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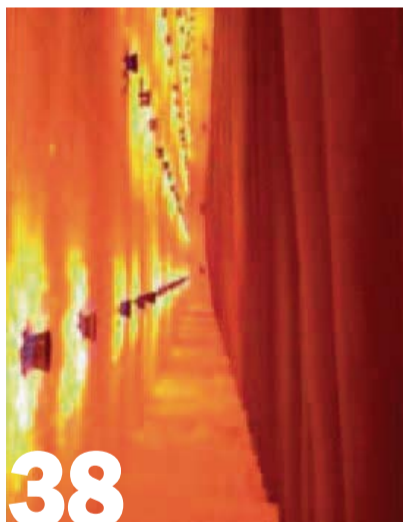
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# nitrogen + syngas

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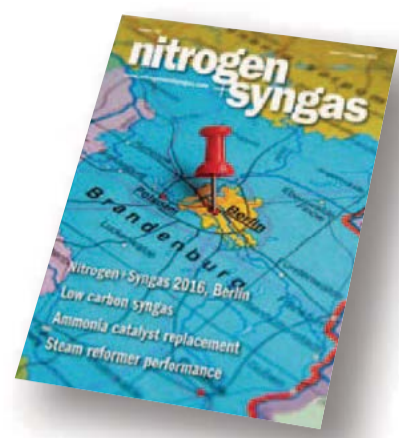
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# Not all doom and gloom



“Demand growth in nitrogen markets is often much steadier than other commodities.”

The news at the start of 2016 seems to have been pretty bad on all fronts, and the business world has been no exception. Commodity prices took a big dive during 2015 – falling by 30% across the board, including 34% for oil and 40% for steel. In virtually all cases the slowdown in the Chinese economy has been to blame, although crude has had an additional factor in Saudi Arabia’s strategic decision not to cut back production in the hope of forcing other competitors out of the market. China’s rapid growth during the 2000s and early 2010s seduced commodity suppliers into thinking that double digit growth could continue indefinitely, and as a result most key markets now find themselves oversupplied as expected demand has failed to materialise at a time when new capacity is coming on-stream.

Demographics have been to blame as much as anything for this – the success of China’s ‘One Child’ policy has led to a rapidly ageing population and a rapid fall in the number of new workers entering the work force, and perhaps a limit to what can be achieved through industrial, investment-led growth. As a result, China faces a period of dislocation as it switches to a consumer rather than industrial-led economy, while any gains from last year’s loosening of the One Child policy will take a decade or more to make themselves felt.

China’s overcapacity has been evident in nitrogen markets as well, especially urea, where prices have recently dropped to their lowest levels since 2010, and closing in on \$200/t – a level not seen for a decade. Urea overcapacity is coming from several different sources; not just China, where capacity additions have been extraordinary, but now also North America, fuelled by the shale gas boom, where CF Industries recently started up its new urea line at Donaldsonville, Louisiana, and Russia and Central Asia, as our article in this issue notes.

But it is not all doom and gloom in 2016. China continues to grow at 6-7% per year, and India, where growth has lagged, has now overtaken China and could yet be the coming power, as its growth has been more balanced and not dependent upon supplying cheap manufactured goods to the rest of

the world. Provided both countries can avoid the so-called ‘middle income trap’ that seems to have engulfed Brazil, they can still be a motor of world growth. The US and northern Europe are also seeing strong growth, although the Eurozone remains burdened by the heavily indebted countries of the south.

Meanwhile, agricultural commodities have also actually picked up in the last months of 2015 on the back of fears of disruption to production caused by El Niño – cocoa is up 10% on the year, and sugar and cotton 5%, and in general agricultural demand works on much shorter cycles than the several years that are normal for ‘hard’ commodities. Demand growth in nitrogen markets is often much steadier than other commodities. IFA is still predicting a 2% rise in nitrogen demand for 2016, and 3% for urea, with industrial markets still providing a boost.

And on the supply side, while the current tranche of new capacity is still working its way through the system, and 2016 is certainly likely to be a poor year for nitrogen producers compared to the bonanza they saw in previous years, it is likely that urea prices don’t have much further to fall – low prices are now starting to see a supply side response, as Laura Cross notes in her Price Trends column on page 6. Looking further ahead, India’s attempts to kick-start new urea capacity building seems to face numerous difficulties, not the least of which is being able to find sufficient natural gas to run the new plants, and India seems set to continue to be a large importer for the world’s urea glut. ■

Richard Hands, Editor

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# Price trends



## MARKET INSIGHT

**Laura Cross**, Senior Analyst, Integer Research, assesses price trends and the market outlook for nitrogen.

## NITROGEN

The state of the nitrogen market in 2015 can be summarised in two words: cyclical weakness. Overcapacity continued to put significant pressure on product pricing, and the supply-driven phase that began with the ramp up of new capacity in 2012 became even more intense. This makes nitrogen supply one of the stand-out themes for the industry in 2016. In the year ahead, a significant amount of new nitrogen capacity is expected to come online, ranging from the obvious locations such as newly-gas-rich North America to the much touted new source of nitrogen demand, sub-Saharan Africa.

Russian producers also have high hopes for their investment potential in 2016, aided by the recent rapid devaluation of the rouble which came largely as a result of the Russian-economy's heavy reliance on oil. Russian fertilizer producers were already competitive with their global counterparts before the currency slide, which began in mid-2014, due to their low production costs and vertically integrated structures. The continued depreciation of the Russian rouble has further strengthened their competitiveness, boosting profit margins and cash flow, and the attractiveness of investment.

Nearly 3 million tonnes of ammonia capacity expansion projects are expected to come on stream in Russia by 2019, including EuroChem's plan to build new nitrogen plants at Kingisepp and Nevinnomysk, while PhosAgro is commissioning a new ammonia plant in Cherepovets and Acron is building ammonia capacity due to start up in 2016. With the continued depreciation of the rouble, these capacity expansion plans look all the more competitive.

Although it has far less promising nitrogen production economics, India has also outlined ambitious targets for increasing its domestic nitrogen capacity. There is some scepticism related to the government's ambitious target of adding 8 million tonnes of urea capacity by 2019 to reach self-sufficiency. In H1 2015, Indian imports increased by 54% year-on-year to reach 3.8 million tonnes. Reports indicate a rather lukewarm response to the latest request for a proposal by the Department for Fertilizer for investors in two new urea plants at Gorakhpur and Sindri – achieving only one bidder each. Our projection for new urea capacity in India is that substantial new urea supply is unlikely. Any new project is likely to have production costs of two or three times the current Maximum Retail Price (MRP) and will lock in substan-

tial additional future production subsidies, albeit offset by lower import subsidies.

In addition to a wave of new capacity in 2015, the nitrogen industry was also tormented by varying projections of the oil market over the course of the year, and more importantly its crucial impact on nitrogen feedstock costs. For what it's worth, the nitrogen market was not alone; 2015 signalled the start of market uncertainty across a range of commodities.

Nitrogen prices remain inherently energy-driven and feedstock costs to the marginal nitrogen producers fell in 2015. The correlation between oil, gas and coal prices, however, is not as strong as it once was. The prices of gas in Europe and coal in China, key metrics influencing the production economics of swing nitrogen production clusters, fell to recent record lows in 2015, flattening the global nitrogen cost curve and shifting the nitrogen industry floor price down. But there is a key distinction between the recent oil price slide and falling nitrogen feedstock costs. By analysing the various energy market cycles in mid-2015 we concluded that falling gas and coal prices actually preceded the oil price crash in late-2014, demonstrating the reduced correlation between the major energy products. This is important because of the current location of the nitrogen market's marginal producers – they are largely positioned in regions where gas and coal prices come with a healthy dose of independence from oil.

It therefore goes without saying that a key metric to watch in 2016 is going to be the changing nature of world energy markets. Analyst consensus forecasts did not foresee the fall to below \$30/bbl oil in January, which has led the year to a shaky start in commodity and financial markets alike. If we consider gas in Europe and coal in China specifically, feedstock costs to producers remain weak, but withstood the extreme declines of the oil market in the last few months.

We have focused on prospects for nitrogen supply in this issue, however, developments in nitrogen demand also carry a great deal of importance – we will cover the major themes for demand in the next issue of *Nitrogen+Syngas*, discussing product substitution, weather patterns and the impact of El Niño, and whether by the end of 2016 capacity will have begun to grow more slowly than demand, prompting an increase in utilisation rates. This development would in turn promise higher nitrogen prices and profits, and perhaps signal a movement towards the next cyclical upturn.

Table 1: Price indications

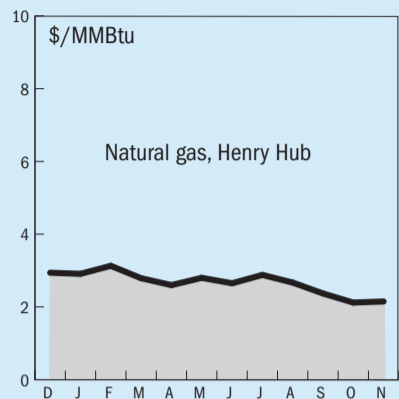
Cash equivalent	mid-Nov	mid-Sept	mid-July	mid-May
<b>Ammonia (\$/t)</b>				
f.o.b. Caribbean	360	405	420	425
f.o.b. Arab Gulf	390-425	420-450	380-400	350-405
c.fr N.W. Europe	410-445	474	455-480	460-480
c.fr India	440-470	463	420-455	410-450
<b>Urea (\$/t)</b>				
f.o.b. bulk Black Sea	258-263	255	262-273	275-283
f.o.b. bulk Arab Gulf*	255-263	266	268-278	308-335
f.o.b. bulk Caribbean (granular)	250-255	264	280	295-305
f.o.b. bagged China	252-255	263	282-287	293-308
<b>DAP (\$/t)</b>				
f.o.b. bulk US Gulf	410	458	470	468-472
<b>UAN (£/tonne)</b>				
f.o.t. ex-tank Rouen, 30%N	183-186	186	195-197	185-190

Notes: n.a. price not available at time of going to press  
n.m. no market \* high-end granular

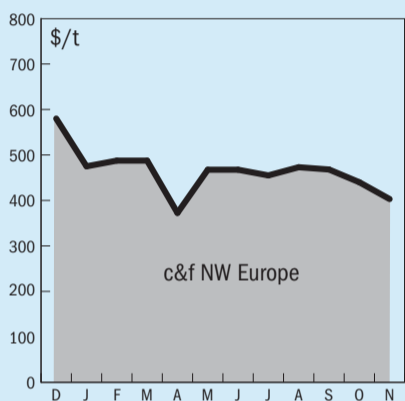
Source: Fertilizer Week

END OF MONTH SPOT PRICES

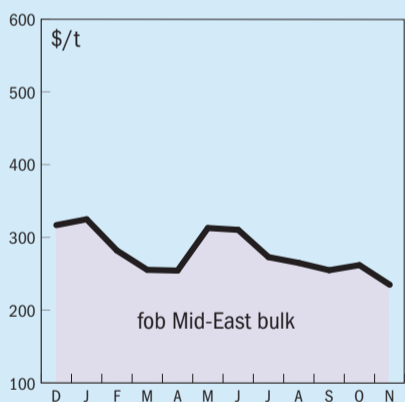
natural gas



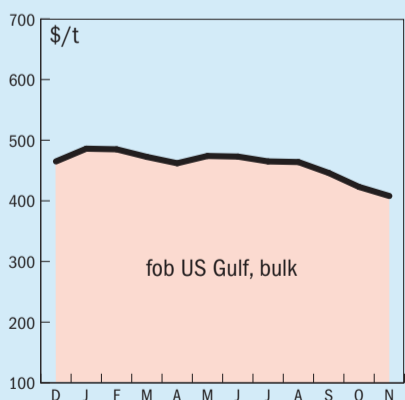
ammonia



urea



diammonium phosphate



MARKET INSIGHT

Mike Nash, Global Business Director, IHS Chemical, assesses the market for methanol.

METHANOL

In North America, the December contract natural gas reference price was settled at \$1.86/MMBtu for Texas, and \$2.30/MMBtu for Louisiana, with Texas down \$0.16 from November. Overall methanol demand is normally slow at this time of year, but we are not seeing the typical restocking demand following the end-year inventory clearances, somewhat surprising given the low prices permeating the methanol market. In company news, G2X held their ground breaking ceremony on January 13th for their Big Lake Fuels site. The project was initially to be a 1.4 million t/a methanol unit with an MTG unit on the back end; but they have opted to delay the MTG plant for the time being given the current crude oil market. Start-up for the methanol unit is targeted for late 2018.

Meanwhile, production across the Americas is estimated to be flat. In North America, IHS Chemical estimates the industry average operating rate in the mid 80% range. Methanex successfully started up their Geismar 2 unit at the end of December and commercial product will start flowing in January. In South America, Venezuelan units are operating at low levels – around 60% of nameplate capacity. Methanex’s Chilean unit remains down to an estimated 30% capacity. Trinidad has seen around 82% on average with the threat of another increase in natural gas curtailment seeming to have faded.

European spot methanol prices fell amid a lack of buying interest and healthy supply in the Atlantic Basin. Demand is stable. The EMethanex unit in Damietta, Egypt, is running after a long period offline due to unavailability of natural gas. A facility in the Black Sea region is temporarily idled due to limited gas availability and a unit in Oman is temporarily shut down due to gas shortages. A unit in Northwest Europe has been temporarily idled due to poor margins. Buying interest in the European spot market has been low, with consumers largely covered by contract volumes. Methanol consumption into derivatives in Europe is stable and there is no significant incremental rise in demand expected this quarter.

