

the motive fluid i.e. ammonia pressure. However, please note that the delta-P of 15.5 kg/cm² [Between HP ejector upstream and HP loop top vent] will not give you a true picture of the available head to move carbamate. You should take the reactor bottom pressure and the motive fluid pressure difference. Normally, in our plant [Saipem technology] it is > 60 kg/cm² [Delta P between reactor bottom and ammonia header].

To check if there is any liquid passing to downstream sections through overflow of the HP scrubber, check its effect on downstream section e.g. sudden decrease in temperature, pressure excursions etc.

Mr Krishan Harit of Qafco in Qatar shares his experiences: Are you sure that your plant is facing a problem due to the HP ejector. Your synthesis pressure tapping is in the wrong location. If you reduce the HP ejector opening then the pressure upstream will increase and more carbamate from the HP scrubber will go to the HPCC, which is good. A HP scrubber level indication is not required; generally this indication is not working anyway.

The best method of control is to use the HP scrubber outlet temperature. If the temperature is low it means your HP scrubber level is high (166-168°C is a good temperature) but if the temperature is 165°C or less it means the HP scrubber level is too high and liquid is going to LP absorber. You can see this by the increase of the LP absorber level opening and the synthesis pressure increase, in which case a closer HP ejector opening will help.

The best place for the synthesis pressure indication is the HP scrubber overflow line or downstream of the HP ejector in the line to the HPCC.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands summarises the discussions so far: In order to have more efficient discussions it would be better to have more information from the start with regard to temperatures, pressures etc.

It is important to know the basic process technology one is referring to as in this case it is important to realise that the HP ejector in a Stamicarbon plant has a different function to that of the HP ejector in a Saipem plant.

The challenges to measure the synthesis pressure in a reliable way, without crystallisation and corrosion risks, is different for each technology. In both technologies one looks for places where these risks are minimal but these places differ between both technologies.

One can easily conclude that, in this particular plant, the pressure measurement, located upstream of the HP ejector, is far from ideal. Depending on plant loads and carbamate flows, the delta-P over the HP ejector can differ significantly (I have seen ranges from 15-35 bars). It is therefore important to have the datasheets for the HP ejector.

For anyone considering a new pressure measurement in your synthesis, please note the presentation of LESER at the AIChE Ammonia Safety Symposium on September 13, 2012 in Chicago. ■

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Plant Manager+

Problem No. 15 Pressure safety valve experiences

Pressure safety valves are critical equipment items in any chemical process plant. In a urea plant, there are additional challenges such as carbamate corrosion and crystallisation risks. These challenges threaten the reliable operation of the pressure safety valves: popping up too early and/or crystallisation of process media in the inlet or outlet lines. Furthermore, these challenges make it very difficult to install spare pressure safety valves, as is common practice in other chemical processes. Sufficient reasons, therefore, to initiate a discussion about experiences with pressure safety valves (PSV) in urea plants.



Mr Mark Brouwer of UreaKnowHow.com starts up the discussion with a simple question: What are your experiences with pressure safety valves?

Mr Mohammad Rastegariyan of PIDEMCO in Iran replies and shares his valuable experiences:

The most critical safety valves are those in the high pressure synthesis loop. In the past we have faced passing problems due

to seat and plug corrosion, which was caused by poor insulation. We subsequently improved the insulation condition.

The second most critical safety valve is the one on the discharge line of the high pressure ammonia pump. Problems were caused due to vibration. We had to replace it with a change over valve (twin safety valve).

Also the safety valve of the CO₂ compressor interstage has popped several times and remained in an open state. We need to mention that the pressure at this stage is higher than normal. We subsequently increased the safety valve set point.

Mr Waqqar Ahmed of Fauji Fertilizer Company in Pakistan also shares his valuable experiences:

We have experienced the following problems:

- Passing problem in the urea high pressure stripper vapour outlet line pressure safety valve: Its frequent passing resulted in severe corrosion of the blow down header. Once it was also gagged. The problem was resolved by proper seat and disc lapping and by proper seat flushing arrangements.
- Pressure safety valve bellow failure at the high pressure carbamate

recycle pump discharge line: The leakage resulted in a forced shut-down of the plant. The main culprit was high line vibration.

Mark comes back with some background information and asks some more questions:

- Does anyone have experience with rupture discs upstream of a pressure safety valve?
- With which vendors do you have experience?
- Does anyone have any experience with a safety valve popping open too soon? Do you have any remedies?
- There is a very nice innovation on the safety valve market: Leser GmbH in Germany has developed a pressure safety valve especially for carbamate service in urea plants, for example:
 - reactor off gas in a Stamicarbon plant;
 - stripper off gas in a Saipem plant;
 - recirculation pressure safety valves;
 - desorber pressure safety valves;
 - HP carbamate pumps, etc.

The Leser pressure safety valve has a continuous flush around its seat, which assures that the downstream side is completely flushed and flushes the seat and stem when the safety valve is in open position so that the stem and seat will not get damaged due to erosion/corrosion of carbamate when the pressure safety valve pops up. As a result, the valve is able to close again properly after opening.

These valves are already in successful operation at several urea plants. In one plant these pressure safety valves opened during an upset condition in the process. When they routinely sent the valve to the workshop for revision, no damage could be observed.

Mr Mahmood Rauf Zafar of Fauji Fertilizer Company in Pakistan introduces an important practical problem and also provides a solution:

Urea plant pressure safety valves are prone to passing problems. This is exacerbated because it is not easy to confirm if a pressure safety valve is passing or not.

The main reasons are:

- PSV seat continuous steaming keeps the downstream line in hot condition. Most of the time, isolation of individual PSV steam is not fruitful due to other PSV's steam back flow from the blow down header.
- Opening of the PSV downstream line drain is also not beneficial due to vacuum in blow down vents.
- Sound checks are also of no help most of the time as steam flow or process flow misguides.

In my experience the only option is lab sampling from individual PSV's downstream open drain. In our plant we have changed the urea stripper vapour outlet PSV angle from ~75° to 90° of the line.

Mr S K Gupta of IFFCO in India provides important information about his experiences with rupture discs:

We have used rupture discs upstream of the following safety valves: CO₂ compressor 4th stage suction PSV and final discharge PSV and the high pressure ammonia pump discharge PSV. Earlier they used to pass frequently (our plant is based on Saipem technology).

We also experience frequent passing of the hydrolyser PSV. We used a rupture disc in the upstream line of this PSV but we experience frequent premature bursting of the rupture disc due to corrosion. The problem remains.

Mark replies and asks for some more information:

Is the hydrolyser PSV in the gas phase? Here condensation of carbamate gasses can cause condensation corrosion problems.

Which material have you applied?

Possible solutions are: use more corrosion resistant materials, proper tracing/insulation, is a flush upstream of the rupture disc possible?

What is your experience with the synthesis PSV – do you have any problems there?

Have you ever experienced premature opening of these PSVs?

Mr Girish Prakash of Tata Chemicals Ltd. in India shares his experiences:

With this arrangement, what is the average life you have achieved for the rupture disc installed? We have experienced premature lifting of the stripper PSV in one of our units (Saipem technology).

Mark asks some more clarifications:

Thanks Girish: How many safety valves do you have at the stripper?

Was the premature lifting during a start-up situation?

Did you have to refurbish the safety valve or did it close again? Which materials did you use for the stem and seat?

Girish replies:

Most of the time it happened during the start-ups and/or immediately after start-ups. We have two pressure safety valves in the HP loop (one on the HP separator and the other on the stripper). This PSV was gagged and was inspected and recalibrated.

S K Gupta also replies:

Yes, the hydrolyser PSV is in the gas phase. The material for the disc and seat is HVD-1 and for the rupture disc it is 316L. Initially the PSV for the hydrolyser was installed at the pipe rack with about 6 m of upstream piping. Frequent choking up of the stream piping was observed. Now we have put the PSV and rupture disc at the hydrolyser body flange. The upstream part of the rupture disc is too short to require flushing. Insulation is already there. We are getting maximum 5 to 6 months life of the rupture disc.

We have also sometimes experienced a passing problem of the HP stripper off gas PSV. We have low pressure steam jacketing for the PSV disc and seat. The steam temperature is 148°C while the stripper off gas temperature is 190°C. We think this steam in the body jacket is contributing to condensation of gasses on the disc and seat and hence promoting condensation corrosion. What is your opinion in this regard?

Mark comes back with his opinion:

I suggest using MP steam for steam tracing in the HP synthesis section. The next question is what is the quality of the tracing installation and insulation? I suggest contacting the company Controls SouthEast who design and supply heating solutions especially for these cases. Are the synthesis PSVs installed directly on the main pipelines? Which materials are applied?

Mr Amrit Ramnanan of IPSL in Trinidad and Tobago asks some questions:

What is your experience as to the cause of the passing of synthesis pressure safety valves? Is it because the seat often gets damaged due to crystallisation due to periodic moments when/if the relief lifts and reseats?

At our site the PSVs are steam jacketed and there is also a steam purge on the outlet piping to prevent crystallisation. But this does not prevent the PSV from passing. On multiple occasions we have experienced passing. What are your experiences as to the cause? What is your experience and opinion of using the PSV with integral steam flush? Do you know of any success stories?

Mark replies:

Yes, the Leser safety valves have proven their success in several Stamcarbon urea plants already. During the AIChE Ammonia Safety Conference in Chicago in September 2012 the details were presented and the technical paper with all the detailed information is now available. ■

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Plant Manager+

Problem No. 16 High pressure scrubber problem

Sometimes a serious problem occurs in a urea plant, which forces the plant to reduce capacity or even forces a shutdown. It is then vital to find the root cause of the problem as soon as possible, especially if the problem repeats. Such a situation happened at Khorasan Petrochemical Company in Iran. Mr Eisa

Norozi started a discussion in the UreaKnowHow.com Round Tables to provide support for his troubleshooting. The following discussion helped Eisa to find the root cause of the problem much more quickly, which clearly proves the added value of modern communication tools like the internet.



Mr Eisa Norozi of Khorasan Petrochemical Company in Iran: We have a Stamicarbon CO₂ stripping urea plant with a capacity of 1,700 t/d which is integrated with a melamine plant. We have been faced with a new problem as described below.

During normal operation, for about 20 minutes, the gas outlet temperature of the HP scrubber decreased from 145°C to 107°C and the pressure in the synthesis section increased to 152 bar (normally it is 145 bar). We immediately decreased the plant capacity and washed the scrubber with the HP steam condensate pump. The situation of the plant went back to normal, but two days after changing the HP carbamate pump the problem was repeated. We couldn't increase the load of the HP carbamate pump because the outlet temperature of the HP scrubber gas line had decreased and the pressure of the synthesis had gone up which meant we were unable to control the plant anymore.

It should be noted that the ΔT in the tempered cooling water loop did not change, water circulation in the loop was normal and immediately after washing the packed bed with the HP steam condensate pump injected at the discharge side of the HP carbamate pump the problem was solved but during any change around of the HP carbamate pump the problem is repeated.

We would like to ask the following questions:

- Do you have any experience with melamine carry over to carbamate (making a polymer on the packed bed in the HP scrubber) causing choking of HP scrubber bed in the urea plant. If yes what is the procedure to solve it?
- Do you have any experience with a partial rupture in the rupture disk in the HP scrubber: What happens when there is a partial rupture?
- Do you have any experience with blockage in the liquid distributor in the HP scrubber with PTFE or other material?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands replies with some questions:

Could you have a problem with clogging of the HP inert valve at the outlet of the HP scrubber? What is the carbamate flow and its composition compared to the design values? Could you have a flooding problem?

Eisa provides some more information:

This is the first case in our plant of a problem where the cause is unknown. As previously described, when steam condensate is injected in the outlet pipe of the HP carbamate pump for approxi-

mately 20 minutes, the problem is solved but after some days especially during any change in the HP carbamate pump situation (for example fluctuation in flow or during change of the pumps) the problem reoccurs. The plant capacity (carbamate flow) does not change but during that time we cannot increase the flow of carbamate because the carryover of carbamate to the LP absorber increases the temperature of the outlet line of the HP scrubber decreases and the pressure of the synthesis increases gradually up to a high condition.

The causes could be the following although we cannot find any clear reasons for these causes:

- Partial opening in the rupture disc because in every overhaul we experienced this and it was repaired by welding.
- Clogging in packed bed with carbamate.
- Melamine carry-over in the off gas from the melamine plant, which makes a polymer in the packed bed.
- Clogging in holes of the carbamate liquid distributor in HP scrubber with PTFE or other material as our HP carbamate pump is reciprocating type.
- Flooding in the HP scrubber, but the cause is not clear.

Currently we cannot increase the carbamate flow, the capacity is decreased and we must drain the carbamate to the ammonia water tank.

Mark asks further:

- I assume carbamate flow has increased after melamine integration... by how much %?
- Has the carbamate liquid divider been adapted for the new flow?
- When did this problem first occur? Since a turnaround?
- Please confirm you have a water/steam flush on the stream close to the high pressure inert valve and, if possible, provide a printout of the DCS screen on the HP scrubber.

Eisa provides more information:

The melamine plant was integrated 8 years ago. Before the problem occurs the rpm of the HP carbamate pump is 145 but afterwards it is about 130 rpm. In this condition we must drain the carbamate. Our procedure is to wash the scrubber bed with HP steam condensate pump while the HP carbamate pump is out of service for 20 minutes which solves the problem and production returns to normal. But as mentioned before after some days with a small fluctuation in the carbamate flow the problem recurs.

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia contributes to the discussion:

I agree with Mark. I suggest the following actions: flush the vent

line at the outlet of the HP scrubber, increase the cooling water temperature across the HP scrubber and analyse the composition of carbamate solution to HP scrubber.

I think this problem has two causes: the scrubber bed is choked or collapsed and the partial condensation is taking place at the bottom part of HP scrubber heat exchanger.

Eisa replies:

Thanks for your advice.

- The outlet line and packing bed are washed with HP steam condensate.
- The composition of the carbamate does not change and it is the same as before.
- The delta temperature in the tempered cooling water loop is normal.
- Please explain more about the effect of condensation in the bottom part of the heat exchanger.

Do you have any experience of melamine carryover to carbamate, resulting in a polymer in the packed bed?

Have you experienced that partial collapse in the rupture disc creates this problem?

We cannot increase the carbamate flow because the temperature of the HP scrubber outlet pipe to the LP absorber would decrease and the synthesis pressure would increase.

Mr Hasan Kashany of KPIC in Iran asks for further clarification:

How much is the LP absorber pressure and HP scrubber level at that time? Are these items constant or decreasing?

Eisa replies:

The LP absorber pressure is constant but when the temperature in the outlet line of the HP scrubber decreases, the pressure of the LP absorber decreases too, because less gas is coming from the HP scrubber and in that time we cannot increase the carbamate flow. The HP scrubber level also decreases.

Mark comes back:

I still feel there might be a problem with fouling/clogging of the high pressure inert valve.

- Do you have a continuous water flush upstream of this valve?
- Are you sure this is not the cause? If yes, why?

For me, it is difficult to imagine the packed bed can be fouled in such a way that it can have these consequences. And if melamine related "polymers" are causing fouling, can they be dissolved so easily with water?

Mr Majid Mohammadian of OCI Nitrogen in the Netherlands:

As I remember, in the top of the HP scrubber there is a block valve then the steam condensate connection that is not continuous and then the HP inert valve and downstream of this valve there is a steam jacket to LP absorber. The main reason for carbamate crystallisation in gas phases is low N/C ratio, so first of all increase the N/C ratio in the synthesis so that you also have a higher N/C ratio in the gas phase.

I believe that you are experiencing crystallisation/choking due to low N/C ratio in the synthesis. Make sure that the HP ejector performance is good and there is no problem with the level in the scrubber, especially when changing the carbamate pump or during fluctuation.

Mr Channing Spears of PCS in the USA provides some valuable suggestions:

How often is the HP inert valve flushed and what percentage is it open? What is happening to the rest of your synthesis section? What is your N/C ratio? Are any other temperatures dropping besides that of the HP scrubber outlet? When I read decreasing temperatures and increased synthesis pressure, my first thought was that you were too high on the NH_3 side.

Eisa replies:

Water flushing of the gas line to the LP absorber and packed bed is done only when there is a problem and in normal condition it is not in use.

I agree with Mark about melamine polymer because it cannot be easily removed with water flushing.

The gas line and HP inert valve are open because during the problem there is carryover of carbamate to the LP absorber and the opening of the gas outlet control valve of the LP absorber does not change.

During water flushing the inert valve was fully open and in normal position it is open more than 80%.

The N/C ratio is about 3.2 according to N/C meter and laboratory analysis and the other temperatures are normal (for example the temperature of the reactor gas line to the HP scrubber is 184°C).

Two questions remain:

- What, if any, is the effect of partial opening in the internal rupture disk in the HP scrubber?
- With temperatures of about 102°C in and 11°C out of the tempered cooling water loop how can we have partial clogging in the packed bed?

Mark replies:

When you have a damaged cylinder (rupture groove part) in the HP scrubber, carbamate gas from the reactor will bypass the heat exchanger part of the HP scrubber and will go directly to the absorber part or gas outlet of the HP scrubber. Your load on the LP absorber will increase. Pure carbamate (i.e. no water) crystallises at 153°C.

- What is the N/C of the recycled carbamate and what is its water content?
- When you see carbamate carryover to the LP absorber during the problem, it makes me think of flooding.
- Is the carbamate flow much larger than design?
- If yes, have the HP scrubber internals been adapted for the larger flow?

With the above suggestions and information Eisa received support to identify out the most likely causes of the problem and has been able to solve it more quickly.

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Problem No. 17 Loose liner or overlay welding?

In urea plants, high pressure equipment typically has a carbon steel pressure-bearing wall, which is protected against carbamate corrosion by means of a protective layer, typically made from 316L UG or 25-22-2 stainless steel.

Typically a loose liner or overlay welding is selected to provide this protective layer. But what is the best choice, what

are the advantages and disadvantages of loose liners and overlay welding and what are the actual experiences in the field?

An important difference between the two options is that an overlay welding leads to a leak before break scenario, while a loose liner leads to a break before leak scenario. Loose liners therefore require a proper leak detection system.



Mr Mark Brouwer of UreaKnowHow.com in the Netherlands starts up the following discussion: What is more reliable/safe/easy to maintain as a protective layer for the carbon steel pressure-bearing walls of high pressure equipment items in urea plants: a loose liner or overlay welding? What are your experiences?

Mr Muhammad Adnan Hanif of Fauji Fertilizer Corporation in Pakistan replies: In my opinion, a loose liner is the more attractive option compared to overlay welding owing to:

- ease of installation especially when the area to be covered is large e.g. a high pressure reactor;
- maintenance is easier and less time consuming as normally a new liner is installed on the old liner without cutting/removing the old one;
- thickness monitoring of a loose liner is easy and preventative maintenance planning is easy as the lining area is normally divided into a number of sections.

Mr Qaisar Abbas of SAFCO, Jubail in Saudi Arabia also shares his experiences: We have both types of protection (loose liner and overlay welding) in different HP equipment items in our TOYO technology urea plant. The domes of the stripper and condensers are protected by overlay welding while the shells of the reactor and scrubber are protected by a looser liner.

It is my understanding that overlay welding of large areas is not practical, therefore a loose liner is preferred. For vessel areas with a difficult configuration (e.g. a dome), overlay welding is preferred. The consequences associated with the failure of a loose liner are more serious than the failure of an overlay welding.

There is not much difference in inspection approaches (VI, UT, DPT) for both techniques, but I agree that repair and maintenance of a loose liner is relatively easy compared to overlay welding.

Mr Waqas Habib, Process Engineering Unit Manager of Engro Fertilizers Limited in Pakistan joins the discussion: Although I am not an inspection engineer, in my view a loose liner is recommended whenever someone wants to design a weep hole detection system. With overlay welding I don't think a weep hole detection system makes any sense. Provided that there is confidence with the quality of the overlay welding and there are no concerns that there will be leakage of carbamate through the overlay welding into the carbon steel shell prior to the next inspection, then overlay welding may be fine.

Mr Sajid Aziz of FFC in Pakistan adds his opinion: In my opinion weep hole detection systems also exist in overlay welding configuration. In such cases the weep hole is provided exactly behind the weld seams because leakages occur from weld seams. A groove is present under the weld seams to lead the leakage towards weep hole.

In a loose liner configuration the life of an equipment item can be increased by providing an additional lining if the retiring thickness is going to be reached, while in the case of a weld-overlay configuration a retiring thickness of sacrificial covering means the end of equipment life.

I suggest that a loose liner is the better option as compared to overlay welding unless you use a material for the overlay welding configuration which has zero corrosion rate along with high erosion resistance.

Mark asks for some clarifications: Sajid, as overlay welding is welded over the complete surface I do not understand your statement the a "weep hole is provided exactly behind the weld seams".

Also, the installation of a second liner on an existing one is not without risks: With a liner one should always assure proper functioning of the leak detection system. Furthermore one loses the chance to inspect the inside of the carbon steel pressure-bearing wall. For your information, overlay welding can be repaired and extra welding could be done or a liner could even be installed on a section with an overlay welding.

Mr Ramin Jafarvand of CCM in Iran shares his experiences: The main purpose of installing the protective layer in HP equipment is protection of the carbon steel pressure-bearing wall. It is important to be aware of any defect in the protective layer and a leak detection system is a solution for this situation. Detection of the leakage in overlay welding is not possible in my opinion and considering the high corrosion rate of carbamate, in the case of any defect in the overlay welding, we will be aware just after the vessel wall failure that is very difficult to repair. In my opinion it is impossible in the field in the case of multilayer vessels. So in my opinion a loose liner is safer than the overlay welding for external wall protection, however overlay welding may be used for the protection of internal parts such as the tubesheet or for media with lower corrosion rate.

Mr S.K. Gupta of IFFCO in India shares his view: Ramin's view is very appropriate. For HP vessels handling corrosive liquid a loose stainless steel liner with a proven leak detection arrangement is

the best option. Early leak detection and then preventative action is a must otherwise the consequences may be disastrous. With an overlay weld arrangement no such leak detection is possible. It is a fact that the risk of a leak in the welds of a loose liner are higher than in an overlay weld but at the same time early leak detection is possible with loose liners.

Mr Javant Panvelkar of Zuari Industries Limited in India introduces an innovative idea: Having overlay welding and having a multi layered shell suitable of leak detection system looks like a very good option. This may be achieved by having the first layer of carbon steel after the welding to be of lesser thickness and then having a leak detection system to pick up any leakage after this first layer of the carbon steel.

Waqas shares more experiences: We have experienced 3 to 5 mm pitting on the overlay welding in the dome area of our urea reactor. In the straight portion, a loose liner is present and has been OK. Since the reactor is part of a CO₂ stripping design, the top section of the urea reactor contains the vapour phase and the solution overflows into the funnel pipe at about 95% height of the reactor. It is strange to find that although some of the liner portion (in straight portion below tangent line) also sees the same vapours but does not contain any corrosion or pitting marks. Only the overlay welding section in the dome area has concentrated corrosion in three regions. Also the corrosion is not uniformly spread throughout the dome. Has anyone else experienced this and found the root cause for it? Is this corrosion due to condensation of corrosive carbamate in the vapor region, due to excessive plant start-ups, due to holding of reactor contents for more than 24 hours, and/or due to formation of the heat effected zone (crystallization of overlay welding material causing the change in metallurgy) during the overlay welding process while manufacturing of the reactor took place? The reactor was first commissioned in 1998 and the last inspection of the dome area was done in 2005. The material of overlay welding and the loose liner is SS-316 L Urea Grade.

Mark replies: We see the following root causes:

- The dome area with overlay welding has poorer insulation than the channel part with the liner.
- Corrosion occurs due to the wrong welding process. In the overlay weld there are areas where welding strips touch each other, which are subjected to two heat inputs during the fabrication process. Sometimes one can see more severe corrosion at these locations.
- Other things to consider are whether overlay welding is easier to repair. When an overlay weld area shows a leak only a small part of the carbon steel wall sees carbamate (leak before break scenario), but when a liner shows a leak a large area of carbon steel sees carbamate.... a catastrophic failure of the carbon steel wall may therefore be less likely with overlay welding. ■

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Problem No. 18 HP stripper passivation air

The high pressure CO₂ stripper, invented by Petrus J.C. Kaasenbrood of Stamicarbon in 1967, revolutionised urea process technology, reducing the energy consumption figures of urea plants by roughly a factor two. Further, Stamicarbon and Sandvik developed the austenitic stainless steel 25-22-2 to handle the relatively tough corrosion conditions in a HP stripper. The HP NH₃ stripper, introduced by Saipem, operates at higher temperatures than a HP CO₂ stripper and materials of construction like titanium and zirconium,

sometimes in combination with 25-22-2 were therefore applied.

Back in 1955, Joseph P.M. van Waes of Stamicarbon made the discovery that austenitic stainless steels could be protected against carbamate corrosion by adding oxygen in the urea process. The oxygen forms a chromium oxide layer protecting the stainless steel and reducing corrosion rates to acceptable levels. This discussion explores the experiences with different amounts of oxygen in the HP stripper in various urea processes.



Mr Le Ngoc Ban of Phu My Fertilizer Plant in Vietnam starts up the following discussion: Can anybody tell me how long we can keep running our urea plant without HP stripper passivation air? Do I need to reduce the plant load or adjust any high pressure process parameters? Are there any warnings? What is your experience? Our plant is Saipem process technology.

Mr Riaz Ahmed Khan of Agritech Limited in Pakistan shares his experiences: Constant supply of passivation air is a must to ensure that a chromium oxide layer is intact on steel. This layer can be damaged during the process so there should always be a supply of air, oxygen in particular, to repair the layer. All plant equipment has a certain degree of resilience, fully austenitic steel is more sensitive than for example duplex steel. You can operate the plant without passivation air for 30-40 mins for austenitic steel and for duplex maybe a little longer. During operation of the plant without air supply, keep an eye on the colour of the melt/product. If there is any change, stop immediately.

Mr Muhammad Farooq of Agritech Limited in Pakistan adds: It is most important to stop the urea plant if air failure has occurred to the synthesis loop. Most licensors recommend a maximum time of 10-15 minutes only, because once oxygen deficiency occurs it will break the chromium oxide layer and, not only the HP stripper, but the whole synthesis loop will need to be re-passivated. This is not only a HP stripper requirement, it is the requirement for the whole synthesis loop. Moreover, monitoring of oxygen should be carried out at the vents of the recovery section to check for explosivity.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands contributes to the discussion: I agree with the critical supply of oxygen to stainless steel or duplex strippers. But with zirconium strippers the situation seems less critical. As I understand it, the first bi-metallic stripper operated without any passivation air with no active corrosion.

Only after corrosion problems (crevice corrosion) occurred did Saipem advise the addition of passivation air to the stripper to reduce the corrosion rates.

Mr Rajkumar Kulkarni of RCF Ltd in India shares his experiences: Plants (Saipem) can definitely be operated without passivation air (on the HP stripper) for a limited period. The requirement is to

keep the stripper bottom temperature below 202°C, as it is known that corrosion is highly pronounced at high temperature of stripper bottom outlet solution.

I do not agree with the times mentioned for sustaining the operation mentioned elsewhere. It is a very complicated phenomenon and very difficult to make an estimate. Better to be safe than end up with a permanent problem.

Le explains further his concern: I think this situation is quite difficult because we really do not know the consequences of running the stripper without oxygen passivation for some time. Some plants are lucky because their licensor gives them recommendations, but others don't. From a process point of view, we try to keep the plant running as long as possible, but the lifetime of equipment items is also important.

I hope that I can learn from more experiences from other plants operating in this situation so that we can run our urea plant more safely and efficiently.

Mr Sam QR of Fertco in India asks a further question: I would like to know the case where indirect air supply is sent to the stripper in the Urea Casale design. How do you monitor the air supply from the reactor pressure control valve to the stripper? In such plants even though the air supply is there to the CO₂ compressor, O₂/air supply is not ensured. Is it assumed that inerts shall go through the vent?

Mark replies: For assuring a passive chromium oxide layer the amount of oxygen in the liquid phase is important, not the amount of oxygen in the gas phase.

Of course under equilibrium conditions there is a direct relation between the amount of oxygen in the liquid phase and gas phase. But do equilibrium conditions exist in all areas in the urea synthesis section?

For example, in a Stamicarbon CO₂ stripping plant oxygen is dosed only via the CO₂ feed. This oxygen meets liquid in the stripper, condenser and reactor and will dissolve more or less in the liquid phase thus taking care of the passive layer.

I believe one needs experience to find the minimum amount of oxygen required in the CO₂, to ensure sufficient oxygen in the liquid phase at the most critical area of the urea synthesis section (in a Stamicarbon plant it is the top of the stripper, where temperature is high and partial oxygen pressure in the gas phase is lowest). I believe a similar situation is valid for a Saipem or UCSA process.

Mr Majid Mohammadian of OCI Nitrogen in the Netherlands returns to the original question: Referring back to Le's original questions, we had the same discussion at the UreaKnowHow.com workshop in Kuala Lumpur at the Nitrogen+Syngas Conference and there were different opinions on that in Saipem plants.

As per their experiences, some stopped the plant after one day, others after 15 minutes or 30 minutes and some stopped immediately after air was cut to the stripper.

At present, what is the procedure in your plant in the case of passivation air failure to the stripper and is there any proven reason for that?

Le replies: In my plant, we don't have a procedure in case of stripper passivation air failure, but some years ago, we faced this situation and at that time we kept the plant running for about 4 hours, until the air compressor restarted. The same year, we opened the stripper for inspection and found a little corrosion in the bottom, but our inspector concluded that it occurred due to not enough air in general, so we doubled the amount of air from that moment. What is your experience?

Majid replies: Actually, in Stamicarbon plants we are not allowed to work without passivation air and in the case that the oxygen percentage is less than 0.5 vol-% in the CO₂ feed for 15 minutes we have to stop the plant. This is the case for 25-22-2 material. For Safurex material we have the same time but the oxygen percentage is 0.3 vol-%.

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran shares his experience: In our Stamicarbon CO₂ stripping plant, the normal operating range of oxygen after the hydrogen converter is between 0.6 and 0.65 vol-%. The oxygen content should not drop below 0.6 vol-%. In this case, according to licensors instructions, when the oxygen content remains below 0.55% for more than five minutes the plant must be shut down, because active corrosion can occur.

Mr Saadat Motamedi of Pardis Petrochemical Company in Iran contributes to the discussion: I think the shut down of the plant is according to the licensor instructions and it depends on the material used in synthesis loop.

Mr Jithendra Kumar of FERTIL in United Arab Emirates shares his experiences: I have had experience in my previous plant, it was Saipem technology, running for more than 12 years without adding passivation air to the HP stripper. Thereafter, frequent tube sheet leakage and corrosion occurred and so we provided passivation air via a dedicated compressor to the HP stripper. The stripper was bimetallic material (2RE69 and zirconium).

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia shares his experience: In our Stamicarbon plant, the passivation air commonly used is 0.6 to 0.7 vol-% but can be varied upon annual corrosion tests of vessels.

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Problem No. 19 High pressure pumps – which is better reciprocating or centrifugal?

In every urea plant, high-pressure pumps are used to pump the feedstock ammonia and carbon dioxide to the high-pressure synthesis section and in most urea plants carbamate liquid is recycled from the recirculation section to the synthesis section.

Traditionally, plant capacities were small, synthesis pressures were high and reciprocating pumps were typically

applied. With larger plant capacities, stripping urea technologies and the development of centrifugal pumps, more and more centrifugal pumps are being applied.

This discussion looks at the various advantages and disadvantages of reciprocating and centrifugal pumps, based on operating experience in the urea industry.



Mr Majid Mohammadian of OCI Nitrogen in the Netherlands starts up the following discussion:

Please share your experiences, ideas and selection criteria for high-pressure pump types in urea plants. Is it a matter of cost, efficiency, capacity, etc.?