In the Middle East, there are a number of supply-side issues, including reduced gas

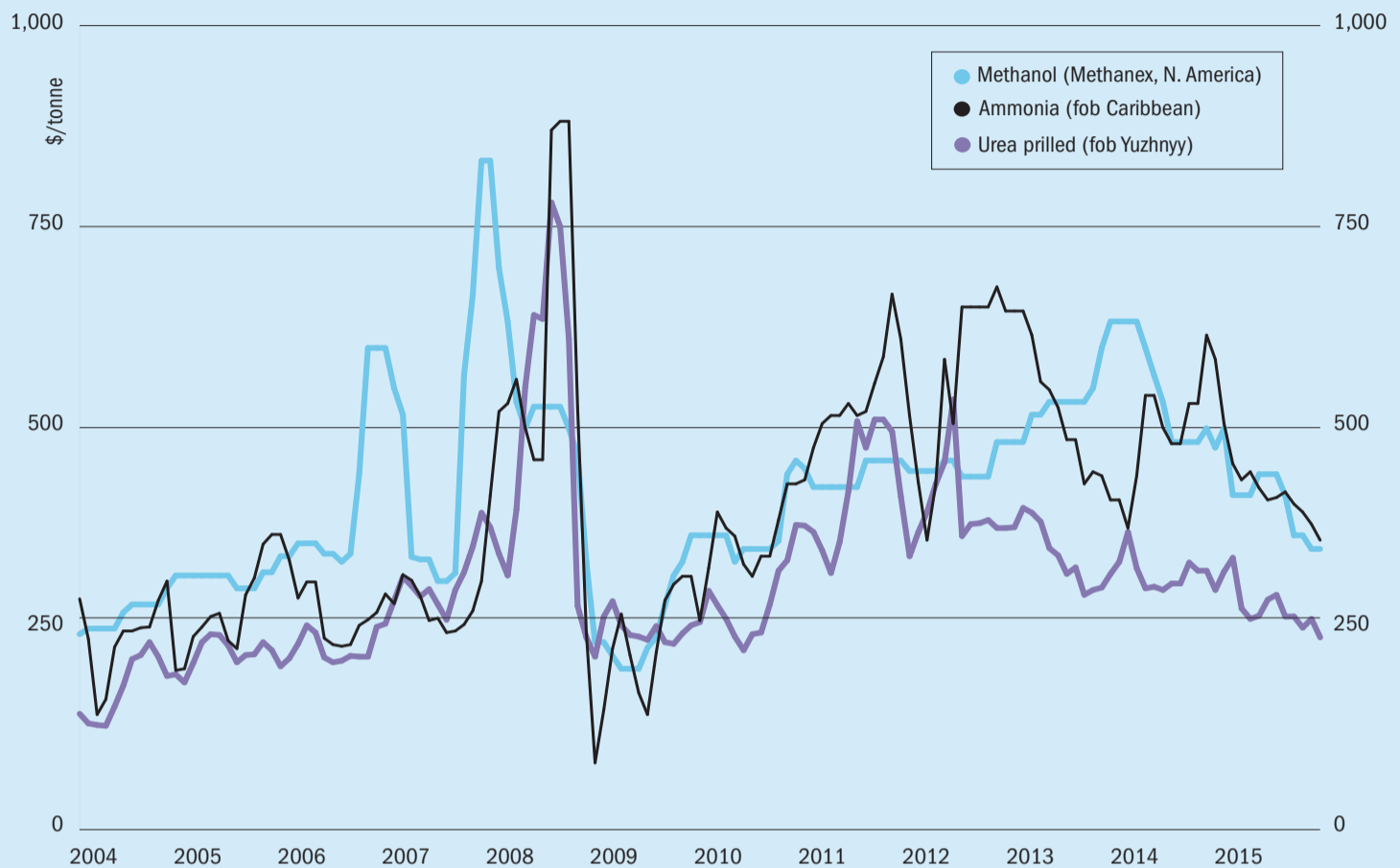
availability and maintenance. This has not, however, had a significant impact on the market, since product is still scheduled to load over January and consumers are largely covered. In addition, length in the US market has boosted confidence in supply in the Atlantic Basin. Saudi Arabian chemical producers are assessing the impact of the energy and feedstock price hikes announced by the Saudi government in its state budget on 29 December 2015. The increases, which apply from the beginning of this year, include 67% for methane and 40% for electricity and water. Sabic estimates that the price hikes will increase its annual costs by around 5%.

In India, lack of buying interest had led to reduced stocks at port in early January, however, imports have since resumed and inventories have been replenished. East Asian markets are stable, with little trading activity. The Chinese Yuan’s devaluation continues to cause a cautious approach to the import market. The general buying activity indicates that major end-users have not yet started to build up their inventory in preparation for the typical operating level during the Chinese New Year Holiday. China’s domestic methanol production is stable. The average operating rate is about 54% of nameplate capacity or 71% of effective capacity. North China is operating at low levels because of air quality controls. Coastal inventory picked up modestly. Demand into the formaldehyde sector has weakened, reflecting a seasonal decline and the government control of operations in some parts of the country due to an environmental emissions audit. Demand into the MTO sector is still very strong with an average operating rate above 80%. Mainstream selling ideas were about \$210/t for January or early February loading. Major end-users are still looking for prices below \$205/t. The import product supply may become tight since several international suppliers are either experiencing unstable operations or are planning maintenance outages.

In Southeast Asia, a decreased level of supply is expected by the end of January with a turnaround at one of Petronas’ plants. However, the spot market remains quiet. A stalemate between buyers and sellers remains in place as buyers look for further price decreases and sellers are holding quoted prices firm. ■

# Market outlook

Historical price trends \$/tonne



Source: BCInsight

## AMMONIA

- There is no immediate relief in sight for the increasingly distressed ammonia market, and prices are not expected to rebound until late February or early March.
- The most imminent driver for strengthening the market is likely to come from the US, where demand is expected to increase ahead of the spring application season and provide some basis for a correction in a global market where the ammonia balance is increasingly trending towards a rather heavy supply overhang.
- This trend is mirrored in other regions, where weak demand is expected to continue until February at least, for example in South Korea where an announcement says no new volumes will be required until that month.
- Producers are restricting supply to halt further falls, most notably OPZ, which decided to take one of its two ammonia units off-line in a bid to cut regional

availability. The firm is not expected to re-start this unit until prices exceed \$300/t.

## UREA

- Oversupply has taken urea prices below \$200/t, \$100/t lower than this time last year.
- Urea prices are expected to remain weak and could move to lower levels before a recovery, as there is no significant improvement in demand expected to emerge in January and February.
- Spring demand in the US, as well as expectations that India could issue a new purchase tender in March or April are expected to be the first signs of improvement, likely to relieve pressure on prices somewhat.
- Any further drop in price levels in January and February are also expected to incentivise buyers, who have been cautious to return to the market so far.
- Worsening economic conditions and currency devaluations in China have placed additional pressure on prices.

## METHANOL

- Methanex's second plant at Geismar, Louisiana has come on-stream in the US, adding another 1.0 million t/a of capacity.
- Methanol consumption is relatively flat and stable in most major markets.
- There are supply upsets in the Middle East but no impact has been seen on the market as yet.
- Likewise the removal of most sanctions on Iran has not so far had much impact on the Chinese market. Ample spot supplies seem to be available from elsewhere, and suppliers still face difficulties with logistics and payments in the short term.
- Low and falling oil prices still seem to be dragging methanol prices lower for the time being.
- Chinese MTO producers remain a bright spot in demand, and there are indications that they will continue to support Chinese methanol prices as conventional crackers take turnarounds in the coming months.



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## UNITED STATES

### Start-up for new CF urea plant

CF Industries' new 1.16-million t/a urea plant, constructed at the company's Donaldsonville Nitrogen Complex in Louisiana, achieved "stable operation" in December, the company says. The urea plant was first started up on November 17th. CF Industries president and CEO Tony Will said that the start-up was a "significant milestone" in the company's capacity expansion programme, and that "Donaldsonville will play a key role as we become an even larger global supplier of nitrogen-based fertiliser. Its increased capacity, along with its unmatched logistics capability, will allow us to serve customers in North America and around the world in the most efficient way possi-

ble, while providing attractive returns for shareholders."

CF is expecting that the associated upstream ammonia unit will come on-stream by the middle of 2016. The ammonia unit will have the capacity to produce 1.1 million t/a of ammonia. Downstream UAN capacity totalling 1.4 million t/a will be operational in 3Q 2016. The Donaldsonville expansions will make the site the largest nitrogen producing facility in the world, according to CF Industries. It is the first new grassroots urea plant built in the US since 1998. CF Industries is also building an ammonia-urea unit at its Port Neal, Iowa site, also due to come on-stream this year. ■

### LSB finances new nitrogen facilities

LSB Industries has completed the sale of \$210 million worth of preferred stock and warrants in order to finance the completion of construction of two nitrogen plants at its facility in El Dorado, Arkansas. The company is building a new 375,000 t/a ammonia plant, which was reported to be 85% complete, and due to be completed in February, with production beginning in 2Q 2016. Work has also been completed on a new nitric acid plant and nitric acid concentrator at the site, which is now up and running and has passed all performance tests according to the company. The total cost of the ammonia and nitric acid plants was put at \$855 million, and the \$210 million raised will be the final tranche of this.

LSB interim CEO Daniel Greenwell said: "We are pleased to have secured the additional financing to complete the expansion projects at our El Dorado facility. These projects remain on schedule, with the nitric acid plant in operation and producing, and the ammonia plant anticipated to enter service in the second quarter of 2016. The budget we articulated in early November remains intact. We continue to forecast a material increase in profitability once the El Dorado ammonia plant is in full production and we look forward to reporting our progress to shareholders."

Elsewhere, LSB says that the ammonia plant at its Cherokee, Alabama facility resumed production on December 24th after a 13-day stoppage for unplanned

maintenance after a leak was detected in a hydrogen vessel. LSB also had to take its urea plant at Pryor down in November to repair a CO<sub>2</sub> pre-compressor, and in December Pryor's urea and UAN plants were shut down in order to repair a leaking joint in the nitric acid plant.

### New ammonia plant for Nebraska?

A company called Fortigen has submitted plans to the city council of Geneva, Nebraska to build a \$75 million ammonia plant. Land has been secured on 20 acres of land on the outskirts of the city in the east of the state, in what is the western side of the US corn belt. The city council has approved Fortigen's application to re-zone the land for industrial use, as it is next door to the town's existing industrial park. The company's plans, which do not disclose the projected capacity of the plant, anticipate beginning construction later this year and completing it in 2017. Fortigen is owned by locally-based property developer Tetrad Corp. of Omaha, Nebraska.

### KBR buys Ecoplanning, Plinke and Weatherly

KBR, Inc. has announced the acquisition of Weatherly Inc., Plinke GmbH and Chematur Ecoplanning Oy from Chematur Technologies AB, for a price of \$24.5 million. Weatherly is a North American-based company with extensive experience providing nitric acid and ammonium nitrate proprietary technologies and services to the fertilizer market. For more than five decades, Weatherly has designed, engineered

and constructed these integral processes in fertilizer complexes worldwide, and has an installed base of 80 nitric acid plants worldwide with approximately 30% market share of installed plants.

Plinke is a German-based company specializing in proprietary technology and specialist equipment for the purification and concentration of inorganic acids used or produced in hydrocarbon processing facilities worldwide, and Ecoplanning, based in Finland, offers proprietary evaporation and crystallization technologies and specialist equipment for weak acid and base solutions.

Stuart Bradie, president and CEO of KBR, said: "KBR's ammonia and Weatherly's fertilizer capabilities result in a powerful combination of industry-leading technologies and Plinke and Ecoplanning enhance the scope of KBR's technology solutions across expanded platforms. We see great opportunity to extend these technologies outside North America to new customers and in revamping units of the existing customer base globally, leveraging KBR's global sales team and EPC capabilities."

## TAIWAN

### Increase in high purity ammonia production

Japan-based Showa Denko (SDK) has increased the production capacity of high purity ammonia at its subsidiary in Taiwan from 2,500 t/a to 3,500 t/a. This brings Showa Denko's total capacity of high purity ammonia to 7,000 t/a. The company has three production facilities located in Japan, Taiwan and China. The facility in Japan produces 1,500 t/a and the unit in China 2,000 t/a. High purity ammonia is a specialty gas used in the manufacturing of electronic materials. During production, the gas forms a nitride film to produce electronic parts, including compound semiconductors such as LEDs and LCD panels.

## RUSSIA

### Uralchem completes ammonia revamp

Russian nitrogen producer Uralchem has completed work to upgrade a large-scale ammonia facility at its subsidiary Kirovo-Chepetsk Chemical Works, at a cost of \$7 million. The work was completed during a maintenance shutdown in October and November. Uralchem says that the work is the largest upgrade to the plant since 1981, and will take ammonia production to 1,750 t/d. The syngas compressor was

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updated to increase its capacity and reduce gas consumption. A gas dehydration unit was also installed to remove water and carbon dioxide from the incoming syngas and help reduce synthesis loop pressure. The use of steam by the compressor turbine and the load on the ammonia evaporators will also be reduced.

Kirovo-Chepetsk Chemical Works director Sergey Momtsemlidze said: "The modernisation work on the ammonia facility increases its capacity and productivity. We expect to obtain a reduction of over 3% of energy consumption, with increases in production consistency, reliability and safety."

### Control systems upgrade at Togliatti

Honeywell Process Solutions (HPS) has completed work on upgrading the controls and safety system at TogliattiAzot's ammonia plant at Togliatti. The project was part of an agreement that will see Honeywell automate all production facilities for TogliattiAzot. Work at Togliatti including replacing the existing pneumatic control system with the advanced *Experion*® Process Knowledge System (PKS) based on C300 and Safety Manager controls. Honeywell also implemented a new control and measurement system, supported by *SmartLine*® pressure and temperature transmitters. The upgrade will help the plant to increase reliability, improve safety and boost productivity.

### Finance secured for ammonia plant

EuroChem has completed a loan arrangement with a consortium of banks for the financing of its new Kingisepp ammonia plant in Russia, part of the EuroChem Group's Phosphorit subsidiary. The 2,700 t/d (1.0 million t/a) plant is based on KBR technology. Construction began in 3Q 2015, with completion and start-up expected in late 2018. The project cost is put at \$1 billion, including utilities, offsites and liquid ammonia storage capacity. Maire Tecnimont won the EPC contract in June 2015.

## CANADA

### AN code of practise now in force

Fertilizer Canada's Agricultural Ammonium Nitrate Code of Practice came into force on January 1st, embodying what Fertilizer Canada describes as "robust industry standards for the safety and security of agricultural ammonium nitrate fertilizer". The Code of Practice is mandatory for Fertilizer Canada members, and parallels federal Canadian regulation outlining best

practices for the safe and secure storage and handling of ammonium nitrate. It is designed to assist shippers, sellers, handlers, customers and end-users to become aware of and to assist in their compliance with the regulatory environment.

"Ensuring fertilizer safety requires coordination among all stakeholders, from facility to farm gate. The Canadian fertilizer industry champions this industry-led, world-class Codes of Practice to manage the risks of fertilizer products throughout their life-cycle," said Garth Whyte, president and CEO of Fertilizer Canada. "Fertilizer Canada and our members continue to work with industry, farmers and government to continuously evaluate and improve the Code of Practice's requirements to ensure the continued safety of our communities."

Educational resources supporting the Code of Practice are available on Fertilizer Canada's website – including the Ammonium Nitrate Security eLearning course, the Ammonium Nitrate Storage Practices Brochure and the On Guard for Canada Program.

### Iffco confirms urea plant now "on hold" again

The Indian Farmers Fertilizer Cooperative (Iffco) has said that its proposed ammonia-urea facility at Bécancour, Quebec, has once again been put on hold until favourable economic conditions prevail, in particular an increase in urea prices, which are currently at their lowest levels since the aftermath of the 2009 banking crash. The project's promoters and investors say that they believe that the project remains fundamentally viable, and that the support of the government of Québec and major institutional partners remains solid.

"We are halting the project; as a result of the current poor rates for urea that is blocking the promoters and investors from attaining the plant's anticipated profitability. We are waiting for the cyclical low point in the price of urea to end, which we expect will allow resumption of the project. In the interim, we continue to work on developing the project and preparing for its re-launch," said IFFCO Canada Director General, Claude Lafleur.

The project was first put on hold in late 2014, because of doubts about securing a long-term gas supply and rising EPC costs – up to \$2 billion from an original estimate of \$1.2 billion. However, it was dusted off again in June 2015, after progress on a gas supply agreement and a change of EPC partner with – presumably – a lower price estimate.

### Worker dies after ammonia leak at Medicine Hat

A man has died following an ammonia leak at the CF Industries Medicine Hat ammonia plant in Alberta on December 7th. A second worker was treated in hospital but later released. Both were contractors engaged in weatherproofing the outside of a large ammonia storage tank, according to the Alberta Occupational Health and Safety Executive. It is believed that a piece of equipment struck a valve on the tank and caused the leak. CF says that the leak was quickly contained, and both men were quickly taken to hospital, but unfortunately one man succumbed to his exposure.

## GERMANY

### Germany launches initiative to buy CERs from nitric acid projects

The German government has launched an initiative to buy Certified Emission Reduction (CER) 'carbon credits' from nitric acid plants in developing nations which have installed nitrous oxide destruction schemes under the UN Clean Development Mechanism (CDM), in order to prevent existing schemes from closing and to encourage new ones. The schemes were often installed based on a carbon price of 20/tonne CO<sub>2</sub> equivalent, which was the prevailing price in 2008, but since then carbon prices have dropped to around 0.60 today as nations wrangle over a new global climate pact and no additional demand for credits emerged beyond initial Kyoto Protocol compliance requirements from the EU and Japan. This has placed the financing of the emission reduction schemes in jeopardy, and so the German government has said that it will buy CERs at higher than market rates to compensate. According to Germany, emission reductions could be incentivised at a cost of just €2.00-2.50/t of CO<sub>2</sub>e, but some environmental consultancies have suggested it could be as low as €1/tonne. The total budget for the German buyback scheme is €20 million per year. Norway has also previously contracted to buy CERs at above market rates from six nitric acid N<sub>2</sub>O reduction projects it deems at risk of closure as part of its compliance with 2020 Kyoto Protocol target.

Nitrous oxide has a high global warming potential and is rated at 350 tonnes CO<sub>2</sub> equivalent per tonne of N<sub>2</sub>O. The Nitric Acid Climate Action Group aims to phase out industrial N<sub>2</sub>O emissions worldwide by

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2020, saving 70-80 million t/a of CO<sub>2</sub>e, around 1% of the mitigation gap the UN says is needed to reach the 2C goal.

**IRAN**

**Shiraz ammonia-urea plant commissioned**

The National Iranian Petrochemical Company says that the third ammonia-urea train at the Shiraz Petrochemical Plant (also known as the Shohadaye Marvdasht Petrochemical Complex) in the country's southwestern Fars Province started up in December. The plant, built by Iran's Petrochemical Industries Development Corporation (PIDEC) has a capacity of 1 million t/a.

**INDIA**

**SPIC begins switch from naphtha**

Southern Petrochemicals Industries Corporation (SPIC) has begun work to convert its Tamil nadu ammonia-urea unit from naphtha feed to natural gas. The plant is one of only three naphtha-based plants remaining in India – all of the others have closed down or been converted, and the government has pressed hard for the remaining three to switch to the cheaper feedstock. SPIC says that the project, which will also involve revamping the 40 year old plant to boost production from 620,000 t/a of urea to 2.0 million t/a of urea, will cost \$880 million. A tie in to a new gas pipeline will also form part of the work.

**Progress stalled on plant re-start**

Plans by the Indian Ministry of Chemicals and Fertilizer to revive the defunct Fertilizer Corporation of India (FCI) Ramagundam ammonia-urea plant appear to have stalled. At issue is proposed collaboration with and investment by the Singareni Collieries Company Ltd (SCCL), lack of a final decision on which is reportedly preventing financial closure for the project, which aims to scrap the old coal-based plant at the site and build a new plant which will use natural gas feed. The new facility will have the capacity to produce 2,200 t/d of ammonia and 3,850 t/d of urea – more than double the capacity of the old unit. A new company – Ramagundam Fertilizers and Chemicals Ltd – was created in January 2015 to manage the project, and work has begun on scrapping the old plant and clearing the site. National Fertilizers Ltd (NFL), Engineers India Limited (EIL) and the Fertiliser Corporation of India Limited (FCIL) have all agreed to be joint venture partners in RFCL, with NFL and IEL each taking a 26% stake and FCIL 11%. However, although SCCL initially indicated it was willing to provide the remaining 37% stake, no final decision has yet been taken by the company. It has been suggested that SCCL could supply 30MW power to the unit, and/or that explosive grade ammonium nitrate could be produced at the site for use by SCCL. Securing an agreed natural gas supply to the plant – which was aiming to startup in 2018 – is also proving to be an issue.

**CORRECTION**

*In a news item in Nitrogen+Syngas 337 (September/October 2015, p11), ('New granulation unit for Yara'), it was incorrectly stated that Stamicarbon was the technology supplier for the urea fluid bed granulation plant. In fact the contract was awarded to Uhde Fertilizer Technology BV. We apologise to all companies involved for the error.*



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## UNITED STATES

### Start-up for second Methanex methanol plant

Methanex Corp. has announced that on December 27th the company successfully produced the first methanol from its newly completed 1.0 million t/a Geismar 2 methanol plant in Geismar, Louisiana. The plant was relocated from the company's production site in Punta Arenas, Chile.

John Floren, President and CEO of Methanex said, "We are extremely pleased to announce that our Geismar 2 plant is producing methanol roughly

three months ahead of our original schedule. We expect the plant to ramp up to full production rates over the coming weeks. With the start up of Geismar 2, we have successfully grown our operating capacity by approximately three million tonnes over the last three years. The total combined cost for the completion of the two Geismar plants is approximately \$1.4 billion. We believe this represents a substantial cost savings relative to a new build plant, and we expect the Geismar 2 plant will

create significant cash flow and value for shareholders." He added; "Our team has done an excellent job of managing the complex and multidimensional undertaking of relocating two plants from Chile to Louisiana. The project was completed ahead of schedule with excellent safety and environmental performance. A sincere thanks to all of the team members throughout the organisation who have worked so hard to achieve this important milestone." ■

### CTL plant mooted for Utah

Revolution Fuels LLC is trying to get a coal to liquids (CTL) development off the ground in Carbon County in the heart of Utah's coal fields. The projected plant would use 750 t/d of coal feedstock to make 1,800 bbl/d of synthetic diesel, jet fuel, LPG and naphtha. Local authorities are keen on the project, which would generate much needed local jobs and demand for the region's ailing coal industry. However, there are concerns over the 300,000 t/a of carbon dioxide that the project would generate, as well as other emissions. Revolution's submission to the local Department of Air Quality proposes that the plant be allowed to release 20.2 t/a of large particulate, 23 t/a of nitrous oxides, 83.8 t/a of carbon monoxide, 9.2 t/a of volatile organic compounds, 1.9 t/a of sulphur dioxide and 8.9 t/a of hazardous air pollutants like mercury. There are also serious doubts as to whether the economics stack up in a low oil price environment. Revolution Fuels claims that its process is more efficient than other CTL technologies, and can be profitable at an oil price of \$35/bbl, but current prices are below even that number.

### Technip awarded contract for hydrogen plant

Technip has been awarded a contract by Air Products to provide technology, engineering and procurement services for a greenfield hydrogen plant in Baytown, Texas. The 3.5 million standard cubic metres per day plant will produce hydrogen and carbon monoxide to be supplied to customers from Air Products' established Gulf Coast Hydrogen and CO Pipeline Networks.

The plant will be built through the global hydrogen alliance between Air Prod-

ucts and Technip. It will feature Technip's proprietary steam reforming technology to produce high purity hydrogen, carbon monoxide and export steam. It will also use the latest nitrogen oxide reduction technology to reduce emissions. Technip's operating center in Claremont, California, USA, will execute the project, which is scheduled for completion in 2018.

Stan Knez, president, Technip Stone & Webster Process Technology, commented: "We are proud that our steam reforming technology is helping customers meet the world's growing energy needs in a reliable, efficient and sustainable manner. We look forward to executing this new project under our global alliance with Air Products."

### NWIW seeks to improve environmental credentials of methanol plants

Chinese methanol plant developer Northwest Innovation Works (NWIW) has sought to allay public concerns about water use as it begins a 12-18 month permitting process for the first of its three proposed shale gas-fed methanol plants for the northwestern US. The company said that as it begins the scoping period in the State Environmental Policy Act process, its engineers have made design modifications which will cut water use by 28%, from 10,000 gallons per minute to 7,200 gallons per minute, by maximising treatment and re-use of waste water.

The \$7 billion methanol plant investment envisages three plants, two of them projected to produce 10,000 t/a of methanol, and the other 20,000 t/a, for export to feed methanol to olefins (MTO) production in China. The 14.4 million t/a of methanol capacity would be an unprecedented investment anywhere in the world. The

plants are to be built at Port St Helens in Oregon, Port Kalama, Washington, and Tacoma, Washington. The Port Kalama site is said to be furthest down the development process, with a draft environmental impact statement underway, and construction is due to begin in 4Q 2016, lasting until the end of 2019. Construction at the largest site, at Tacoma, is currently provisionally scheduled to begin in 4Q 2017. NWIW has selected Johnson Matthey as methanol technology supplier, using the company's ultra-low emission (ULE) reforming technology, which reduces CO<sub>2</sub> emissions by up to 75% compared to more traditional reforming processes.

NWIW was created by China's Clean Energy Commercialisation Co., in partnership with other Chinese companies Double Green Bridge and the Chinese Academy of Science Holding Co., as well as other private investors in H&Q Asia Pacific.

## VIETNAM

### PetroVietnam considering methanol JV

PetroVietnam says that it is in discussions with Canada's Methanex Corporation and South Korean construction company Samsung Construction and Trading (C&T) over the construction of a new joint venture methanol plant in the country. Senior executives from all three companies attended a meeting in Hanoi to discuss details on the plan, according to PetroVietnam. PetroVietnam is considering using gas from the Ca Voi Xanh gas field as feedstock. At the moment the size of the proposed plant has not been disclosed, but Methanex was previously involved in studying constructing a 1.6 million t/a \$1 billion methanol plant in southern Vietnam in 2009.

## UNITED ARAB EMIRATES

### Agreements signed for GTL commercialisation

At a meeting in Dubai in November small scale GTL technology supplier Gas Technologies LLC, aka GasTechno, signed an agreement with the Chinese-based Future Trends International Group to establish a \$2 billion Sharia compliant, green-certified equipment leasing and carbon asset fund that will focus on financing GasTechno's gas flaring emission reduction projects worldwide. Earlier in the month, the company signed a manufacturing agreement with air separation and gas processing systems provider GENERON to use their custom engineering and fabrication capabilities to manufacture GasTechno's *Mini-GTL* system. *Mini-GTL* is a modular skid-mounted system to convert stranded natural gas or associated gas that is currently flared into higher value fuels and chemicals. Gas flaring from oil producers is subject to increasingly stringent environmental regulations and programs such as the World Bank's "Zero Routine Flaring by 2030" initiative.