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran replies: In my experience, centrifugal pumps are better than reciprocating pumps for the following reasons:

- With a centrifugal pump flow control to the synthesis section is better than with a reciprocating pump because, with a centrifugal pump, flow is controlled by a flow control valve with minimum fluctuations, but with a reciprocating pump the flow must be controlled by changing the plunger speed.
- Start up is easier with a centrifugal pump compared to a reciprocating pump.
- The maintenance cost with a centrifugal pump is lower than with a reciprocating pump.
- Some parts of the plunger packing that is used in a reciprocating pump can be damaged and enter the synthesis section, where they may block the liquid distributors of the HP scrubber and also in the top of the HP stripper, causing them to malfunction. This risk doesn't exist with a centrifugal pump.
- From an environmental viewpoint, a reciprocating pump has more leakage than a centrifugal pump. A centrifugal pump can meet the most stringent environmental regulations.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands adds some arguments to the discussion and asks a question: Reciprocating pumps are better with respect to investment costs and efficiency, but what about reliability?

Mr Saadat Motamedi of Pardis Petrochemical Company in Iran shares his experiences: Change of flow during operation is easier with a centrifugal pump and is possible with minimum flow fluctuations.

Majid comes back: Do you have any price figures for the same capacity for both types? Do you have power consumption figures for both types at the same capacity? As regards reliability, I think that centrifugal pumps are more reliable due to easier operation, less maintenance and better safety.

Mr Mahmood Zafar of FFC Mirpur Mathelo in Pakistan contributes to the discussion: A reciprocating pump is far better than a centrifugal one for the following reasons:

- Costs: A centrifugal pump requires two auto valves in the discharge, one for feed and the other for recycle to maintain minimum flow during low plant load. This means that a recycle line with other accessories are also required. A reciprocating pump requires only one auto valve for feed isolation.
- A centrifugal pump always needs more power than a reciprocating pump for the same flow.
- A centrifugal pump requires more seal flush water flow than a reciprocating pump for carbamate service.
- The change over of a reciprocating pump is relatively easy compared to a centrifugal pump and causes less disturbance to the process. I have experience with both pump types for carbamate service.

Mr Muhammad Farooq of Agritech Limited in Pakistan shares his opinions and experiences: The following points are well known for pump selection:

- Manufacturers generally provide a chart that indicates the zone or range of heads and flow rates that a particular pump model can provide. Before selecting a pump model, examine its performance curve, which is indicated by its head-flow rate or operating curve. The curve shows the pump's capacity and also indicates pump size, type, operating speed and the pump's best efficiency point. The pump operates most cost effective when the operating point is close to the best efficiency point (BEP).
- A centrifugal pump has varying flow depending on pressure or head, whereas a reciprocating pump has a more or less constant flow regardless of the pressure. A centrifugal pump loses flow as the viscosity goes up but a reciprocating pump actually increases flow.

In my opinion a centrifugal pump is preferable for carbamate liquid. This is based on our 13 years experience of trouble-free operation with a centrifugal Ebara pump. Maintenance costs are negligible, whereas in the case of a reciprocating pump it requires a lot of maintenance of the plunger, packing, gear box problems, pump stoppages and safety matters. Carbamate centrifugal pumps are much easier to handle and easy to change over in operation.

An overview of the differences between centrifugal and reciprocating pumps is given below:

- The most obvious reason to use a reciprocating pump is when you have a high viscosity application. It is common knowledge that a centrifugal pump becomes very inefficient at even modest viscosity. However, there are many other reasons to select a reciprocating pump over a centrifugal other than high viscosity. In fact reciprocating pumps are very commonly used for thin liquids like ammonia and solvents.

- A simple rule of thumb is you should consider using a reciprocating pump whenever you might be operating a centrifugal pump at other than at the best efficiency point (BEP). Of course the further away from the BEP you get the more likely a PD pump will be a better choice. This can typically happen at low flow conditions, modest to high head conditions, or any type of elevated viscosity. As you can see from the efficiency curve it takes more horsepower to operate a centrifugal outside of its BEP. This horsepower has a cost, the initial cost of the larger motor plus a higher life cycle cost in energy consumed. Often the reciprocating pump will have a lower initial cost as well as a lower operating cost.
- Another reason to use a reciprocating pump would be if the application has variable pressure conditions. A centrifugal pump will travel up and down the curve, which can cause process problems. A reciprocating pump will give near constant flow that makes it possible to match the flow to the process requirements. The desire to have constant flow is the reason that a reciprocating pump is the pump of choice for metering applications.
- Obviously, if there is changing viscosity in the application the reciprocating pump is the best choice. As can be seen from the charts, viscosity has a major impact on the centrifugal pump performance. Even a small change in viscosity, like 200-400 SSU, has a large impact on the centrifugal.
- Reciprocating pumps generally can produce more pressure than centrifugal ones. This will depend on the design of each pump but pressures of 250 psi (580 feet) are not unusual for a reciprocating pump with some models going to over 1000 psi (2,300 feet). This is a significant difference between the two principles. The capability for a reciprocating pump to produce pressure is so great that some type of system overpressure protection is required.
- Generally speaking, pumps tend to shear liquids more as speed is increased and the centrifugal is a high speed pump. This makes the reciprocating pump better able to handle shear sensitive liquids. Shear rates in reciprocating pumps vary by design but they are generally low shear devices, especially at low speeds. Internal gear pumps, for example, have been used to pump very shear sensitive liquids. It is important to contact the manufacturer for specific information on shear rates and application recommendations.
- By their nature, reciprocating pumps create a vacuum on the suction side so they are capable of creating a suction lift. The standard ANSI centrifugal does not create a vacuum so it cannot lift liquid into the suction port. There are self-priming centrifugal designs that can lift liquid an average of 15 feet. This corresponds to a vacuum of 13" Hg. Wetted reciprocating pumps (a pump that is not full of liquid but with some liquid in it) can often reach vacuums of 25 to 28" Hg. So a reciprocating pump is the logical choice when there is a suction lift required.
- As mentioned earlier, reciprocating pumps tend to run at lower speeds than centrifugal ones. This will have an impact on seal life, so reciprocating seals tend to last longer than seals in centrifugal pumps. In addition, to assure adequate seal life a

centrifugal will typically require one of the seal flush plans. A reciprocating pump, because of its lower shaft speed typically does not need an external flush plan. Also, generally speaking, low speed mechanical devices tend to operate longer than high speed mechanical devices.

- At certain combinations of flow and pressure centrifugal pumps are inherently inefficient, due to the design of the impeller and the short radius turn the flow must make. These applications are generally under 100 gal/min but particularly under 50 gal/min. A reciprocating pump, by contrast, is very well suited for low flow conditions. Centrifugal pumps, by contrast, tend to do very well in high flow conditions.

Mr Muhammad Adnan Hanif of Fauji Fertilizer Company Limited in Pakistan shares his opinion: I agree with Mehmood and I think reliability is the only argument that favours a centrifugal pump.

Majid replies: Do you have experience with reciprocating pumps for large plant capacities (>3000 t/d)?

Mr Faraham Jafarvand of NEWJCM Turbomachinery Manufacturing Co. in China provides valuable information: I agree with those that are of the opinion that a centrifugal pump has a higher reliability compared to a reciprocating one. Despite the higher efficiency of a reciprocating pump, it usually requires a lot of maintenance compared to a centrifugal pump. Regarding power consumption, I have figures for Ebara pumps for a 3,250 t/d urea plant. HP ammonia pump: 139 m³/h and dP = 171 bar leads to a driver power of 1200 kW (of course, the required power is considerably less than this amount; about 1035 kW), efficiency = 65%. HP Carbamate pump: 84 m³/h and dP = 147 bar leads to a pump rated power of 600 kW and a driver of 700 kW.

Mark replies: I can give an indication on price: 50 m³/hr centrifugal HP carbamate pump, power driven, 2004: 600.000 Euro ex works.

Mr Muhammad Sarfraz of Fatima Fertilizer Ltd. in Pakistan gives his opinion: I agree with those supporting centrifugal pumps but one major factor may also be considered during the initial investment phase: capital cost. A reciprocating pump is very competitive compared to a centrifugal one.

Mr Meer Salman Muddasar of Engro Fertilizer Ltd in Pakistan shares his experiences: We are operating a 21 years old relocated urea plant with reciprocating pumps for both ammonia and carbon dioxide. We have faced several issues over the years but a reciprocating pump is much easier to operate, e.g.:

- During startup feed into the reactor, if the carbon dioxide compressor is centrifugal type, the flow in and out would surge with changes in pressure of the synthesis loop.
- For plants with power and steam limitations reciprocating pumps, power driven, are fine.
- Very fine and easy control of the N/C ratio since a centrifugal pump disturbs the flow with change in synthesis loop pressure. ■

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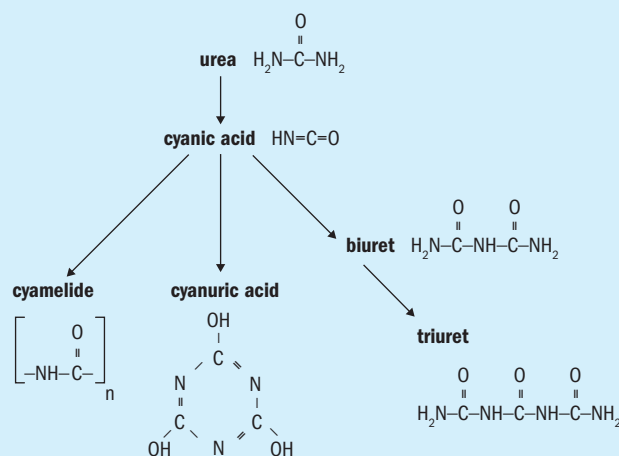
Problem No. 20 Unblocking pipelines after a urea plant shutdown

In several sections of a urea plant pipelines, and sometimes heat exchangers, can easily become blocked below a certain temperature and crystallisation can take place. In the evaporation section, urea melt is present which crystallises at about 133°C when in the pure form.

Urea can also polymerise to form biuret, triuret and poly-urea with high crystallisation temperatures when urea is kept for a prolonged period at high temperatures.

The internals of other equipment can also suffer from these problems (see picture below, showing poly-urea in the internals of a vacuum separator).

In the recirculation section, ammonium carbamate is present which crystallises at about 153°C maximum when in the pure form. In the high pressure synthesis section, urea and carbamate mixtures are present. During normal operation the



Above: Possible reaction products formed from urea during exposure to high temperature.

risk of lines and heat exchangers becoming blocked is small, however during a plant shutdown blocking can occur when the right measures are not taken.

In other industries, applying heat may be a normal procedure when solids block a pipeline or heat exchanger. In a urea plant it is a little more complicated – when solid ammonium carbamate is heated up it decomposes into gaseous ammonia and carbon dioxide. When urea is heated up, however, it partly decomposes into gaseous ammonia and isocyanic acid and partly reacts to produce other products like biuret, triuret and other poly urea molecules, which have even higher melting points. Pure biuret, for example, has a melting point in the range of 185-190°C and pure triuret 231°C. Alternative measures are therefore required to unblock pipelines and heat exchangers. This discussion looks at which measures to take and what to do when a line is blocked and is a follow-up of an earlier Round Table Discussion.

Mr Manzoor Ahmad Taraqfadar of Bangladesh Chemical Industries Corporation in Bangladesh introduces the problem of blockages in urea plants after shutdown:

After a shutdown, when we are ready to start-up, on several occasions we have experienced a line blockage. We are not sure whether the white material is ammonium carbonate or ammonium carbamate. Normally this blockage can be removed by hot water, but when water washing is not possible without dismantling the pipeline we have tried flame heating of the pipe from the outside by removing the insulation. However, most of the time it was unsuccessful and it was necessary to dismantle the pipeline.

I would like to know the following:



- What is the white material?
- How to avoid its formation after a shutdown?
- What is the standard method for unblocking?

Previous discussions have provided a number of explanations to these questions:

The white material may be urea or ammonium carbamate, which solidify upon cooling or when they have sufficient time to solidify. Biuret, triuret and other poly-urea components are also white materials, so it is not possible to identify which solids are formed from the colour.

A possible cause of blockages can be the improper flushing of the sections during the shutdown procedure. After shutdown, all sections should be thoroughly flushed with hot condensate.

To avoid blockages, it is always recommended that, in each shift, the drains of all critical lines subject to blocking are checked. If the line has tracing it should be in service and it should be ensured that the steam trap isolations are removed and they are in service.

One method to unblock lines is to heat either directly by injecting condensate or steam into the pipeline or by indirect heating via a flame or steam outside the pipeline. If the blockage can be located it can be targeted at that point. If the point of blockage is not known, the flanges in the line can be opened and direct heating can be applied. However, care should be taken when unblocking the pipelines in a urea plant by flame heating because overheating or localised heating may cause thinning and damage of the pipeline as has been experienced during the unblocking of a reactor drain line. Heating up austenitic stainless steels like 316L UG or 25-22-2 and duplex steels can adversely affect the corrosion resistance of these materials. For example, heating 316L UG above 500°C will increase the corrosion rate as chromium will form chrome carbides Cr₂₃C₆ with any carbon present in the steel along the austenite grains. This causes depletion of chromium from the austenitic grains, which reduces the protective passive film.

Another option is to dilute the solution (in cases where the solution is being held) with condensate.

Other critical areas where blocking can occur:

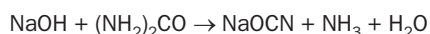
The same thing happens mostly in urea melt lines where solidification is quick after shutdown if the drain line is also found to be blocked.

Another critical area is high concentrated (99%) urea heat exchangers in the vacuum stage. Concentrated urea solution if allowed to remain stagnant at higher temperature in the heat exchanger can result in the pyrolysis of urea to form biuret/triuret. This problem can arise say if 99% urea pumps lose suction for any reason, steam to the heat exchanger is open and solution is stagnant in the heat exchangers for some time. Removal of this sort of blockage is not possible by steaming.

Other solutions for unblocking:

Mr Rajkumar Kularni of RCF LTD in India offered an interesting suggestion to identify areas where the temperature has dropped due to problems. He suggested providing skin temperature measurements for the most probable and difficult to access points. This is a low cost solution which can immediately detect any problem, and can also help in isolating/identifying the problem area (blockage) thus helping to solve the problem quickly.

Mr Jayant Panvelkar of Zuari Industries Limited in India introduces another solution: We have a melter in our urea crystallisation section and during shutdowns we circulate 10% caustic solution at 95°C to remove biuret deposition. Urea can react with caustic soda forming sodium cyanate, ammonia and water according to the following reaction:



Biuret, triuret and other poly-ureas also dissolve very well in urea melt and many urea plants apply urea melt flushing connections

to get rid of this fouling. Of course these flush lines need an after flush of water to avoid urea blocking.

Follow up discussions:

Mr Tarun Batra of National Fertilisers Limited in India continues the discussion by sharing his experiences: We experienced a blockage problem in the second stage evaporator heat exchanger, following stagnant 99% urea melt at high temperature. Blocking is most likely due to condensation products of urea/polymerisation products.

Due to the low heat transfer caused by some completely blocked tubes, we can only manage operations with an increased steam pressure.

Mr Rajan Khular of Chambal Fertilisers & Chemicals Ltd. in India contributes with his experiences: The first thing that is taught to someone new to the urea plant is of course the crystallisation temperature of urea melt. Flushing and re-flushing to ensure clearance of all critical headers is a must for trouble free start-up of urea plants. Reaching a stage requiring local gas heating indicates total negligence immediately after plant stoppage. In fact the only two things of concern immediately after shutdown are the thorough flushing of the vacuum section and the discharge line of the carbamate feed pump.

Typical carbamate blocking of the ammonia condenser in a Saipem urea plant, due to carryover of carbon dioxide from the medium pressure absorber, can be cleared by repeatedly filling and draining hot water on the shell side of the condenser.

Mr Waqas Habib of Engro Fertilizers Limited in Pakistan comes up with two possible causes: If the blockage has occurred, even after adequate flushing, consider the following:

- There may be an opportunity that allows the reaction of ammonia and carbon dioxide vapours even after the flushing is complete.
- The pipe is not steam traced or if it is traced, the tracing steam valve is either closed or its steam trap is blocked.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands draws attention to a related article in the E-Library of UreaKnowHow.com: "1931 Davis Ind Eng Chem Liquid Area of Urea-Ammonia-Carbon Dioxide System", which indicates the crystallisation temperatures of various synthesis mixtures.

Mr Ease Norozipour of Khorasan Petrochemical Company in Iran provides recommendations and a new solution for unblocking: Besides the points already mentioned, I think the following items should also be considered:

- tracing and isolation must be checked and in service;
- flushing of lines must be performed carefully before and after each shutdown;
- check valves in the pipelines must be checked to ensure they are working properly;
- the isolation valves must be checked to ensure they are working properly;
- direct flame heating is not good for unblocking. We have used a thermal stress relief device to control the temperature. ■

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Problem No. 21 Vibrations in the discharge line of the HP reciprocating carbamate pump

In all urea plants, high pressure pumps are used to pump the feedstock ammonia and carbon dioxide to the high pressure synthesis section and in most urea plants carbamate liquid is recycled from the recirculation section to the synthesis. Often reciprocating pumps are used for these services and

sometimes vibration problems occur in the discharge pipeline. This Round Table discussion considers the possible causes and solutions of this problem. It soon becomes evident that many possible causes can play a role and an easy and quick solution does not always exist.



Mr Mark Brouwer of UreaKnowHow.com in the Netherlands initiates a discussion by asking how vibration problems of the discharge carbamate line from the HP reciprocating pump can be reduced.

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran comes up with four possible causes:

In my opinion, the vibration can be caused by the following:

- the piping supports may be loose;
- internal defect in the pump e.g. in the plunger, internal circulation etc.;
- internal defect in the check valve in the discharge pipe near the HP scrubber;
- partial plugging of the outlet pump strainer.

Mr Muhammad Farooq of SAFCO in Saudi Arabia shares his experiences: In my opinion the following points may be considered:

- design of piping, (insufficient diameter, improper layout);
- stress during initial fabrication;
- improper supports;
- impact of recycle line vibration on pump itself;
- external impact such as vibration due to nearby rotary equipment items or any structure.

To find the root cause of the vibrations it is important to know whether it is a design fault due to improper isometric drawings or poor fabrication. Then measures can be taken accordingly. Adjustment of loose supports/proper hanging, clamps and a support audit can lead to better results.

Mr Muhammad Umar of FCC in Pakistan asks a question for further clarification: Before making any comments, are orifices installed in the discharge piping and what type of reciprocating pump is it?

Mr S.K. Gupta of IFFCO in India makes an important suggestion to clarify the problem: In addition to the above, in order to rule out the possibility of vapour locking, the suction side operating conditions and suction piping, fittings, pressure drop etc. should also be checked.

Mr Premysl Ververka of Chemoprojekt in Czech Republic asks some further questions and suggests priorities in possible causes: What do you mean by installation of the restriction orifice on the discharge of HP reciprocating pump? Do you have experience of that? The design of the pipe supports is important for

sure. But I still think that the most important item is the design of the discharge resonator, which based on the reflecting principle eliminates the pressure wave from the reciprocating pump. That is or should be the main concern of the pump manufacturer.

Mark adds his recently gained information to the discussion:

I have learned that possible causes could be:

- a leaking valve, in which case all suction and discharge valves along with stuffing boxes should be checked;
- incorrect pipe sizing or booster pumps sizing;
- triplex and quintuplex pumps have flow variations of 23% and 7.1% respectively.
- That natural frequency can easily be attenuated with correctly sized pulsation dampeners and that should be done by the engineering firm with help from the pump manufacturer.

Mr Luis Noque of Bunge in Argentina shares his experiences: In Argentina we have five high pressure carbamate pumps and three high pressure ammonia pumps. These all are Uraca pumps and discharge pressures are around 210 to 215 bars. We have switched to new Uraca pumps (purchased in 2010) with ball resonators in the discharge of the pumps and vibrations reduced considerably.

Another aspect to review and to reinforce are the supports of the pipelines, these must be tight.

Mr Kashif Naseem of SABIC in Saudi Arabia shares his knowledge: HP reciprocating pumps can have vibration issues in the discharge piping, even if the size of the line size is designed properly and stress analysis is OK, if there is no pulsation dampener or there is improper mechanical support.

Mr Reza Mollaei of Pardis Petrochemical Complex in Iran discusses some possible problems with pulsation dampeners:

Variations in the pressure and flow in the discharge lines of reciprocating pumps cause problems in piping systems such as vibration. A pulsation dampener is a device that can reduce these problems. However, possible problems with the dampener can be:

- tearing of the pulsation diaphragm;
- long distance between the pump discharge nozzle and pulsation dampener
- wrong calculation in sizing of the pulsation dampener.

Mr Majid Mohammadian of OCI Nitrogen in the Netherlands asks another question: What is the effect of the number of plungers? Can we say more plungers will reduce the vibration because of a lower rpm?

Mr Raja Fawad Ashraf of Pak Arab Fertilizers in Pakistan also asks a relevant question: Is there any effect of high ammonia content in the liquid? I am specifically asking with regard to the Saipem process. There must also be an effect on the suction liquid temperature.

Mr Aslam Muhammad of FFC in Pakistan introduces timing as a possible root cause: If vibrations have been present since commissioning, then most probably there are mechanical defects but if these started after some time then it may be due to flow disturbances - look at the check valve in the discharge line, concentration of fluid may also be considered, mean temperature and concentration of handling fluid or some obstruction due to mechanical parts like damaged gaskets / cloth in suction eyes of the impeller or other material. Foreign material is most probably the cause if it started at a later stage.

Mark adds more of his recently gained knowledge: A discussion with an expert lead to the following conclusions: Referring to the question: What is the effect of the plunger's number? Can we say more plungers will reduce the vibration because of lower rpm?

If you were to draw a straight line, which represents discharge pressure you would see that a triplex pump has flow variations 6.1% above and 16.9% below this line (23% total) and quintuplex 1.8% above with 5.3% below (7.1% total). Additionally the frequency in which these variations occur will be different between 3 plunger and 5 plunger pumps. For example crankshaft rpm 115 with 3 plungers equals a frequency of 5.75 Hz but the same rpm with a 5 plungers pump is a much higher frequency 9.58 Hz. This lower flow variation and higher frequency makes the 5 plungers by definition a smoother machine. But there is a lot of added cost using 5 versus 3, for instance larger power end with larger crankshaft and intermediate bearings along with more valves and stuffing boxes to maintain. 3 plungers are always preferred because of their simplicity. Only if one cannot meet customer capacity requirement do we go to quintuplex.

And referring to the question: Is there any effect of high ammonia content in the liquid (in particular for the Saipem process)? There must also be an effect of suction liquid temperature. For sure the ammonia content is critical. Due to heat and pressure the ammonia will change to the gaseous state, which is not good for a pump.

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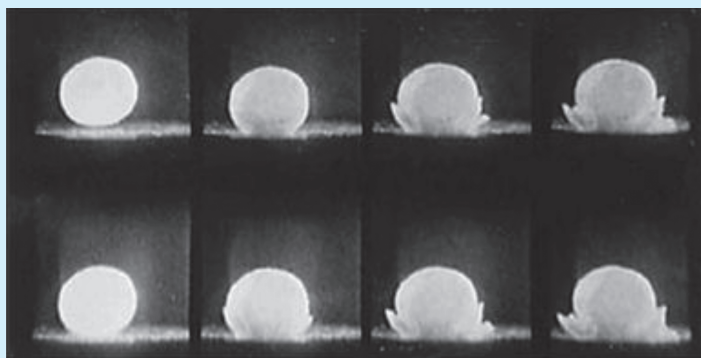
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Problem No. 22 How to strengthen prilled urea product

Approximately 75% of all urea plants worldwide still produce prills. Compared to granules, prills are smaller in size and have lower strength, leading to more dust problems during handling. Furthermore, prill strength is reduced, particularly during hot and humid weather conditions and when plant loads are increased. This Round Table discussion reports on the experiences of different plants and the solutions available to increase the strength of urea prills.

Right: High speed record of impact of a 2mm urea prill at impact velocity of 10m/s. Framing speed: 100,000 frames per second.



Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia initiates the following discussion: We have prilled urea and its strength is very weak. We often have too much undersize. What are the causes of weak prill strength and how can it be improved?

Mr Le Ngoc Ban of Phu My Fertilizer Plant in Vietnam confirms the problem: We have the same problem. Dust in our prilled product (size < 1mm) sometimes exceeds 1% even when we reduce the prilling bucket speed to nearly overflow speed.

Mr Salman Islam of DHCL in Pakistan provides an answer and asks a further question: One reason for excess fines could be improper cooling of urea prills and high bucket speed. Which type of prill tower do you have in your plant?

Kashif replies: Our plant has an induced draft prill tower.

Le also replies: Our plant is using a natural draft prilling tower and a tattle prilling bucket.

Mr. Les Farbotko of Incitec Pivot Limited in Australia shares his experiences: I am not sure if your problem is broken prills and dust, or tiny prills. We experienced both of these problems several years ago, and spent a lot of time and effort trying to improve our prill quality. To make harder prills (i.e. prill which do not break as easily), we found the main factors that made a big difference to us were:

- Moisture content: Prills made with 0.3 wt-% H₂O or less were noticeably harder than prills with 0.5 wt-% H₂O.
- Formaldehyde: Adding 0.3 wt-% formaldehyde to the urea melt upstream of prilling makes stronger prills. (It is preferable to use urea-formaldehyde concentrates rather than aqueous formaldehyde solutions, for safety reasons)

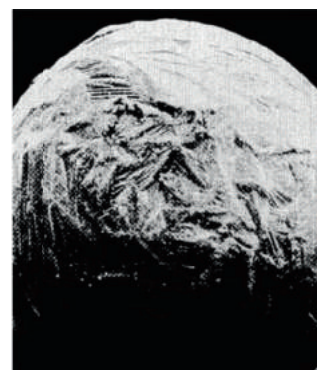
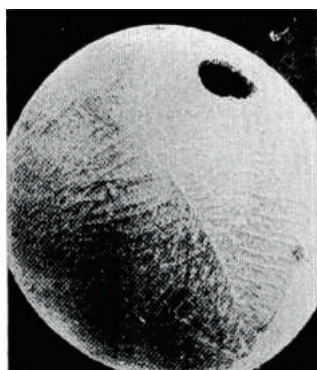
I am reliably told that prill seeding also makes prills harder, although I can't say how much difference it makes.

To make prills with a more uniform size distribution (fewer tiny prills, and less oversize prills which form eggshells and break into dust easily), you need to pay close attention to your prilling buckets. In aluminium buckets, the holes gradually corrode or erode,

becoming larger over time and requiring periodic replacement. This seems to be worse if your prilling temperature is high. We also discovered that it is not easy to drill thousands of uniformly sized holes in a prilling bucket. There is a degree of variability in hole diameter that is related to the amount of care taken, the sharpness of the drill bits and the drilling technique.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands contributes to the discussion: As regards strength of prills we need to distinguish:

- Crushing strength (how much weight a prill can withstand before it crushes)
- Impact strength (with which speed a prill can hit a wall before it is destroyed)
- Adding Formaldehyde has some positive effect on the crushing strength (+10-20%)
- Seeding technology improves the impact strength of prills as it ensures that the prills consist of many crystallites.



Above left: Normal prill with cavity due to solidification from outside to inside. Above right: Seeded prill with many crystallites.

Therefore, if your prills are subjected to a lot of handling, or if the prills have to drop from high elevations during handling, seeding technology is beneficial. It should also help to reduce fines at the bottom of the prill tower. Finally, adding sulphur or sulphates also increase the crushing strength.

Mr Janusz Ma kowski of ZCh Police in Poland shares his experiences: Regarding the buckets, I agree that tuttle buckets are very sensitive and if you use them you have to check the condition of the buckets:

- Check that the geometry of the bucket doesn't change over a long time in operation.
- Check if holes of the bucket have still the same diameter.
- Check if the holes are still sharp (not rounded after long time of operation)

If the bucket is not perfectly round, or if the diameters of the holes vary a lot, you will produce an excessive amount of dust. Check also that the bucket isn't tarnished by impurities e.g. corrosion products. In this case the bucket should be replaced for cleaning.

Adding 0.3 wt-% formaldehyde to the urea melt is a very good idea. Have you ever used a vibropriller (vibrobuckets)? What is your biuret content in final product?

Mr Son Nguyen of PVFCCO in Vietnam shares his experiences:

We have a Snamprogetti plant with a natural draft prilling tower. Our product has a moisture content of: 0.27-0.3 wt-%, prill temperature of 50-55°C, biuret content of less than 1 wt-%, urea melt temperature to prilling bucket of 136-137°C and we use a Tuttle aluminum prilling bucket.

We always keep process data as close as possible to the designed values by Snamprogetti but we have found that the dust level in our product is still high: about 1% product can go through a 1 mm screen during the Lab test (screen amplitude = 65), then we tried to improve our prill strength.

While inspecting our product at the bottom of prilling tower, we detected some ammonia smell but unfortunately our lab isn't able to measure the NH_3 content in the urea melt to the prilling bucket. We thought that the free NH_3 in the urea may cause our prills to be weaker by creating capillaries during the crystallization of the urea prill and by getting rid of it we could make our product stronger.

In our plant, we have a H_2SO_4 injecting system with the initial purpose to reduce the NH_3 content in the off gas of the prilling tower and we thought it could help in getting rid of free NH_3 . We tried injecting some H_2SO_4 into the urea melt going to prilling tower, varying the amount of H_2SO_4 at several levels. Then we took samples to measure product strength and we got the following results:

- NH_3 smell at the bottom of the prilling tower is reduced.
- Dust at the bottom of the prilling tower is reduced.
- Specific crushing strength of all samples with H_2SO_4 reduced than without (2 mm size).
- Common crushing strength of samples with H_2SO_4 is generally higher than without.
- Specific impact strength of all samples with H_2SO_4 is higher than without (2 mm size).
- Common impact strength of all samples with H_2SO_4 is worse than without.

These results mean that our product after H_2SO_4 injection is somehow better than before when in static condition, i.e. in storage, but is worse during transportation, when erosion effects takes place,

resulting in increased customer complaints of dust.

We are now thinking of using formaldehyde to solve the problem.

Mark introduces some other solutions: Have you considered applying seeding technology to improve the impact strength? If your customer accepts urea with sulphur or ammonium sulphate, you could also consider adding some ammonium sulphate to the melt. This could be done for part of the urea production by installing Rotoform technology. Another advantage of installing Rotoform technology is that the load on the prilling tower can be reduced to produce larger prills.

Mr Girish Prakash of Tata Chemicals Ltd, Brabala in India asks for some clarifications: Can you provide approximate values for the extra investment per ton for using the pelletising process? Also, as far as I am aware, farming in this part of Asia is mostly by manual means, farm sizes are small, urea application to the crop is mostly by hand and farmers do not prefer large granules for this type of application.

Mark replies: Producing specialties means higher margins. The Rotoform product is harder and contains extra nutrients required by specific crops. The size of the Rotoform product can be chosen freely in a wide range. The Rotoform technology is also very environmentally friendly.

Mr Ahsan Muhammad Sarfraz of Fatima Fertilizer Ltd in Pakistan joins the discussion: My understanding of the problem is that if the moisture and free ammonia contents of prills are kept in range, strength figures would be higher. The FFCL plant is running at 112% load and fines are around 0.2-0.4% with APS around 2.05-2.1 mm using a tuttle bucket and natural draft prilling tower. Crushing strength is around 840~850 g.

Girish asks a question: Crushing strength of prills without formaldehyde or any other type in excess of 800 g seems to be great. Can you please elaborate how you measure the crushing strength?

Mark provides an answer: In our Gallery you can find one simple and easy way to measure crushing strength: One takes ten prills from a certain size distribution (after sieving) and measures the crushing strength. Then one takes the average value. There is more sophisticated equipment also of course.

Mr Malik Sohail of Safco in Saudi Arabia asks for some clarifications: You mention that sulphur/ammonium sulphate can improve the strength, can you tell me the concentration of sulphate in the final product? Also is there any adverse effect of sulphate in product and is there any upper limit of sulphate in product?

Mark replies: AS concentrations typically vary from 20-50 wt-% depending on crop needs.