Speaking at the signing in Dubai, Dr Farzam Kamalabadi, chairman and president of the Future Trends Group said: "I have been involved for over 30 years in the oil & gas business, and have recognised that flared gas was polluting our environment and especially those communities living near this wasted valuable resource. To me flared gas has always been 'paying huge amounts to pollute the world', whereas GasTechno is the reverse – clean the world while earning good money for the investors. Over the past six years I have watched GasTechno grow from an idea to prototype to an actual viable, technology that can change the world... I encourage nay urge the World Bank, the United Nations, other sovereign governments, national oil companies, and other major players to join us in supporting the Zero Routine Flaring by 2030 initiative."

## UZBEKISTAN

### GTL plant "still under development"

The state-owned Uzbekneftegaz National Holding Company says that it is still trying to develop the Oltin Yo'l gas to liquids (GTL) plant in spite of setbacks and falling oil prices, and that it will continue to work during 2016 on the creation of a consortium to

finance the construction of the plant, which is estimated to cost \$5.6 billion. Negotiations to build a banking consortium began in September 2013, but so far talks with potential consortium members have failed to reach concrete agreements because of the prevailing international prices for oil and downstream hydrocarbons.

The GTL plant will process 3.5 bcm per year of gas and produce 865,000 t/a of diesel fuel, 395,000 t/a of naphtha and 11,200 t/a of LNG. Financing was to be 30% provided by the joint venture equity partners in Uzbekistan GTL, and 70% from

bank loans. Sasol Synfuels and Petronas were signatories to the project's founding documents in 2009, with Sasol providing its own GTL technology for the project. However, Petronas reduced its share in the joint venture in 2011 to 11%, leaving Sasol and Uzbekneftegaz with 44.5% of the company. Sasol in turn dropped its share to 25.5% in 2013. Hyundai Engineering & Construction was awarded the \$2.3 billion EPC contract in December 2013, and site preparation at Shurtan began in January 2014, but no plant construction has begun as yet. ■



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# People

**Henrik Stiesdal** and **Anders Heine Jensen** have been elected to the board of directors of Haldor Topsoe A/S, expanding the total number of board members from ten to twelve. Stiesdal has been a major figure in the Danish wind power industry. In 1978, he co-designed one of the first wind turbines representing the so-called "Danish concept", which one year later was licensed to Vestas. After working as a consultant to Vestas, he joined Bonus Energy (later Siemens Wind Power) where he became chief technology officer. He retired as CTO of Siemens Wind Power in 2014. Anders Jensen is CEO of Burmeister Wain Scandinavian Contractors A/S a global developer, contractor and operator of medium to large-scale power plants. He has been a driving force in adding renewable and waste-to-energy technologies to the company's conventional diesel-fuelled power plants. The strategy has been executed with considerable success, resulting in seven biomass-based power plants in the UK.

Henrik Topsoe, chairman of the board of Haldor Topsoe A/S, said: "we're very pleased to welcome Henrik Stiesdal and Anders Heine Jensen to the board. We have been able to attract two very capable and experienced people, who understand the nature of the business we're in, especially the essential energy sector. Yet, they

have also shown the ability to drive necessary change and execute the business strategy. I'm sure that they will be a very positive addition to the board and I know that the entire board looks forward to working with Anders and Henrik."

Austin Powder Co. has promoted **Andy Velo**, former construction and maintenance manager of the US Nitrogen project, to plant manager as the \$200 million industrial chemical plant in Greeneville continues to ramp up. Velo has shown exceptional leadership skills and attention to detail in his role as construction and maintenance manager, according to Austin Powder Chief Financial Officer Jim Boldt. A graduate of the Naval Nuclear Power School, Velo has served as a nuclear submarine officer in the Navy and an instructor at the Virginia Military Institute. He has previous private industry experience with Archer Daniels Midland Co. and as lead for corporate wide maintenance and reliability at Rentech Nitrogen Partners. He replaces former plant manager **Justin Freark**, who has been promoted to director of nitrogen engineering projects for Austin Powder. US Nitrogen will begin production this year, with the nitric acid plant due to start up in January or February. The ammonia plant and ammonium nitrate solution (ANSOL) plant will begin production in the following months.

LSB Industries has appointed **Mark Genender**, **Jonathan Bobb** and **Joe Reece** to its board of directors. Board members **Charles Burtch** and **Robert Butkin** have resigned. With the three new independent directors, the board has increased from 13 to 14 directors. Genender is a managing director at an affiliate of Security Benefit and also serves on the board of Media Rights Capital. Bobb is a member of the corporate investment team at an affiliate of Security Benefit. Reece is president of Helena Capital LLC, a merchant bank with offices in New York and Los Angeles, that he founded in 2015.

Mangalore Chemicals and Fertilizers Ltd (MCFL) says that **N Suresh Krishnan**, previously with the Zuari Group, has assumed the position of managing director as from January 1st. Krishnan, a chemical engineer, has worked at Zuari Group since 1986, and replaces **Deepak Anand**, who continues to be a non-executive director of MCFL. Zuari acquired an additional 36% stake in MCFL in May 2015 and became a majority shareholder in the firm, with a 53% controlling stake. Zuari has made additional board appoints to MCFL, including **Akshay Poddar** and **Kapil Mehan**, and in October appointed **Arun Duggal** as MCFL chairman and Krishnan as managing director of the company effective from January 1st 2016.

## Calendar 2016

### FEBRUARY

29 – March 3

Nitrogen+Syngas Conference,  
BERLIN, Germany  
Contact: CRU Events,  
Chancery House, 53-64 Chancery Lane,  
London WC2A 1QS, UK  
Tel: +44 20 7903 2444  
Fax: +44 20 7903 2432  
Email: conferences@crugroup.com

### MARCH

15-17

IFA Global Technical Symposium,  
NEW DELHI, India  
Contact: IFA Technical & SHE Committee,  
28 rue Marbeuf, 75008 Paris, France  
Tel: +33 1 53 93 05 00  
Fax: +33 1 53 93 05 45/47  
Email: ifa@fertilizer.org

### APRIL

18-20

SynGas 2016, TULSA, Oklahoma, USA  
Contact: SynGas Association  
Tel: +1 225 922 5000  
Web: www.syngasassociation.com

### MAY

30 – June 1

84th IFA Annual Conference,  
MOSCOW, Russia  
Contact: IFA Conference Service,  
28 rue Marbeuf, 75008 Paris, France  
Tel: +33 1 53 93 05 00  
Email: ifa@fertilizer.org

### JUNE

9-10

26th IMPCA European Mini-Conference,  
PORTO, Portugal  
Contact: IMPCA, Avenue de Tervueren 270  
Tervurenlaan, 1150 Brussels, Belgium  
Tel: +32 (0) 2 741 86 83  
Fax: +32 (0) 2 741 86 84  
Email: info@impca.be

23-24

International Fertilizer Society Meeting,  
BUDAPEST, Hungary  
Contact: International Fertiliser Society,  
PO Box 12220, Colchester, CO1 9PR, UK  
Tel: +44 1206 851819  
Email: secretary@fertiliser-society.org

### SEPTEMBER

18-22

AICHe Ammonia Safety Symposium,  
DENVER, Colorado, USA  
Contact: AICHe Customer Service  
Tel: +1 800 242 4363/+1 212 591 8100  
Fax: +1 212 591 8888  
Email: xpress@aiche.org

18-23

Ammonium Nitrate/Nitric Acid Conference,  
EINDHOVEN, Netherlands  
Contact: Hans Reuvers, BASF, Karl  
Hohenwarter, Borealis  
Email: johannes.reuvers@basf.com/  
karl.hohenwarter@borealisgroup.com



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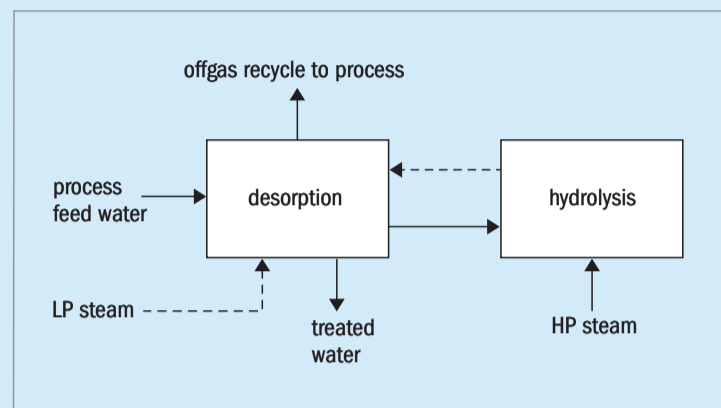
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# Plant Manager+

## 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/cm<sup>2</sup>g 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 m<sup>3</sup>/h at 3,500 t/d load;
- hydrolyser pressure 34 kg/cm<sup>2</sup>g 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/cm<sup>2</sup> and 1.8-2 kg/cm<sup>2</sup> 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 m<sup>3</sup>/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/cm<sup>2</sup> 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.

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**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 CO<sub>2</sub> is generally separated at the CO<sub>2</sub> 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?

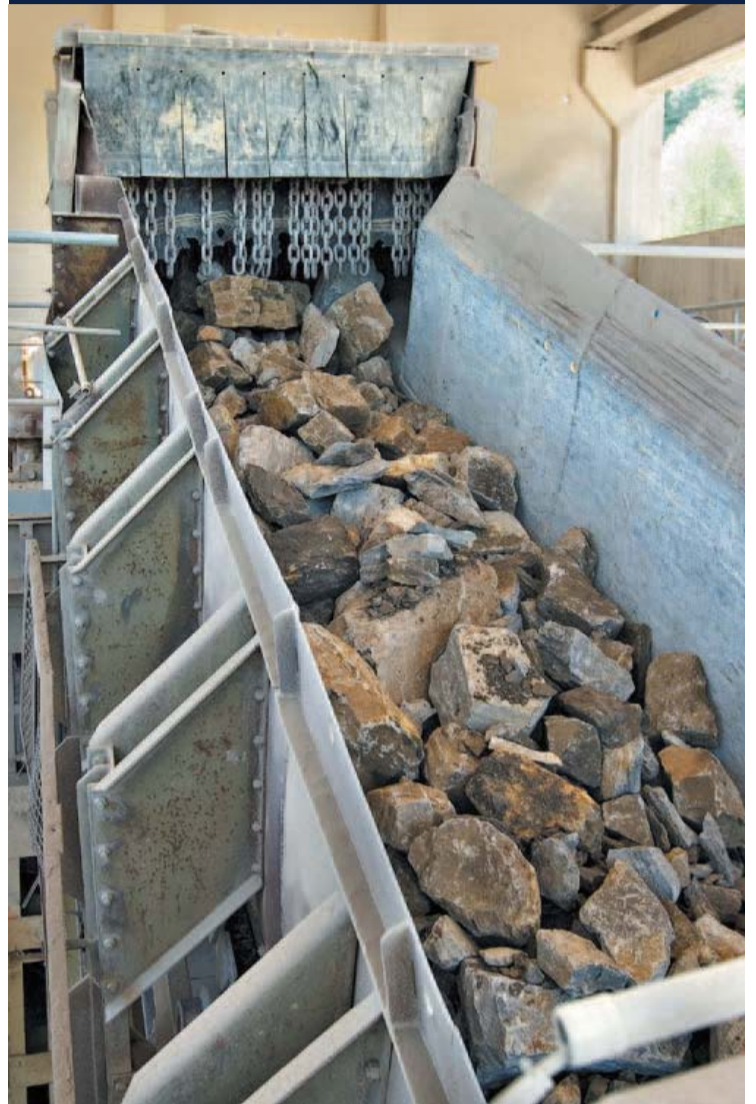
**Mr Nikhil Kamat from India asks a question:** What should the ammonia content and urea content in the treated waste water be so that it can be used as boiler feed water?

**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.

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.

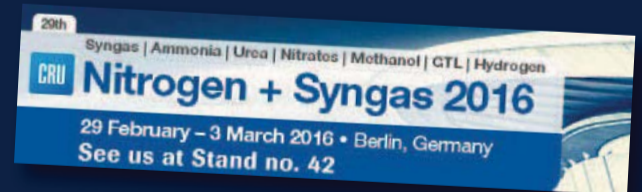
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# Low carbon syngas

In the wake of the Paris Agreement on climate change, *Nitrogen+Syngas* looks at potential ways of reducing the greenhouse impact of syngas and downstream product production.

The United Nations' Paris Agreement on Climate Change (COP21), finally signed in December last year after years of on-off wrangling, has focused global attention on the need to cut emissions of greenhouse gases (GHGs) in order to keep climate change to a 2% rise in global temperatures (compared to pre-industrial levels). A binding treaty signed by 195 countries, and the successor to the Kyoto Protocol that has dominated thinking on the subject since 1992, it will require stringent cuts in carbon emissions in order to meet 2050 targets. Signatory countries have all submitted binding climate change action plans, and it seems certain that carbon-intensive industries like ammonia and methanol production will start to see increasing pressure to lower emissions.