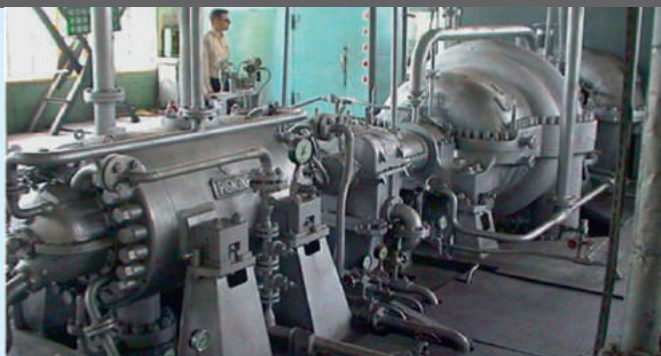
I think the strength already increases at lower concentrations. S and N fertilizer combined give a better crop yield according to several field tests. ■

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Plant Manager+

Problem No. 23 CO₂ compressor capacity limitation

The CO₂ compressor in a urea plant is a unique and interesting high-pressure rotating piece of equipment. CO₂ is compressed from more or less atmospheric conditions to supercritical conditions. The CO₂ is saturated with water, mixed with air and sometimes contaminated with the absorption solution from the ammonia plant. This means that its behaviour is not always easy to predict and having sufficient experience is very important. As the CO₂ compressor is often a limiting factor in the urea plant, how best to debottleneck it is always an interesting topic.



Mr Faisal Ghadoor of Fertil in the UAE initiates the following discussion: We are operating a CO₂ compressor at a suction temperature/pressure of 50°C/0.5 kg/cm²g and a discharge pressure of 139 kg/cm²g. The limitation is the third stage pressure (67.7 kg/cm²g), which is quite near to the PSV setting (70 kg/cm²g).

Can anybody share their experience or give their technical opinion on the following: If we bypass the chiller and allow a higher gas temperature of 58°C and a higher suction gas pressure of 0.62 kg/cm²g, what would the impact be on the interstage pressures/temperatures if we keep the discharge pressure constant at 139 kg/cm²g by adjusting the rpm of the machine?

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia replies: Your increase in temperature will increase the energy consumption of the turbine/motor and also increase the interstage temperature. There are compressors in the world operating at 0.85 to 1.0 barg suction pressure and temperatures of 38 to 50°C, but the drawback will be that more energy will be required in the ammonia plant CO₂ desorber due to the higher pressure.

Faisal replies: The pressure at the CO₂ desorber is 0.68 kg/cm²g. We are cooling the CO₂ gas in a chiller from 60 to 50°C, but it is causing a pressure drop, leading to 0.5 kg/cm²g pressure at the compressor suction.

Mr Girish Prakash of Tata Chemicals Ltd in India asks for a clarification: The pressure drop across the chiller seems to be high. Is it possible to reduce the pressure drop across the chiller?

Faisal replies: No, that is not possible.

Mr Chander Mohan Goutam of NFL in India asks for some more information: What are the suction and discharge pressures of each stage?

Faisal replies: T°C/P kg/cm²a: 1st stage suction: 51/1.5, 1st stage discharge: 195/5.4, 2nd stage suction: 37.5/4.3, 2nd stage discharge: 203/18.6, 3rd stage suction: 29.5/18, 3rd stage discharge: 201/68.6, 4th stage suction: 37/68.3 and 4th stage discharge: 152/141.

Muhammad Kashif asks for some clarification and suggests a possible solution: Please define the Nm³/h flow value of the machine and the power. If you use the scrubbing system for cooling the CO₂ I suggest you change your first inlet cooler to a cooler with a coolant media to cool the CO₂. This will reduce your pressure drop and decrease the energy consumption.

Mr Muhammad Farooq of Sabic-Safco in Saudi Arabia provides his analysis: From the data it seems that the discharge temperatures across your CO₂ machine is too high, which is affecting the capacity. In order to improve the machine you need to decrease the discharge temperature at each stage, especially at the first stage by installing a chiller. In my view the suction temperature can be reduced from 51 to 20°C. We have already done it with our CO₂ compressor. Is the 4th suction temperature correct?

Mr Javed Gujjar of Agritech Limited in Pakistan also finds some of the data odd: Normally there is a concept of LP/HP casings in high pressure centrifugal compressors. These casings are designed to keep the machine away from the thrust. I have experience with two machines and have seen that almost 25% of the total pressure load remains on the LP casing and 7% remains on the HP casing. But according to your data you are making 12% of total pressure in the LP casing and 88% in the HP casing, which will definitely put some limitations for margin of PSVs on the HP stages. Has your third discharge pressure always been so or has it changed since it was first started up?

Mr Ahsan Muhammad Sarfraz of Fatima Fertilizer Ltd in Pakistan contributes his ideas and suggestions: What I conclude from your query is that you want to increase the load across the compressor while the major limitation seems to be across the third stage which is already operating near to the maximum discharge pressure. Besides this limitation can you tell me if there is sufficient margin available in your turbine and compressor speed?

In my opinion, as your fourth suction is lower than the critical pressure you can afford to lower the fourth suction. The focus should be to shift more of the load on the fourth suction, which will really prove helpful in increasing the overall load with little increase of the third discharge pressure. This is generally covering the limitation across the third stage and the rest is taken as normal.

Mr Rajeev Gupta of GPIC in Bahrain shares his experiences:

I would not recommend doing anything without considering the stage specific surge curves as any change in these process variables will cause the operating point to shift and can cause instability in operation of the machine. Also, the prolonged usage of the machine since its commissioning might have caused a drift in the original surge curves supplied by the manufacturer. I think it will be best to consult the supplier and maybe you should carry out a surge test in co-ordination with the manufacturer to evaluate the present characteristics.

Javed also warns of some risks: Decreasing the fourth suction temperature from 37°C by increasing the cooling water flow rate across the exchanger means you will be approaching the critical temperature of CO₂, which is 28°C at the mentioned pressure. Any instability in heat transfer could lead to dry ice formation in a worst case scenario. Therefore, I think it is wiser to remain at these conditions and consult with the manufacturer of the machine rather than risk dry ice formation.

Faisal replies: All temperatures are correct. Yes, there is a chilling facility to bring down the interstage temperatures. We can reduce the suction temperature further; but the concern is that in doing so, the compressor capacity will be increased and the interstage pressure further increases leading to PSV popping. The flow through the compressor is 28,000 Nm³/hr and power is ~7,800 kW. Machine rpm: 7,000.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands asks for a clarification: Were the pressures and temperatures you provided for each stage actual figures? What are the design figures?

Girish provides a solution: If the problem is specifically limited to the third stage there could be internal recycling within the HP barrel. It should be opened and all clearances should be measured.

Faisal replies: The design conditions stage-wise are (kg/cm²g): Suction/Discharge: 0.4/5.3, 4.8/19, 17.1/67, 63/146 at 35,000 m³/hr 1st suction flow.

These have been operating at the following conditions in previous years: Suction/Discharge: 0.44/4.5, 3.6/17.1, 16.6/60.5, 60.2/146 at 31,500 Nm³/hr and 6,915 rpm.

Note: Maximum allowable speed is 7,185. The stage efficiency is definitely an issue. The third and fourth stage calculated efficiencies are 59 and 48% respectively.

Chander comments: Your problem will not be solved with the option you suggest because by increasing the suction pressure, the interstage pressure ratios adjust in such a way that there is no possibility of any reduction of the third stage discharge pressure.

Mr Faraham Jafarvand of NEWJCM in China asks for further clarification: Is the HP casing rotor a back to back arrangement? Is there any balance piston for the HP casing? Is there any flow meter in the balance line? Do you have any idea about the recent compressor's efficiency compared to previous years?

Muhammad Kashif also asks for more information: What is the flow rate of the CO₂ machine?

Faisal replies: Flow rate is normally 28,000 Nm³/hr with CO₂ of average molecular weight 42 at the inlet (with some inerts of course). In the later stages, additional cooling had been added at the interstages that is causing more pressure drop. For the third and fourth stage, differences may be a type error; consider it 67 in both cases with 0.2 kg/cm² differential as being experienced now. Compressor efficiencies in the third and fourth stage have dropped over the last 6 months. We don't have any flow measurement for the HP casing balancing line.

Girish draws a conclusion: Based on the facts, it appears likely that there is some internal recycling.

Muhammad Farooq agrees and offers a solution: I agree with Girish's conclusion that internal recycling is resulting in low efficiency of the CO₂ machine. Increased clearance around the labyrinth can lead to increased temperatures across the stages despite additional chilling, so the high pressure case needs overhauling.

Faraham also agrees and provides another solution: Based on the information provided, I agree that internal recirculation is the root cause of the problem. Internal recirculation can occur through the intermediate labyrinth between two stages or the balance piston. If you had a flow meter in the balance line it would show you the increased bypass flow through the high pressure part to the low pressure part. Your maintenance people should check the clearances and change the labyrinth or balance drum if necessary.

Girish also offers a solution: As an improvement you can install a differential pressure transmitter, if not already present, to monitor the compressor performance continuously.

Mr Muhammad Adnan Hanif of Fauji Fertilizer Company Ltd in Pakistan answers Faisal's question and provides another option to solve the situation temporarily: Is there any strainer downstream of the third stage discharge before the fourth stage suction? If yes, it should be checked as it may be creating a restriction, which has increased the overall 3rd stage discharge pressure.

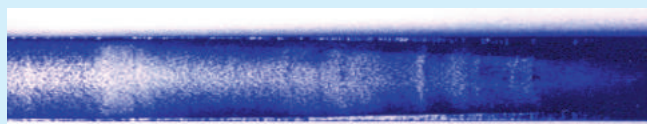
To answer your original question, increasing the suction pressure to reduce the third stage discharge pressure (keeping final pressure constant) will not work. Centrifugal compressors work like a train. If one stage does less work then other stages have to do more and vice versa (if there is margin available in their design compression ratios). However, you may try reducing the suction pressure. As the third stage efficiency is already lower, there is a possibility that reducing the suction pressure may reduce the third stage pressure and the fourth stage may increase its work load (if there is margin available in its compression ratio) to keep final pressure constant at the cost of a higher energy value. ■

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Plant Manager+

Problem No. 24 Why is the minimum urea plant load 60%?

It is commonly known that any urea plant has a certain minimum plant load, typically 60% of its design capacity. But why is this so critical for urea plants? Several equipment items and valves in urea plants could have limitations when operating at a minimum load and there is always the risk of active corrosion.



Active corrosion due to a flooding HP stripper tube.



Mr Omid Fayezi of Pardis Petrochemical Company in Iran initiates the discussion: We operate a urea plant of Stamicarbon design with a pool condenser and a design capacity of 3,250 t/d. I have a question about our start-up procedure. We always start our plant at 60% of total capacity (according to our licensor's recommendation). What is the background for this recommendation, is it due to plant process reasons or corrosion?

Mr Habib Najim of State Fertilizer Company in Iraq provides a process reason: During start-up of our urea plant (Stamicarbon process) we start with an ammonia excess – 100% NH_3 feed and 60% of CO_2 flow to get a N/C ratio of 2.9-3.0 until we have overflow in the reactor. We then decrease the N/C ratio to 2.0 by decreasing the NH_3 flow to 60% before gradually increasing plant load up to 100%.

Mr Nawal Gupta of Kribhco in India follows with a corrosion reason: If the plant load were below 60%, some of the stripper tubes would run dry and there would be no falling film in those tubes. This will result in higher skin temperature of those tubes resulting in severe corrosion.

Mr Shoaib Minhas of FFBL in Pakistan provides another process reason: Our ejector at the discharge line of the HP ammonia pump opening/suction power is also limited below 65% plant load.

Mr Rajan Khular from Chambal Fertilizers & Chemicals in India joins the discussion: I believe you will get the answer to your question once you refer to the "turn down ratio" for which the plant was designed by the licensor.

Mr Easa Norozipour from Khorasan Petrochemical Company in Iran gives his opinion: I think there is minimum plant load of 60% for the following reasons:

- HP control valves in the synthesis loop cannot work properly below 60% load.
- If the plant load is lower than 60%, the distribution of liquid in HP stripper tubes would be lower than the normal value and some of tubes may operate with a very thin film, leading to higher tube skin temperatures, resulting in severe corrosion.
- With lower than 60% plant load, the residence time of the solution in the synthesis will be higher than normal resulting in increased biuret formation in the synthesis loop.

Mr Muhammad Adnan Hanif from FFC in Pakistan makes a statement: In my opinion, the main reason for having a minimum plant load of 60% of nameplate capacity is because the flow controlling system i.e. control valves are sized to remain 60-70% open max at design load i.e. 100% of nameplate capacity (to have enough margin to cope with emergencies). So in order to keep product specifications at the optimum/required level these controlling systems should be operated at their recommended values, i.e. >30 %.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands joins the discussion: I think corrosion risks are the main concern when defining a minimum load percentage. The HP stripper tubes need a minimum liquid level to assure all will be wetted. Also the HPCC and HP scrubber have liquid/gas distributors at the inlet.

Muhammad replies again: In my opinion, stripper tubes are designed for saturated steam temperature at design pressure of the stripper- in our case the design temperature is 235°C for a design pressure of 30 bar. To protect the stripper tubes from higher than design temperatures, a steam to stripper valve closure interlock is also provided.

Mark responds: I agree that the mechanical design for the stripper tubes is like that, but that does not mean one would have excessive corrosion rates under those conditions. Further, the design P and T do not say anything about a minimum liquid load on the tubes to keep them wetted.

Muhammad adds: Keeping all tubes wet is the requirement for equal heat load distribution to utilise all the available heat transfer area in the form of tubes. At reduced load operation, the heat load on the stripper is already on the low side due to lower mass flow of components which have to strip off in stripper i.e. carbamate, NH_3 , CO_2 etc.

Mark clarifies further: No, I mean a minimum liquid level in the top is needed to assure all tubes will get a more or less equal amount of liquid. When one tube gets less liquid than another, more gas will enter this tube, which means that even less liquid will be able to enter the tube leading to a dry tube. CO_2 will start to show a preferential flow pattern. Some tubes will be dry and others fully wetted, so tubes where hardly any CO_2 will be able to enter. Temperatures in these full tubes will rise leading to significantly higher corrosion rates. Stamicarbon is able to adapt the design of the liquid divider to realise a lower minimum load of for example 40% if required. ■

Plant Manager+

Problem No. 25 High temperature in top of HP scrubber

Sometimes, when a process condition changes in a urea plant, for example, an operating temperature, the cause is not clear. Such a situation happened at Kermanshah Petrochemical Company in Iran. Mr Reza Keyhani started a discussion in the UreaKnowHow.com Round Tables to aid his troubleshooting. The following discussion helped Reza to find the root

cause of the problem much more quickly. Mr Keyhani experienced a high temperature in the top of the high pressure (HP) scrubber. The function of a HP scrubber is to reduce the ammonia content in the inert stream coming from the high pressure synthesis section by washing the inert stream with the recycle carbamate and cooling it against cooling water.



The HP scrubber reduces the ammonia content in the inert stream.



Mr Reza Keyhani of Kermanshah Petrochemical Company in Iran initiated the following discussion: The normal temperature at the top of the HP scrubber in our Stamicarbon pool condenser urea process is about 112-115°C and the O₂ required in the outlet of the hydrogen converter is 0.59 vol-%. If the temperature in the top

of the HP scrubber increases to 145°C but the overflow temperature of the scrubber is normal (166°C) what is the cause? Is it due to inerts or is there another explanation?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands replies: I assume you mean the gas temperature in the outlet of the HP scrubber.

The temperature of a liquid phase depends on the system pressure and its composition. If inerts decrease, the system pressure increases and the liquid temperatures increase. If the N/C ratio of a gas increases, the gas temperature will increase. So in your case I assume the inert valve has been opened leading to a higher N/C ratio in the gas outlet of the HP scrubber (higher N/C and lower inerts) and the lower inerts lead to a higher liquid overflow temperature. You state your normal value of the liquid overflow temperature is 166°C, which seems rather high to me.

Mr Ahmed Selim of Alexandria Fertilizers Company (ALEXFERT) in Egypt shares his experiences: In our plant (Stamicarbon falling film HPCC technology), the gas outlet temperature from the HP scrubber reaches 150°C, while the temperature of the liquid overflow reaches 172°C. I think that the N/C ratio is higher than normal?

Mark replies and asks for some more clarifications: It looks like it. I also think the load on the HP scrubber is high (is the position of the inert vent valve wide open?), so the inert percentage is low.

Ahmed replies: You are right, our plant load is about 112% and the inert valve opening is 70% which confirms what you said about increasing system pressure.

Reza comes back: The normal value of the HP scrubber liquid overflow is 165°C according to design. When we experienced problems of high pressure in the LP section the temperature (overflow) was 155-158°C. We tried opening the inert valve to reach 164-165°C

in the overflow and this action helped to us to realise a closed LP vent valve. But the temperature at the top of the HP scrubber increased to 140-142°C, when it should be 112°C according to design. I think it is because we have more inerts than normal. The inert valve is more open and more gas (with inerts) is exiting the inert valve which increases the average temperature of the gas.

Reza asks for further clarification: For the N/C ratio, I think you mean the composition of the gas in the top of the scrubber is the cause of the high temperature. If the N/C ratio is more or less than optimum there is less condensation in the synthesis section and less LP steam production. The remaining gas from the reactor flows to the HP scrubber and condenses there leading to energy loss here as the condensation heat is lost to the cooling water system. Is this the reason (more NH₃ and CO₂ going to the top of the scrubber) that this temperature is high? Were we right to open the valve to 80%?

Ahmed replies: By increasing the opening of the inert valve, the residence time in the HP scrubber will decrease (the velocity of gases from the top of the HP scrubber will increase) so the gases will be vented from the HP scrubber without being condensed leading to an increase of the N/C ratio in the vent of the scrubber and an increase of the overflow temperature from the HP scrubber as a result of the increase in temperature of the gases vented from the HP scrubber.

Mr Saadat Motamedi of Pardis Petrochemical Company in Iran shares his experiences: In our Stamicarbon pool condenser urea plant the gas outlet temperature from the HP scrubber is 130°C and the temperature of the overflow from the HP scrubber reaches 160°C. The opening of the inert vent valve is 32% and the plant load is 96%. I therefore think the inert level in your plant is high.

Mark provides some clarifications: Please take into consideration the following:

- The N/C ratio of a gas in equilibrium with a liquid in urea synthesis always has a higher N/C ratio when one operates at top ridge line conditions or on the ammonia rich side.
- The temperature of a liquid is very much influenced by the inerts content in the gas. The liquid overflow temperature gives a direct indication of how much inert is in the gas phase. The liquid temperature is the condensation temperature at the sys-

tem pressure (so not at the total pressure, as you know total pressure = system pressure + inert pressure). So if the total pressure is 140 bar and the inerts percentage is 5% (7 bar), the system pressure is 133 bar. When the inerts percentage is 10% (14 bar), the system pressure is 126 bar. The liquid overflow temperature is at 133 bar, higher than 126 bar.

So the liquid overflow temperature can be between 155-170°C depending on the inerts percentage. The more the vent valve is open, the lower the inerts percentage. The influence of N/C ratio on the liquid overflow temperature is less than the inerts percentage.

Reza concludes: So, in our plant, with an 80% opening of the inert valve and good HP scrubber overflow temperature, the opening of the vent valve is greater than normal because we had a higher than normal level of inerts in the synthesis and, to remove the inerts, we opened the vent valve to 80% which led to an increase in the temperature in the HP scrubber top.

Mark replies: Yes, and please note that even if one has a low inerts content in the feed, one is free to open the inert valve further than normal. The position of the inert valve can be used to control the inerts percentage in the top of the reactor. Maybe you can reduce the inert amount by reducing the inerts in the feed. There is a technology to reduce inerts in the CO₂ by implementing an additional flash step in the CO₂ washing. Also, the inerts in the NH₃ can be reduced by flashing the ammonia first to atmospheric pressure. In a Saipem synthesis there is no gas/liquid separation in the reactor as

in a Stamicarbon synthesis. In a Saipem synthesis section therefore there are fewer options to control the inert pressure in the reactor.

Mr Omid Fayezi of Pardis Petrochemical Company in Iran contributes to the discussion: In our plant (Stamicarbon pool condenser technology) the gas outlet temperature of the HP scrubber is 130-135°C. An increase of the temperature at the top of the HP scrubber depends directly on the N/C ratio. If the ratio is higher than 3.2, then the temperature at the top of the HP scrubber increases. The temperature also depends on the opening of inert vent valve: if the inert valve is higher than 60%, then the temperature increases. I think the temperature at the top of the HP scrubber also depends on the amount of H₂O in urea and carbamate solutions: if there is an increase of H₂O, the temperature increases. In addition, the flow rate of the recirculating cooling water should not be decreased as this will increase the temperature.

Mr Ahsan Sarfraz of Fatima Fertilizer Company in Pakistan shares his opinion: This is mainly attributed to a higher N/C of the reactor off gas, which means your ammonia slippage from the reactor is higher than design.

Mr Majid Mohammadian of OCI Nitrogen in the Netherlands contributes to the discussion: I think that in Reza's case they have a normal overflow temperature (166°C) but the off-gas temperature is higher than normal so he wants to know how he can adjust both temperatures according to the plant specified data. I think it is possible to adjust the temperatures with the N/C ratio and system pressure. ■

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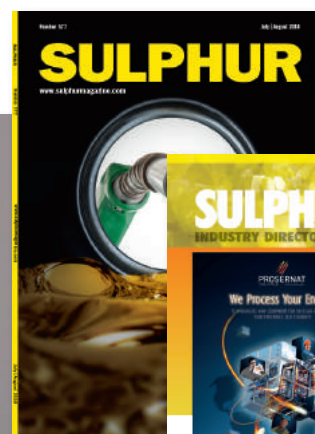
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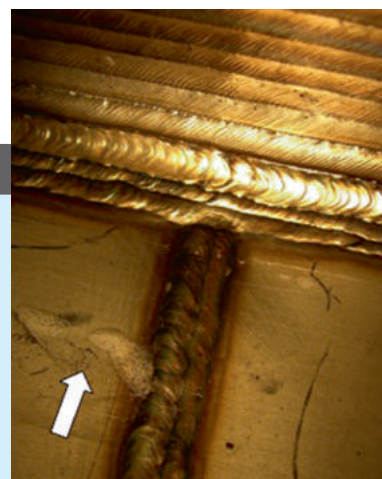
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Plant Manager+

Problem No. 26 How to find a leak in a liner

High pressure urea synthesis equipment consists of a carbon steel pressure bearing wall, which is protected by a protective layer. Carbamate can corrode carbon steel with a rate of 1000 mm per year. The protective layer can be a loose liner or weld overlay. Loose liners require an accurate and reliable leak detection system in order identify in time

that there is a leak in the liner. Once a leak is confirmed, the only advice is to stop the plant and repair the leak. But how can the leak be located? It is not always easy to find a small leak. There are several methods: helium leak test, air soap test, ammonia leak test, etc, but which method works best for small leaks?



Mr Mark Brouwer of UreaKnowHow.com in the Netherlands starts the Round Table discussion:

A leak in a liner is identified by a leak detection system. According to your experiences what are the best ways to find the precise location of the leak?

Mr Chris Boyda of Agrium Inc. in Canada shares his experiences:

We recently experienced a liner leak in our HP stripper. Initially, we tried to use a pressurised ammonia test to locate the leak. This test required putting a small amount of pure ammonia in between the liner and the vessel shell and controlling the pressure to ~5 psig. On the vessel interior we tried to locate the leak by using ammonia sensitive paper (blue print paper), and ammonia sensitive spray.

Due to the very small size of the leak, this test failed numerous times. At this point we tried using helium. The helium test was set up where a vacuum pump was used to apply a vacuum to the leak detection ports, and small amounts of helium were swept over all internal welds until we detected helium on the gas chromatograph on the vacuum side. This helium method was very successful in helping to locate the leak location and did not risk over-pressuring the cavity between the liner and the shell.

Mr Srivatstava Prabhat of Tata Chemicals Limited in India shares his experiences: Leak detection in HP equipment liners especially in a stripper is a little tricky compared to a reactor because some of the weep holes connected to the liner are independent. In the case of the reactor all weep holes are interconnected. For identification of a leak in the liner we have used a method in which we pressurised the shell side with low pressure steam maintaining a pressure of 0.5 kg/cm² (selecting a weep hole close to the leak point.) Then, by touching the inner surface we were able to detect heat in the area of the leak.

By pressurising the shell side with air, maintaining a pressure of 0.5 kg/cm² with the same weep hole, and applying a rich soap solution in the selected area from the inside, with the help of an air bubble leak test method the leak source point can be identified.

Another method is the helium leak test method. This is a good method but it needs time. Pressurising with ammonia in my opinion is not a suitable method for identification of a leak because of the safety risks for the person entering the vessel. In addition, welding is difficult in the presence of ammonia so

removal of ammonia is also important. If the leak is near to a single point liner ammonia removal can be difficult and time consuming.

If someone has a more suitable method, please share as this is very useful and almost all fertilizer plants face this problem.

Mark asks for some clarifications: What do you mean by a single point liner, do you mean that the liner compartment contains only one leak detection hole? Normally each liner compartment has two leak detection holes so that flushing is possible with a gas from one hole to the other. This is normally valid for all kind of HP equipment.

Please elaborate on your statement that a stripper is a more tricky job than a reactor. Please confirm that with the shell side you mean the area between the liner and the carbon steel pressure wall.

Mr Girish Prakash of Tata Chemicals Limited in India provides some answers: Probably, what Prabhat means is that for a Saipem reactor and stripper the weep hole arrangement is slightly different. Also there are a few weep holes, which are not connected to others.

The helium leak test has proved to be a good alternative in our experience.

Mark provides further information: Stamicarbon's advises (see E-newsletter dated 24 January 2011) that there are two recommended methods for finding a leak: the bubble test and the ammonia leakage test.

In cases where the leak is large enough, a bubble test is an appropriate method. The detectable leak rate using the bubble method is in the range of 10-3 std.cc/s (standard cubic centimeters per second).

For smaller leaks an ammonia leak test is advised. Ammonia is introduced behind the liner with a maximum pressure of 0.03 barg. After a period of 8 to 10 hours leaks with a leak rate as small as 0.02 to 0.03 cc/hr becomes detectable. The detectable leak rate is about 1000 times better compared to the air bubble test; 10-6 std.cc/s.

Srivatstava replies to the earlier questions: Principally weep holes of all HP equipment items must be interconnected at at least one point. But in our stripper I have checked many times and found that some of the weep holes in the stripper are independent although they are in the minority.

The reason I say that strippers are more tricky it is more difficult in the bottom portion specially if the leak occurs from the joint of exchanger tube sheet and bottom dome of stripper shell.

Yes, the shell side means the area between the liner and the carbon steel pressure wall.

Mr Amrit Ramnanan of IPSL in Trinidad and Tobago shares his experiences: In the TEC ACES HP synthesis loop equipment the following techniques are employed to determine the location of a liner leak after the leak detection system has flagged a problem:

- review equipment drawing for liner welds associated with detection hole;
- Use dye penetrant inspection of liner weld(s).

What else can be done if the leak is still not found?

Mr Son Nguyen of PVFCCO in Viet Nam replies: You can apply steam to the weep holes and then measure the conductivity as condensate comes out at the outlet to determine fairly accurately where the leak is.

Mark replies: The disadvantage of putting steam or condensate behind the liner is that one can introduce stress corrosion cracking risks to the carbon steel. Several times severe cracks have been found in the carbon steel pressure wall on the inside (liner side) and in all these cases the end user followed the practice of putting steam or condensate behind the liner.

Son responds: Am I right in saying that stress corrosion will only take place if you cannot maintain a continuous flow of steam or condensate?

Mark replies: SCC is caused by a combination of chemical substances and metals at certain conditions.

For example, nitrates with carbon steel or chlorides with stainless steel at certain temperatures and stresses.

Also carbonates with carbon steel can cause SCC. I am not aware that assuring flushing condition will avoid that. Why you think so?

Son reacts again: You can see many steam drums and pipelines are made by carbon steel as well.

Mark replies: Yes, but in HP urea equipment carbamate and carbonates can enter when there is a leak.

You can find more information about stress corrosion cracking in a publication by National Physical Laboratory, which you can download from UreaKnowHow.com's E-Library.

The point I want to make is that in several cases cracks have been found on the liner side of the pressure bearing carbon steel wall and a connection with flushing with steam or condensate cannot be ruled out.

Mr Ashish Kumar Panda of OMIFCO in Oman contributes to the discussion: I have experience of leak detection in a urea reactor with ammonia vapour. The ammonia vapour after pressure reduction (as recommended by equipment manufacturer) is charged through one of the weep holes of the suspected leakage area. Other weep holes in the particular segment are kept plugged. Wet phenolphthalein paper is pasted inside the reactor. Leakage is detected by development of pink colour on the leakage point. This procedure is very effective in detecting very small leakages. Please ensure proper air purging inside the vessel to avoid accumulation of ammonia vapour. Breathing apparatus inside the vessel is a must. ■

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Problem No. 27 How do you clean the scraper in a prilling tower?

A scraper rotates above the floor of the prilling tower and moves the prills from the bottom of the prilling tower to the conveyor belt. At higher plant loads and/or under summer conditions the prills are not completely solidified and are still soft when reaching the bottom of the prilling tower. These soft prills clog up the scraper forming a hard

layer of solid urea. This kind of fouling can also build up on the walls and ceiling of the prilling tower and at regular intervals cleaning becomes necessary. But how can this be done safely? This Round Table discussion starts with a description of a related safety incident as reported in UreaKnowHow.com's urea incident database.



Mr Mark Brouwer of UreaKnowHow.com in the Netherlands starts the Round Table discussion:

The incident: During 2010 overhaul activity employees were in a prilling tower for cleaning to remove lumps on the scraper's arms. A steel structure with a wooden platform on top was used for safety of the employees. A big lump fell down onto the steel structure breaking the wooden platform. The shoulder of one employee was crushed without major injuries.

Mr Muhammad Umar of FFC in Pakistan shares his experiences:

Yes, it's true one should never enter the tower even with protection without washing prilling tower top beams.

Mr Jawad Aslam of FFC in Pakistan adds: Falling lumps during cone cleaning is quite a frequent occurrence and it is not always possible to do beam washing together with the scraper cone cleaning. Has anyone used a protection system to protect employees from falling lumps?

Mr SK Gupta of IFFCO in India joins the discussion: Thanks for initiating an important topic having serious safety concerns. Urea deposition on the scraper arm, hard cake formation on the scraper floor, urea lump disposition on the bucket floor bottom ceiling and

on top louvers are a common phenomenon in almost all prilling towers. Periodically we have to remove these deposits for better prill quality. One can only minimise the depositions and reduce the frequency of cleaning, but one cannot get rid off it permanently. In our case deposits are cleaned manually (without using water) by taking back end shut down. Due to the limitation of the urea solution tank capacity we have to finish the cleaning operation within max 4 to 5 hours. Also due to time constraints, only the top louvers or the scraper floor is cleaned at any one time. While cleaning the scraper arm/floor our staff use crash helmets for safety to protect them from falling lumps from the louvers/bucket room bottom ceilings. We know that a crash helmet does not fully protect a person from large falling lumps from a height of 70 to 80 meters. We have faced several near misses during this activity. In industry there are examples of unreported fatal injuries during this operation. We tried using a movable steel canopy but it did not prove to be at all practical. During back end shut down for cleaning operations inside the tower temperature changes which initiates the dislodging of urea lumps from louvers/ceilings etc.

Mark adds pictures of a successful solution of this safety problem developed by an anonymous urea producer: Here are some pictures of a solution to minimise the risks associated with the cleaning of the scraper (June 2014).



Muhammad replies: This topic really is a serious safety concern and we are also facing problems of lump formation on top beams and one side of the tower wall. As mentioned by others, it is not possible to carry out wall and beam washing every time you plan for cone cleaning so we take the following precautions:

- Be aware of which side of the tower lumps are present and avoid working direct under that side.
- We have made a movable steel structure under which the worker carries out cleaning. We place the structure inside the tower cone and then the worker can move it as required.
- Any worker going inside the tower should have a full body harness along with rope and a standby man to rescue them in the case of any mishap.

Mr Prabhat Srivastava of TATA Chemicals Limited in India contributes to the discussion with his experiences: Certainly scraper floor cleaning is one of the toughest activities in the urea plant, which is a big safety concern. I agree with Mr Gupta that due to the limitation of the urea solution tank this activity needs to be completed in 4-5 hours max.