## Methanol production

The amount of GHGs emitted in producing methanol can vary hugely according to the syngas generation technology selected and the choice of electrical or steam turbine drive for compressors and pumps. Johnson Matthey has performed an analysis for a US-based methanol plant based on various options<sup>1</sup>;

- Steam-methane reforming (SMR)
- Steam-methane reforming with maximum CO<sub>2</sub> addition (SMR + CO<sub>2</sub>)
- Combined reforming, using steam-methane reforming and autothermal reforming (CR)
- Gas heated reforming and autothermal reforming (GHR + ATR)

The analysis assumes a typical US natural gas feed composition and a 5,000 t/d methanol plant. A comparison of the natural gas efficiency, electrical power consumption and carbon dioxide emissions for the four flowsheets is shown in Table 1. These flowsheets are based on driving all compressors and large pumps with steam-driven turbines and utilising imported electricity to drive the air cooler fans and smaller pumps only. This results in the min-

Table 1: GHG emissions from 5,000 t/d methanol plant based on minimal electrical import

Case	Overall gas efficiency, LHV (GJ/t)	Electricity use (MWh)	CO <sub>2</sub> e emissions (t/h)
SMR	32.6	5.0	92.8
SMR + CO <sub>2</sub>	31.6	5.0	144.9 (80.9)*
Combined reforming	30.8	3.6	71.7
GHR + ATR	31.0	4.5	74.0

\* = based on captured CO<sub>2</sub>

Source: Johnson Matthey

Table 2: GHG emissions from 5,000 t/d methanol plant based on maximum electrical import

Case	Overall gas efficiency, LHV (GJ/t)	Electricity use (MWh)	CO <sub>2</sub> e emissions (t/h)
SMR	32.4	13.4	90.4
SMR + CO <sub>2</sub>	31.4	12.9	142.8 (78.8)*
Combined reforming	30.7	8.3	70.9
GHR + ATR	25.5	90.5	13.9

\* = based on captured CO<sub>2</sub>

+ = ISBL plant only

Source: Johnson Matthey

imal electrical import to the inside battery limits (ISBL) plant without the addition of a turbo generator (the ISBL plant refers to the methanol unit only, and does not include utilities other than the air separation unit). For the case with carbon dioxide addition, two cases are shown; the figure in parenthesis shows the net emissions based on using captured CO<sub>2</sub> as a feedstock.

However, as an alternative option, it is also possible to minimise the amount of natural gas burnt in the auxiliary boiler by maximising the number of compressors that are driven by electric motors, allowing an improvement in the natural gas efficiency of the ISBL plant as well as reducing the carbon dioxide emissions. The values in Table 2 are based on maximising the import electricity while maintaining the minimum load on the auxiliary boiler. Of

course, if the electricity can be provided from a renewable source then this could be a better option from a GHG standpoint.

As can be seen, the carbon dioxide emissions in Table 1 move in line with the natural gas efficiency of the flowsheet, with the exception of the SMR + CO<sub>2</sub> flowsheet. This stands to reason because (aside from the CO<sub>2</sub> addition option), natural gas is the only carbon input into the plant, with methanol and CO<sub>2</sub> emissions the only output. Therefore any carbon in the natural gas not converted to methanol will eventually leave the plant as a carbon dioxide emission. The SMR + CO<sub>2</sub> flowsheet is the exception to this, as additional carbon is added to the process, which helps improve the natural gas efficiency at the expense of increasing the carbon dioxide emissions from the ISBL plant. The increase in CO<sub>2</sub> emissions for

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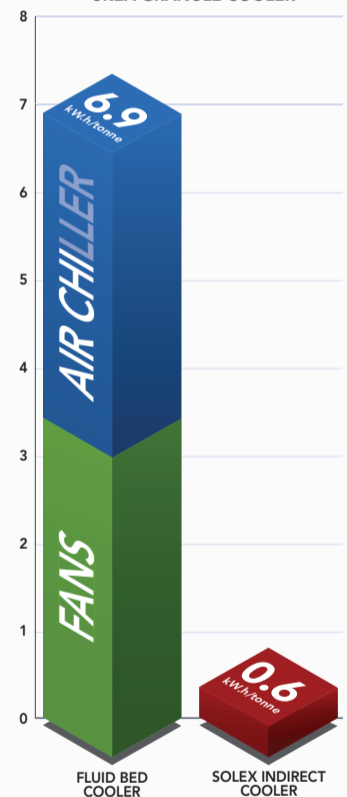
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Table 3: GREET numbers for minimum electrical import

Stage	SMR	SMR+CO <sub>2</sub>	CR	GHR+ATR
Natural gas to plant (gCO <sub>2</sub> e/MJ methanol)	13.0	12.6	12.3	12.4
Methanol plant (gCO <sub>2</sub> e/MJ methanol)	23.1	36.0 (20.1)	17.8	19.2
Methanol storage (gCO <sub>2</sub> e/MJ methanol)	1.3	1.3	1.3	1.3
<b>Sub total</b>	<b>37.4</b>	<b>49.9 (34.0)</b>	<b>31.4</b>	<b>32.9</b>
<b>Electricity</b>				
Typical N America (gCO <sub>2</sub> e/MJ methanol)	0.76	0.76	0.54	0.67
Renewable mix (gCO <sub>2</sub> e/MJ methanol)	0.005	0.005	0.003	0.004
<b>Total (N America mix)</b>	<b>38.1</b>	<b>50.7 (34.8)</b>	<b>32.0</b>	<b>33.6</b>
<b>Total (renewable mix)</b>	<b>37.4</b>	<b>49.9 (34.0)</b>	<b>31.4</b>	<b>32.9</b>

Source: Johnson Matthey

Table 4: GREET numbers for maximum electrical import

Stage	SMR	SMR+CO <sub>2</sub>	CR	GHR+ATR
Natural gas to plant (gCO <sub>2</sub> e/MJ methanol)	12.9	12.5	12.2	10.2
Methanol plant (gCO <sub>2</sub> e/MJ methanol)	22.5	35.5 (19.6)	17.6	3.5
Methanol storage (gCO <sub>2</sub> e/MJ methanol)	1.3	1.3	1.3	1.3
<b>Sub total</b>	<b>36.7</b>	<b>49.3 (33.4)</b>	<b>31.2</b>	<b>15.0</b>
<b>Electricity</b>				
Typical N America (gCO <sub>2</sub> e/MJ methanol)	2.03	1.95	1.23	13.7
Renewable mix (gCO <sub>2</sub> e/MJ methanol)	0.012	0.012	0.008	0.083
<b>Total (N America mix)</b>	<b>38.7</b>	<b>51.3 (35.4)</b>	<b>32.4</b>	<b>28.7</b>
<b>Total (renewable mix)</b>	<b>36.7</b>	<b>49.3 (33.4)</b>	<b>31.2</b>	<b>15.1</b>

Source: Johnson Matthey

the SMR + CO<sub>2</sub> flowsheet is due to both the increase in natural gas fuel required in the reformer because of the reduced lower heating value (LHV) of the methanol loop purge gas, as well as an increase in CO<sub>2</sub> concentration in the recycled fuel from the methanol loop and distillation.

Therefore, any CO<sub>2</sub> injection flowsheet aside, the better the natural gas efficiency of the ISBL plant, the lower the CO<sub>2</sub> emissions. If captured CO<sub>2</sub> is used as a feedstock to the ISBL plant for CO<sub>2</sub> injection flowsheets then Tables 1 and 2 show that the net CO<sub>2</sub> emissions fall back in line with this trend.

Comparing Tables 1 and 2 shows that for the SMR, SMR + CO<sub>2</sub> and CR flowsheets there is no significant scope to maximise the electrical import while maintaining the minimum auxiliary boiler load. The SMR, SMR + CO<sub>2</sub> and CR flowsheets all generate HP steam as a way of cooling the process

gas after reforming. This steam is a useful by-product of the cooling process because it can be used to power the turbines of the large compressors on the plant. In addition, all flowsheets have an auxiliary boiler, whose primary purpose is for start-up and shutdown. In normal operation the boiler is kept running but it has a minimum turndown and so this steam also has to be utilised within the ISBL plant. After all this steam has been consumed, the additional power requirements of the smaller compressors is minimal, and hence there is no real benefit in switching from steam turbine driven to motor driven compressors for reducing the ISBL plant emissions and improving the natural gas efficiency.

In contrast, the GHR + ATR flowsheet uses the high temperature process gas to provide heat for the reforming reaction in the GHR, which then allows all

the compressors and large pumps to be electrically driven if required. The ability to decouple the power requirement for the compressors and large pumps from the ISBL plant, and the fact that the GHR + ATR flowsheet does not contain a SMR, means that the carbon dioxide emissions of the ISBL plant can be reduced significantly for normal operation, as shown in Table 2.

### Impact of renewable electricity

Of course, the imported power means that the source of the carbon dioxide emissions generated by producing the electrical power is transferred from the ISBL plant to the existing power producers. When building a new methanol plant in the US, this is advantageous as the emissions required for a Title V environmental permit are only those for the new plant and do not include those for the existing producers supplying the import electricity. Therefore, in areas where greenhouse gas (GHG) emissions are restricted, the GHR + ATR flowsheet with imported power offers the best flowsheet for reducing GHG emissions for the ISBL plant and also for providing a natural gas efficient flowsheet.

Importing electricity allows the ISBL emissions to be reduced but it doesn't give a complete representation of the carbon intensity of producing methanol using the GHR + ATR process. For certain states in North America and Canada, for example California, there has been a drive to reduce the carbon intensity of the fuels they use and this has resulted in the implementation of legislation in California called the low carbon fuel standard (LCFS). This standard looks at the total carbon emissions of a fuel from well to wheel and so tries to capture the total carbon intensity of that fuel over its whole life cycle.

California uses software developed by the Argonne National Laboratory called GREET in order to calculate GHG emissions for its LCFS. Tables 3 and 4 use the GREET software to generate well to wheel product values for the four cases discussed above. Table 3 assumes the minimum electrical import into the ISBL plant, and Table 4 the maximum. As can be seen, each flowsheet also has two calculations for GHG emissions based on the emissions of the electricity supply used; a 'standard' US mix of supply, including 41% coal, 26% natural gas, 20% nuclear and 12% renewables and biomass, and a

'standard renewable' mix, assuming 67% hydroelectric, 4% geothermal power, 25% wind and 4% biomass-based generation.

The GHG emission values in Table 3 follow much the same pattern as Table 1. This is because for the minimum electrical import flowsheets, the contribution to the GHG emissions from the imported electrical power is minimal, and so the total emission figures are dominated by emissions from transporting the natural gas to the ISBL plant and from the ISBL plant itself.

However, the GREET GHG emission values in Table 4, for flowsheets with the maximum electrical import, show a very different trend. For the SMR, SMR + CO<sub>2</sub> and CR flowsheets, moving to the maximum electrical import actually increases the overall well-to-product GHG emissions compared to the values in Table 3 when using the typical North American electricity mix, and only a small reduction when using the renewable electricity mix. This is compared to the GHR + ATR flowsheet which shows a reduction in GHG emissions of 15% and 54% when using the typical North American electricity mix and the renewable electricity mix respectively.

The reason for the increase in GHG emissions for the SMR, SMR + CO<sub>2</sub> and CR flowsheets when using the typical North American electricity mix centres around the plant heat integration and utilisation of the steam from the auxiliary boiler. For these flowsheets the generation of HP steam in the reformed gas cooling train means that there is only sufficient heat remaining in the reformed gas to provide approximately 55% of the distillation duty, with the remaining duty provided by LP steam. There is therefore a large LP steam demand, which typically has been satisfied by using MP steam in back pressure turbines, with the LP steam header topped up by letting down a small amount of MP steam. This therefore maximises the amount of work performed by the MP steam. When, however, the compressors driven by these turbines are switched to motor driven, the LP steam demand remains the same and so the short fall in LP steam is made up by letting down more of the MP steam. This then results in the use of MP steam becoming less efficient, and so the GHG emissions for the combined ISBL plant and import electricity actually increase.

For the GHR + ATR flowsheet, the LP steam demand is small because all the distillation duty is provided by the reformed gas train cooling, so the flowsheet does not

Table 5: Well to wheel GHG emissions for various fuels

Fuel	Vehicle operation gCO <sub>2</sub> e/MJ	Well to product gCO <sub>2</sub> e/MJ	Total gCO <sub>2</sub> e/MJ
<b>Methanol M85+15% reformulated gasoline</b>			
SMR, N America elect import	26.6	36.7	63.2
SMR, Renewable import	26.6	35.0	61.5
SMR+CO <sub>2</sub> , N America elect import	26.6	47.3 (33.8)	73.9 (60.4)
SMR+CO <sub>2</sub> , Renewable import	26.6	45.7 (32.2)	72.3 (58.7)
CR, N America import	26.6	31.3	57.9
CR, Renewable import	26.6	30.3	56.8
GHR+ATR, N America import	26.6	28.1	54.7
GHR+ATR, Renewable import	26.6	16.6	43.1
<hr/>			
Reformulated gasoline, 10% ethanol	66.3	25.0	91.3
Low sulphur diesel	75.7	17.1	92.8
Compressed natural gas	57.6	18.6	76.2
Liquefied petroleum gas	64.7	12.5	77.2
Ethanol E85	12.6	57.7	70.4
Gaseous hydrogen	0.8	94.5	95.5
Fischer-Tropsch diesel	73.1	36.5	109.6
Electric vehicle (N America mix)	0	174.4	174.4

Source: Johnson Matthey

need to incorporate back pressure turbines to satisfy the LP steam demand. Therefore, switching the compressors from turbine to motor-driven does not mean additional MP steam has to be let down to the LP steam level and so removing the steam driven turbines has a direct impact on the load of the auxiliary boiler, in proportion to the increase in electrical load and hence allows a total reduction in emissions.

For the GHR + ATR flowsheet, running all the compressors, pumps and air coolers on imported electricity shows a modest saving on the GHG emissions if the supplied electricity is from the grid with a typical North American electricity mix. However, using a renewable energy source to provide the electrical import power to the plant has a significant impact on the GHG emissions, with the emissions over half that of the CR flowsheet, which has the second best emission figures. The GHR + ATR flowsheet is the only flowsheet that doesn't generate HP steam as a by-product of the process, allowing a large portion of the energy requirement of the ISBL plant to come from electricity import. This in turn allows a large portion of the energy required to make methanol to come from a renewable source.

### Low carbon fuel?

While just over half of the methanol market goes towards traditional chemical production like formaldehyde, acetic acid etc, the faster growing sectors of the methanol market are represented by demand for fuel uses, particularly blending into gasoline. In addition to calculating the well to product GHG emissions using GREET, it is also possible to go one step further and calculate the well to wheel value, which allows methanol as a fuel to be compared to all the other available transportation fuels. Table 5 shows the comparison between the methanol well to wheel carbon emissions and some of the other standard fuel types. What Table 5 shows is that methanol as a fuel has a lower carbon intensity than gasoline over its full life cycle, irrespective of which flowsheet is used to produce the methanol. It also highlights that methanol as a blendstock for gasoline is less carbon intense than using ethanol, unless non-captured CO<sub>2</sub> injection is used on the flowsheet.

The conclusion seems to be that due to the way HP steam is generated in the SMR, SMR + CO<sub>2</sub> and CR flowsheets it is not possible to easily incorporate renewable electrical energy into the process to

enable a reduction in carbon intensity of methanol. The heat integration in the GHR + ATR flowsheet allows the flexibility to significantly increase the electrical power input into the ISBL plant, however, which not only allows a large reduction in the GHG emissions from the ISBL plant but also allows a total reduction in the carbon intensity of the process over its entire life cycle and significantly so if the source of electricity is from renewable energy.

From well to wheel, methanol produced from natural gas provides a significant reduction in GHG emissions when compared to standard gasoline, and even when compared to ethanol, methanol shows a modest reduction in GHG emissions and emphasises why methanol is such a good supplement to gasoline fuel for the reduction of GHG emissions.

### Other 'low carbon' options

The Methanol Institute (MI) has commissioned a report on pathways to renewable methanol<sup>2</sup> which examined a number of ways of producing methanol, including gasification of municipal or industrial waste, gasification of biomass, and using hydrogen generated from renewable electricity to combine with captured carbon dioxide, as well as other options. Industrial wastes can be a very broad topic. In the Netherlands, BioMCN is currently producing approximately 66 million gallons per year (200,000 t/a) of methanol using by-product glycerine from biodiesel production as the feedstock. In Sweden, Chemrec operates a small plant producing methanol from black liquor, a by-product of forest waste, with onward conversion into demthyl ether (DME) for transportation fuel. Iceland's Carbon Recycling International uses electricity generated from geothermal energy to electrolyse water and generate hydrogen which is then combined with captured CO<sub>2</sub> to generate methanol. Varmlands Metanol in Sweden is a biomass gasification-based plant using forest waste. Finally, Blue Fuel Energy in Canada is aiming to use a combination of hydrogen from electrolysis (using hydro-electric power), recovered CO<sub>2</sub> and natural gas to drive methanol to gasoline (MTG) production. Table 6 shows the estimated reduction that can be achieved in GHG emissions on a well-to-wheel basis using these processes.

The primary pathways for producing some of these renewable forms of methanol all rely on waste feedstocks – waste biomass, municipal waste, industrial waste, industrial emissions, and existing sources

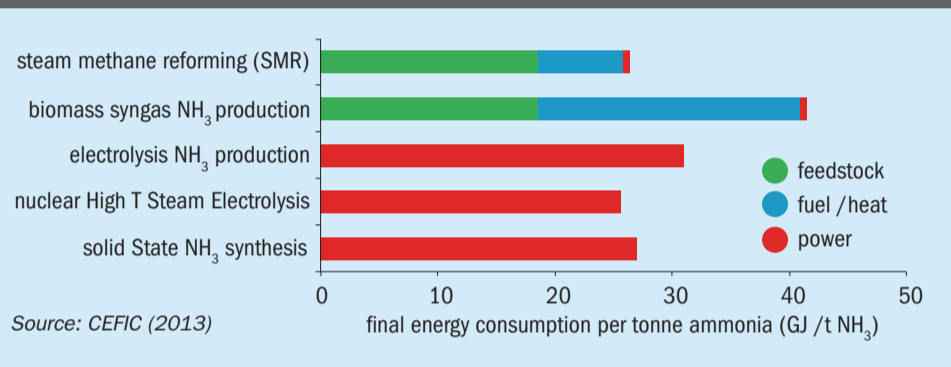
Table 6: Well to wheel GHG savings for renewable methanol technologies\*

Producer	Process	Reported reduction
BioMCN	Glycerine from biodiesel	78%
Blue Fuel Energy	Gas/renewable H <sub>2</sub> mix, recovered CO <sub>2</sub>	65-84%
CRI	Renewable H <sub>2</sub> , recovered CO <sub>2</sub>	85%
Chemrec	Gasified biomass (paper pulp waste)	95%
Varmlands Metanol	Gasified biomass (forestry waste)	80-90%

\* relative to conventional gasoline

Source: Methanol Institute

Fig 1: Final energy consumption of steam methane reforming compared to potential substitute technologies



Source: CEFIC (2013)

of CO<sub>2</sub>, and there is some question about the scalability of these feedstocks to the large-scale implementation that would be required to replace a methanol industry that currently stands at close to 80 million t/a. However, the MI report notes that in the US alone, the total potential biomass resource for biofuels is estimated to be as much as 1.6 billion dry tons per year by 2030, equivalent to 790 million t/a using presently available gasification technology. This does not however factor in the cost of gathering and processing the biomass, which has long been one of the big issues for biomass-based production, and clearly it works best when combined with existing biomass-based industries like paper manufacture. It could be that a much more energetic implementation of some form of carbon pricing might be required to make some of these routes commercially viable even considering virtually zero cost for feedstock at source.

### Ammonia

The foregoing has mostly been aimed at methanol production. For ammonia production the options for syngas generation are more limited. In particular, for example comparing the analysis conducted by Johnson Matthey, there is no scope for using CO<sub>2</sub> from captured sources unless a downstream step to urea production is also considered.

While most (55%) ammonia production goes to make urea, the remaining 45% of ammonia does not. Likewise the stoichiometric hydrogen requirement for ammonia production means that steam methane reforming remains the preferred syngas generation method even at large scales, and while newer plants tend to be much more efficient than older ones, with a typical energy use of 28GJ/tonne ammonia produced, compared to 35-40GJ/t for older plants, there is a theoretical maximum efficiency to the process of 18.6 GJ/tonne ammonia which cannot be exceeded. At a best guess, future improvements might bring energy consumption down to 24-25GJ/t ammonia.

The Ecofys consultancy prepared a report<sup>3</sup> for CEFIC in 2013 which looked at the potential for increasing the efficiency of ammonia production. In addition to options for improving heat integration, process control, motor systems etc, it also looked ahead towards 2050 at potential alternative methods of production, and the results are shown Figure 1. The baseline figure comes from assuming continued improvement in SMR technology down to 26GJ/tonne ammonia produced, with an option for carbon capture and storage. Other options are biomass gasification, ammonia production using hydrogen generated by electrolysis – still practised at a couple of older plants around the world – and some novel



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technologies being investigated by the US Department of Energy, including a high temperature nuclear reaction and the solid state ammonia synthesis technology, which uses only electricity, with a target use of 7-8 MWh/t ammonia. As can be seen, the report leans towards SMR continuing to be the most efficient method, possibly with carbon capture and storage, although that technology too remains unproven on a large scale at the moment. And of course, no attempt was made to calculate relative costs, which for current biomass and electrolysis-based plants remain far higher than conventional SMR.

### A way forward?

Remarkable new technologies aside, at the moment the main options that the industry seems to be proposing for lowering carbon emissions seem to be threefold:

Firstly; gasification of biomass or other waste streams that might otherwise be incinerated. The difficulty with this is mainly one of the energy density of the biomass and hence the amount of gathering and pre-processing that must be performed beforehand, and of course the costs associated with it. The amount of biomass required to replace a typical world-scale ammonia or methanol plant would be larger than that used in even the largest biomass-based power plant today, and its replicability on a large scale remains very much open to question.

Secondly, carbon capture and storage (CCS). This seems to have been a magic wand waved at a lot of GHG-generating processes, from energy generation to energy-hungry industrial processes like cement production, but aside from reinjection for enhanced oil recovery (EOR), actual uses of CCS are currently still few and far between, and carbon dioxide's corrosive nature could also make this an expensive option. EOR also depends upon there being a convenient reservoir to use, which limits its applicability. As with biomass, there are certainly niche uses for this, but it is hard to imagine CCS being used for every single ammonia and methanol plant in the world.

The third option seems to involve use of renewable electricity, and here there may be light at the end of the tunnel. On the one hand, as it is rolled out on a larger and larger scale, so the cost of renewable energy is continuing to fall, and solar energy in particular is now roughly at grid parity in western Europe. And on the other hand, wind, wave and solar power represent very variable power generation capacity, unsuited to providing the kind of base-load power that modern economies require. On days when the wind is blowing, Denmark can export power to the rest of Europe, but on days when it is not, it is a major net importer from coal, nuclear and gas-based capacity in Germany and France. Generating hydrogen for use in ammonia or methanol production could be a productive use of this capacity.

Probably the industry will need to develop smaller scale, more modular production to make the best use of these lower carbon technologies. There are signs that this is already happening. But if we are to meet the ambitious 2050 targets embedded in the Paris Agreement, we could be looking at a very different industry in 35 years time. ■

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Left: EuroChem's Novomoskovsky plant.

# New nitrogen in the east

A look at plans for new nitrogen capacity in Russia and the other countries of central Asia.

**W**hile there has been a slowdown in nitrogen capacity building in the Middle East, and India has found it hard to get new plants started, there are still some bright spots for new construction. North America has grabbed many of the headlines, with a plethora of new projects on the back of cheap shale gas availability, alongside ready-made domestic demand, but Central Asia and Russia are also seeing major new investment. Integer Research calculates that nearly 3 million t/a of ammonia capacity expansion projects are expected to come on stream in Russia in the period 2016-2019, including EuroChem's new nitrogen plants at Kingisepp and Nevinnomysk, and a new ammonia plant for PhosAgro at Cherepovets. JSC Acron is also building ammonia capacity due to start up in 2016. The continued depreciation of the rouble continues to make capacity expansion plans look globally competitive even at a time of overcapacity and low product prices.

## Economies

The economies of the region had been recovering from the banking crisis of 2008, but in the past couple of years have faced some major headwinds. For Russia, the fallout over the dispute in Ukraine continues to cast a shadow, as sanctions imposed by the US and Europe in 2014 continue to bite. As well as restrictions on banking and money transfers, several major oil and gas companies lost access to US and European technology and services, restricting the development of new oil and gas reserves and pipelines for companies like Lukoil, Gazprom, Transneft etc.

But falling oil and gas prices have themselves also had a significant effect, being responsible as they are for much of the GDP and especially foreign exchange earnings of resource-rich countries like Russia. On the other hand, the crash in the rouble caused by these two pressures has also

been a major bonus for Russian nitrogen producers. Russian fertilizer producers were already quite competitive globally because of low feedstock costs and vertical integration, but the depreciation of the rouble has further strengthened this competitiveness, boosting profit margins and cash flow, and of course the attractiveness of investment.

Meanwhile, US Secretary of State John Kerry said recently at Davos that he foresaw the potential for major sanctions against Russia to be lifted over the coming months. The US and Russia have seen some common ground recently on the fight against the Islamic State in Syria and Iraq and nuclear sanctions on Iran, and with a ceasefire in place in Ukraine between the government and Russian-backed rebels there are hopes for an agreement there too.

## Natural gas

Oil and natural gas are the mainstays of the economies of several of the countries of the region. Gas production in the region is dominated by Russia, but Uzbekistan, Turkmenistan and Kazakhstan are also major producers, and Ukraine and Azerbaijan also have significant gas production. As gas is the key feedstock for nitrogen production, this has helped the region become one of the major global centres for ammonia and urea production.

## Russia

BP's annual review of natural gas shows that Russia's proved natural gas reserves stood at 32.6 trillion m<sup>3</sup> by the start of 2015. This represents 17.4% of the world's natural gas reserves. However, this is a significant downward revision from the figure a few years ago, and places Russia in unaccustomed second place in terms of reserves, behind Iran. Production was 578 billion cubic metres (bcm) in 2014, a 4% fall on the previous year. More instructive is that this figure is almost exactly the same as for 10 years ago, and in general Russian gas production has stagnated, in a period when global gas production has risen overall by 30%, and in, for example Qatar, has more than quadrupled.

Russia remains dependant on natural gas for 55% of its energy. In spite of some

attempts to develop private gas production in Russia, state producer Gazprom is still responsible for 72% of Russia's natural gas production, owns and operates the entirety of the country's natural gas pipeline network, and has a monopoly on gas exports. The company represents over 20% of Russian tax revenues.