We have a conical scraper floor and we have also faced a lot of challenges. We are now using water to clean the scraper. Since we have a conical scraper floor cleaning is easier and by the end of cleaning not a single drop of water is left on floor, but the effectiveness for a flat scraper needs to be checked.

One safety measure I can suggest is that when we allow a man inside to clean the scraper arms we use a "rugby dress" which has

a helmet, shoulder guard, back guard, leg guard and hand gloves. This is much easier than having a movable cage inside.

Mr Sadanand IM of Mangalore Chemicals & Fertilizers in India introduces another solution: After installation of PP sheets on the scraper arms our build up is negligible.

Mr Pradeep Pednekar of RCF Ltd Thal in India joins the discussion: We are operating a Saipem urea process with 2,300 t/d load after capacity enhancement from 1,725 t/d design plant load. We wash the scraper floor once every three months. The temperature of the prills is 60°C. The prilling tower height is 90 m with free falling height of 65 m and a bucket rpm of 380-400 rpm. Ambient air temperature is max. 40°C. There is a weekly routine schedule for changeover of bucket and crown washing (fortnight) in spite of good prill quality to avoid overflow of bucket and to obtain quality product. The time required for scraper floor cleaning is 5 hours from prilling to prilling.

Mr Prem Baboo of National Fertilizer Limited in India also shares his experiences: Urea deposition on the scraper arms due to an overflowing prilling bucket can be avoided by increasing the rpm of the prilling bucket. If the overflow is not stopped by increasing the rpm then the bucket must be changed. During scraper cleaning waste oil is applied to the scraper arms after removal of the urea deposition to make subsequent cleaning/descaling of the arms easier. The temperature of the urea melt should be 136-137°C. ■

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Problem No. 28 How to avoid hot prills in the prill tower bottom

Hot prills can easily cause caking because heat transfer and moisture transfer take place due to the temperature difference between the hot prills and the other prills. Caking is the process of building crystal bridges between the urea particles. Caking leads to lump formation and consequently dust formation when these lumps are crushed. This all leads to poor

product quality and customer complaints, but how can hot prills be avoided?

A Round Table discussion about an incident during the cleaning of a scraper (see Plant Manager+ Problem No. 27) triggered a subsequent discussion on how to avoid hot prills; hot prills obviously cause more fouling of a scraper.



Mr Mark Brouwer of UreaKnowHow.com in the Netherlands starts the Round Table discussion:

The incident: During 2010 overhaul activity employees were in the prilling tower for cleaning the lumps on the scraper's arms. A steel structure with a wooden platform on top was used for safety of the employees. A big lump fell down on

steel structure breaking the wooden platform. The shoulder of one employee was crashed without major injuries.

Mr Kirittkumar Patel of IFFCO in India shares his experiences:

We are operating a 1,650 t/d urea plant of Stamicarbon design. The prill tower floor is equipped with a rotating scraper unit to collect and drop urea prills onto the bottom conveyor through a slit in the floor aligned to the conveyor. We observe that the urea prills get deposited on the top of the rotating arms of the scraper resulting in the need to stop the prilling unit. During summer the temperature of the urea prill is higher (approximately 105 to 110°C) than during winter (95°C depending on ambient conditions). We have to break the lumps deposited on the scraper arms at least once a week or three times in a fortnight. The frequency increases during the monsoon. Is there any solution to avoid this urea deposition on the rotating arms? How can we prevent this problem?

Mr Abdul Raouf of FFC in Pakistan contributes to the discussion:

In my opinion your prill temperature at the prill tower bottom (I assume you mean this temperature) is very high and needs to be reduced to at least around 75°C maximum in the hot humid season. We are operating a 1,925 t/d Saipem plant (currently operating around 2,300 t/d) and experience the same problem. In the hot humid season, we use a prill bucket with a smaller average prill size and the cleaning frequency of the scraper arms is around once every two months. The prill temperature at the prill tower bottom is about 70-75°C while maintaining maximum possible draft through the prill tower (limit is acceptable moisture content in product). The ambient temperature here is around 45-48°C maximum and humidity about 80-85% maximum.

Mr Majid Mohammadian of OCI, The Netherlands also shares his experiences: I have the following figures from one of our plants which does not have any problems with the scraper arms:

- Prill bucket speed: 235 rpm
- Product humidity: 0.27% as average
- Maximum ambient temperature: 50°C

- Prill average size: 94% between 1-2.4 mm
- Plant capacity: 1,500 t/d.

Please let me have the above data in your plants.

Kirittkumar replies: In our plant, we have the following parameters:

- Prill bucket speed: 188 rpm
- Product humidity: 0.4% average
- Max ambient temperature: 46°C
- Prill average size: 1 to 2.4 mm: 95 %; Less than 1 mm 2 % average
- Plant capacity: 1,650 t/d

Note, we have a prill cooling system (fluidised bed cooler) downstream of the prill tower conveyor which reduces the product temperature from 110°C to 60°C.

Mr Salman Islam Shahzad of Dawood Hercules Chemicals, Ltd replies:

In Dawood Hercules Chemicals Ltd, we are also facing this problem but its intensity is not as severe as indicated by you. The temperature of the prills can be reduced by producing proper draft in the prill tower (we have a natural draft prill tower). Reducing the prill size by increasing the bucket speed will also reduce the prill temperature because a high prill temperature will force the prill to absorb moisture even at low humidity. Try to reduce it below 70°C. Repeated bucket overflow due to blockage will also cause urea build-up on the scraper arms.

Majid provides more information: I fully agree with Salman and think that it is better to reduce the prill tower bottom temperature as much as possible (less than 70°C).

In our plant, the prill tower bottom temperature is normally around 65 to 70°C. We then reduce the temperature to 40-50°C for storage using a prill cooler. Maybe you can reduce the temperature by increasing the speed of the bucket?

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran shares his experience:

Urea build-up on the scraper arms is a normal phenomenon but not every week or every month, it occurs approximately once every six months and its cause is water and urea dust.

To reduce the temperature of urea at the bottom of prill tower, I suggest the following:

- Increase the bucket speed.
- Increase the air flow to the prill tower if there is induced fan and clean the fan blades every shift and change the blade angle in summer time.
- Decrease the urea melt temperature from the concentration unit as much as possible, target near 137°C.

- Check the urea melt jacket pipe steam pressure for controlling the urea melt temperature to the bucket.
- Clean the bucket holes.

To reduce the moisture content of the prills, I suggest:

- Increase the urea concentration vacuum system
- Put in service FES System to remove air humidity for the fluid bed cooler.

Mr Meer Salman Muddasar of ENGRO fertilizer in Pakistan replies: To control the prill temperature to avoid frequent cake build-up on scrapper arm, set a frequency for cleaning the top louvers of the prill tower. The draft should be well maintained. One can also install an online draft calculator at the prill tower bottom for natural draft prill towers.

Mr Ali Zama Khan of Kribhco Shyam Fertilizer Limited in India makes a suggestion: If the urea prill temperature increases, first the top louvers should be checked because the air path can be restricted due to deposition of dust on the top louvers.

Mr Pradeep Pednekar of RCF Ltd Thal in India joins the discussion: We are operating a Saipem process urea plant with a 2,300 t/d

load after a capacity enhancement of 1,725 t/d design load plant. Our frequency for washing the scraper floor is once every three months. The prill temperature is 60°C. The prill tower height is 90 m with a free-fall height of 65 m and a bucket speed 380-400 rpm. The ambient air temperature is max. 40°C. There is a weekly routine schedule for changeover of bucket and crown washing (fortnight) in spite of good prill quality to avoid overflow of bucket and to have quality product. The time required for scraper floor cleaning is five hours from prilling to prilling.

Mr Prem Baboo of NFL in India adds his valuable experience: Urea deposition on the scraper arms due to an overflowing prill bucket can be avoided by increasing the prill bucket speed. The temperature of the urea melt should be 136-137°C.

The bucket capacity is also an important factor: for 3,600 t/d TX 434 series is required having about 10500 holes of 1.1 mm diameter. If the temperature of the prills is higher than 55°C then the summer bucket is used. In our plant we are producing 3,500 t/d and with the bucket using TX 434 no problem is observed. If the bucket is choked scraper arm deposition is sometimes observed within 5-6 days due to the bucket overflowing. Our normal scraper cleaning frequency is 20-22 days.

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.

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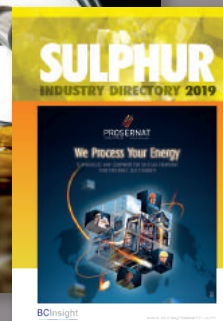
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Problem No. 29 Stripper versus decomposer: What is the difference?



In 1967, Mr Piet Kaasenbrood (picture left) of Stamicarbon invented the CO₂ stripper. The CO₂ stripper revolutionised the urea process industry as it reduced the synthesis pressure from about 200 bar to 140 bar and reduced the energy consumption of a urea plant by about 50%. The CO₂ stripper decomposes the unconverted ammonium carbamate coming from the reactor at the same pressure as that in the reactor. Therefore no water needs to be added to recycle this carbamate to the reactor, which is beneficial for the conversion in the reactor.

The phase diagram of the system NH₃-CO₂-Urea-H₂O (see picture) shows that by stripping with CO₂ one is able to

significantly reduce the ammonia content in the reactor outlet while at the same time reducing the temperature. Minimum temperatures are vital to keep corrosion rates under control and to allow economical materials of construction to be used. A decomposer on the other hand only removes the excess ammonia from the reactor solution until the azeotropic composition is reached. During this process the temperature increases. It is for this reason that in a CO₂ stripper with a bottom temperature of 170-175°C 25-22-2 stainless steel can be used, while in a Saipem stripper, with a bottom temperature of 204-212°C, titanium or zirconium needs to be applied to handle the high temperature.

Mr Kessla Belkacem of AOA in Algeria initiates the Round Table discussion:

I have a question: I would like to know what the difference is between decomposing and stripping?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands replies:

These terms are frequently mixed up. Theoretically, stripping is decomposing with the help of a stripping gas. So a CO₂ stripper works with CO₂ as a stripping agent and is a stripper. An NH₃ stripper is in my view more a decomposer than a stripper although one could argue that the decomposed carbamate acts as a kind of stripping agent.

Mr Malik Sohail of SAFCO in Saudi Arabia gives his view: Actually the purpose of both the stripper and decomposer is the same, the decomposition of ammonium carbamate. If this decomposition takes place mainly by partial pressure motive then it is called stripping and if this motive is heat then it is called decomposing.

Mr Nawal Gupta of KRIBHCO in India contributes to the discussion:

If ammonium carbamate is dissociated into NH₃ and CO₂ gases by reducing the pressure of the solution, it is called decomposition. If the same is achieved by reducing the partial pressure of one of the components (that of NH₃ in the case of CO₂ stripping or that of CO₂ in the case of NH₃ stripping), it is called stripping.

Mr Kashif Naseem of SABIC in Saudi Arabia provides his view:

Decomposition: Carbamate is formed in the reactor and passes to the stripper. With heat of steam this reaction is reverted back to ammonia and CO₂ with breakage of its bond.

Stripping: The formed ammonia and free ammonia are forced to come out using CO₂ as the stripping agent.

Mr Thota Raju of Nagarjuna Fertilizers and Chemicals Ltd. in India adds to the discussion:

In my view we normally heat the carbamate solution to reverse the reaction towards the reactant side (i.e. NH₃ and CO₂). The NH₃ and CO₂ will be in the liquid phase and in order for them to go to the gas phase their saturation pressure at those conditions should be greater than partial pressure of the respective in gas phase. In a stripper the gas phase pressure is high so suppose that

in a Saipem process the excess ammonia and formed ammonia is present in a higher quantity (i.e. high mole fraction) than as per the vapour liquid equilibrium, it will start vaporising. It first increases its partial pressure in the gas phase and since the system pressure is constant it decreases the CO₂ partial pressure then stripping starts.

Mr Azad Panchal of GNFC in India asks a similar question: In the Saipem urea plant the stripper has the same function as the MP decomposer, why is one called a stripper and the other a decomposer?

Mr Nimesh Maurya of KRIBHCO in India replies: In the HP stripper the decomposition is carried out by stripping action using Henry's law in the presence of excess ammonia while in the medium pressure decomposer decomposition is carried out by elevated temperature at reduced pressure only. There is not any stripping action.

Azad comments: But the stripper is also operating at high temperature by using medium pressure steam and at a reduced pressure of 145 kg/cm², while the reactor operates at 160 kg/cm².

Nimesh replies: In the HP stripper part of the ammonium carbamate is decomposed due to elevated temperature, but the decomposition is mainly achieved by stripping action so it is called a stripper.

Mr Shashank Sharma of Tata Chemicals Ltd. contributes: In the stripper, 80% of the ammonium carbamate is decomposed into NH₃ and CO₂ by the stripping action of NH₃ vapours occurring due to Henry's law as the NH₃ is present in excess. Since the main action here is decomposition by stripping it is called stripper. In the MP decomposer the decomposition takes place due to flashing and the increase in temperature of the ammonium carbamate solution in the medium pressure section, as a result of which it is decomposed into NH₃ and CO₂.

Mr Prem Baboo of NFL in India gives a detailed reply: Definition of a stripper: To reduce the partial pressure of the product by swamping the system with one of the reactants, which considerably reduces the partial pressure of the other reactant without changing the total pressure. Either CO₂, NH₃ or both can be used as a stripping agent.

Definition of a decomposer: The process based on the first principal of decrease in pressure and increase in temperature followed by a series of decomposition stages where the reactor discharge is treated successively at lower pressure.

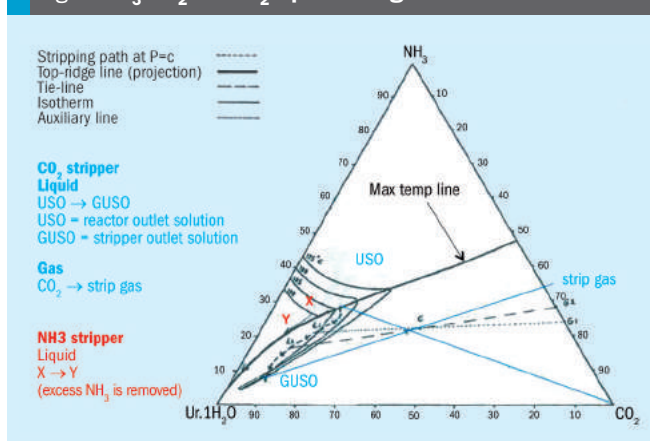
The delta P for a decomposer is greater than for a stripper. There is no delta P between the reactor and the stripper, however in a Saipem process there is small delta P.

With the decomposer the solution flashes due to the large difference of pressure and with the stripper the base is Henry's law of partial pressure and mass transfer is also involved.

Decomposition is based only on high temperature and low pressure, decomposition occurs due to let down, the water recycle is greater. Stripping is based on the partial pressure. In the Stamicarbon process CO_2 is introduced in the stripper, which is advantageous because CO_2 increases P1 CO_2 to P2 CO_2 , so P1 NH_3 will be reduced to P2 NH_3 to maintain constant total pressure as $\text{PCO}_2 + \text{PNH}_3 = \text{Total Pressure}$. Now $K_1 = (X_{\text{NH}_3}^2 \times X_{\text{CO}_2}) / X_{\text{Carb}}$ at particular temp K1 is constant so when X_{NH_3} is reduced to keep K1 constant X Carbamate will be reduced much faster by decomposition as X_{NH_3} appears in the equation with power of 2.

Decomposition is favoured by low pressure but energy is required to recycle the decomposition products back to the reactor. Also, at low pressure more water is evaporated during decomposition and this water will enter the urea reactor along with the recycle stream and adversely affect the conversion. Thus if the decomposition is carried out in a single stage near atmospheric pressure the carbamate formed during recovery at the same pressure will carry a lot of water. Considering these factors, decompo-

Fig 1: NH_3 - CO_2 -urea- H_2O phase diagram



sition is carried out in a number of stages.

In a stripping process the first stage decomposition and recovery is done at the reactor pressure which permits heat to be recovered at high pressure and also results in energy saving the for returning the recycle streams to the reactor. However, in the Saipem process there is a difference in pressure between the reactor and stripper, so there is the additional advantage of differential decomposition.

Mark adds to Prem's explanation: The phase diagram above helps to explain the difference between the CO_2 stripper and NH_3 stripper (or HP decomposer).

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Problem No. 30 What is the best stainless steel for urea?

Ammonium carbamate is an intermediate product in the production of urea from carbon dioxide and ammonia. Ammonium carbamate is very corrosive under the synthesis conditions in a urea plant. The various process licensors have developed different solutions to assure sufficient safety and reliability levels. In the early days titanium and 316L Urea Grade stainless steels were applied. The process conditions in the high pressure stripper are

the most severe, i.e. the highest temperatures and lowest oxygen partial pressures are present. A new stainless steel 25-22-2 with a higher chromium content was developed specifically for the high pressure CO₂ stripper. Super duplex materials like Safurex[®] and DP28W[™] and Omega-Bond[™] (a combination of zirconium with titanium) are the latest developments and have proven to be better than earlier materials, but what is the best material?

Mr Janusz Maćkowski of Zch Police in Poland starts the Round

Table discussion: What is the best material for the stripper (tubes and liner) and the other synthesis equipment in a urea plant: Safurex[®], Super duplex, DP28W[™] (Sumitomo), bimetallic with Zr/25-22-2, any others? What is the actual level of oxygen they need? What is the best material for the urea reactor liner?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands

responds: Maybe titanium should also be on your list. In addition, Saipem has developed a new type of tube material for the stripper (Omega-Bond[™]). Looking at the “best” materials I believe several different aspects are important:

- intrinsic resistance against corrosion (for example zirconium is better than 25-22-2)
- need of oxygen for passivation
- weldability (here zirconium is much worse)
- sensitivity to problems like erosion, H₂ embrittlement, chloride SCC, crevice corrosion (bimetallic) etc.
- price
- references.

Considering all these, Safurex[®] is in my opinion the best choice for urea synthesis conditions. It also has the potential to work under zero oxygen conditions.

Janusz replies: Thank you for your reply. Omega-Bond[™] is bimetallic (Ti/Zr). I know that Safurex[®] is very good. Super duplex with tungsten is also very interesting (DP28W[™]). I think this material could have similar features to Safurex[®] (or even better) but it is quite a new material (very few references in comparison to Safurex[®]). Does anyone have experience with this material?

Mr Waqqar Ahmed of Fauji Fertilizer Company Ltd. in Pakistan joins

the discussion: Saipem's technology uses 316L Urea Grade (UG) or Modified (Mod) and 25-22-2 and 0.35 vol-% of oxygen in CO₂ feed is sufficient for passivation. We have experience of both titanium and bimetallic (25-22-2/Zr). Titanium has excellent corrosion resistance but it has erosion problems. With additional passivation air at the bimetallic urea stripper bottom, we found no problem even when operating at 205-206°C. 3 ppm of oxygen at the stripper bottom is enough for passivation of 25-22-2 tube sheet and tube ends. Safurex[®] has excellent corrosion resistance, but can it sustain its corrosion resistant properties at temperatures up to 205-206°C without passivation air? With Omega-Bond[™] strippers Saipem claims the stripper bottom can even be operated at a temperature of 210°C.

Mr Fabiano Parizotto of Petrobras in Brasil shares his experi-

ences: Take care with Safurex[®], because one can find corrosion in machined parts which have suffered during the fabrication process. This corrosion phenomenon is called “stern face corrosion”. This corrosion occurs due to the break of grains, which reduces the corrosion resistance. Check parts such as the hole in tubes, dividers or clips to attach trays, etc.

Mark replies: It was indeed true that the first generation Safurex[®] was more sensitive to cross cut end (or stern face corrosion). In the later generations Safurex[®] this problem has been solved. Further, a Safurex[®] stripper was successfully in operation at the Saipem urea plant of Profertil in Argentina and is now in operation at Agrium Redwater in Canada, so yes Safurex[®] can also handle 205-206°C. I do not know whether these plants had extra passivation air injection on the stripper.

Mr Jan Cervenka of Chemoprojekt Nitrogen in Czech Republic asks

some questions: Is it possible to protect the machined parts Fabiano refers to, e.g. by some kind of grinding (mechanical way) or pickling (chemical way)? What can be done at the workshop (after machining/welding) and what can be done with already corroded surfaces?

Mr Eddie Canadilla of SAFCO in Saudi Arabia adds:

How does Safurex[®] handle stern face attack (or cross cut end attack) where other urea grade stainless steels are not so effective?

Mark replies: Grinding or pickling can be used to remove oxides, which occur after machining or welding. Removal of these oxides is important in order that a proper passivation layer can built up as explained in one of Giel Notten's Technical Papers (for example UreaKnowHow.com Mechanical Paper February 2009). This is applicable for austenitic stainless steels like 316L Urea Grade (UG) or 25-22-2 usually applied in urea plants. Also after severe active corrosion of these materials it is advisable to apply pickling to remove these excessive corrosion products and enable the build up of a proper passivation layer. Safurex[®] is a duplex stainless steel, a mixture of austenite and ferrite material and more specifically the Safurex[®] structure is such that there are austenite islands in a ferrite matrix. Passivation is therefore not necessary any more (Safurex[®] urea plants may not need oxygen for passivation). In first generation Safurex[®] this structure had not been optimised at cross cut ends. In later generations it was improved and better fabrication procedures were developed to minimise the problem of cross cut end corrosion.

Mr Muhammad Kashif Naseem of SABIC in Saudi Arabia shares his experiences: Toyo Engineering Corporation (TEC) uses DP28W™ material for the stripper tubes, the tubesheet carbamate side and the liner in the top and bottom chamber of this stripper in ACES21 technology. There is also very good experience with this material and it shows better corrosion resistance.

Janusz requests more information: I would like to know the corrosion rate for different urea stainless steels, especially for the following: 316L UG, 25-22-2 and Safurex®.

Mark replies: All corrosion rates strongly depend on temperatures. In addition, we should make a distinction between passive corrosion rates and active corrosion rates.

As a rough guide passive corrosion rates of the tubes in a high pressure CO₂ stripper are:

BC.05 (25-22-2): 0.06-0.07 mm/yr

BE.06 (Safurex®): 0.05-0.06 mm/yr

Further, the sensitivity for active corrosion for the various materials is as follows: 316L UG > 25-22-2 > Safurex®. And then we can distinguish for chloride SCC: Safurex® not sensitive and 316L UG and 25-22-2 sensitive.

Janusz asks: Does anyone know the corrosion rate of 316L material for a HP CO₂ stripper and reactor? Can AISI 904L (1.4539) material be used in urea synthesis (especially for renovating valves)?

Mark replies: 316L material or 316L UG can be used efficiently in urea reactors. Corrosion rates vary between 0.2 and 0.05 mm per year on stream (pyos). Whether 316L can be used depends on the amount of alloying elements. Cr and Mo play a very important role and of course the carbon content, the higher the Cr content in combination with a low carbon content gives the best results and that is obvious when you review the chemical composition of the urea grade 316L types. A regular 316L may end up with corrosion rates of over 0.5 mm pyos and in the worst case up to 2 mm per year. Also welding 316L will have a negative effect on selective corrosion as there is weld decay and sigma phase. As regards 316L in strippers, the wall temperature in a stripper is far too high for even a Urea Grade 316L type, the first HP CO₂ strippers had 316L UG tubes and the tubes lasted only a few months. This material is completely inadequate for stripper tubes. Lliner overlay welds and internals can be made in Urea Grade 316L but with the addition of sufficient oxygen. Cross cut end attack is greater for all austenite and austenitic-ferritic (duplex) materials higher than surface corrosion. For duplex this attack is greater due to the micro structure of these materials. When discussing Safurex®, the surface attack looks higher because the surface attack is negligible here. The cross cut end attack is not higher than for other duplex materials.

AISI 904L is not a good material for urea service, it is relative low in Cr and very high in Ni. It can be compared with Sandvik type 2RK65 and is useful in a seawater environment

Mr Pradeep Pednekar of RCF Ltd Thal in India shares his experiences: We have Saipem technology in our urea plants 3 x 1,725 t/d design capacity. In Saipem plants 316L UG and 25-22-2 (2RE69) are used and 0.35 vol-% oxygen in the CO₂ feed is sufficient for passivation. Previously we were using titanium strippers. Titanium has excellent corrosion resistance but it has problem with

erosion. With bimetallic strippers passivation at the bottom of the stripper is a must. With additional passivation air at the bimetallic urea stripper bottom, we experienced no problems even when operating at 205-206°C. 3 ppm of oxygen at the stripper bottom is sufficient for passivation of 25-22-2 tube sheets and tube ends. For high pressure carbamate condenser tubes 25-22-2 is the best.

Janusz asks some more questions:

- What is the safe minimum thickness of the reactor liner with 316L UG stainless steel?
- What level of nickel (Ni) in urea solution from the urea reactor (316L UG) is normal and what level of nickel is emergency?
- What is an average lifetime of urea reactor liner (316L UG)?

Mr Prem Baboo of National Fertilizers Ltd. in India shares his experiences: In our Saipem urea plant, commissioned in April 1987, the reactor liner thickness is still 6.8-7.1 mm. If one regularly checks the weep holes, performs maintenance during the shutdown (dye penetrant test and pitting maintenance), etc. then the life of urea reactor can be increased. Passivation air flow i.e oxygen in the 3rd stage compressor should not be less than 0.35%. The top temperature of the reactor should not exceed more than 190°C. The level of nickel (Ni) in urea solution from the urea reactor (316L UG) is 40-45 ppb. Below is an overview of the various materials applied in carbamate service in urea plants.

Titanium: Titanium is used in the relatively pure state. It has excellent corrosion resistance but is one of the more costly materials and more difficult to weld. The corrosion resistance of titanium is due to the impervious oxide film i.e. 100% TiO₂ film on the surface. Titanium is resistant to stress-corrosion cracking and erosion corrosion, but is susceptible to crevice corrosion in stagnant chloride solutions. Titanium was used for the lining and tube material of the high pressure NH₃ stripper in Saipem urea plants. Commercially pure titanium exhibits a high resistance to pitting attack. Titanium has also been used for the lining of urea reactors in TEC urea plants. The advantages are that less passivation air is required compared to other stainless steels and the HP NH₃ stripper bottom temperature in the urea plant can be kept at temperatures of up to 210°C while with a bimetallic stripper the temperature cannot be raised beyond 207°C. The disadvantages are that titanium is not maintenance friendly due to the difficulty in welding and erosion of tubes ends resulting in bypassing in the stripper due to tube end over ferrules badly eroded by carbamate solution.

Zirconium: The corrosion resistance is because of the ZrO₂ layer. Zirconium is more expensive than titanium (roughly twice that of titanium).

316L UG and 25-22-2: These are tailor made grades of austenitic stainless steel to suit required specifications of licensors. The rate of corrosion as per the Huey Test is as follows:

Material	Corrosion rate (mm/yr)
Zirconium	0.005
Titanium	0.06
25-22-2	0.3
316L UG	0.6

Duplex stainless steels: Duplex steel is characterised by a micro-structure containing both a ferritic phase with a BCC crystallographic structure and an austenitic phase with a FCC structure. The ferritic phase is normally 40-60%, mainly introduced in the wrought alloys by a careful balance of the critical alloying elements. It has a higher strength and better resistance to chloride stress corrosion cracking. ■

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Problem No. 31 Insulation of high-pressure flanges in a urea plant



The intermediate product in the production of urea from carbon dioxide and ammonia is ammonium carbamate. Ammonium carbamate is very corrosive under the synthesis conditions in a urea plant. It is good practice to minimise the number of flange connections in the high pressure urea synthesis section. However

some flange connections will be unavoidable. Typically two types of flange connections are applied in the high pressure urea synthesis section: ring type joints and lens ring joints. Once a flange connection leaks it is almost impossible to stop the leak by re-tightening the bolts. Due to the leak a crevice is created in which the oxygen dissolved in the carbamate containing liquid will be depleted and passive corrosion will become active corrosion. Active corrosion rates of stainless steel can be >50 mm per year and this means that in the case of a leaking flange connection one has to stop the plant immediately and service the leaking flange connection. This raises the question whether the flange connection should be insulated.



Mr Easa Norozipour of Khorasan Petrochemical Company in Iran starts up the round table discussion: What do people recommend as regards insulating the high pressure flanges in the high pressure section in a urea plant?

Mr Prem Baboo of National Fertilizers Limited in India responds:

Insulating flanges is a generic term of pipe flange joints with requirements in sealing performance of buried steel pipes and insulation properties of electro-corrosion pipes. It includes a pair of steel flanges; easy to manufacture, strong, high pressure flange-type insulating coupling with wide separation between exposed metal parts. The insulating coupling is made up of two metal half flanges supported on a non conductive cylindrical non-metallic member. The space between the two vertical ends of the half flanges is wound in typical fashion with plastic impregnated glass fibres to provide the necessary working strength.



Easa asks further: Thank you for your reply, but my query is about insulation of high pressure flanges in the high pressure section of a urea plant for heat resistance, not for electrical isolation. If the flanges are covered by insulation

leakages cannot be identified via a visual check. In this case the flange and bolts will be corroded (see picture).

Prem replies: In our plant the high pressure urea section flanges are non insulated so that a minor leakage can be easily detected

at an early stage. The first sign of a leakage is vapour escaping, which can be easily seen by the rotating shift operator. The flanges are tightened when there is a leakage so it is not possible to have insulation on the flanges. Regular nut bolt cleaning of these flanges is routine practice in our plant checklist. Vessel manhole cover flanges are also non insulated so that leaks can be detected.

Easa asks further: Many thanks Prem. Do any other experts have any recommendations for the insulation of high pressure flanges?

Mr Shoaib Minhas of FFBL in Pakistan shares his valuable experiences:

Apart from visual inspections, in my opinion no insulation at the flanges will lead to atmospheric corrosion, surface corrosion, moisture ingress and corrosion under insulation due to improper waterproofing of insulation. In our plant all flanges are covered with proper insulation and waterproofing.

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands joins the discussion:

Find below the philosophy of one expert in the industry: "Some of our plants have insulation over the flange joint and others have left flanges that are non insulated. For flanges in carbamate gas service, I like to insulate the flanges to reduce the risk of condensation corrosion (refer to picture). For flanges in liquid service, I like to keep flanges uninsulated to keep the stud bolts visible so that a small leak doesn't go unnoticed



and seriously corrode the bolts.” Others apply a leak detection tube in the case of insulated flanges.

Mr David Smith of Startupdynamics in Canada shares his experiences: I have also worked in urea plants, which do both. My preference is to “not” insulate high pressure flange connections in the urea synthesis section. The flanges have a very large metal mass, which retains the heat from the line. In my opinion, the amount of condensation corrosion caused by a cold spot from reduced temperature would be small. The bigger concern would be early detection of leakage from the ring joints. I think most of the bolts used are some type of ferrous material, which may corrode if a leak begins.

Normally a leak is not difficult to determine, based on the ammonia smell or build-up of crystals. Should a leak occur, the first action that happens is to remove the insulation anyway to determine the size of the leak. If it is unmanageable, the plant is stopped and drained, but if it is a small leak, it is tightened or if unsuccessful, flushed by steam to avoid any build-up of corrosion material until the gasket can be replaced. We have used permanent and removable insulation, we have put leak tubes inside the

insulation to detect an early leak, but personally I want to know about leaks as early as possible.

Mr Ali Salman Bokhari of Pak-American Fertilizers (Pvt.) Ltd. in Pakistan contributes to the discussion: As per TEC standards, all these flanges must be insulated. The reason behind this may be the following: Generally lens rings are used as a connector in high pressure piping but in some cases titanium gaskets are also used. During start-ups and shutdowns there is a possibility that titanium gaskets may undergo stresses due to uneven expansion of metals. This may weaken the gasket, which at some later stage causes leakages. Insulating the flanges will reduce the extent of uneven expansion of metal by reducing a significant delta temperature, which exists between metal and ambient temperature. The same standard exists in the case of manholes, which are insulated, generally at all plants.

Mr Pablo Gastón Schulz of Profertil in Argentina gives his opinion: We do not insulate flanges in the high pressure section so that bolts do not loosen with temperature, avoiding leaks and the need to tighten them in service.

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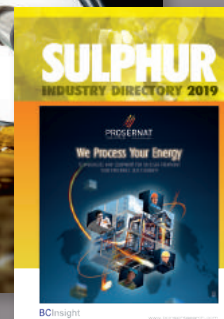
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Problem No. 32 Temperature drop at bottom outlet of HP stripper in a Saipem urea plant

The stripper in a urea plant is a key item of high pressure equipment affecting the overall performance of a urea plant. There is a significant difference between the Stamicarbon CO₂ HP stripper and the Saipem NH₃ HP stripper when judging its performance. The bottom outlet temperature of a Stamicarbon CO₂ stripper is normally relatively low (170-175°C) and when the performance of the HP stripper reduces (meaning lower stripping efficiency) this temperature will increase



indicating an increase in the ammonia content in the bottom outlet of the stripper. However, in a Saipem NH₃ stripper the situation is totally different. The stripper bottom outlet temperature increases with a higher stripper efficiency and an operator in a Saipem urea plant tries to operate the urea plant at the maximum bottom outlet temperature. The maximum value is determined by the acceptable corrosion rates, for example, 204°C for a bimetallic stripper.

Mr Ngateno Utomo of PKT, Indonesia starts this round table discussion with a practical problem: We operate a Saipem urea plant with a design capacity of 1,725 t/d. The plant is 13 years old. During start-up the temperature at the HP stripper outlet can reach 202°C at 100% plant load (flow of steam 38 t/h), but after a few days the temperature drops to 194°C (and flow of steam drops to 20 t/h). We have tried trimming all operating conditions but the temperature cannot be increased even when we reduce the plant load to only 50%. We tried to block in and restart the plant, but the condition reoccurred. Can anyone explain what the problem is with the stripper?

Mr Prem Baboo of National Fertilizers Ltd in India replies: The reduction in steam flow indicates restriction of the steam, for example due to one of the following reasons:

- Check the conductivity of the steam condensate for any leakage. If the conductivity has increased (200-1000 micro mho/cm) there is leakage in a tube or tube sheet and the shell might be pressurised with vapour causing restrictions of steam flow.
- Check the steam condensate drum level
- Check the stripper bottom level

I am fairly confident the first reason may be your problem. If a leakage is found, shut down immediately to avoid further damage/corrosion of the carbon steel shell side and the carbon steel tube sheet.

Ngateno responds: Thank you for your suggestions. We have checked and tried to increase and decrease the level in the stripper bottom and steam condensate drum level, but there is no effect on the temperature. The conductivity of the steam condensate is also normal.

Prem offers further advice: If the above points are fine, check the steam to the stripper control valve. Sometimes the internals are damaged and physically show normal opening but actual passage is blocked due to denting of the stem or foreign material blocking the control valve passage.

Ngateno replies: We have checked the steam control valve and then rechecked it by opening the bypass but it has no effect.

Mr Thai Son Phan of Phu My Fertilizer plant in Vietnam joins the discussion: Did you check the temperature into/out of the stripper steam condensate separator? I favour the temperature outlet. If it is not perfectly saturated (219°C at 22.5 bara) its heat exchange capacity decreases and the stripper bottom outlet temperature decreases gradually.

Mr Ali Salman Bokhari of Pak-American Fertilizers (Pvt.) Ltd. in Pakistan provides his support: In my view, you need to check the following:

- Do you have any vapour balance line installed between shell side of stripper and steam condensate (saturation) drum?
- Purge the shell side of stripper during normal operation and observe whether there is any positive change in stripper performance, more specifically steam consumption.
- Have you ever conducted a leak test of the stripper liquid distribution system and was it satisfactory?
- What type of ammonia feed pump is installed at your plant?
- Have you observed any coloration of product – yellowish or brownish?
- Have you observed some sort of oil layer or foam in the absorbers?
- What is the performance of other steam heaters at your plant?

Mr Umesh Kottara of Mangalore Chemicals and Fertilizers Ltd. in India contributes with his recommendations: Check that the HP stripper shell side vent is opened sufficiently to vent the inerts present in the steam. Check the quality of the steam introduced. We had a similar problem due to bubbling of the carbon dioxide feed (we have a CO₂ stripping process) into the urea solution at the stripper bottom due to a gasket leakage. We stopped the plant and found the gasket leaking as expected.

Fouling of stripper tubes may be another reason – adopt an appropriate chemical cleaning procedure for this, like EDTA.

Mr S.K. Gupta of IFFCO in India shares also his valuable experiences: I assume the stripper in your plant is bimetallic. What is the diameter of the tangential holes of the ferrule? In our experience a larger hole diameter solves the problem you indicated.

Ali returns: It is very interesting to know that you are increasing the hole size of the ferrule. What is the procedure?

SK replies: We have four urea strippers of the same heat duty in operation at our site. Initially we had two urea streams with titanium strippers. After an expansion of the site two more urea streams were added. The new urea streams have bimetallic strippers. We experienced the problem of temperature drop in the bimetallic strippers. We tried all the suggestions mentioned in this forum but to no avail. Saipem took notice and enlarged the ferrule hole diameter for their new bimetallic strippers. A few years back we replaced our aging two titanium strippers in the old urea streams with new bimetallic strippers. We have not experienced the problem in the new strippers. As per the advice of Saipem we are also planning to increase the ferrule hole diameter of the old strippers. Nowadays we are managing it by cutting off the feed for five minutes whenever there is problem in temperature drop.

Mr Pablo Gastón Schulz of Profertil in Argentina shares his experiences: We also operate a Saipem plant and have extensive experience with stripper heat transfer inefficiency. This year we had an abrupt trip due to a positioner failure in the stripper bottom level control valve. After this, the heat transfer efficiency in the stripper had a small step decrease, yet after each restart (we had two after this event) the problem got worse. Then, in just one week the stripper efficiency decreased in such a way that we were forced to shut down, since the plant could not be operated any more.

Analysing the DCS trends, we managed identify the most probable cause: liquid maldistribution in the tubes. One key symptom was the noise in the MP steam flow, which got worse with time. The fact was that during this emergency trip, the ferrules became loose, due to the pressure buildup within the tubes. We performed a leak test during the turnaround and got an order of magnitude more leakage than allowable.

Since the male inserts of the ferrule were deformed (slightly, not visible), they could not be fixed in position by the metal grid. So we installed Teflon washers in between the ferrule and the tube, to tighten the ferrule. This allowed us the smallest leakage ever registered in our plant, and since then the stripper efficiency boosted up.

Yet for the sake of being sure before you shut down the plant, I suggest you take a systematic approach:

Steam side:

- Check for inerts in the shell. Open the vent.
- Check actual level in steam reservoir of the stripper (V109). If the shell is partially flooded you loose heat transfer area.
- Check the PV that controls the steam pressure, also the transmitter (in case the controller is failing)

Process side:

If you had a cold start up, if too much carbamate was present at the time of overflow of the reactor, a high pressure is built up within the tubes and despite the composition eventually becoming OK, this disruption in liquid distribution cannot be reversed by any load reduction. The only way we have found to reverse this is by stopping all flow (we have a procedure for this). The key is that when having a hot restart the composition is OK from the very beginning so there is no disruption. You only have to be very careful to keep the NH_3/CO_2 ratio in the normal range.

Also make sure that the problem is not coming from another area. Check the delta T of the reactor, and sample the outlet to verify conversion and N/C ratio. Check the actual N/C ratio by having an instrument specialist verify CO_2 and NH_3 measurements. When we had this problem typically there is more water in the pro-

cess since the decomposition is shifted to the MP and LP sections.

Be careful not to allow the PVO20 (MP steam to stripper steam drum) to be saturated to 10%, along with the MP decomposer TIC. We made this mistake and overloaded the LP decomposer, and this led to EO8 (CW condenser) eventually plugging, leading to a shut down. This was because the proportion of gases from the LP decomposer and gases from waste water treatment column was distorted, making the solution less aqueous. During shutdown, check that the distributor toroid weld is not damaged, also check the serrated overflow. This could also lead to maldistribution of liquid.

We also had the gas orifice of the ferrules enlarged by Saipem along with moving the tangential holes so that the liquid level increases. You are definitely facing the same problem we had.

Ali replies: I agree with your comments. Actually uneven opening of the hand control valve controlling the reactor level (due to any reason - instrument malfunction or mis-operation) causes a hammering impact on the liquid distribution system resulting in displacement of ferrules. We have also faced this problem in our TEC plant during early years of operation.

You must be familiar with ferrule and swirl operation. Actually stripper efficiency is based on the film uniformity of the urea solution in terms of the thickness in the falling film heater and film thickness is dictated by the velocity of the urea solution through the tubes, which in turn is dictated by the liquid head and swirls hole size. That is why hydrolysis occurs in the stripper and fluctuates with fluctuations in plant load. In extreme conditions it may lead to dry run operation, which is a nightmare situation.

Mr Akash Deep Mawkin of National Fertilizers Limited in India shares his suggestions: Probable causes for not achieving the stripper outlet temperature:

- stripper level is actually running very high (touching the tube sheet and restricting the vapour passage through tubes);
- steam condensate separator level is very low (no saturated steam is going to stripper);
- restriction in steam inlet to stripper;
- reactor outlet pipe having leakage (inside the reactor);
- poor stripper liquid distribution (may be due to the ferrule tangential portion being damaged due to thinning);
- temperature and flow indicators are malfunctioning (this can be checked if there adverse effect on MP section).

Mr Zeeshan Shoaib of Fauji Fertilizer Company Limited in Pakistan provides his input: After reading all of the above discussions, in my opinion the condition of the ferrules needs to be checked. Improper film generation may cause poor thermal efficiency of the stripper. Sometimes the ferrules nozzle holes are blocked or ferrules were out of place causing gas bypassing, indicated by a high top stripper temperature. In addition, it is important to maintain the proper NC ratio in the system.

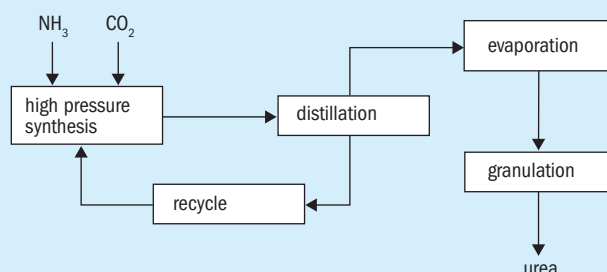
During start-up, don't let the stripper temperature exceed 198 to 200°C at 60 to 70% plant load. Raise the temperature of the stripper bottom gradually. If high temperature MS steam is given too quickly hot spots may appear on the inner wall of the stripper tube and cause channeling.

This problem may occur due to poor indication of the level transmitter that misleads the stripper bottom holder level controller. A high level may cause channeling. ■

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Problem No. 33 Excessive amount of carbamate solution from recirculation section

The recirculation or distillation section in a urea plant where unconverted carbamate is separated from the urea/water mixture is a critical section of a urea plant that influences the overall performance of the plant. Too much carbamate solution recycle back to the synthesis section will increase the amount of water in the synthesis and will negatively influence the reactor conversion figures. Fouling in the recirculation section can sometimes cause these operational problems, but how can fouling be identified as the root cause of a problem?



Mr Janusz Maćkowski of ZCh “Police” in Poland kicks starts the Round Table discussion with the following problem: We have quite an old Stamicarbon plant designed in 1976. The gas outlet pipe from the low pressure rectifying column is connected to the gas outlet pipe from the first desorber of the waste water treatment section and together these are connected to the low pressure carbamate condenser (LPCC). There is no reflux condenser. We have a problem with an excessive amount of carbamate solution in the LP carbamate condenser level tank, which leads to overflowing of the level tank. Is it possible (as a result of some kind of equipment damage) that:

- an additional amount of solution goes through the gas pipe from the rectifying column to the LP carbamate condenser?
- and/or an additional amount of solution goes through the gas pipe from the waste water treatment section to the LP carbamate condenser?

Has anyone experienced such a problem?

Mr Mark Brouwer of UreaKnowHow.com in the Netherlands shares his experience: Yes, I have seen excessive liquid entrainment from the low pressure rectifier to the LPCC due to fouling (iron oxide scaling) of the holes in the trays in the rectifier. What is the urea content in the carbamate recycle?

Janusz replies: We don’t have trays in the rectifying column, we have a bed with rings. Some (not all) of the carbamate solution samples show a very high urea content, up to 29 wt-%.

Mark responds: But you do have a liquid divider on top of the bed – that is what I meant by trays.

29 wt% is a sure indication of entrainment from the rectifier. Do you have a tangential inlet or sprayer for the liquid inlet of the rectifier?

Mr Akbar A of Safco in Saudi Arabia shares his view: We have also experienced the same problem, but with a new Stamicarbon plant where a separate reflux condenser was installed for the desorber column. At the moment when the level of the LPCC level tank shoots up, collect a sample and analyse it for urea. If you find a high urea content, it will be clear that urea solution carryover is occurring from the LP rectifier column only. 29 wt-% urea is too high and may affect the synthesis conditions and HP scrubber, as well as the HPCC equipment.

In a new Stamicarbon urea plant this problem can be solved temporarily by increasing the pressure in the rectifier, but in your case you don’t have a separate condenser for the desorber so this increase in pressure will disturb the desorber performance.

Do you have orifice plugs in the tubes at the bottom of the



Blocked orifice plugs in circulation heater bottom and top liquid divider blockage.

rectifier? If the internal diameter of these orifice pugs has reduced, that can also cause carry over.

Janusz answers: Yes, we do have something that could be called a liquid divider over the bed. We don't have a tangential inlet. And we don't have orifice plugs in the heater tubes.

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran shares his experiences: We have experienced this problem twice in the low pressure rectifier column. After opening the column we saw that the packing bed was choked with corrosion material. Another one was choked in the heater tubes. The failure of the level transmitter in the rectifier tower can also be another cause.

Mr Kashif Naseem of SABIC in Saudi Arabia contributes with his experiences: Possible sources for this is choking of the orifice plugs in the bottom of the rectifying column circulation heater and the packing.



Packing support plate clogged with corrosion products.

Mr Akbar Ali of SABIC/Safco in Saudi Arabia joins the discussion: For a better understanding of the phenomenon of carry over due to blockage in the top liquid divider and bottom orifice plug, see pictures on previous page, captured after opening the rectifier column following the same problem of solution carry over. After removing these bottlenecks the plant conditions improved.

Janusz replies: In my case the heater was clean but the packing support plate was almost completely clogged with corrosion products – see picture above.

Mr Muhammad Farooq of SABIC/Safco in Saudi Arabia agrees with Mark: I agree with Mark's observation about the rectifying column. However, a higher recycle rate can be reduced gradually by closing the reactor off gases to the low pressure section provided that the synthesis pressure is in a controllable range, in this scenario the control valve opening should be checked. Although it is not the cause of urea carryover, it may reduce the intensity of the higher recycle rate. ■

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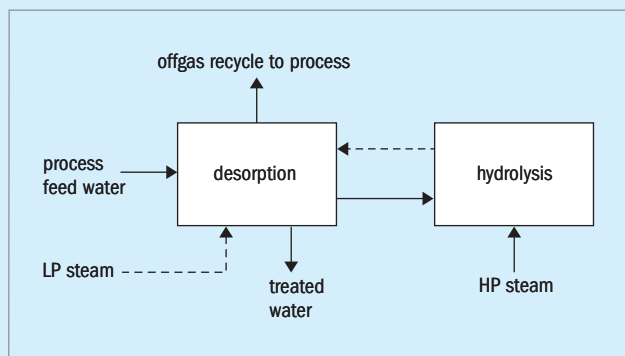
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Problem No. 34 Reuse of off-spec waste

The waste water treatment section in a urea plant processes the water containing $\text{NH}_3\text{-CO}_2$ and urea from the vacuum system in the evaporation section. It delivers an almost $\text{NH}_3\text{-CO}_2$ -urea-free purified process condensate that is suitable for reuse outside the urea plant. This process condensate can be discharged with a content of 1 ppm of urea and 1 ppm of ammonia. With such negligible values of pollutants, three targets are simultaneously realised which contribute to reducing the cost of urea: specific consumption of ammonia is decreased, environmental pollution is reduced and the possibility to reuse the process water is guaranteed for several technical purposes.



Mr Niraj Nimje of RCF Thal in India kicks off this Round Table discussion with a practical and troublesome problem: In our Saipem urea plant, after the booster ejector and condenser washing in the vacuum section, we are experiencing problems with the concentration in the waste water section feed tank and the off grade condensate is wasted as effluent. What methods can be used to treat this off-spec condensate to make it BFW grade? I have heard of using a polishing unit for such cases but what is the maximum concentration that is permitted? What are the exact arrangements required to ensure quality?

Mr Prem Baboo from NFL company in India shares his experience: We have carried out the following modifications to the vacuum sections:

- crown flushing of 2nd stage vacuum separator (cone flushing) with 99.7% urea solution to minimise the water load and to minimise prilling diversion;
- condenser flushing with waste water (waste water to distillation), a 2" line taken from the distillation tower feed pump for flushing and to minimise the water load in the waste water section;
- water monitoring and water balance in each shift.

We receive waste in the DM plant containing 1.0 ppm urea and 2-3 ppm ammonia and this is suitable as DM water. We maintain the following parameters:

- distillation pressure top 1.8 kg/cm²g and a temperature of 112°C; bottom temperature of 132°C; low pressure steam to distillation flow is 11.0 t/h;
- waste water flow about 80-82 m³/h at 3,500 t/d load;
- hydrolyser pressure 34 kg/cm²g and temp 232°C; steam flow to hydrolyser about 5.0 t/h;
- waste water analysis 5.5 to 6.0 wt-% ammonia and 0.9 to 1.0 wt-% urea.

Mr Nagaraju from Notore Chemical Industries in Nigeria asks a question: Does the waste water from the 2nd desorber contain such high levels of ammonia and urea? In our plant it used to be around 100 ppm urea and 50 ppm ammonia.

Prem replies: This high concentration of ammonia and urea cannot be taken in demi-water, it means your hydrolyser is not working properly, the hydrolyser outlet urea should be less than 1-2 ppm. Please clarify your hydrolyser pressure, temperature and desorber temperature etc.

Mr Kashif Naseem from SABIC in Saudi Arabia shares his idea and asks a question: I agree with Prem that waste water with an ammonia and urea concentration of 1~3 ppm is acceptable for use as BFW after passing through a polisher.

Like Prem you are using this in boilers as boiler feed water, have you observed any corrosion in the boiler or steam line and what is the status of ammonia and urea in CBD?

Niraj provides further information: Prem, we perform the same activities that you mentioned and we are getting BFW quality water without any polishing unit. Our hydrolyser and distillation units are working fine at 25 kg/cm² and 1.8-2 kg/cm² respectively, but after crown washing, condenser booster ejector washing, or during any upset, the waste water tank concentration changes (temporarily) and off-spec wastewater is generated.

During such situations we increase the steam to the hydrolyser and distillation column. After a revamp our waste water load increased from 40 to 50 m³/h so it is sometimes difficult to maintain the quality of water generated after performing activities mentioned. As we don't have any polishing unit, we are looking forward to it, and want to convert even small quantities of off spec waste water into boiler feed water.

Prem replies: For BFW water ammonia is not a problem because the ammonia is added to raise the pH of the BFW. Only urea is a problem due to a problem of your hydrolyser. Hydrolysis is a function of temperature and you cannot raise the temperature beyond 223°C at a pressure of 25 kg/cm² with a revamp. You should revamp this section by either increasing the residence time or raising the pressure.

Please note the residence time of your hydrolyser has been reduced after the revamp.

Mr Mang Tân Phong from Petro Vietnam Ca Mau fertilizer company in Vietnam raises another question: Our plant uses Saipem technology for the urea synthesis and TEC technology for the fluid bed granulation. Our MMU section didn't work well so we decided to stop operating the MMU section and feed formaldehyde directly to the pump suction line. In this case our waste water after treatment still contains formaldehyde (more than 500 ppm). The parameters at the top and bottom of distillation are, respectively 4.2 bar and 130°C, 4.7 bar and 157°C. The condition in the hydrolyser is 31 bar and 230°C. How does formaldehyde at concentrations above 500ppm affect the BFW, and how can it be treated?

Mr Mark Brouwer from UreaKnowHow.com in the Netherlands replies: Formaldehyde will be converted into formic acid in the hydrolyser. Depending on how the purified process condensate is reused, additional measures may be needed to avoid corrosion problems. For example reuse as cooling water make up may be possible without further treatment, while reusing it as boiler feed water for 40 bar steam will require further purification. One could send the purified process condensate to the inlet of the demi water section where the carbon filter and ion bed exchangers will take care of the formic acid.

Prem replies: Best answer given by Mark, the formaldehyde and methanol are controlled by the ammonia plant: the formaldehyde is formed in the LT CO shift converter and methanol is formed in the HT CO shift converter. Formaldehyde and methanol which comes with CO₂ is generally separated at the CO₂ compressor inter-stage cooler with separators. In the urea plant nothing can be done apart from controlling the compressor inter stage temperatures to maximise the amount of methanol/formaldehyde separated by the separators.

Mr Majid Mohammadian from OCI in the Netherlands shares his idea: If you are injecting the formaldehyde as an anti-caking agent for the granulation/prilling section, it should normally be injected to the discharge of P8 and not P6, in this case you will have much less formaldehyde in your waste water treatment section. Can you clarify why you are injecting it to P6?

Mr Nikhil Kamat from India asks a question: What should the ammonia content and urea content in the treated waste water be so that it can be used as boiler feed water?

Prem replies: In our plant, the treated water from the distillation tower contains about 2-3 ppm ammonia and 0.5-0.8 ppm urea. This treated water is sent directly to the DM plant (cation/anion/mixed bed) and then used as BFW. The turbine condensate and steam condensate are sent to the DM plant i.e. cation unit for iron removal only and then used for BFW. ■

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Problem No. 35 Urea plant preservation

Preservation covers the method of protection and preventative maintenance carried out on equipment and systems before and after they are put into use. In power plants, fertilizer units and refineries, expensive equipment like turbines, chillers, power generators, boilers, condensers, panels etc. begin to rust and corrode when in storage or during shutdown or even when in operation due to the presence of excess moisture in the air. Proper downtime procedures are extremely important. In this round table discussion, preservation and its importance is discussed by urea plant experts.



Above left: Failure due to carbon build up.

Above right: Corrosion on the HP flange in the urea plant.

Mr Nauman Talat of Pakarab Arab Fertilizer in Pakistan starts the round table discussion: We are planning to preserve our urea plant for a period of six months to one year using air. Please share your experiences in this regard.

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran replies: As you know, special materials of construction are used for the equipment in urea plants. My recommendations for long term preservation of the plant are, if possible:

- wash the equipment well with demi-water;
- purge and dry with nitrogen;
- use nitrogen blanketing with positive pressure.

Mr Mark Brouwer of UreaKnowHow.com from The Netherlands offers his advice: In addition, take care that no salts (chlorides, nitrates, carbonates) can enter the tube side, shell side and the area between the loose liner and the pressure bearing carbon steel, so also plug the leak detection holes. Control a positive pressure with a manometer.

Mr Assadollah Rastegarpour of NEBCO from Iran shares his experiences: There is always a risk of accumulation of carbamate and urea in dead points, low points, around ejectors nozzles, and especially in the HP stripper. Ensure that these are flushed with boiler feed water and take samples from the drains to check for traces of urea and carbamate. If you decide to pressurise the system, blank it with a blind flange. I recommend blanking all control valves in the outlet of the reactor and HP stripper. In order to reduce the risk of control valves seizing up, take important and special valves to the shop for checking and to store them in good condition.

Mr Shiraz Chandio of Engro Fertilizer from Pakistan joins the discussion: We are also facing a similar situation at our plant due to gas shortages nowadays. After complete and thorough flushing of the vessels and piping, we preserve all the vessels and piping with a

nitrogen blanket with positive pressure and continuous flow (where pressure blanket is not possible). We have also preserved the cooling water system after a shock dosing of chemicals as recommended by our process guys. Steam headers are also preserved with nitrogen.

Mr Majid Mohammadian of PIDEMCO from Iran shares his experiences: Take care to preserve the rotary equipment too. My suggestion is that if you have power and cooling water (small requirements), keep lube oil systems for the CO₂ compressor, ammonia and carbamate pumps in operation or on a regular basis and, for other rotary equipment, turn their shaft on a regular basis.

Easa adds: If you are planning to preserve the plant over the winter take care that equipment doesn't freeze, especially rotary devices. Any residual water inside devices can freeze and cause a rupture. We experienced this in our plant during the winter season (-20°C), even though we purged the equipment with nitrogen some items were forgotten.

Mark offers some more recommendations: Below are some recommendations when operating a urea plant at low loads, different loads, with many shutdowns and with a long shutdown:

Control/limit temperature changes during plant start-up/shutdowns and for frequent load variations. During these changes big stresses are put on the liners.

Operating at low plant loads can be critical for the HP stripper, HPCC and HP scrubber in cases where gas and liquid at the inlet of these HX are not well distributed. This is critical to avoid higher corrosion rates.

In the HP stripper each tube needs a minimum amount of liquid. When the liquid dividers are older, the liquid holes become bigger and the liquid level at low loads can become too low to assure that each tube receives the minimum amount of liquid. In the HPCC and HP scrubber a gas cushion should be present below the gas distributor at the inlet.

Changes in plant operating and monitoring procedures for prolonged low load operation: Take daily nickel analysis in the urea melt and also establish a baseline nickel level during normal operation, so you know when the nickel content become too high. Carrying out more frequent inspections would also seem to be a good idea.

Preservation of equipment's during prolonged shutdowns: In general pay attention to chlorides and nitrates. Ensure that they cannot enter the equipment and/or the leak detection system.

An increased risk of corrosion due to frequent and prolonged shutdowns is especially valid for equipment in a marine atmosphere.

As a result of "breathing" due to cyclic temperature changes infiltration of chloride containing vapour is likely to occur. These chlorides may cause stress corrosion cracking in austenitic stainless steels and pitting/crater type attack in carbon steel equipment. To avoid the ingress of chloride containing vapour, the equipment should be made inert with nitrogen (0.3-0.5 bar gauge). Avoiding the ingress of chloride containing moisture is of utmost importance for the shell side of HP heat exchangers like the HPCC, stripper, HP scrubber with austenitic stainless steel tubes in carbon steel tube sheets. Chloride containing moisture in the gaps (crevices) of the tube to tube sheets cannot be removed by flushing with chloride free condensate or demi water.

Special attention is required for the increased risk of CUI (corrosion under insulation) due to frequent and prolonged shutdowns. Cyclic temperature changes are most detrimental with respect to CUI and need specific preventive measures like painting and maintenance at the (aluminum) insulation cover sheeting to avoid ingress of moisture. It has to be emphasised that the increased risk of atmospheric corrosion should not be ignored.

Mr Nasir Hussain of Pak Arab Fertilizers Multan from Pakistan describes the planning for their urea plant: We are planning to preserve stainless steel equipment and piping with instrument air and use nitrogen for carbon steel material.

Mr Ali Salman Bokhari of Pak-American Fertilizers in Pakistan asks a question: Please explain the significance of providing a gas cushion in the HPCC and scrubber below the gas distributors at the inlet. What measures should be taken for preservation of control valves and other instrument items?

Mark replies: This is only valid for low plant load conditions to assure a good gas/liquid mixing before condensation of the gas. Otherwise condensation corrosion can occur with 316L UG material. ■

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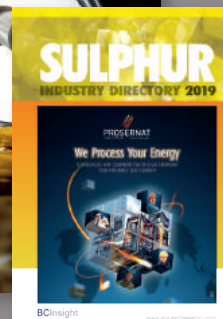
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Problem No. 36 Change in colour of urea product



Stainless steel owes its corrosion resistance to the presence of a protective chromium oxide layer on the surface. As long as this layer remains intact, the metal corrodes at a very low rate. If the passive layer in stainless steel is damaged, active corrosion starts in aggressive corrosive environments. Stainless steels exposed to carbamate containing solutions in the urea synthesis section can be kept in a passive state by adding a minimum amount of oxygen. If the oxygen content drops below this minimum, active corrosion starts, which can be identified by a shiny silver colour on the surface (see picture). Adding oxygen and maintaining a sufficiently high oxygen content in the various process streams are prerequisites for preventing excessive corrosion of the equipment and piping. Once active corrosion occurs, the corrosion rate is extremely high (>50 mm/year) and will not stop even if more oxygen is supplied to the process.

Mr Akbar Ali from SAFCO, Saudi Arabia starts off the discussion:

What are the possible causes for a change in urea product colour e.g. a dull white colour?

Mr Mark Brouwer from Ureaknowhow.com, The Netherlands,

provides the first answer: I know of two causes: oil and corrosion products (iron oxides). Check the nickel content in the prills and compare it to the normal level and/or check for high oil consumption somewhere. A low level in a tank could indicate that oil is passing to the downstream sections.

Mr Nasir Hussain from Pak Arab Fertilizers Multan in Pakistan joins the discussion and provides further information based on his experiences:

Oil and corrosion products are the most common causes of a change in prill colour. Another reason can be if the urea solution is held for a longer time in the urea melt storage tank. When we recycle this urea with fresh urea melt it results in a colour change. To check for corrosion products samples can be collected at the exit of the reactor, stripper, medium pressure decomposer, low pressure decomposer etc. to determine the exact location.

Mr Faisal Ghafoor from Fertil, United Arab Emirates shares his experience:

The presence of methanol in the CO₂ from the ammonia plant can also cause a colour change; this can be observed with a new LTS catalyst.

Mr Sam QR from Fertco company in India confirms Mark's recommendation:

As Mark said, it is mostly the corrosion product you will see after a stoppage or load reduction. Check for iron and nickel in the product.

Mr Malik Sohail from SAFCO in Saudi Arabia shares his experience:

Yes the possible causes of product colour change have been provided by Mark. Of these I believe that during plant operation the causes are a high oil content from the pumps and compressor and/or an abrupt drop in the recovery level and concentration sections. This can be indicated by the level glass of the final separator. Foaming in the first stage evaporator can be seen from the level glass. When ammonia is imported from ammonia storage it is possible for it to have a high oil content.

Mr Muhammad Farooq from SAFCO in Saudi Arabia asks Akbar a question:

Is this a permanent change of colour of the urea product or did you observe it during foaming or excess ammonia at the concentrator and rectifying column?

Akbar summarises and provides more information on the issue:

Thank you for your inputs. We experienced this problem during a start-up after holdup of urea solution in the reactor for 48 hours. Just before shutdown we encountered a problem of increased H₂ slippage up to 1.4% (for 15 days) with CO₂ from the ammonia side; due to a problem in the ammonia plant, the delta temperature across the hydrogen reactor approached 68°C.

We suspect that during this time it was not possible to manage the air flow control properly, as seen from the oxygen content trend downstream of the hydrogen reactor. This would have resulted in active corrosion which probably caused the change in product colour. After three days of operation the original colour was resumed.

Mr Majid Mohammadian from PIDMCO in Iran shares his opinion:

Contamination with impurities from the recycle tank or urea formaldehyde tank (in case of use) could be another cause.

Mr Muhammad Kashif Naseem from SABIC in Saudi Arabia gives his recommendations:

From my experience, product colour can change due to the following:

- high oil contents in product;
- high iron contents;
- carry-over of CO₂ system absorbent to the synthesis loop.

Please confirm the passivated air range of between 0.6-0.8 vol-% in CO₂, which is dependent upon the N/C ratio and the material of construction of the HP synthesis system. During a colour change measure the oil, Fe and Ni content in the product at different sample points. During the hold-up time whether in the reactor or the urea solution tank, the product colour always changes during start-up.

Mr Victor Rengel from FertiNitro in Venezuela asks a new question:

I would like to take advantage of this discussion to ask about the normal limits for the nickel content in urea during reliable plant

operation. In the last few days we had a plant shutdown with a delay of three days for recuperation. Yesterday after start-up we noted a yellow colour in the urea granules. We checked the nickel content, which was 4.44 ppm, much higher than the normal average of 0.02 ppm. We have stopped feeding liquid urea from the tank. We would like your opinion or advices about the high Nickel from the point of view of damage to the passivation layer or equipment corrosion.

Mark replies: In the case that your passive layer is not fully intact and as a result there is active corrosion, the active corrosion will continue even if you increase the oxygen content.

If you have active corrosion somewhere, you will see an increasing trend of your nickel level. There is no normal limit, every plant has its typical normal nickel level depending on materials used etc.