Gazprom faces challenges in spite of its size and significance. Facing issues in European markets over oil index-linked pricing and the Ukraine dispute, it had embarked upon a much-vaunted 'pivot to Asia' in order to supply gas to the hungry Chinese market. However, for the moment Europe remains its main customer. The 'Power of Siberia' pipeline to northeastern China is unlikely be

completed before 2020, and no agreement has yet been reached on a second pipeline either from western Siberia or from Sakhalin Island. Meanwhile Russia's LNG plans have also been receding as global gas market conditions have worsened, with Novatek's Yamal LNG project now the only Russian project likely to produce gas before the end of the decade. With Europe diversifying its own gas supply away from its prior dependence on Russian gas, Gazprom may have to accept more changes from its previous insistence on oil indexation, as it did in March 2015.

On the nitrogen side, Russia remains the dominant producer in the region. Progressive industry consolidation in Russia

has seen more than half of production fall into the hands of three companies; EuroChem, UralChem and TogliattiAzot. Together with JSC Acron, Sibur, PhosAgro and Minudobreniya Rossoh, these seven companies now produce 92% of ammonia in Russia.

### Kazakhstan

Kazakhstan is the third largest holder of gas reserves in the region, but most of it is associated gas in the large Tengiz and Karachaganak oil fields. Actual volumes of gas produced have tripled in a decade, but two thirds of the gas is immediately re-injected into oil wells to keep the oil

Fig 1: Nitrogen capacity in Russia and the CIS



<p><b>Russia</b></p> <ul style="list-style-type: none"> <li>1. EuroChem (Nevnyomyssk)</li> <li>2. EuroChem (Novomoskovsk)</li> <li>3. UralChem (Berezniki)</li> <li>4. UralChem (Perm)</li> <li>5. UralChem (Kirovo-Chepetsk)</li> <li>6. Sibur (Kemerovo)</li> <li>7. TogliattiAzot (Togliatti)</li> <li>8. KuibyshevAzot (Samara)</li> <li>9. JSC Acron (Novgorod)</li> <li>10. JSC Acron (Dorogobuzh)</li> <li>11. Minudobreniya Rossoh (Rossoh)</li> </ul>	<ul style="list-style-type: none"> <li>12. PhosAgro (Cherepovets)</li> <li>13. AO Salavat (Salavat)</li> <li>14. JSC Angarsk (Angarsk)</li> </ul> <p><b>Ukraine</b></p> <ul style="list-style-type: none"> <li>15. Odessa Port Plant (Odessa)</li> <li>16. Concern Stinol (Gorlovka)</li> <li>17. DniproAzot (Dniprodzerzhinsk)</li> <li>18. RovnoAzot (Rovno)</li> <li>19. Severodonetsk Azot (Severodonetsk)</li> <li>20. OJSC Azot (Cherkassy)</li> </ul>	<p><b>Kazakhstan</b></p> <ul style="list-style-type: none"> <li>21. KazAzot (Aktau)</li> </ul> <p><b>Turkmenistan</b></p> <ul style="list-style-type: none"> <li>22. Turkmenchimiya State Concern (Tedzhen)</li> <li>23. MaryAzot (Mary)</li> </ul> <p><b>Uzbekistan</b></p> <ul style="list-style-type: none"> <li>24. UzKimyoSanoat (Navoi)</li> <li>25. UzKimyoSanoat (Ferghana)</li> <li>26. Maxam-Chirchik (Chirchik)</li> </ul>	<p><b>Belarus</b></p> <ul style="list-style-type: none"> <li>27. GrodnoAzot (Grodno)</li> </ul> <p><b>Tajikistan</b></p> <ul style="list-style-type: none"> <li>28. TajikAzot (Sarband)</li> </ul> <p><b>New plants</b></p> <ul style="list-style-type: none"> <li>29. EuroChem (Kingisepp)</li> <li>30. EuroChem (Nevnyomyssk)</li> <li>31. JSC Acron (Novgovod)</li> <li>32. PhosAgro (Cherepovets)</li> <li>33. Turkmenchimiya (Garabogaz)</li> <li>34. Uzkimyo Sanoat (Navoi)</li> </ul>
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flowing. The reason for this is mainly lack of gas processing capacity at the Karachaganak gas processing plant, and considerable volumes of gas actually cross the border to the Russian Orenburg gas plant. As Kazakhstan's gas is often highly sour, it requires considerable processing to turn it into usable sales gas.

Last year the major oil fields were due to be joined by another, super-giant field at Kashagan. This huge offshore oilfield – the largest oil deposit outside the Middle East and the fifth largest in the world – has been under development for many years and run several times over budget, but it finally began production in 2013 year. Almost immediately, however, the project was forced to shut down due to sour gas leaking from the pipeline connecting the wellheads to the onshore processing plant. An inspection revealed extensive corrosion and the entire length of pipe is now in the process of being replaced. Production may restart later this year. Phase one, however, involves large scale reinjection of the gas (estimated at around 80% of production), and the country's overall gas output may not increase drastically.

## Turkmenistan

Turkmenistan has seen its estimated gas reserves leap in recent years, from 2.3 tcm in 2004 to 17.5 tcm in 2014, making it the second largest regional holder of reserves (after Russia) and the fourth largest in the world. The country had a hard time monetising this bonanza however, due to having little domestic use for it and being dependent upon access to Russian pipelines for export. The country has however attracted considerable interest and indeed investment cash from countries such as India and China, both of them desperate for additional sources of natural gas. Gas production and processing is ramping up at the huge Galkynysh facility, which began production in 2013 and aims to be producing 20 bcm per year in 2020.

On the export side, China has financed the Central Asia – China pipeline, which connects Turkmenistan and China via Uzbekistan and Kazakhstan. This pipeline consists of three lines; Line C with an annual capacity of 25 bcm went on stream in May 2014. Lines A, B and C between them have a total capacity of 55 bcm per year of natural gas. Turkmenistan plans to increase this to 80 bcm per year by 2020 by constructing Line D.

Table 1: Ammonia production, consumption and exports, Russia and CIS countries, 2014 ('000 t/a)

	Production	Consumption	Imports	Exports
Belarus	873	919	57	11
Russia	12,029	9,039	0	2,990
Kazakhstan	90	103	13	0
Turkmenistan	400	400	0	0
Uzbekistan	1,207	1,195	0	12
Ukraine	2,454	1,881	0	573
<b>Total</b>	<b>17,053</b>	<b>13,537</b>	<b>70</b>	<b>3,586</b>

Source: IFA

There are also plans for a 33bcm line – TAPI – the Turkmenistan-Afghanistan-Pakistan-India pipeline, to run from Galkynysh across Afghanistan and Pakistan to India, but although the route has been surveyed and gas offtake and supply agreements signed, the complex politics and security situation of the region make many sceptical about whether this will ever get built.

Turkmenistan has two nitrogen manufacturing sites, run by Turkmenhimiya State Concern. At Tedzhen, 200,000 t/a of ammonia and 350,000 t/a of urea capacity was completed in 2004, but the largest complex is MaryAzot at Mary, where 400,000 t/a of ammonia and 635,000 t/a of urea are already operational. A new plant is also under construction at Garabogaz (see below).

## Uzbekistan

Uzbekistan also has considerable gas reserves – of a similar order to Kazakhstan's – and it is the third largest regional producer after Russia and Turkmenistan. Considerable volumes are exported via the Central Asia-China pipeline, but considerable volumes of gas are still flared in the country, and there is room for extra production. Like Russia. Uzbekistan has an ageing gas infrastructure and has seen gas production relatively stagnant over the past decade.

## Ukraine

Ukraine has similar sized reserves, but its gas production, at 18.6 bcm per year, runs considerably lower than the country's overall gas demand of 38.4 bcm. Because of this, Ukraine has traditionally taken gas from Russia while acting as a transit country to Europe. While this traditionally gave

Russia considerable leverage over Ukraine, since Russia's occupation of the Crimea, Ukraine has found its own counter in cutting off energy supplies to the peninsula. The energy relationship remains extremely fractious – Ukraine halted gas purchases from Gazprom in July 2015 after Ukraine and Russia failed to reach a deal on gas deliveries for the third quarter of the year. Ukraine has since been importing gas via reverse flows from the EU, mainly from Slovakia, and running down its storage, but recently there seems to have been another breakthrough on gas pricing with Gazprom and the taps may be turned on again. All of this makes life extremely difficult for ammonia producers within Ukraine.

Ukraine's nitrogen industry is mostly in the hands of Group DF (via its Ostchem holding company), which owns four of the six producing locations, at Rivne, Cherkassy, Severodonetsk and Stirol/Gorlovka. It also markets product from the state-owned Odessa Port Plant (OPZ) at Yuzhnyy. The final producer, DneproAzot, is leased to fellow Privat Group company and gas supplier Ukrnafta. Most of these plants have avoided direct consequences of the fighting, but the two largest, Severodonetsk and Stirol are both in the east of the country and near to the combat zone.

## New production

In Russia, EuroChem has the most ambitious expansion programme, intending to build four new nitrogen plants over the next ten years, three in Russia and one near Zhanatas in Kazakhstan (a fifth in Louisiana, USA seems to have been shelved for now). Maire Tecnimont has been invited to submit EPC proposals for all five of the plants. Work on the first two is under way; an ammonia-urea facility in Nevinnomysk

with a capacity of 2,700 t/d (890,000 t/a) of ammonia and 3,500 t/d of urea, and an ammonia plant on the Gulf of Finland at Kingisepp near St Petersburg with a further 2,700 t/d of ammonia, which would also include an additional project to integrate urea production. The EPC contracts were signed last year and the two plants are due for completion in 2018. KBR is providing the technology for the Kingisepp ammonia plant, where construction began in 3Q 2015.

JSC Acron is also building a fourth ammonia plant, due to start up in 2016 at its Novgorod side. The 700,000 t/a unit is using Haldor Topsoe technology, and will increase the group's ammonia capacity by 65%. Acron says that the plant will be more efficient in terms of gas consumption, enhancing fertilizer output at its existing plants, and providing a base for future expansion. The plant will also have access to the BCT port terminal to export ammonia.

In 2013, PhosAgro began construction of a new, more energy efficient ammonia plant with a capacity of 760,000 t/a at its Cherepovets site. Mitsubishi Heavy Industries is building the plant, using Haldor

Topsoe technology, with completion due for 2017. The plant will allow the company to become self-sufficient in ammonia and allow for future downstream production, according to the company.

Uralchem is expanding production via revamps. A revamp was recently completed at the Kirovo-Chepetsk ammonia plant which took capacity to 1,750 t/d, and Stamicarbon is working on increasing urea production at Uralchem's Perm site by 40% (250,000 t/a) by 2019 using *Urea2000plus* technology. Togliatti Azot is also revamping all seven of its ammonia plants.

In Turkmenistan, state concern Turkmenkhimiya is building a new ammonia-urea unit at Garabogaz, with 2,000 t/d of ammonia and 3,500 t/d of urea, with completion set for 2018. HaldorTopsoe and Saipem are the technology suppliers here, with UFT providing the urea granulation plant.

Finally in Uzbekistan, Navoiyazot, the chemical production arm of state-owned Uzkimyosanoat, has awarded the EPC contract for its new gas-based ammonia-urea fertilizer plant at Navoiy to Mitsubishi Heavy Industries (MHI) and Mitsubishi Corporation.

The plant will produce 2,000 t/d of ammonia and 1,750 t/d of granulated urea, with a surplus of around 1,000 t/d of ammonia.

### Impact of new capacity

Table 1 shows the region's production and consumption of ammonia by country. Russia operates over two thirds of all ammonia capacity, and its nearest rival Ukraine has seen production cut back due to the conflict and gas shortages and costs. Ukraine's production in 2014 was only about 60% of its normal annual output. The new plants being built, together with upgrades and revamps, will add 3.5 million t/a to Russian ammonia capacity by 2018, about half of it with associated downstream urea capacity, but much destined for the merchant market. Another 1.5 million t/a is under development in Turkmenistan and Uzbekistan, 80% of it with downstream urea capacity. Taken together, this 5 million tonnes of ammonia is the largest capacity addition outside of China, and promises to more than make up for losses from Ukrainian production. For the moment, the cheap rouble is making regional capacity here extremely competitive. ■



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**N**ow in its 29th year, the Nitrogen+Syngas Conference returns to Berlin's Estrel Centre this February to, as usual, explore the impacts of key market trends, feedstock outlooks, project updates and supply and demand forecasts, as well as showcasing more than 40 technical papers covering the nitrogen industry from syngas generation through to finished fertilizers.

There is a subdued tone to the industry, as product prices have fallen to a global cost floor set by Chinese urea producers. This in turn has been due to the recent nitrogen investment boom which has oversupplied the market and depressed prices. However, nitrogen producers in the US, Europe and Russia are all enjoying lower production costs at the same time as low oil prices feed through into natural gas markets, with costs down 15-20% in Europe and 30% in Russia, where the weak rouble has provided a remarkable boost to competitiveness.

There is further uncertainty on the horizon: economic output from the emerging markets that have driven nitrogen consumption growth over the last decade is slowing, and the market is now waiting to see what the decelerating Chinese and Indian economies, as well as recessions in Russia and Brazil, have in store for nitrogen demand in 2016.