Victor provides further information: After stopping the feed from the urea liquid tank the nickel level is decreasing back to its normal value, therefore is no active corrosion.

Mr Nimesh Maurya from KRIBHCO in India shares his opinion: As mentioned earlier, the colour change of the urea prills is due to two main reasons: corrosion and oil contamination.

When oil seals of the ammonia compressor or carbon dioxide compressor leak oil into the system, it can be diagnosed by viewing the glass window of the MP urea holder and LP holder. If there is any foaming it means oil is coming into the system.

If oil is entering with liquid ammonia it can be removed by draining the ammonia receiver tank for some time. When you drain it, lumps of oil will come out. The ammonia receiver tank needs to be drained regularly until the leakage problem is solved.

Akbar comes back with more information: The intent of this post is to share our problem related to blocking in and its impact on product quality at one of unit (1,800 t/d Stamicarbon technology with fluid bed granulation). We are facing a frequent problem regarding off specs product due to its dull white colour, this is a common thing now whenever urea solution is kept under blocking in for 24 hours or even less than 24 Hrs.

During a recent shut down (April 29, 2014), urea solution was kept for about 25 hours for blocking in. After start up, for more than two days product was off spec due to a dull white colour. Regular analysis for Fe and Ni in the final product was carried out and it was found that the Fe content was very high (up to 5 ppm compared to the normal figure of <0.1 ppm), but in all samples the Ni content was < 0.1 nppm which is acceptable and normal for us.

We checked the synthesis parameters before and during blocking in as below:

- O₂ to stripper as per analyser was 0.66 vol-%;
- reactor temperature during blocking in came down to 140°C;
- synthesis pressure came down to 54 kg/cm²;
- N/C was operating 2.98 to 3.0;
- CO₂ feed was cut first as the CO₂ compressor was tripped, NH₃ feed was cut after 3 minutes;
- When product becomes normal, Fe in final product was analysed and found to be normal <0.1 ppm;
- the material of the reactor liner, down comer and trays is 316L UG (BC.01).

We are investigating this incident and seeking technical assistance concerning this problem. ■

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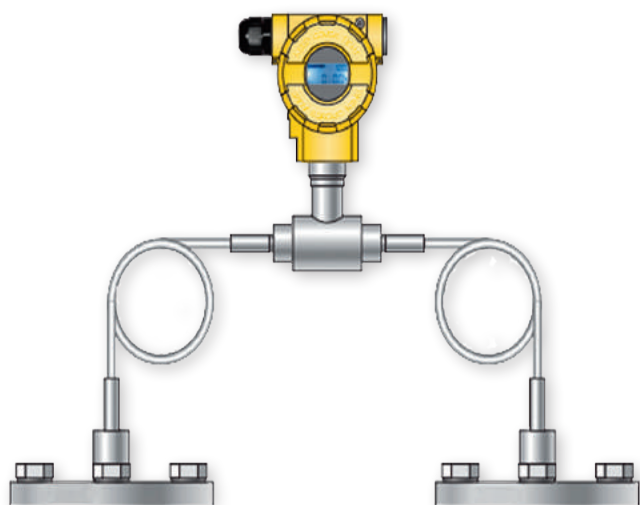
Plant Manager+

Problem No. 37 Faulty reactor pressure transmitter

Pressure transmitters have become essential parts of virtually all urea plants. Pressure measurement undoubtedly forms an important measurement in industrial process measurement and control. Pressure transmitters convert an applied pressure into an electrical signal, which is then transmitted to a device

such as a computer, programmable logic controller, recorder, or digital meter to analyse the electrical signal and utilise it to display, record, or modify the device's pressure.

A schematic of a pressure transmitter is shown in the picture below left.



Mr Ab Samad Noraini from **Petronas Fertilizer, Malaysia** initiates a discussion of a familiar problem in urea plants: Our plant uses Saipem technology and we have experienced frequent faults with our reactor pressure transmitter. Because of this we often need to by-pass the protection of the trip logic of urea reactor.

Currently we are using a capillary type pressure transmitter. Does anyone else have similar problems and experiences that they can share and advise on modifications to solve the issue?

Mr Waheed Ahmad from **FFC, Pakistan** is first to respond: Different options are available to eliminate this problem. Please specify the position of the transmitter. How many times have you experienced tripping or failures?

Mr Girish Prakash from **Tata chemicals Ltd, India** joins the discussion: Reactor pressure transmitters are prone to failures but with certain precautions you can ensure a longer life. One of the easiest methods is to ensure proper working of tracings. In no case should they be in an isolated condition once start-up is under progress or the reactor is under draining.

Mr SK Gupta of **IFFCO, India** shares his experience: The reactor pressure transmitter installed in the CO₂ line, just after the control valve in the CO₂ line near the reactor, is always giving us problems. You can install the transmitters in a horizontal position by rotating the spool piece between the control valve and the reactor. This will reduce the frequency of failure.

Mr Mark Brouwer of **UreaKnowHow.com, The Netherlands** provides some more suggestions:

Other options to improve the reliability are:

- measure the reactor pressure in the ammonia line
- when measuring in a carbamate line, use tantalum as membrane material

Mr Gopala Krishna of **Safco, Saudi Arabia**, provides more information: You mentioned you are using a capillary type transmitter but the most reliable transmitter is the pad type. Also when the transmitter is faulty its sensor element should show the minimum value; this measure can be taken by instrument people. Secondly, the high pressure reactor reflects the high pressure of the compressor, hence the reactor high pressure trip can be bypassed or reactor pressure influences more or less MV-01 (separator pressure) so the reactor high pressure trip can be shifted to the separator pressure trip.

Recently an accurate and reliable pad type pressure measurement has been developed, which is successfully in operation since September 2011 and is able to measure the pressure even in high pressure carbamate gas lines.

Mr Javant Panvelkar of **Zuari industries limited, India** shares his experiences: We have the impulse tubing type pressure transmitter on the ammonia feed line and it is working fine. The only time it has gone wrong is during shutdowns when the reactor is in a boxed up condition, due to carbamate entering the impulse lines. This is easily cleared by flushing the lines. As previously mentioned, new technologies are available, but I feel the problem with the pad type is that if it fails it is not possible to replace it online. Two pads probably will have to be used? We flush the transmitter lines once a month.

Mark provides more information: In stripping plants it is an advantage to measure the synthesis pressure as close as possible to the safety valves, i.e. in the carbamate gas line. This new development makes that possible without risks of crystallisation and corrosion.

These special pad type pressure measurements are in operation since Sept 2011 without any problem.

Mr Kashif Naseem of **SABIC, Saudi Arabia** gives his suggestions: The most suitable place for this pressure transmitter is the ammonia feed line to the reactor. You can use a double pressure transmitter diaphragm type and make both at different pressure frequency. So make the trip point when both will appear.



The Badotherm extended type diaphragm seal with all wetted parts in zirconium.

“**Pressure transmitters**
have become essential
parts of virtually all
urea plants

Mr Ismail Ahmad of Ruwais Fertilizer, United Arab Emirates shares his experiences: We are using Stamicarbon technology and face a similar problem. However, we are going to replace the existing unit with a remote seal (extended diaphragm) type. It will be installed directly to the pipe, and the sensing diaphragm will be at same level as the piping surface, thus preventing the crystallization problem. Based on feedback from other plants using similar technology, the problem is resolved by using this transmitter. We will be installing the new transmitter this coming September.

Mark provides more information: Badotherm has successfully developed a safe and reliable membrane type of pressure and level measurement suitable for all urea process technologies. On stream times of already nearly two years have been achieved without any issues. This means significant costs can be avoided and additional profits can be gained with the innovative Badotherm diaphragm seals mounted to transmitters.

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Problem No. 38 Flares in urea plants

Flares in urea plants are designed to improve the site's safety performance by capturing and burning ammonia emissions from safety release points. This will prevent/reduce ammonia emissions from entering the environment and in turn reduce ammonia risks. Flares are becoming more common in new urea plants, but are they the optimum solution to improve safety and reduce environmental risks?



João Amilton from Petrobras, Brazil initiates this round table:

Recently I read about flare stacks to reduce ammonia emissions in Profertil, Argentina. Our manager wants to know more about flares to handle the discharge of pressure safety relief valves of the high pressure synthesis section because he is considering installing one in our plant. Is this kind of flare available and is it a good idea?

Mark Brouwer from UreaKnowHow.com replies to the question:

The relief case of a synthesis pressure safety valve has major consequences and requires careful investigation into how to reduce the likelihood of pressure safety valves from popping up and how to reduce the consequences. Installing a flare system is one option. Other options include installing instrumental safeguards (high pressure switch) and applying pilot operated safety valves. For your information please refer to the paper from Joey Dobree, Stamicarbon presented at the 2012 Asian Nitrogen+Syngas Conference, which gives a comprehensive view on the environmental consequences of a flare system.

João asks another question: Thanks Mark, do you know of any plants where the flare system is operating now?

Mark replies: There is a flare system in operation at the Profer-til's Saipem plant in Argentina as already indicated. There is also a flare system in operation at PIC's Stamicarbon plant in Kuwait.

B Suresh from NFCL, Kakinada, India provides more examples of flares in operation in urea plants: In many urea plants in India, ammonia safety valve discharge and vents are connected to blow down headers and the header is elevated to the prill tower height i.e. around 100-110 m. But, continuous flaring is used in the vent stacks of ammonia storage tanks and there are flares in the front end and back end of ammonia plants.

Dading Abdul Kadir from PFK S/B, Malaysia, provides another example of an operating flare: There is a flare system operating in our urea plant at PFK Sdn.Bhd. in Malaysia. There is a high pressure flare stack for relief valves, one for medium pressure vent and one stack for low pressure vent and tanks.

Noureldeen Abbas from Petrochemical Industries Company, Kuwait provides information about a dedicated ammonia flare in a urea plant:

We have installed a flare system in our urea plants in Kuwait (PIC) and connected all safety valves and vent stack to a dedicated flare system. All ammonia sources whether continuous or intermittent are connected to the flare system and it is working well.

Mark provides conclusions from two papers that were presented at the 2012 Nitrogen+Syngas conference and gives new suggestions: Two interesting papers about flares in urea plants were presented during the Nitrogen+Syngas Conference in Athens. The main conclusions were:

- Flares introduce new risks (crystallisation and explosive mixtures in the headers) and more environmental concerns (NO_x versus NH₃).
- Do not simply specify a flare system in your inquiry for a new urea plant, define which continuous and discontinuous emissions are acceptable.

In my view there are better alternative solutions than a flare such as:

- design the piping around the centrifugal HP pumps for higher pressure to delete the safety valves on these pumps;
- use high pressure switches in the synthesis section to reduce the risk of blowing of synthesis safety valves;
- use emergency separators or absorbers;
- disperse gaseous ammonia at safe locations.

Several urea plants have been safeguarded according to these alternative solutions.

Ramchandra Nesari, a freelance consultant from India shares his experiences: I have seen flare stacks installed in some Saipem urea plants in the Middle East. These plants have separate flare stacks for continuous and non-continuous emissions. There is a separate flare stack for blowdown from safety valves. They are operating satisfactorily, but you have to provide a small quantity of natural gas as fuel for the pilot burners. ■

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Plant Manager+

Problem No. 39 Warehouse curing for urea granules

Good storage and housekeeping practices are always important to ensure a safe workplace. Where possible, fertilizers should be stored in closed storage to protect the product from the weather (sun, rain etc.). The ideal storage conditions are:

- Enclosed building capable of protecting the product from the weather and sunlight
- Temperature between 5 and 50°C
- Protect the fertilizer from moisture, which can cause lumps and dust, affecting spreading capabilities
- Good stock management e.g. carried out on a first in first out basis.



Muhammad Kashif Naseem from SABIC, Saudi Arabia starts this round table discussion: Can anyone explain the warehouse curing phenomenon for granulated urea product and the maximum time required for this, particularly in the summer season in the Gulf region with a product outlet temperature of around 50°C?

Mark Brouwer from UreaKnowHow.com replies: Intentional aging or curing of fertilizer in a storage pile prior to bagging or bulk shipment is referred to as curing. Chemical reactions that cause caking bonds proceed to near completion during the curing period. The heat of reaction retained in the curing pile speeds up the completion of the reactions. After curing there is reduced tendency for additional bonds to develop. What is your experience of the time required for curing?

Akbar Ali from SAFCO-SABIC, Saudi Arabia raises a good question: In urea granulation the curing time is 60 to 72 hours. Can we understand from your comments that the length of curing time is due to chemical reactions causing caking bonds or is there any other reason behind this? Have you any literature on curing time for urea granules showing how we can decide on the curing time for a specific product temperature?

Muhammad Kashif Naseem from SABIC, Saudi Arabia replies: Almost three days is considered normal, but can we reduce this or is there any method available to reduce this warehouse curing time?

Easa Norozipour from KHPC, Iran answers the last question: As we know, curing is a phenomenon for stabilisation of a humid and high temperature product like urea. In this case, I think the curing time also depends on the following:

- water content in the final product;
- amount of ammonia content in the product;
- temperature of the final product;
- the delta temperature between warehouse and environment;
- height of the urea pile in the warehouse;
- relative humidity of the environment;
- size of the final product.

All of these items can influence the curing time of the final product. In our experience three days is the normal time.

Zaheer Abbas from SAFCO, Saudi Arabia shares his experiences: In my experience, our final check is the temperature differential between the urea granules and warehouse ambient conditions.

Whatever the humidity outside, it will finally affect the temperature differential. If we are able to maintain the recommended temperature differential ($\Delta T = \pm 5^\circ\text{C}$), the curing time will be automatically adjusted. I would recommend taking some temperature measurements before deciding on the curing time for any warehouse. Other contributing factors are final cooler efficiency and the difference between wet bulb and dry bulb temperature inside of warehouse.

Mark Brouwer from UreaKnowHow.com provides more information: Another important consideration is that caking behaviour is in part determined by the amount of dust and fines in the product.

Easa Norozipour from KHPC, Iran shares more information: Sometimes near the Gulf the humidity is very high and we have experienced a lot of water droplets in and around the urea belt conveyer. As we know, there aren't any warehouse and belt conveyer channels with 100% sealing. Therefore the relative humidity of the environment can increase the caking tendency. The mesh size (dust) of the final product is also an important factor as mentioned already by Mark.

Akbar Ali from SAFCO-SABIC, Saudi Arabia asks a question: Do you have any written information regarding this subject? I could not find anything in the Stamicarbon documents.

Janusz Maćkowski from ZCh"Police", Poland provides a reference: At the Tenth Stamicarbon Urea Symposium 2004 (Round Table Presentation) there was an interesting article "Improvements in the physical quality of urea prills". I think it is also related to this topic.

Nick Sutherland from SOLEX Thermal Science Inc., UK provides some useful information: As we know, urea (and all other fertilizers) are hygroscopic and as such if sent to the storage area while still at a temperature above that of the local ambient conditions any moisture in the air will be attracted to the product, this is obviously a problem in climates where the humidity is high and if the product has even a small amount of dust then "caking" is likely to occur.

To reduce this situation I recommend you consider the Solex Bulk Flow Cooler which can cool the product closer to the ambient temperature with the added benefit of not generating any further dust, in fact the unit, in conjunction with a de-dusting system, can reduce the dust load.

Muhammad Kashif Naseem from SABIC, Saudi Arabia contributes to the discussion: Product dust formation is influenced by:

- moisture or humidity;
- differential temperature of atmosphere and product;
- free ammonia in product;
- strength of product;
- pressure in final product system (prilling tower + granulator scrubber);
- frictional resistance of the conveying system.

Methods to prevent dust formation include:

- UF-85;
- Urecoat/Ureasoft;
- seeding technology;
- suitable temperature and pressure in final concentrator.

Mark Brouwer from UreaKnowHow.com provides more information: Freshly produced urea granules need some kind of “curing time” to reach their final anticaking quality. Normally 1 or 2 days will do the job.

The product leaving the granulator has been screened to remove the undersize and oversize particles in order to produce an end product of typically 95% between 2 and 4 mm. That means that your end product is a mixture of granules from 2 mm up to 4 mm diameter. That seems to be very close but in terms of weight per granule the difference is huge. The weight of a 4 mm granule is about 8 times higher than the weight of a 2 mm granule.

Because of this big weight difference, a 4 mm granule will be more difficult to fully dry out than a 2 mm granule. The result is that a 2 mm granule has a lower moisture content than a 4 mm granule. The moisture figure you get from the laboratory during production is the average moisture of the granule mixture. You can easily check this by determining the moisture content of the fines recycle product and of the oversize before crushing. You will find a very big difference.

When you dump your freshly produced end product on a pile in storage, this moisture difference will slowly disappear: finer product will pick up moisture from the bigger product. This moisture “migration” will cause some caking in the fresh pile. Once the moisture migration is finished and you break the slight caking (during shipping) this initial caking will not happen again.

Of course that does not mean that your product will be 100% cake-free: the normal rules remain, low moisture is better than high moisture, low temperature is better than high temperature, a low pile is better than a high pile, etc.

Adel Tabei from Pardis Petrochemical Company, Iran raises new questions: Is it possible for the caking phenomena to occur during shipping (at high humidity condition) after sufficient curing time of urea in the warehouse? Or if we have a sufficient curing time in the warehouse, will that prevent caking during shipping? Or are these not related?

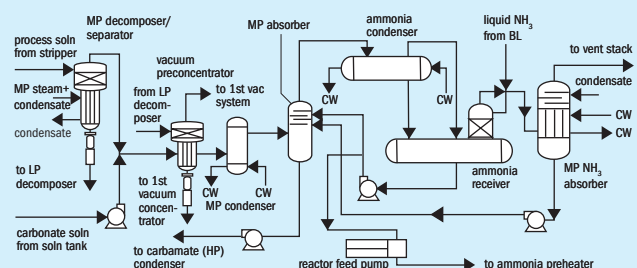
Gholamali Soroush from Shiraz Petrochemical Complex, Iran replies to the raised question: Besides the drying time, the urea curing process depends on humidity, temperature, piling height, and so on. Even if there are no changes in the temperature and humidity (good sealing) from the warehouse to the destination, there may be some reduction in urea quality that reduces the urea crushing strength. Urea powdering (degradation) is related to the granule formation step and the quality of the anti-caking agent. In some destinations, the environmental dew point (humidity and temperature) may have less impact on the urea than the warehouse. However, if urea encounters a reduction in temperature and an increase of humidity from loading port to destination, some moisture can become trapped in the urea granule. Humid air can also diffuse into the urea granule leading to caking. For these reasons, besides the control of all the usual parameters, I suggest using good quality urea formaldehyde, and spraying anti-caking agents after the granulation step to ensure customer satisfaction. ■

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Plant Manager+

Problem No. 40 Medium pressure absorber in Saipem urea plant

The function of the medium pressure (MP) absorber is to separate pure ammonia from ammonium carbamate before both of these streams are recycled to the synthesis section. Control of the process conditions of the MP absorber is one of the key control parameters in a Saipem urea plant. CO₂ carryover to the ammonia receiver is not permitted in the process as it will create corrosion problems and upset conditions in the ammonia recovery and pumping system. This round table discussion considers the problems and solutions in this process section.



Nasir Hussain from Pak Arab Fertilizers Multan, Pakistan starts the round table discussion: Please tell me about problems with the medium pressure absorber in a Saipem urea plant. What is the allowable CO₂ carryover to the ammonia receiver?

Siddharth S from TATA Chemicals Ltd, India replies: High temperatures above the first tray and high levels and/or partial choking of the trays may cause CO₂ carryover from the MP absorber. If the problem persists there could also be some passing between the joints of the tray segments and inlet pipe sparger joints (gasket may be damaged). Proper sealing on the joints is a must. This may be checked during shutdown by observing the water retention period on the trays (normal retention time for water should be more than 20 minutes). During normal operation there should be no CO₂ carryover to the ammonia receiver.

Girish Prakash from TATA Chemicals Ltd., Babrala, India shares more experiences: Level fluctuations in the column during start-up/upset conditions are very common for this equipment. You need to pay attention to the actual liquid level in the column.

Mohd Saiful Mohd Sofian from PC FK, Malaysia raises a question: Has anyone implemented any improvements in this unit? There is a patented technology claiming to be able to eliminate this problem. Does anyone have any more information?

Girish Prakash from TATA Chemicals Ltd., Babrala, India explains what he has done in his plant: We have made internal improvements without any external help. If you have any particular thing in mind, please share so that we can also benefit.

Azad Panchal from GNFC, India asks a question: Can anyone tell me why the upstream temperature is kept as high as 88-90°C in the MP absorber?

Prem Baboo from National Fertilizers Ltd, India answers the questions and shares some documents: The MP absorber is the heart of the Saipem urea synthesis section. This is the vessel where pure excess ammonia is separated from ammonium carbamate. To operate at the right temperature is key for good performance of this absorber. The top temperature must be maintained at 42-43°C, while the bottom temperature is maintained at about

72-80°C. If the bottom temperature is below 70°C the ammonia will not evaporate and goes to the HP carbamate pump leading to cavitation risks. For the complete troubleshooting process refer to the UreaKnowHow.com website.

Muhammad Umar Munir from Pakarab Fertilizer, Pakistan provides more information and asks another question: The MP absorber (C-1) at our plant has a bottom temperature of ~82-84°C. The middle tray temperature is also high i.e. 60°C but the top tray temperatures are okay. I have requested the lab results and will share them with you but according to the sheet prepared by Prem Baboo, there is a probability of CO₂ carryover. The corrective action recommends checking the E-7 temperature. I carried out this check and it is running at 82°C (E-7 outlet).

My question is: What is the optimum temperature at the E-7 outlet? Also, is there any other reason that could contribute to this observation?

Prem Baboo from National Fertilizers Ltd, India replies to the question and shares a valuable document: Good question. The bottom temperature 82-84°C is not a problem – it is slightly higher than the design temperature of 75°C. It is beneficial because the higher the temperature the more ammonia is evaporated from the bottom to the top and the less recycle there is. The top temperature is OK. The 2nd tray temperature being higher is not a problem and is likely due to the fact that you have more water going to C-3 (inert washing column). This ammoniacal water is increasing the 2nd tray temperature.

The E-7 (MP condenser) process outlet design temperature is 78°C and the cooling water outlet design temperature is 66°C. E-7 is the critical equipment item, where control of sedimentation of the cooling water is important which is influenced by the opening of control valve TV 101. If the TV 101 opening is reduced there is a chance of sedimentation and scaling of phosphate/silica. In this case you must control the flow by opening TV 105.

Below are some points for your attention:

- Ensure sufficient cooling water flow in E-7, to avoid sedimentation.
- The cooling water outlet temperature of E-7 should not exceed 66°C as at higher temperatures scaling takes place.
- The inlet temperature of the cooling water to E-7 should be no less than 35°C to avoid crystallisation on the shell side (carbamate solution).

- The outlet temperature of E-7 (process) is optimum at about 78-80°C.
- The bottom temperature of C-1 should be 75-82°C.

Recently we carried out a modification to C-3 (inerts washing column): ammoniacal water was taken from E-8 (LP condenser). The following advantages were noticed:

- C-1 temperature control;
- reflux to C-1 reduced by 2 m³/hr. i.e. reduction of recycling flow;
- LP pressure control and the total cold water flow to C-4 (LP inerts washing column) is stopped;
- V-3 (L.P carbonate solution tank) level control;
- P-7 (inerts washing ammoniacal pump) now stopped the solution transfer by deferential pressure of MP and LP;
- power saving due to stopping the motor.

Muhammad Umar Munir from Pakarab Fertilizer, Pakistan provides more figures and information: That clarifies a lot. I'll come back once I get lab results, but in the meantime I would like to show you the operating conditions from this morning:

The tray temperatures are okay so I am not expecting any carry over and the higher middle temperature is probably due to the same phenomenon as the bottom one.

Temperature, °C	Design	Operating
Bottom	70-75	82.0
Middle	45-50	55.0
Tray 1	45-50	49.0
Tray 2	40-46	45.0
Tray 3	40-45	42.0
Vapour overhead line	40-45	47.0

These are the results of the MP absorber bottom effluent: NH₃ 44 wt-%, CO₂ 26.7 wt-%, H₂O 29.3 wt-%.

The tray temperatures corresponding to these results are: Bottom: 86°C; Tray 1: 54°C; Tray 2: 47°C, and other temperatures are the same as given by you.

Apparently, there is an increasing trend of C-1 bottom temperatures. Moreover, the water content seems to be on the high side with relatively less ammonia. This means there is minimal chances of P-2 cavitation but what impact does a higher water content have?

Prem Baboo from National Fertilizers Ltd, India shares more information: The higher percentage of water may be due to disturbances in the common section. If your top temperature of C-1 is maintained there is no problem at all. The main sources of water for the ammonium carbonate solution tank are:

- Common section (waste water section) reflux accumulator ammonia wt-% is low: design is 35 wt-% but in practice 32-35 wt-%.
- Your LP section pressure is low resulting in water carryover to V-3 (ammonium carbonate tank), the pressure/temperature equilibrium not matching.

- Distillation tower (C-2) feed check ammonia wt-%
- Check water balance of vacuum section (i.e. steam to booster ejector)
- Cold water to C-3/E-11 (inerts washing column) is increased.
- Check also upstream.

If you have sufficient margin in TV 101 then reduce the outlet temperature to 70°C. But the inlet temperature must be watched – design is 40°C but you reduced to 35°C.

Shahbaz Soomro from FFC, Pakistan joins the discussion: What about flushing of the C1 trays? Were there no problems of C1 trays choking after this modification?

Prem Baboo from National Fertilizers Ltd, India replies: After this modification the temperature of the 3rd tray (from top) came down drastically. High temperature favours the carryover of CO₂. To date, there is no problem of choking and this modification is recommended by Saipem. In India, two plants have been implemented with this scheme.

Manikanta Vema from Kribho Fertilizers, India asks for further information: Thank you for the information about your C3/E-11 modification. I would like to know what the overall effect is on the total steam as a result of this modification apart from the power saving of the P-7 motor?

Prem Baboo from National Fertilizers Ltd, India replies: The following advantages were observed:

- Water reduced in the process by 1.0-1.5 m³/hr.
- Reflux reduced in C-1 (MP absorber) by about 2.0-3.0 m³/hr.
- Overall (steam to distillation, steam to LPD, etc.) LS (low pressure steam 4.0 bar) reduced 2.0-3.0 t/hr. Also LS generation in E-5 (HP carbamate condenser) increased by 1+1 =2 t/hr.
- Waste water feed reduced by 1.5 to 3.0 m³/hr.
- KS (105 ata) reduced by 0.5 to 1.0 t/hr.

The above modification was only carried out in line-II Plant (expansion), line-I modification approval under progress.

Manikanta Vema from Kribho Fertilizers, India asks a new question: After this P-7 modification, have you faced any problems with C-1 tray temperatures, because the P-7 water helps to flush the trays to avoid partial choking?

Prem Baboo from National Fertilizers Ltd, India answers to the question: After P-7 (aqueous ammonia pump) stopped we did not experience any problems of trays choking. This ammoniacal water was taken from tray No 2 (from top). Due to absorption the temperatures of the 2nd, 3rd & 4th trays (from the top) were running higher, so the possibility of CO₂ carryover was greater than now. Due to the higher tray temperatures the reflux quantity was also higher.

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Plant Manager+

Problem No. 41 Leakage of a high pressure flange connection

Among the many problems that threaten the daily operation of a urea plant, fluid leakage is perhaps the most common, especially the leaking of high pressure flanges in the urea synthesis section. A high-velocity jet emerging from a flange due to a failed gasket, a pinhole in an elbow, corroded threads of a level transmitter, etc., are familiar scenes that the maintenance crew encounters and must rectify.

Isa Norozi from Khorasan Petrochemical Company, Iran initiates this roundtable discussion:

We are experiencing corrosion on the bolts of the high pressure flange in the HPCC liquid line to the urea reactor. 3 out of 12 bolts were corroded and we are

going to replace these with new ones. The corrosion happened via a small leakage from the flange inside the insulation sheet. The process pressure inside the pipe is 145 bar and HSE groups have not given permission to replace the bolts. The bolts are close together. Does anyone have any experience or recommendations for replacement of this type of bolt during normal operation?



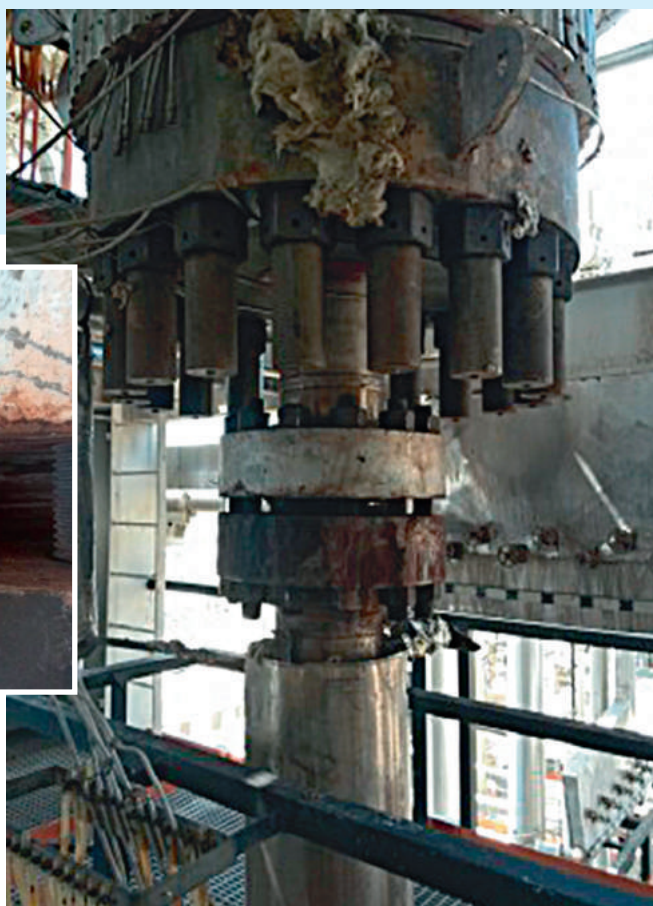
Mark Brouwer from UreaKnowHow.com, The Netherlands replies: There is only one course of action – stop the plant and replace the lens ring and bolts.

Prem Baboo from National Fertilizers Ltd, India offers his advice:

It is not possible to replace the corroded bolts while the plant is running, it is very risky. It may be possible to stop the leak by clamping and applying a cold weld. Mechanical problems in early satellites have sometimes been attributed to cold welding. Our stripper bottom manhole cover flange has had leakages on many occasions and they were stopped by applying cold weld material. Furmanite is the worldwide innovator and leader in comprehensive on-site and on-line plant and pipeline maintenance. The Furmanite seal has been invaluable in keeping our unit up and running, please don't take any risk.

Isa Norozi from Khorasan Petrochemical Company, Iran provides

more information: For your information, the very small leakage happened inside the insulation sheet but there is no longer any leakage from the flange. As you see in the pictures, one of the bolts was severely corroded and the others were slightly corroded. Can we continue in this way or do we need to replace them with new ones?



Mark Brouwer from UreaKnowHow.com, The Netherlands shares more experience on this topic:

The fact that the leak has stopped does not mean the problem has been solved! On the contrary, carbamate will be present in the crevices between the lens ring and flange and as oxygen will be depleted, active corrosion will start. Only time is needed to cause a potential deadly failure. Please note that active corrosion occurs in stainless steel materials at rates of > 50 mm/y. The application of cold clamps and/or Furmanite on carbamate leakages is very risky for the same reason. One can never ensure that there will be no crevices where carbamate can be present causing active corrosion to stainless steel parts. I repeat, there is only one course of action recommended: Stop the plant and repair the leak.

Yudhistira Perdana Putra from PT Pupuk Kaltim, Indonesia

shares his advice: The leakage may have stopped due to solidification of the urea carbamate solution. It may seem to have stopped but corrosion may still be occurring at the flange face of the HPCC. It is much too risky to keep the plant running.

Isa Norozi from Khorasan Petrochemical Company, Iran provides

more information: Now that the leakage has stopped we have

seen small bubbles of gas escaping from two points of the flange, but not continuously. The insulation of the flange is open and the leakage is under visual check by operators and inspection groups and hasn't shown any sign of change up to now.

Mauricio Medici from M&M-CT Company, Argentina also shares his experiences: I agree with Mark and other colleagues here, leakage of HP equipment or lines, are always under discussion. Nobody wants to shut down the plant, especially plant managers, but although the leak appears to have stopped, corrosion may continue, as you mention this gas leak indicates that crevice corrosion in the lens rings/flanges is occurring (oxygen depletion), on the other hand carbon steel bolts are in great danger, and it's difficult to check the back side or the inner area of nuts.

The recommendation of performing a shut down soon is the best option, to avoid injuries and major loss. Try to keep the area clean of solid carbamate. Never replace bolts while the plant is running!

M Hassan from a fertilizer plant in Pakistan provides more advice: Keep the area clean on a daily basis. Apply steam at the location of the leakage so that build of carbamate does not occur

and you are not blinded. Maybe try slightly tightening if the leakage rises may help.

Don't replace the bolts while the plant is running. The condition of the bolts does not look too bad from the pictures (maybe close up it is a different story).

Never go for cold box up on this service and pressure. Whoever did it was very lucky to avoid a disaster.

Isa Norozi from Khorasan Petrochemical Company, Iran provides an update: Thank you for all your recommendations. We had a short shut down because of a gas compressor failure in the ammonia plant. During this time we have changed the bolts. Only one bolt was corroded on its outer face.

Syed Ali Salman Bokhari from Pak-American Fertilizers, Pakistan also shares his opinion: I must say "Nip it in the bud". Although it may be a painful decision, it is better to stop the plant and take the corrective actions as per standard operating procedures. A little production loss is much better than huge man and equipment loss.

Khalid Khattak from FFC Goth Machhi, Pakistan also comments: Box up is to be avoided in HP equipment. Don't do it. ■

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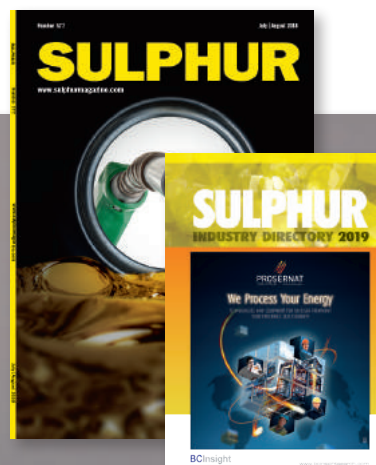
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Problem No. 42 Coated urea fertilizers

Coating urea prills (granules) with a water-insoluble, semi-permeable, or impermeable (with pores) material delays the release of nitrogen from the urea. Urea is highly soluble in water, but the solubility of coated urea is dependent on the coating material, its thickness, and the coverage and uniformity of the coating on the granule. As the urea is gradually released from the coated granule, it is exposed to

the same chemical and biological processes as non-coated urea. Coated urea provides a safer and more efficient form of urea by:

- safeguarding against nitrogen losses through volatilization and degradation;
- slowing down leaching due to its slow-release characteristics;
- improving stability in fertilizer blends.

Amirhossein Hadian from Khorasan Petrochemical company, Iran starts the roundtable discussion: I am researching coated urea fertilizers – controlled release fertilizers consisting of urea prills, coated with less-soluble chemicals such as sulphur, polymers, etc. Does anyone have any suggestions for coating urea prills to slow down dissolution rates when used on farms?

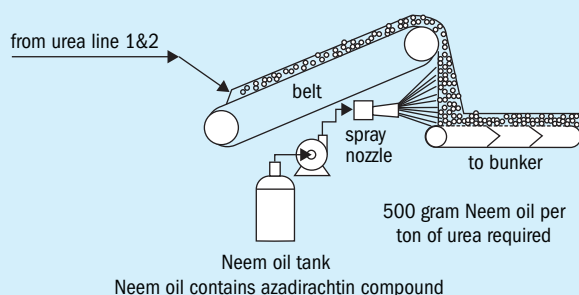
Luis Nogue from Bunge, Argentina shares his information: There is a company called NAQ Global who specialise in anti-caking and anti-dusting agents. They are present in Asia, Middle East, North America you can see more info on their website.

Prem Baboo from National Fertilizers Ltd, India provides more information: We are also producing neem (azadirachtin) coated urea. It is well known that neem has great potential for increasing the efficiency of urea. Indian agricultural scientists were the first in the world to establish neem as a nitrogen regulator. National Fertilizers Ltd., Vijapur started using it in October 2004.

Neem-coated urea has the following advantages:

- Neem cake when applied along with urea retards the nitrification rate for about two weeks and thus more nitrogen remains available as ammonium for a long period.
- 350 ppm of neem oil content is being maintained at NFL Vijapur as per the guidelines under section 20(a) FCO.
- Useful as a germicide to help crop decaying.
- Neem product namely neem oil, cake, neem bitters concentrate and neem oil were identified as good nitrification inhibitors.

Fig 1: Spraying arrangement in NFL Vijapur



Advantages:

1. Urea efficiency increased
2. Caking problem reduced
3. As a germicide
4. Crushing strength improved



PHOTO: PIXELFUSION3D /ISTOCKPHOTO.COM

Neem coated urea fertilizer has become very popular with Indian farmers.

Development of process for coating urea prills

NFL has carried out development work for the coating of urea in fertilizer plants, where plant conditions require a new approach to use neem cake or neem bitters for urea coating. The work was divided in two parts: (1) developing a suitable neem product which could easily be sprayed on urea prills in the final product stage and (2) development of a suitable arrangement for spraying neem product after leaving the prilling tower.

Amirhossein Hadian from Khorasan Petrochemical company, Iran asks for more information: We are interested in neem products. How much dust reduction can we expect by spraying neem oil? We currently spray Uresoft-150 after the urea fluidised bed, can we substitute it with neem oil?

Prem Baboo from National Fertilizers Ltd, India provides more information: Neem coating is being carried out in the final stage, i.e. before bunkering because neem oil has a bitter smell and it is

immediately loaded in bags. When farmers apply urea on the farm, the rats and rabbits vacate the field. It is also a germicide. Neem coating definitely reduces dust generation. You can switch from Uresoft-150 to neem. Crushing strength is also improved by neem e.g. before coating 650 gm/prill and after neem coated 670 gm/prill. You can apply neem after the urea fluidised bed.

We started using neem coating in 2004 as a trial run, nowadays there is increasing demand from farmers. At present, it represents about 40% of total production by National Fertilizers Ltd. Fertilizer companies, including Tata Chemicals & Fertilizers, Babrala and Indo Gulf Fertilisers, Jagdishpur also produce neem-coated urea.

Vinay Gandhi from GSFCL, India provides more information: GSFCL, Vadodara, Gujarat, India has also started production of neem coated urea prills. The response from farmers is overwhelming.

George Van Bommel from Energy Consultancy UREA, The Netherlands shares new information: There are at least 10 suppliers of various urea agents, which all claim various improvements of properties. The question is: Which property do you want to improve, and how much do you want to pay for it per ton of prill or granule...

Read chapter 16 of the Fertilizer Manual, then make your choice of the following products (suppliers) in no specific order (others may also be available):

- NovoFlow (Novochem)
- UreaSoft (Kao)
- UreFlo (Filtira)
- Galoryl (Armaz)
- AC Fert 100 (Kephass)
- Flotigam (Clariant)
- UltraCoat (SSCI)
- UreCoat (Neelam)
- Fertibon products
- FA1665 (Forbon)

Amirhossein Hadian from Khorasan Petrochemical company, Iran raises new questions: When neem oil is sprayed onto urea prills, do the coated prills have a special odour and does it cause problems for storage and workers in bagging?

Prem Baboo replies: Neem coated urea does have a bitter smell but it is not pungent, and poses no problem for any workers, nor harm to health. In India about 11 plants are producing neem coated urea and there have been no problems with loading and handling.

Mark Brouwer from UreaKnowHow.com, The Netherlands provides supplementary information: Neem also acts as an anti-caking agent. ■

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Problem No. 43 Increased dust content of urea granules

Dust formation in the granulator can adversely affect stream time. There are several reasons for increased dust in the fluid bed granulator. No formaldehyde or less formaldehyde injection, lower bed level in the granulator, lower concentration of

urea melt to the granulator, a higher pressure in atomisation (sprayer) air and a higher pressure in the urea melt header are all factors which can play a role. This roundtable discusses increased dust level and its causes.

Fateme Ahangarani from Razi Petrochemical Company, Iran starts the discussion: These days we have a problem in our urea granulation plant and the amount of product urea dust has increased. Which parameters are important for decreasing the dust content? We try to control the concentration of urea formaldehyde at about 0.5 wt-% but the problem still persists.

Naceem Kashif from SABIC, Saudi Arabia replies: Dust can increase due to the following factors:

- high ammonia content in product;
- higher moisture content;
- higher temperature;
- low strength.

Mark Brouwer from UreaKnowHow.com, The Netherlands asks some questions: Do you see an increase in the temperature of the product granules leaving the granulation section? If yes, from what temperature to what temperature? Has the inlet urea melt concentration changed?

Majid Mohammadian from PIDEMCO, Iran shares his ideas: If the amount of dust is greater than normal check for the following items:

- low content or poor quality of urea formaldehyde;
- low level in the beds;
- low concentration of urea melt feed.

Chandra Mohan from Nagarjuna Fertilizers and Chemicals Ltd, India offers some advice: You could try contacting GPIC, Kingdom of Bahrain for more details/advice. As far as I know, the GPIC granulation unit is the world's best running unit.

Abbas Nouraldean from Petrochemicals Industries Company, Kuwait makes some suggestions: If the dust increase is in the final product, you may need to check the air flow and levels in your first and second bed coolers. When the level in the final bed cooler is high and the air flow is reduced, dust will be carried over with the final product. You need to increase the air flow a little and decrease the bed level. If the dust is high in the granulator scrubber then you need to check the temperature and urea melt concentration as it might be high.

By the way, increasing the urea formaldehyde concentration to more than 0.45 wt-% will not help much in decreasing the dust formation.

Mark Brouwer from UreaKnowHow.com, The Netherlands asks for more information: When you talk about a dust problem, what do you mean? Is the fines (<1 mm) content in the product high? Are there any caking problems? Does the product show caking behaviour?

Mohammad Farooq from SABIC-SAFCO, Saudi Arabia shares his opinion on this subject: By dust we typically mean fines which are less than 1 mm in urea product. In my opinion, if the amount of fines (<1mm) increases to more than 1.5 wt-% of the product at the exit of the prilling tower and the product is to be stored there is an increased possibility of caking. Caking will start within a few days and the situation will get worse with the passage of time. However if the product is packed directly, the problem can be minimised.

Reza Keyhani from Kermanshah complex, Iran asks a new question: What is the effect of the granule temperature on this?

Jeremy Feser from Agrium, Canada asks for clarification: Are we talking about granulation or prilling?

Mark Brouwer from UreaKnowHow.com, The Netherlands replies and provides more information about drum granulation: We are talking here about fluid bed granulation. I believe you are talking about drum granulation. In a fluid bed granulation unit a high bed temperature leads to a softer product and thus more dust formation and a shorter run length.

Dusty urea product often leads to uncomfortable discussions between a urea producer and their clients. It also leads to caking and causes unpleasant and even hazardous working conditions for any employee working with such a product. Additionally, dusty urea can cause damage to crops, leading to reduced yields in the agricultural sector.

Dusty urea product is typically caused by operation parameters of the evaporation section in the urea melt plant, operation parameters of the granulation section, storage conditions and logistic procedures. In most of these cases an end-of-pipe solution is the most efficient and cost-effective solution to improve the product quality and produce premium-grade urea. The Van Bommel Dedusting Technology (VBDT) offers such a solution.

The VBDT was developed in 1992, and has been in continuous operation ever since at a modern fluid bed urea granulation plant in North America. The original VBDT design was a one-line unit with a design capacity of 200 t/h. It was capable of breaking up soft lumps with a diameter larger than 8 mm, taking out fines and broken product (90 µm-1.0 mm), and removing dust and ultra-fines (1 µm-90 µm). The design made it possible to guarantee an "on-spec" premium-grade urea granular product of between 1.0 mm and 4.0 mm at variable feed rates.

The line was expanded in 1994 to 500 t/h. The VBDT has also been implemented at various DAP & MAP plants and has the option to perform cooling of the product and refresh warehouse air to improve visibility during unloading and loading operations. ■

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Problem No. 44 Rise in HP CO₂ stripper outlet temperature

In the high-pressure CO₂ stripper of a urea plant, which is essentially a shell-and-tube heat exchanger, the incoming carbon dioxide feed flows counter-current to the urea solution leaving the reactor.

On the shell side, the high-pressure stripper is heated with steam. The off-gas of the high-pressure stripper, containing

the feed carbon dioxide along with additional carbon dioxide and ammonia from the dissociated carbamate, is then fed into the carbamate condenser or pool reactor. This round table discussion is about the high-pressure CO₂ stripper and how to solve the problem of a high temperature in the bottom of the stripper.

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India initiates this round table discussion:

In a CO₂ stripping process, what could the reason be for an increase of the stripper liquid outlet temperature from 176°C to 182°C?

The stripper off gas temperature has reduced from 186°C to 183°C. The stripper vent on the shell side is more open than normal so that inerts can be vented and heat transfer will be more efficient. There is no fluctuation in the stripper level or steam drum level.

Majid Mohammadian from OCI Company, The Netherlands replies and asks for some further information:

There are several reasons for high temperature in the stripper bottom: high level at the stripper bottom, poor CO₂ distribution, poor liquid distribution, too high steam pressure, poor reactor composition etc.

In your case when the delta temperature between the liquid inlet and liquid outlet is low, about 1°C, it indicates poor liquid distribution in the top of the stripper, resulting in poor stripping. If you check the lab results you will find poor stripper efficiency as well. Please provide the lab results of the stripper inlet and outlet.

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India provides more information:

When the stripper outlet temperature was 182°C, the reactor outlet composition was 30.5 wt-% ammonia and 24.9 wt-% CO₂ and the stripper outlet composition was 10.1 wt-% ammonia and 11.2 wt-% CO₂. Urea is 33.3 wt-% at the inlet of the stripper and 54.1 wt-% at the stripper outlet.

The level in the stripper bottom is measured by RADAR indicator and is controlled by a level control valve. The level control is found to be accurate. The steam drum pressure is maintained at the normal value of 18 kg/cm²g at a low load.

Majid Mohammadian from OCI Company, The Netherlands returns to the discussion:

The CO₂ percentage in the reactor outlet (24.9 wt-%) looks wrong, it is normally between 17 and 19 wt-%.

The stripper as per the lab data is working with 75% efficiency (design efficiency is 80%).

Poor liquid distribution at the top of stripper is the reason for high temperature at the bottom of the stripper (less delta T around the stripper) and consequently poor efficiency.

My advice is to stop the plant, open the stripper and check the liquid dividers, holding plates, Teflon rings and fix the problem.

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India asks further questions:

The stripper outlet temperature is found to be dropping day by day (it has dropped to 178°C which is almost back to the normal value) as the steam pressure of the stripper increases above 20 kg/cm². What is the reason for the normal bottom temperature after increasing the stripper pressure? Has anyone faced the same kind of problem? What actually happened inside the stripper?

Maksud Alam from KAFCO Company, Bangladesh asks for more information:

Could you please tell us about the following points:

1. Throughout the process of increasing steam pressure was the plant load the same?
2. Did you notice any change in the low-pressure section and process condensate treatment load before and after?
3. Do you have a condensate level control on the steam side (not the saturator level but the stripper shell side level)?

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India replies:

1. At a higher plant load the steam pressure used to be 19.20 kg/cm² but now for the same plant load the steam pressure is 20.30 kg/cm². If the steam pressure is dropped then the low-pressure system load increases.
2. Yes, the low-pressure system is increasing and more gas is venting from the low-pressure system to the scrubbing system.
3. There is no condensate level control on the stripper shell side. Level control is there only for the saturator.

Mark Brouwer from UreaKnowHow.com, The Netherlands provides more information:

I am also thinking that there may be issues with the liquid distribution:

1. Have you ever experienced fouling in the top of the HP stripper?
2. What is the age/condition of the liquid dividers?
3. Did these problems occur shortly after a turnaround?

Sandesh Patil from Mangalore Chemicals and Fertilizers Company replies to Mark's questions:

1. We have never experienced fouling in the new stripper.
2. The top ferrules were replaced last year and the stripper was replaced in 2006.

3. This problem occurred after starting the plant after a short shut-down of 6 days. The turnaround took place in May 2016 and after starting the plant in June 2016, the plant has been running smoothly for the last four months.

Maksud Alam from KAFCO, Bangladesh replies with more information and some words of caution:

As described before, poor liquid distribution leading to poor stripping efficiency may be the root cause of your problem, which you have overcome by increasing the steam pressure. But please bear in mind that some tubes may have a thicker layer of film and some may be thinner/dry. In either case the higher steam temperature will contribute to higher corrosion rates.

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India replies and raises new questions:

At present the stripper shell side pressure is maintained higher compared to the normal pressure at the full load. If there is a problem in the liquid distributor, how many days can the plant be kept in operation at this higher stripper pressure? Tubes whose liquid distributor is not working properly may be overheating and there will be higher corrosion in those tubes. (Stripper outlet analysis is carried out weekly for Ni, Cr and Fe and it is within the design limit). Is there any other way we can identify the higher corrosion rate in those tubes where the liquid distributor may not be working properly so that the plant can be shut down before the stripper tubes reach final breakdown?

Mark Brouwer from UreaKnowHow.com, The Netherlands asks a new question regarding the material of construction:

What is the material of the stripper tubes?

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India replies:

The stripper tubes are made of Safurex.

Bob Edmonson from a contracting company in Canada gives his point of view:

I agree with the others who have concerns regarding the liquid distribution. Your plant is quite new so it's hard to imagine why, but if one or more bolts that secure the hold down plates are loose, due to corrosion, a plate can lift and the liquid divider tubes can rise up somewhat. If that happens, you will see stains on both sides of the Teflon rings when you are in the stripper next time. The risk is that these tubes can (will) corrode more rapidly if 25-22-2 so it might be worth checking the ppb nickel in the stripper outlet liquor if this happens again.

Ali Ancaza from Igsas, Turkey raises new questions:

- How long has the stripper steam pressure been like that?
- How is the level in the stripper steam drum?
- How long has the stripper steam temperature been like that?

Mark Brouwer from UreaKnowHow.com, The Netherlands shares his experience:

Although with Safurex tubes there is no risk of active corrosion, it makes sense to check the liquid distributor system as soon as possible.

Ali Ancaza from Igsas, Turkey shares experience from their plant:

We have experienced the same problem in our system in 2002. In order to help you, I need to know the following values:

1. What is the CO₂ input temperature to stripper?
2. What is the output temperature of stripper urea?
3. What is stripper vapour pressure and steam temperature?
4. What is the steam drum level of the stripper?
5. What is the low pressure recirculation pressure?

Salam Malih from North Fertilizer Company, Iraq offer his advice:

1. Check the CO₂ content in the stripper outlet and check the performance of the level measurement of the stripper as there is the possibility of CO₂ slippage.
2. Check the ratio of O₂ in CO₂.
3. Check the HP steam consumption of the stripper.
4. Check the synthesis pressure.

Sandesh Patil from Mangalore Chemicals and Fertilizers Company, India answers Ali's questions:

1. CO₂ input temperature is maintained at 135°C.
2. Stripper urea outlet temperature is 179°C.
3. Steam temperature of shell side of the stripper is 215°C and the steam drum pressure is maintained at 20.30kg/cm²g (which vapour pressure do you need?)
4. Level in the steam drum is maintained at 45%.
5. Which recirculation pressure do you need – the carbamate recirculation?

... and responds to Salam's questions as follows:

1. At the stripper outlet the CO₂ is 10.82 wt-% which is close to the design value of 10.50 wt-%.
2. Oxygen is maintained at 0.55 vol-%. Oxygen analysis at the outlet of the H₂ converter is carried out daily.
3. HP steam consumption for stripper for full load is normal (almost 44-46 t/h)
4. Synthesis pressure is maintained at 144.5 kg/cm²g.

Ali Ancaza from Igsas company, Turkey provides some suggestions to reduce the stripper liquid outlet temperature:

Gradually increase the stripper steam level of the drum up to 55-60%. According to the recirculation pressure, increase the pressure of the drum by 21.5-22 kg/cm². The outlet temperature of the stripper solution will decrease.

Kashif Naseem from SABIC, Saudi Arabia shares his experience:

Check your stripper liquid distribution system which is the main cause for it.

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.

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Problem No. 45 Effect on reactor liner in case of reactor hold up

In the case of a failure in a urea plant, when the problem is expected to be solved within a short period, the high pressure synthesis section is typically blocked in (referred to in this article as reactor hold up). All input and output streams are closed and the synthesis section remains partly filled with a solution consisting of ammonia, CO₂, water, ammonium carba-

mate and of course urea. Several risks can occur during this blocked in situation: 1) as the temperature slowly reduces crystallisation could occur; 2) active corrosion could start as no fresh oxygen is supplied to the synthesis during blocking in and; 3) an explosive gas mixture could occur as hydrogen and oxygen are present.



Sandeep Gochar of GNFC in India initiates a discussion on this topic: I am working in a Saipem technology urea plant. I would like to know what effect keeping the reactor in hold up conditions after a shutdown will have on the reactor liner. Usually Saipem recommends draining the reactor if the shutdown is longer than 48 hours.

Prem Baboo of NFL India replies: Generally, the new practice in our plant is to have reactor hold up solution for no more than 48 hours. The reactor must be drained within 48 hours. Some corrosion was observed due to the low CO₂ purity (98.0 vol-% or less). Secondly the ammonia feed is not pure; it has some oil contents including some sulphur. Chlorine is also present in the passivation air. Our practice is as follows:

- feed liquid ammonia every 15 hours;
- drain the reactor within 48 hours;
- ensure a proper NH₃/CO₂ ratio;
- ensure purity of CO₂ (reduce hydrogen and organic matter slipping from the CO₂ removal section);
- during start-up introduce NH₃ feed 10-15 min prior to CO₂ feed.

Sandeep asks for some clarifications: Feeding liquid ammonia every 15 hours is something new for us but we will definitely look into it. Suppose we have reactor hold up for 48 hours, how does it affect the reactor liner thickness? One more question, which area (bottom, middle, top) of the reactor liner is more prone to thinning in the case of hold up or is it affect uniform throughout the reactor?

Prem replies to Sandeep: The top part of the reactor lining is more prone to thinning. If you feed ammonia then the reduction in thickness at the bottom of the reactor liner is always less than at the top. The solution must be drained within 48 hours. Do not exceed 48 hours. If you have a planned shutdown, first stop the CO₂ feed and then after 8-10 minutes stop the ammonia feed.

Mark Brouwer of UreaKnowHow.com in the Netherlands asks Prem some additional questions: What is the best practice during blocking in of a Saipem synthesis with regard to the following:

- Position of MP vent valve (to avoid accumulation of hydrogen);
- Steam pressure on stripper;
- Do you monitor the temperature in the reactor to assure no CO₂ leaks in?
- When you add ammonia every 15 hours, do you reduce the level in the HP level tank? As the ammonia enters the reactor via the HP ejector, is there a risk that the HP level tank will be empty?
- How much time should expire after start up before another blocking in of 48 hours is allowed?

Prem replies to Mark:

- Whenever there is a plant shutdown the MP vent is in auto mode, the excess pressure caused by inerts (H₂/CH₄/N₂/O₂/Ar and some ammonia, etc.) is released and then the valve closes automatically, i.e. during shutdown the MP vent valve is in closed condition (after the hydrogen and all inerts have been vented out).

- During shutdown the steam pressure is zero on the stripper shell side.
- Yes, the temperature of the reactor must be more than 126°C and is generally run at about 140-165°C.
- Yes, the level in the HP level tank is reduced. If there is no ammonia left, take it from ammonia storage. If it is unavailable for any reason (shutdown of equipment etc.) it is not a problem. We try to drain the reactor within 40 hours for equipment health. There is no harm in draining out the HP loop solution, the effluent (urea, ammonia and CO₂) will be recovered after shutdown. Draining means no effluent will drain out into the open. It should be collected in vessels (ammonium carbonate, urea solution tank etc.). These tanks are at atmospheric pressure and low temperature so there is less corrosion. Equipment health is always better than production loss.
- Another blocking in of 48 hours is permitted once the reactor is receiving feed and all of the solution has been replaced with fresh solution and with passivation air.

Mark asks for some more information: Have you ever experienced passing of the CO₂ valves resulting in CO₂ entering the reactor during blocking in? This would lead to higher temperatures and possibly active corrosion.

What is the reason for adding some ammonia every 15 hours? Does it reduce the risk of corrosion or crystallisation?

Prem replies: As regards CO₂ leakage, there are two feed valves: one is a motor operated angle valve (MOV) and the other is a pneumatic valve also angle (needle valve), so there is less chance of leakage.

When the feed is stopped, stop the CO₂ compressor immediately and there will be no risk of leakage due to the compressor tripping.

If the compressor is operating, the discharge pressure may be reduced to less than the system pressure (reactor pressure) so that there is no risk of leaking.

It is important that the operator stops the CO₂ feed first and then the ammonia to prevent CO₂ leaks.

Ammonia addition reduces the risks of both corrosion and crystallisation.

Sandeep asks another question: What is the logic behind the reactor temperature not being allowed to go below 126°C.

Mark replies to Sandeep: Two reasons are given:

- risk of crystallisation;
- to reduce the temperature shock when the plant is started up again.

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Problem No. 46 CO₂ cooler failure

CO₂ coolers in the inter stages of CO₂ compressors typically cool down the CO₂ stream by means of cooling water. It is preferable for the cooling water through the tubes to have a minimum velocity to avoid fouling and corrosion issues. This is not a problem after the first and second stage when CO₂ pressures are still relatively low. However, after the third stage the CO₂ pressure is about 80 bar, too high (meaning too expensive) to have the CO₂ on the shell side. Vendors specify coolers with the cooling water

on the shell side and the CO₂ through the tubes. This however introduces several failure mechanisms: As cooling water velocities cannot be guaranteed to be above a minimum value in the complete shell, fouling can occur. Also, chlorides accumulate (even when chloride levels are relatively low) and cause chloride stress corrosion cracking in austenitic stainless-steel tubes. In this type of cooler choosing duplex as the material of construction for the tubes is the best choice for a reliable heat exchanger.

Irfan Rashid, working in Operations at FFBL in Pakistan starts the discussion with the following question: Does anyone have experience of CO₂ cooler failure where failure occurs after the third stage of the CO₂ compressor?

Mark Brouwer of UreaKnowHow.com in the Netherlands replies:

Please share your experiences with us as I think it will trigger more discussions.

Irfan continues: We have a Dresser Rand design CO₂ compressor which takes CO₂ at 0.34 kg/cm²g and compresses it to 145 kg/cm²g in four steps. We have a CO₂ cooler after each compression stage. Recently we have been experiencing leakage problems in our CO₂ cooler after the third stage. It receives CO₂ at 76 kg/cm²g. Due to this problem, we replaced this exchanger but the new exchanger collapsed in a hydrotest, after just two years of service. I would like to know what types of problems can cause this type of failure.

Mark comes back with a question: Which materials of construction were applied?

Irfan replies: The tubes are stainless steel and the shell is carbon steel. In the old cooler the baffles were made of carbon steel but in the new cooler we specified stainless steel for the baffles to avoid any galvanic corrosion effect.

Ramchandra Nesari, a freelance consultant in India shares his valuable experiences: I have had experience of CO₂ cooler failures in three urea plants. We had frequent failure of the third stage cooler in one stream but in the other two the failures used to occur in second stage cooler.

You have provided some details about your CO₂ cooler in the third stage. You mentioned that the tubes are of SS 304 whereas the shell and baffles supporting the tubes were of CS initially. You then replaced the baffles with SS material to avoid galvanic corrosion effect. By doing this, you have removed one of the major causes of tube failures. We had similar observations in our CO₂ cooler failures. After replacing the baffle material with SS 304, we overcame the failures. So I would like to know whether you are still facing tube failures after replacing the baffle material. Please also provide details about the tube arrangement – whether it is a straight tube or U-tube bundle. Often, vibrations occur in U-tube bundles if the cooling water flow is above a certain limit.

Normally in the third stage cooler a temperature control valve is provided at the discharge of cooling water flow to limit the CO₂ temperature above a critical temperature. So, if the failure is due to vibration problems, then the design of the baffle spacing needs to be looked into.

Koorosh Lieravizadeh from the process engineering department of Shiraz Petrochemical Complex in Iran shares his valuable experiences:

We have also experienced some failures, not only of the tubes, but also on the shell of the third stage cooler of the CO₂ compressor. The cooler type is a fixed tube sheet.

Faraham Jafarvand of the Engineering department of NEWJCM Turbomachinery manufacturing co. in China provides his expert opinion:

Everybody may have their own ideas about the reasons for failure, but I think the following information is necessary to investigate the root cause of failure:

- Type of heat exchanger (TEMA class).
- Location of failure in the tubes: it would be very useful if you could provide a photo of the failed tubes as the crack direction and other features may indicate the type of failure. If a photo is not available please describe the crack feature according to your own observations (direction of crack, location of the cracks in the tube with respect to tube sheet or baffles or U bend in case of a U-tube, uniformity of failure in different tubes, surface morphology of failed parts, buckling of tubes if any...)
- Type of temperature control you are using to control the CO₂ temperature: The CO₂ condition in third stage cooler outlet is near the critical point and there is a probability of formation of CO₂ condensate if the temperature falls below a certain level. So please provide some info about the temperature control method you are using.

Irfan provides further information: We carried out a detailed investigation of this failure and the probable cause is strain induced intergranular corrosion. Our exchanger is a U-tube type, and baffle design/spacing is not a problem.

Harun Idrees, Technical Services of Fatima Energy Limited in Pakistan shares his experiences: At our plant, we have a brand new third stage intercooler with duplex tubes and after in 1.25 years of operation we have not faced any problems. ■

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Problem No. 47 Active corrosion during blocking in

This discussion is about the risks of active corrosion or failure of passivation during a blocking-in situation of the high pressure synthesis section of the urea plant. It touches on many aspects searching for a possible cause of active corrosion during such a situation. The final outcome is of significant importance for all urea plants worldwide.

Cory Holt, process engineer at CF Industries in Canada, initiates the discussion: Could anyone share their expertise with respect to urea process passivation and the possible modes of failure and has anyone ever worked at a plant where passivation has failed. Our facility has recently shown physical signs of passivation failure in our reactor although all process data shows that we had oxygen in the system and proper passivation. We recently have come out of a scheduled shutdown and upon starting up we had a higher than normal N/C ratio as well as periods where we sat full in our HP loop while maintenance worked on some start-up issues.

Mark Brouwer of UreaKnowHow.com, the Netherlands, asks for some further clarification: Did you block in the synthesis while solving the start-up issues? If so, for how long and how many times and what was the interval between the block-ins?

Cory replies: There were a couple of issues and we attempted to start up three or four times. The longest period was a 22 hour period at which we know our N/C ratio was greater than 4.

For the first start-up we were nine hours into start-up when we went down for six hours. Then we attempted the second start-up over a three hour period and went down for five hours. The third start-up was over a two hour period and then we went down for 22 hours. We attempted the fourth start-up over a 22 hour period when we were again forced to go down due to our leak detection system ringing in exposing a leak. When we shut down to look at the leak we realised our vessel had been stripped of its regular iron oxide coating due to passivation. Upon vessel entry we realised we had many more pinholes and advised that they were a result of active corrosion at accelerated rates. This tends to point a finger at loss of passivation but our DCS trends show that we had oxygen in our system for the duration of the start-ups.

We are wondering if our high N/C ratio had something to do with losing passivation in the reactor or if it was consumed by a contaminant, perhaps something from turnaround cleanings.

Mark provides his experience: I've never heard that a high N/C ratio causes active corrosion. On the contrary, Saipem states a high N/C ratio reduces corrosion rates.

Blocking in is always a situation where hot carbamate solution is in a vessel and no new oxygen can enter. The existing oxygen will be consumed by the normal passive corrosion and a chromium oxide layer will form as a passive layer. When the blocking in time is too long the amount of oxygen becomes insufficient and active corrosion starts.

Stamicarbon and Saipem claim that blocking in times should be limited to maximum 72 hours.

Your remark of a contaminant consuming oxygen is interesting,

although I have never heard of that phenomenon.

Does anybody else have similar experiences of blocking in right after start up and seeing active corrosion?

To see many new pinholes after such a short time seems strange to me. One should realise that a pinhole itself is anyhow an area of low oxygen refreshment even if the bulk liquid has sufficient oxygen. That its growth is accelerated so much seems odd.

Further stop-start and start-stop situations cause big stresses on the liner due to temperature differences between the liner and the carbon steel wall and weaker areas could create a leak.

Cory responds: Thank you for your expertise, it does allow us to eliminate our N/C ratio concern. The active corrosion is odd to us too but we had the reactor inspected and did some repairs during the turnaround which would not have missed these pin holes. We have found between 30 to 50 significant pin holes in just the bottom head alone and are inspecting the remaining zones now.

Muhammad Farooq of SABIC-SAFCO in Saudi Arabia asks some further questions:

- What is the material of the liner of your urea reactor and how long has it been in operation?
- What is the inspection frequency period?
- What procedure did you apply for the repair of previous pinholes and is it your practice that defects are removed first time? Did you monitor the liner thickness ever year?
- What is your feeding sequence and ammonia feeding temperature at start up?
- Did you analyse the temperature profile in a recent start-up of the urea reactor, especially at the reactor bottom?

Cory answers Muhammad's questions:

- The lining material is 316-SS and when we have made repairs we use 25-22-2 SS as a weld material after grinding out the cavity. This liner is approx. 35 years old and the reactor was scheduled for re-lining in 2015.
- We inspect on every scheduled maintenance outage which is every three to four years but due to other maintenance issues this has been every two years lately.
- Repairs are always done as explained in my first point. As mentioned in the previous discussion, we were just coming out of a turnaround where minor repairs were done and we had Stamicarbon do a pre-start-up inspection. They are currently doing our inspections now as well.
- We usually warm the HP loop by feeding CO₂ and steam (CO₂ having oxygen from air blowers @ 0.6 vol-%) until we achieve 270°F (132°C) at the top of the reactor. Then we feed in ammonia.

- Yes, we looked at the temperature profile and actually realised we achieved the fastest heat up we have ever seen. Usually the heat up takes three to four hours but we almost achieved 270°F (132°C) within two hours. We are currently trying to prove whether the instrumentation was working properly as we cannot explain the accelerated temperature increase.

We are currently looking at our DCS data to see if during our start-up attempts and block-ins we mismanaged the pulling of feed sequence or if somehow our instrumentation failed to show us loss of actual air. Initial data pulled has shown we were adding the right amount of air to our CO₂.

A question arising from this is whether circulating ammonia through the HP loop without CO₂ (which means no oxygen) may have somehow disrupted the passivation layer?

Muhammd Kashif Naseem of SABIC in Saudi Arabia shares his experience: I think your heating rate is very high. Keep it around 30°C/hour and use fresh air instead of a mixture CO₂/air for heating time passivation. I think the CO₂ mixture during heating with steam as water is creating the corrosion issues.

Both materials SS316-L UG and 25-22-2 SS are OK. Your licensor can advise you further with regard to materials.

We also operate a plant that is 30 years old. Every two years we inspect the plant and replace the liner material in the areas with the highest corrosion rates (vapour section). The original material was SS316-L UG and the top portion has been relined with 25-22-2 SS.

Cory comes back: I agree that the heat up rate is too high. We usually stay at around 30 to 50°F for the heat up. With our process it is impossible to have the heat up rate that we had with just steam or CO₂ and we are not sure why it got as high as it did. We have our vendor involved to find out how we reached such a high heat up rate (almost 107°F/hour). We looked at our instrumentation to see if it was functioning fine and it looks like it was.

Not sure if the heat up rate has anything to do with active corrosion. All of our process data shows we had passivation (oxygen). We are wondering if we had a contaminant that gave us an exothermic reaction that affected the passivation and give us a lot of heat?

Mark shares his experience and asks some further questions:

Heat can be generated via condensation of steam and by the formation of carbamate from NH₃ and CO₂. What are the pressures where you see a high temperature increase? Do you see a temperature increase at all thermowells or only certain ones? I do not see that circulating ammonia without CO₂ would cause problems with the passive layer. Is there any chance that chlorides or sulphur could have entered the synthesis? These components cause problems to the passivation layer.

Cory replies: When the temperature took off, the loop pressure was roughly 1.4 bar (80°F/24°C) and increased to approx. 4 bar (240°F/115°C) within 1.5 hours. We have seen the temperature increase on all four thermowells in the reactor as well as the overhead gas line. The CO₂ flow was around 1,130 kg/h during this time. So far we have not found any indication of chloride or sulphur contamination.

Mark continues the discussion: Do you have graphs of the various temperatures versus time?

Passivation of stainless steels can be hindered by heat tints (result of welding) or by fouling caused by corrosion product (iron

and chromium oxides) or metal parts (grinding).

Once active corrosion of carbamate starts it cannot be stopped and one needs to stop the plant, drain and re-passivate the surface.

Is it possible that heat tints and/or fouling is playing a role?

Essa Norozipour of Khorasan Petrochemical Company in Iran shares his experience: All equipment in the synthesis section requires at least two hours of oxygen injection for passivation. If the plant is started and then stopped before two hours of passivation has been carried out, the synthesis cannot be blocked in and must be drained completely and re-passivated. Did you check the interval of the start-up, shutdown and block in of your plant?

Cory replies: We do have trends, I have been trending every reliant DCS tag looking for discrepancies.

It may be possible that we have had grinding or welding contaminants as we did do some repairs during the scheduled turnaround before these start-up events. This has been discussed and we are looking into it. What baffles us still is the significant heat up rate.

We have looked at the time intervals and discussed them with our vendor and everything looks like it was in place and confirmed with our DCS data showing we had oxygen.

Mark responds: It is not easy to control the temperature rise at the beginning. I believe this occurs in other plants where the temperature increase is higher than the recommended 30°C/h.

Muhammad Farooq shares his experience: I also agree that the temperature rise during start-up when the pressure is 20 bar and temperature is more than 100°C (after passivation and heating) increases quickly especially during the first hour. However it is normal start-up behaviour in many plants.

My other observations are that the reactor liner is very old and needs early replacement.

The issue of passivation and heating of the synthesis loop needs to be sorted out with the vendor's help. It is not clear what the impact is on other equipment such as the stripper, carbamate condenser and scrubber.

The inspection frequency needs to be increased to once per year.

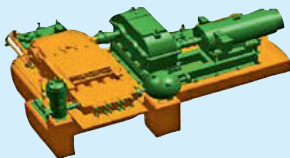
Note: This above case was presented during the licensor's conference and the lessons learned are:

1. Any time the urea plant goes down and the loop is not drained, monitor the vessel temperatures for any heat increases. If the temperatures increase by more than 20°F, the synthesis loop must be drained immediately.
2. Ensure positive isolation of CO₂ when the loop is blocked in by monitoring the loop pressure. If the loop pressure starts increasing, and the temperature in the vessel remains stable, operators can investigate according to their own judgment. If the vessel temperatures also start increasing, the loop must be drained down as above.
3. Ensure operators close the isolation valve downstream of HIC-1201 (CO₂ inlet to reactor).
4. Modify the urea plant start-up procedure to include lab analysis of the stripper bottoms for metals found in the stainless steel liner, specifically Fe, Ni, and Cr. The first sample must be taken within 12 hours of start-up and the second 24 hours later. If the values are stable or decreasing, no action is required. If the values are increasing, the plant will be shut down and drained. ■

Plant Manager+

Problem No. 48 Seal systems of high pressure reciprocating pumps

There is a lot of misunderstanding about the purpose and functioning of the seal systems (stuffing box) of the high pressure (HP) ammonia and HP carbamate reciprocating pumps. Some believe that lubrication is the main purpose, while others feel it is all about sealing. Both views are partly true. And for the HP ammonia pumps the



Pump unit KD 827.

objective is also to maintain the possibility to retighten the packing when ammonia starts to leak through the stuffing box. The stuffing box consists of the plunger, the casing, the high and low pressure packing assemblies, the glands and the seal system, either oil or water. This round table discussion is all about the experiences of various end users.

Mr Mien Pin Chin from Asean Bintulu Fertilizer in Malaysia

initiates the discussion: Can anyone tell me why our Uraca HP reciprocating carbamate pump requires two seal-water injection points for the plungers – one nearest to the plunger, which is supplied by a centrifugal pump, and another injection point nearest to the discharge, where the seal water is injected by an attached injection pump (positive displacement (PD) type)? Why can't we just use the seal water from the PD injection pump? Why add another injection point supplied by a centrifugal pump? As for the NH_3 pump plunger seal oil, why can't we use water? Will it cause corrosion problems in the discharge piping?

Mr Mahmood Rauf Zafar from Fauji Fertilizer Company in Pakistan

has a similar question and explains his issues: I would also like to change the HP ammonia pumps plunger sealing to water instead of seal oil for the following reasons:

- The seal oil recovery system is large i.e. three oil tanks, one centrifugal pump, two seal oil return lines and one supply line.
- The recovery of ammonia losses from seal oil is difficult – ammonia is being vented locally. Land contamination by oil drained from vessels during plant shutdowns and during vessel handover is difficult.
- Poor performance of heat exchangers due to oil contamination. Would anyone like to share their problems with seal water or any available literature? Which is more cost effective, oil or water?

Mr Muhammad Farooq from SABIC-Safco in Saudi Arabia replies:

It needs to be considered on a case by case basis as there are questions around both carbamate pump water seal systems and ammonia pump oil seal systems:

HP ammonia pump: Many pump vendors use a water seal system for their ammonia pump (PD). Why? Will it cause corrosion? Is it due to the quality of the water or something else? The ammonia pump oil seal system can be changed to a water seal system. It is economical and ammonia can be recovered. I would be interested to hear more about Mahmood's experiences as we don't have any ammonia losses or oil contamination issues.

HP carbamate pump: Sealing from the PD pump on the discharge line may be to counter the change of discharge pressure as the centrifugal pump cannot meet the pressure requirements because the system has a PD Uraca pump, whereas the centrifugal pump is only used to counter carbamate leakage from the plunger sealing and the pressure requirement is less as it is only a seal leak.

Mr Girish Prakash of Yara (former Tata Chemicals Ltd.) in India

shares his experiences: Water sealing on ammonia pumps is now a standard feature. It has plus points in that it eliminates expensive lube oil consumption and problems of occasional oil carry-over in the process leading to faulty level gauge indications in the medium pressure absorber. Sometimes even product quality may be affected due to this. The negative aspect is a slight increase in the waste water section load due to additional ammonia water from the plungers. Periodic tightening of the plunger packing plays an important role in keeping the ammonia content in the outlet condensate under control. Only steam condensate/boiler feed water should be used for plunger sealing/flushing.

Mahmood has another question: Please share the boiler feed water flow requirement and temperature. We have Uraca ammonia pumps.

Mien also comes back with more information: For the ammonia pump, the idea of changing from seal oil to seal water is not readily accepted by some people and therefore I hope to get more clarification. My questions are:

- When you mention that water sealing is a standard feature on ammonia pumps does that mean new pumps have water sealing or were you able to modify the existing pumps by changing their seal system from oil to water?
- Are there any differences in the material for construction for pumps using seal oil compared to those using seal water?
- For the seal water, does demin water have to be used? Our desorption system is already overloaded due to a number of revamps and the idea was to use ammonia water from the ammonia water tank so that no extra water is introduced into the system.
- For those using seal water for the ammonia pump, is there any effect on the urea conversion at the reactor? (The point made by other engineers is that the more water that goes to the NH_3 water tank, the more water will be sent back to the synthesis, reducing conversion).
- Were you able to handle the extra ammonia water without pumping more to the synthesis via the carbamate pump?

Girish replies: The boiler feed water flow requirement is roughly around 0.11-0.15 $\text{m}^3/\text{h}/\text{plunger}$. So if you have three plungers then the total requirement is around 0.45 m^3/pump . The temperature should be around 40°C. You can modify the existing oil seal pump to a water seal pump. You should preferably use clean and cold condensate for sealing purposes.

Mr Mark Brouwer UreaKnowHow.com in the Netherlands shares his experiences: Most important is to use solid-free water. If it contains some ammonia it is not a problem. It is therefore better to have stainless steel lines for the water supply. The stuffing box also needs to be changed when switching from oil to water seal.

Muhammad shares his experiences:

HP carbamate pump: Judging from your information it means that your PD injection and centrifugal pump are both in service when the carbamate pump is in operation? That means that the cylinder arrangement is such that it requires sealing at both ends as one pump may not be sufficient to lubricate (water seal) the complete cylinder. Moreover, most probably there are outlets at both ends. If you want to keep one pump in service then you might need to modify the cylinder sealing arrangement (a mechanical expert can give a better opinion).

HP ammonia pump: For the HP ammonia pump many agree that a water seal is a better solution than an oil seal. We are also planning to change one of our PD ammonia pumps from oil to water seal in the near future. The water quality (steam condensate) is important. Most vendors adopted water seals in the 1990s and initially they had oil sealing. As far as material of construction is concerned we need to contact the vendor. We believe, however, it requires only stuffing box/lantern ring modifications. The provision of a filter may also be considered for seal water injection applications. The leakage rate is minimal having possibly a very minor impact on the desorption section. And since water is removed from the desorption section as process condensate, in my opinion, it is not going to affect the reactor conversion.

Mahmood provides his experiences: In my opinion, ammonia pumps with water sealing have the following advantages over oil sealing: No recycling arrangement is required (oil passing into the process cannot be eliminated), whenever you open exchangers they will be clean, no environmental problem of vessels draining, no inventory problem with water, and finally improved exchanger capacity especially in the process condensate treatment section. In my experience reducing the number of equipment items and machines is helpful for both plant operation and maintenance.

Mien replies: As regards the effect on urea conversion, the argument made was that currently we are using oil sealing so only oil gets entrained in the HP stripper, HPCC and finally the reactor. If we switch to water, water will be entrained in the synthesis instead so we will have more water in the mixture and it will reduce the urea conversion. Will this actually happen? What urea conversion is achieved in plants that are using water as their sealing water? Is there any reduction?

Mark explains the difference between water seals for ammonia and for carbamate pumps: Please be aware that there are two types of water systems. For reciprocating HP NH_3 pumps one applies a water seal system: water acts as a seal between packing rings on the process side (high pressure side) and packing rings on the atmospheric side of the plunger (low pressure side). Any water entering the process side is caused by leakage through the high pressure side packing rings, which is a minimal amount and does not visibly influence the conversion figures in the reactor. For reciprocating HP carbamate pumps a water flush system is applied: here water needs to flush along the spindle between the high pressure side and the low pressure side in order to wash

away carbamate crystals. Here one can expect more but still a limited amount of water entering the process side.

Girish replies: I fully agree with you, however, in case of the ammonia feed pump, the contaminated seal water quantity theoretically is close to $0.5 \text{ m}^3/\text{pump}$ (for 3-plunger pump) but in practice (for higher packing life) it is close to $0.75\text{--}0.8 \text{ m}^3/\text{pump}$ and often compromises are also made for higher ammonia content in the seal water outlet. All water collected is processed in the waste water section and increases the ammonia load in the top section of the column. As a result, very marginal increase in process water recycle to the synthesis section is observed. Its impact on conversion is very small and cannot be measured for all practical purposes.

Mr Joseph Geronimo of Chemac Inc. in United States replies to Mien's original questions: Carbamate as we know is a difficult medium being highly corrosive and quite abrasive. As such in order to prolong packing and plunger life, Uraca has incorporated a timed flush injection in their carbamate pumps. That means the small PD pump coupled directly to the main pump crankshaft must be installed so injection occurs when the main pumps plungers are in suction stroke. When timed correctly the small PD pump injects a small amount of condensate directly on the plunger while it is retracting to BDC (bottom dead center). The small PD pump must only overcome suction pressure to inject condensate which will flush clean the plunger and also prevent carbamate from contacting the packing. This design works and packing life should be one year or more depending on how well you maintain your pump. Regarding seal oil for low pressure packing, this is an old design however many customers prefer this due to the fact that the packing will last longer sometimes up to two years or more. The seal oil must be metered such that you only overcome the suction pressure so oil can enter the chamber. Adding too much oil is a waste. If you want to eliminate the oil flush you will need new stuffing boxes designed for condensate only.

Mr Janusz Maćkowski from ZCh Police in Poland joins the discussion: We would like to change from an oil seal system to a water seal system for reciprocating HP NH_3 pumps. Which European companies are able to make such modifications and where has this already been done?

Muhammad replies: You can check with Peroni, an Italian company and they can also provide you with references for these modifications.

Janusz asks for some more information: How can we check if lubricating oil for rotating equipment (especially HP ammonia pumps) may be used in urea plants? What is the correct specification (requirements) for such lubricating oils?

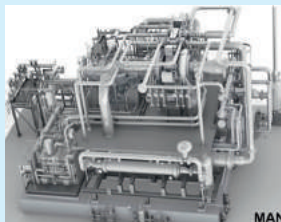
Mark replies: In the past Stamicarbon had a specification for the oil quality requirements for use in urea plants. What is important here is the residue content of the oil when heated (simulation of stripper conditions). Have you had oil fouling problems? Optimised designs have been developed to avoid oil fouling in the top of the HP stripper, radar level measurements and in control valves (stuffing box).

Janusz responds: We have found oil fouling on the top of the stripper in the area of the liquid divider ferrules. I am wondering whether this contamination could have come from the CO_2 compressor by accident. ■

Plant Manager+

Problem No. 49 Reverse rotation of CO₂ compressor

The CO₂ compressor of the urea plant is an expensive piece of critical rotating equipment installed without any spare position. Its reliability is therefore of prime importance. Sometimes reverse rotation occurs which can damage the internals of the compressor. What are the causes and what are the remedies to avoid reverse rotation of the CO₂ compressor? There is a lot of misunderstanding around this subject. Here we share the experiences of various end users.



Bhawna Dangi of NFL Vijaipur in India starts the round table discussion: What harm can reverse rotation of the CO₂ compressor do and how can it be prevented?

Niranjana Murthy of Mangalore Chemicals and Fertilizers in India first asks for some further information: Which type of compressor are you referring to and why is it rotating in reverse direction?

Bhawna replies: When a centrifugal compressor trips, the higher pressure still existing at the discharge side of the centrifugal compressor will equalise with the lower pressure that is always present at the suction side of the compressor. This situation can cause reverse flow. Some plants have reported reverse rotation phenomena in the CO₂ centrifugal compressor.

Gholamali Soroush of Shiraz Petrochemical Complex in Iran shares his valuable experience: In the CO₂ compressor outlet, there are three valves (check valve, shutdown valve and bleed valve) which should avoid reverse flow. By depressurising the compressor outlet line, one prevents reverse rotation. The shutdown valve and bleed valve are automatic valves, which are actuated by trip signals and are in the discharge outlet upstream of the non-return valve (NRV).

Ajay Singh of Chambal Fertilizers in India contributes to the discussion: Reverse rotation of the compressor may damage internals, such as bearings, seals etc.

CSK provides some valuable input: In general CO₂ compressors have four reverse flow protections in the discharge line: 1) trip shutoff valve, 2) vertical NRV, 3) horizontal NRV and 4) pressure release valve. However, reverse gas flow through the compressor is possible, by passing of the level control valve of the third separator or by passing of the NRV at the suction side of the third stage. These may cause the reverse rotation of the compressor. The compressor is not designed for reverse rotation, and it can cause bearing damage and misalignment. To overcome this problem, the pressure release can be linked (open) with the trip logic of the compressor.

Pawan Verma of IFFCO in India shares his valuable experience: Reverse gas flow through the CO₂ compressor is only possible when NRVs provided in compressor are passing and the vent

control valve size provided in the final discharge line and at the third discharge line size are insufficient to discharge the volume trapped. This problem of reversal flow may be overcome by regular servicing of the NRVs and by increasing the size of the vent valve in the compressor.

Prem Baboo of National Fertilizers Ltd, in India contributes to the discussion: We also experienced reverse rotation in our urea line-II in 1997. Reverse rotation is an unwanted phenomenon in a centrifugal machine as it is always associated with severe radial vibration and axial displacement which may lead to damage of the bearings and rubbing of seals with the stationary components. Bearing failure is a definite possibility. Seals are not rated for this rotational speed. Internal compressor components may become dislodged. There is a high possibility of damaging vibrations during reverse rotation. Coupling is rated for the rotational speed as long as the train does not become energised. Rotor overspeed can cause excessive stress of the bearing (journal/thrust). Standard face seals may be adversely affected by overspeed and rings may be rotation sensitive. To safeguard against reverse rotation in CO₂ compressors the following changes have been made:

1. During shutdown, anti-surge valve HV-62 (provided from the fourth discharge to the first suction) and the second stage vent HV-61 (provided from the second discharge to the atmosphere) are both open. In addition, HV-63 (provided from the second discharge to the first suction) logic has been changed so that it opens during tripping. In addition, the time taken to open HV-61 has been decreased.
2. The final discharge vent PV-3 logic was changed to open during tripping of the machine. Capacity of CV should be sufficiently high.

Kashif Naseem replies: Reverse rotation can be avoided by installing kickback from casing to casing and installing the NRV at the discharge of the final stage.

Ali Salman Bokhari of Agritech Ltd. in Pakistan joins the discussion: In my view, reverse movement of the shaft is only possible in case of high poly-tropic/discharge head or in simple words "surging". Once the compressor is tripped due to actuation of the surge counter security, reverse movement is almost impossible because:

- kickbacks will automatically open and equalise the pressure between the suction and the discharge ends of the compressors;

- generally auto valves at the compressor discharge open automatically to relieve the discharge line;
- additional protection in terms of NRVs is also available;
- gas in the compressor at the discharge side does not have much force to first stop the compressor, nor does it have much capability to provide the force required for causing reverse rotation.

In case of reverse rotation, what is important point is the speed of the compressor i.e. high speed will definitely cause vibrations and damage to seals.

Sometimes there are some design constraints regarding relieving the third stage pressure through the fourth stage and uneven depressurisation of inter-stage piping and equipment. This uneven depressurisation generates enough torque to rotate the shaft in the reverse direction. Latterly, the problem was resolved by instantaneously opening the 2-1 recycle valve with application of the solenoid valve along with the second stage vent and by modifying the interlock system.

In my view, there should be no reverse rotation in the compressor if there are no design constraints. Anti-surge, vent and non-

return valves are provided in order to protect the machine from such problems in worst case conditions.

Let me share our experience as regards design constraints. We have a turbine-driven centrifugal CO₂ compressor comprising LP and HP casing with each casing consists of two stages. The original equipment manufacturer is MHI (Japan). The anti-surge system is provided by CCC (USA) consisting of 1-1, 2-1 and 4-3 kickbacks. Under normal operating conditions the machine is designed to raise the pressure from 0.9 kg/cm².g to 178 kg/cm².g. A few years ago, we did an emergency shutdown due to a problem in our national gas headers. The compressor tripped due to actuation of low pressure security at the first suction. This was surprising because everything was going smoothly as per standard operating procedures. Latterly it was found that the 2-1 kickback opens rapidly letting down the pressure from 44 to approx. 0.9 kg/cm².g in order to meet the flow requirements at the first suction. This sudden opening results in the formation of dry ice, even after the injection of hot gas and restricts the flow passage downstream of the kickback. This design constraint was resolved by some counteractive measures. ■

This series of discussions is compiled from a selection of round table topics discussed on the UreaKnowHow.com website. UreaKnowHow.com promotes the exchange of technical information to improve the performance and safety of urea plants. A wide range of round table discussions take place in the field of process design, operations, mechanical issues, maintenance, inspection, safety, environmental concerns, and product quality for urea, ammonia, nitric acid and other fertilizers.

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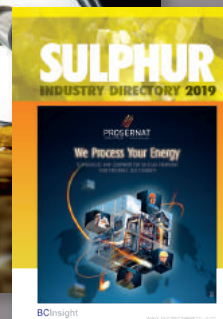
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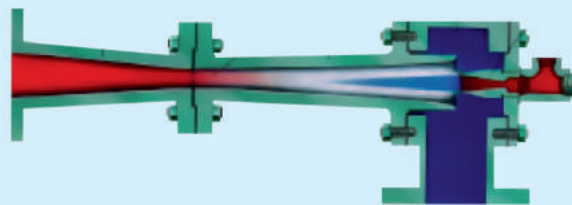
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Problem No. 50 Low vacuum pressure in the second-stage evaporator

Most urea plants operate a two-stage evaporator section. Both stages operate under vacuum pressure conditions and both pressures and temperatures are critical to achieve the right urea product quality. It is not always easy to determine the exact cause of non-optimum values for certain process parameters like, for example, low vacuum pressure in the second-stage evaporator. Many factors can influence this vacuum pressure as discussed below.



Source: Graham Corporation, USA

Mohit Bayer of CFCL Kota in India starts the discussion: In our plant we are experiencing a problem with the vacuum pressure in the final concentrator which is very low (it is now 105 mm Hg, whereas before it was 60 mm Hg). We have done all types of flushing. We have checked that air is not being sucked in through the flange connections. We have insured that the condensate outlet lines of surface condensers are not choked. The instrumentation is also okay. The urea sample moisture content is 0.56 wt-% which is not very high compared to before. The plant is TEC ACES technology running on two trains with a common prilling tower. The problem is in one train only.

- Are there any other parameters that can show that the vacuum pressure is really low?
- To what extent will this very low vacuum pressure affect the product quality?
- How can we insure that the final concentrator heat exchanger tubes are not leaking?
- What will happen to the steam consumption of the heater of the final concentrator when the vacuum pressure is low: will it increase or decrease?

The actual cause is still unknown. We have performed a lot of flushing and tried to clear the path of vapour. We have also performed a lot of polymer washing, and insured that the vacuum is not broken anywhere.

Kashif Naseem asks for some further clarifications and provides some valuable suggestions: What are the evaporator parameters? Check that the flushing valves are fully closed. Purge your surface condensers. Flush the bottom bottles of the surface condensers and check the ejectors (maybe the downstream line is choked).

Mohit replies: We have checked all of the things you suggest and all are clear. The evaporator parameters are as follows: shell-side steam pressure 2.8-3 kg/cm²g; tube-side inlet urea solution temperature 131°C; tube-side outlet urea solution temperature 136°C; tube-side pressure 50 mm Hg; flow of urea solution 60-65 m³/h; temperature of urea outlet solution is controlled by steam pressure; urea inlet to this evaporator comes from the first evaporator that is operating on 150 mm Hg vacuum, its vacuum is taken by an ejector that is in the vapour outlet of a surface condenser (say No. 3), uncondensed vapour of this condenser No.3 goes to surface condenser No.1 (condenser for first stage evaporator) whose vacuum is taken by an ejector in its downstream line; sur-

face condenser No.1 operates at 150 mm Hg and No.3 at 50 mm Hg, these two ejectors are in series; the vacuum of the first evaporator is okay.

Majid Mohammadian of OCI Nitrogen in the Netherlands shares his experiences: Vacuum in concentrators is mostly the result of cooling in the condensers, performance of the ejectors and the concentration and temperature of the urea solution. Sometimes partial crystallisation in the condensers may occur leading to a bad vacuum pressure. If the urea solution concentration is correct my suggestions are as follows: First raise the condenser temperature in the cooling water return line to about 45 to 50°C for a short time and check that there is no partial crystallisation in the condenser. Secondly check the ejector internal and its performance.

Mohit replies: We have already done what you suggest many times, we call it condenser hot flushing, but it has not helped.

Muhammad Farooq of SABIC/SAFCO in Saudi Arabia offer his suggestions: As the moisture content is quite high, it looks to me like the final concentrator may be overloaded. I would suggest checking the following: urea solution concentration in the urea solution tank downstream of the low pressure decomposer/flash tank – compare it with the design value in the mass balance (percentages of ammonia and water are important); urea solution concentration after the first evaporator/concentrator (percentage of ammonia in wt-ppm); condition of the final ejector line-up to the condenser – polymer deposition will hamper the vapour path to the surface condenser.

Does the vacuum pressure change when you carry out flushing of the final ejector line up to the condenser for 1-2 hours with hot media? Finally check physically/internally the nozzle of the final ejector of the final concentrator.

Mohit replies: The vacuum pressure in the first concentrator is within the normal range, so there is no chance of more ammonia and water in the low pressure decomposer outlet. The conditions in the back end are also the same as before. We have also done polymer flushing and because it may choke the vapour path it is done with the help of molten urea, but it makes no difference. As far as the ejector is concerned, there is no provision for flushing the ejector of the final concentrator.

Siddharth from Tata Chemicals in India shares his experience:

I would like to share an improvement we made to our urea plant before shutdown of the plant during the annual turnaround of 2012. Before the turnaround, we were faced with the problem of huge deposition of polymer in the first and second stage separators of the vacuum sections. In view of this problem we started dome flushing of both separators with urea melt on a daily basis for 15 days before doing the shutdown. Even after shutdown of the plant we continued this flushing for more than 8 hours, recirculating the melt through the recovery pump. The result was splendid as much less deposition of polymer was observed in the separators and we have saved a lot of water and reduced the amount of disposal of polymer and manual work, which had been necessary earlier. In my opinion the circulation of urea melt for separator dome flushing is an important and useful activity.

Sam QR from India joins the discussion: Do you have a demister in the vapour outlet of the second stage evaporator and if so where is the pressure measurement taken, upstream or downstream? Have a routine for boiling out the second stage evaporator with 72 wt-% urea solution (once every two months). Be careful not to contaminate the evaporator condensate with urea solution, which will affect the hydrolyser. Based on the volume of the second stage evaporator/inlet solution flow rate, determine the time required to fill the second evaporator and fill the evaporator with urea solution. This will greatly help in removing poly-urea.

Evgeniy Shishkin of Koch Industries in the United States asks for further information: We are experiencing similar issues. Do you have any more information on what you mean by poly-urea and the procedure for flushing it out with urea solution?

Mark Brouwer of UreaKnowHow.com replies: Poly-urea refers to polymers of urea like biuret, triuret etc. These are formed quite easily in the second stage evaporator and dissolve easily in urea melt. Nowadays, many urea plants apply a flush system with urea melt (from discharge urea melt pump) on the second stage separator. It is of course important that these small flush lines can be flushed out when they are not in use.

Aslam Muhammad of Fauji Fertilizer Company Limited in Pakistan provides another suggestion: Please check the ejector downstream nozzles for choking or dislodgement.

Zeeshan Shoaib of Fauji Fertilizer Company Limited in Pakistan shares his experience: I will add just two more suggestions as per our own experience as we have faced similar problems. Map the temperature of the ejector and condensers from outside and observe any change. It will help to rule out the health of the ejector and condenser. Measure the cooling water flow to the vacuum section and the condensers. Back flush the condensers from the cooling water side online after by-passing the feed of vacuum section at reduced plant load. We have implemented these ideas and the poor vacuum pressure problem diminished.

Prem Baboo of National Fertilizers Ltd in India joins the discussion: The problem of second stage vacuum pressure might be due to a leakage in the vacuum system i.e. flanges of heat exchangers, condenser sight glasses, all equipment flanges. Flushing of the condensers is a temporary phenomenon, after flushing it can be improved further. There is relationship between the vacuum pressure and the separator outlet temperature. Various parameters are related to vacuum. For example if vacuum pressure is pulled out then the temperature will come down and the steam requirement increases. The moisture content in the urea product is also affected by weather conditions. The steam pressure can be increased to improve the vacuum ejectors. If there is sufficient margin in the first stage vacuum then it can be improved subject to the outlet temperature of urea and according to the concentration and temperature graph. If the outlet temperature of the first vacuum is more than 120°C then it can be increased to 225-240 mm Hg so that the load on the second vacuum is shifted to the first vacuum. The detection of leakages in the vacuum system can be checked. The procedure is tricky: two or three persons are required to check for leaks, break the vacuum pressure and all ejectors steam valve should be closed, slowly closing the condenser cooling water outlet valve. In so doing, the system pressure will increase to more than 750 mm Hg and any vapours will leak out. Observe and note down the leakage points and open the cooling water valve immediately. ■