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# Nitrogen+Syngas 2016

## Nitrogen+Syngas 2016 Conference programme

### Monday 29 February

- UreaKnowHow.com pre-conference workshop

### Tuesday 1 March

#### COMMERCIAL & MARKET OUTLOOKS

- **Global gas outlook**  
Patrick Heather, Senior Research Fellow, Oxford Institute for Energy Studies
- **Global nitrogen outlook**  
Alistair Wallace, Principal Consultant, CRU
- **Producer case study to be confirmed**
- **Panel discussion: Challenges and opportunities in Iran**  
Stephen Zwart, Licensing Manager, Stamicarbon
- **Freight market outlook**  
Christian Rynning, Bery Maritime

#### AMMONIA CATALYSTS AND OPERATIONS

- **Breaking frontiers in ammonia plant operation**  
Stephanie Michelle King, Haldor Topsøe A/S
- **Catalytic solutions to improve efficiency; making more for less**  
Matt Cousins; John Brightling, Johnson Matthey
- **Small-scale ammonia production to minimise transport risk and cost**  
Dr. Bernd Keil, ThyssenKrupp Industrial Solutions AG

#### AMMONIA REVAMP STRATEGIES

- **Reinvigorate your plant**  
John Pach, Johnson Matthey
- **Exergy efficiency improvements in ammonia production by combined use of Clariant wustite based catalyst AMOMAX-10 and Casale reactor design**  
Filippo Pellacani, Clariant; Ermanno Filippo, Casale
- **Make your vintage plant modern and profitable**  
Sergio Panza, Casale SA

### Wednesday 2 March

#### REFORMER MANAGEMENT AND PERFORMANCE

- **Steam reformer overheating: absolute protection now available**  
Dan Barnett, BD Energy Systems
- **Thermal cycling damage in reformer tubes**  
Daniel Drabble; Charles Thomas, Quest Integrity NZL Ltd., Peter Tait; Baher El Shaikh, Methanex
- **Operating at peak performance with Topsøe reformer management**  
Kim Braad Carlsen, Haldor Topsøe A/S

#### TURBOMACHINERY

- **Engro's 50 Years experience of operating, maintaining & uprating turbo machines**  
Haseeb Bukhari, Engro Fertilizers
- **Increase of production and energy savings on ammonia plants based on total replacement of turbomachinery equipment**  
Petr Polak; Vladislav Polednik, Siemens
- **Up-to-date approach to modernisation of high-speed steam turbines in ammonia and methanol production lines**  
Robert Werner, Alstom Power Sp. z o.o.

**Nitrogen+Syngas 339** | January-February 2016

Nitrogen+Syngas 2016 Conference programme continued

**COMPRESSORS**

- **Significant reduction of the ammonia prime cost and operational specific energy consumption of the synthesis-gas centrifugal compressor**  
Aleksandr Liubimov, Entechmach RPC LLC
- **Air compressor and related equipment corrosion in ammonia plants: A hidden problem**  
Alireza Orooji; S.Sajjad Hosseininia, Pardis Petrochemical Company
- **Modernisation of synthesis gas compressor drive steam turbines in fertilizer plants using cutting edge technologies**  
Tomoyuki Nishikawa; Norihisa Wada, Mitsubishi Heavy Industries Compressor Corporation (MCO)

**EMISSIONS REDUCTION AND SYNGAS OPTIMISATION**

- **Implementation to flue gas recycle in steam reformer furnaces: From design to operation**  
Bernd Breuckner, ThyssenKrupp Industrial Solutions AG  
Paul Tawiah, Christopher Pigott, Shell Canada
- **Achieving ultra-low NOx emissions in methanol downfired reformer applications**  
Rex Isaacs; Ryan Roberts, Zeeco, Inc
- **Modelling and optimising syngas production**  
Benjamín Monge, DVA Global Energy Services

**NEW DEVELOPMENTS IN UREA AND MELAMINE PRODUCTION**

- **UFC manufacture for urea production**  
John Pach, Johnson Matthey
- **An approach to innovation: UFT and Shell Thiogro sulphur enhanced urea**  
Rafael Garcia; Charles Ingoldsby; Cyrille Allais, Shell Thiogro Technologies, Shell Canada Services. Harald Franzrahe, UHDE Fertilizer Technology B.V.
- **Seamless integration of melamine plant into a nitrogen based fertilizer complex**  
Alberto Di Amicis, Eurotecnica

**UREA OPERATIONS**

- **Maximum load test at a large scale urea plant in the Netherlands**  
Eelco Mostert, Stamicarbon
- **The Snamprogetti™ SuperCups Solution**  
Ugo Avagliano, Saipem SpA
- **Engro 40 years' experience of total recycle Urea-1 Plant**  
Abdur Rehman Choudhary, Engro Fertilizers

**AMMONIA AND UREA INTEGRATION**

- **Improved economics of ammonia-urea fertilizer plants via process integration**
- **Dr. Yevgeny Makhynya, ThyssenKrupp Industrial Solutions AG**
- **Ammonia and urea integration**
- **Improved economics of ammonia-urea fertilizer plants via process integration**  
Dr. Yevgeny Makhynya, ThyssenKrupp Industrial Solutions AG
- **Integrated approaches to improve the overall profitability of fertilizer complexes: A review of options available and experience gained**  
Ermanno Filippi, Casale SA
- **Commissioning experiences on ammonia and urea plant complexes**  
Mümin Hacimusalar, Independent consultant

**FERTILIZER FINISHING**

- **Adopting new technology to improve product quality**  
Khurram Saleem, Fauji Fertilizer Company Limited
- **Preparation of synthetic granulated ammonium sulphate**  
Nenad Zečević, Petrokemija d.d.
- **NAK Azot achieves world-class granulated calcium ammonium nitrate**  
Igor Makarenko, Solex Thermal Science Inc.

**Thursday 3 March**

**CO<sub>2</sub> REMOVAL AND SYNGAS OPERATIONS**

- **The art and science of CO<sub>2</sub> recovery at low operating pressures**  
Syed Ali Raza Sani; Muhammad Mushfiq Hussain, Engro Fertilizers Limited
- **Reducing CO<sub>2</sub> slip from the syngas unit of an ammonia plant**  
Ralph Weiland, Optimized Gas Treating, Inc., Eddy Cooper, Agrium Carseland Nitrogen Operations
- **Milling of pre-reformer catalyst and incident investigation/troubleshooting**  
Abdul Jabbar, FatimaFertilizer
- **Improved uniformity of flue gas flow in downfired steam methane reforming utilising the StaBlox reformer tunnel system**  
Joseph Quintiliani, Blasch Precision Ceramics Inc., Joe Price, BD Energy Systems

**ENERGY EFFICIENCY**

- **Hydrogen for the IOCL Paradip Refinery: Experience from design to commissioning**  
Dr. Nicolas Diaz, ThyssenKrupp Industrial Solutions AG  
Jamal Ferguson; Troy Raybold, Praxair Inc.
- **Applying Six Sigma techniques on neglected areas to optimise ammonia plant energy efficiency**  
Arsalan Naeem; Ali Javed Akhter; Farooq M. Saleem, Engro Fertilizers Limited
- **Results from the application of a pressure energy recovery turbocharger-based hydraulic system in an ammonia plant**  
Max Shirazi, Energy Recovery

**NITRIC ACID**

- **Casale and Chemoproject Nitrogen collaborate to build a new nitric acid plant: A project overview**  
Iacopo Cerea, Casale Group
- **Service options for colourless start-up and shutdowns of a nitric acid plant**  
Dr. Paul Kern, ThyssenKrupp Industrial Solutions AG
- **MPAC's first anniversary – and already a success story**  
Christian Goerens, Umicore AG & Co. KG (Platinum Engineered Materials)
- **Bimetallic tubes for nitric acid applications**  
Daniel Gullberg, Sandvik Materials Technology

**ENSURING SAFE PLANT OPERATIONS**

- **Ammonia synthesis converter reaction loss followed by fire incident**  
Saqib Raouf; Muhammad Hashim; Abdul Ghani; Safdar Javed; Muhammad Imram; Farahan Ahmed, Fatima Fertilizer Company
- **Best practices of HSE to achieve ammonia/urea plants T/A-2015 objective "No Injury And No Fire"**  
Mubashar Butt, Fauji Fertilizer Company Limited
- **Return of experience on the efficacy and reliability of fibre optic ammonia leakage detection systems**  
Daniele Inaudi; Roberto Walder, Smartec SA

**PLENARY SESSION – INCIDENT ANALYSIS**

- **Failure due to hydrogen embrittlement in steels**  
Ahad Ahmad Satti, Saudi Arabian Fertilizer Company
- **Foaming of Catacarb CO<sub>2</sub> removal system leads to methanator runaway reaction and expander fire incident**  
Muhammad Hashim; Abdul Ghani, Fatima Fertilizer Company
- **Failure of outlet pipeline of the reactor in urea plant**  
Mohamad Ebrahim, Abu Qir Fertilizer Company

# Getting the merger bug



Last year saw the largest number of corporate mergers and acquisitions since 2007. While on the nitrogen side the news has been dominated by the CF Industries-OCI merger, a number of other key deals were also done or are in the offing.

According to Deloitte’s mergers and acquisitions (M&A) index, 2015 saw the largest number of company takeovers since 2007, with a total value of \$4 trillion. And more than \$1 trillion worth of these were cross-border deals, of which a third were transatlantic partnerships between North American and European companies, while there have also increasingly been deals between Asian and European companies, led by China and Japan.

The chemical, petrochemical and fertilizer industries were no exception to this. Years of high commodity prices have left many organisations with large bank balances. The oil industry perhaps has some of the largest chequebooks – according to Bloomberg, ExxonMobil tops a list of the six largest publicly traded oil firms in terms of available stock and cash for acquisitions, with \$320 billion available, while Chevron and BP follow with \$65 billion and \$53 billion in stock and cash available, respectively.

Large bank balances are joined by cheap debt due to low interest rates (and an expectation that these may begin to rise soon), while a falling market offers opportunities for rationalisation and a need to retrench in the expectation of falling commodity prices. Some very large names have been involved in the M&A game recently. Shell is looking towards

a \$47 billion takeover of BG Group, while the Dow-DuPont deal could be worth \$130 billion – the largest deal in the chemical industry ever, dwarfing the previous record, AkzoNobel’s \$17 billion acquisition of ICI in 2008.

Dow and DuPont’s merger will be followed by a three-way split, into agriculture, speciality chemicals, and materials science. The agriculture segment will be worth \$18 billion based on 2014 sales, according to IHS. In their commentary on the deal, they noted: “The combination of Dow and DuPont’s agricultural businesses likely presages further consolidation in the agriculture segment. The cyclical downturn in agriculture has made deals in agriculture all but inevitable – a sentiment confirmed by nearly all senior executives at the big-six agricultural chemical firms. Until this DowDuPont was announced, the top ranks of the big six in seeds and crop protection chemicals have remained unchanged since Novartis and Zeneca combined their agriculture businesses to form Syngenta in 2000, although significant consolidation has occurred in recent years among tier-2 producers, who typically focus on crop-protection chemicals.”

Deepak Karthikeyan of Frost & Sullivan argued that “the cost advantages that can be achieved in the agro business will be significant for the combined entity...

[which] will surely become a formidable competition to the likes of companies like Monsanto.”

## Nitrogen mergers

The largest M&A news in the nitrogen sector last year was undoubtedly the \$8 billion proposed merger between US-based CF Industries and Dutch resident OCI Nitrogen, which is due to close in the middle of this year. The move came after Norway’s Yara unsuccessfully tried to buy CF Industries in 2014. CF Industries, previously virtually exclusively North American-based, will gain access to OCI’s global distribution network, across Europe and via its Egyptian presence, while OCI, previously with only a toehold in the US via the Beaumont, Texas ammonia/methanol plant, but now with its own new plant under construction in Iowa, will gain valuable market connection in the US market. CF Industries had previously made a move into Europe by buying up the remaining 50% in GrowHow UK, and acquiring two more nitrogen facilities in Britain. According to the two companies, they will also be able to gain “process expertise and access to existing critical spare parts inventory pool reduce capital requirements with increased production” and “reducing costs and optimising product mix offerings for our customers in the United Kingdom



and Europe through coordination between the Geleen, Netherlands, facility and our UK facilities; being able to more efficiently serve the global market from our enhanced production footprint.”

CF and OCI also between them have the three largest new project developments in the US via the expansions at Wever, Donaldsonville and Port Neal, and following the completion of these plants the merged entity will come to dominate the North American UAN market, claiming a total capacity of 8.7 million t/a, or around 60% of US UAN capacity. Given that the US imports total 3.5 million t/a and the new plants will supply 80% of that, it will place the CF-OCI company

Slightly smaller in the scheme of things was the \$333 million buyout by Coffeyville Resources (CVR) of Rentech Nitrogen. CVR runs a petroleum coke-based nitrogen complex at Coffeyville, Kansas, and Rentech Nitrogen the former Agrium plant at East Dubuque, Illinois. Their merger claims 1.2 million t/a of the US UAN market, placing it ahead of Koch, Agrium and PCS Nitrogen, and – with the completion of the CF-OCI deal – leaving that market consolidated with just five main domestic suppliers.

The collapse of the Yara-CF Industries merger deal meanwhile leaves Yara – which had been on an M&A spree of its own in recent years, from the 2007 takeover of Finnish international fertilizer giant Kemira to its 2013 purchase of Bunge in Brazil – still looking for partners.

### Technology companies

On the technology side, as noted in our Nitrogen Industry News section this issue, KBR has agreed to buy Weatherly Inc. ,Plinke GmbH and Chematur Ecoplanning Oy from Sweden’s Chematur Technologies AB, at a price of \$24.5 million. North American-based Weatherly has extensive experience providing nitric acid and ammonium nitrate technologies and services to the fertilizer market.

Last year saw Switzerland’s Casale SA acquired neighbouring Austrian-based Borealis’ proprietary process technologies – bought in turn by Borealis from France’s nitrogen producer GPN in 2013. Casale took GPN’s nitric acid, ammonium nitrate and urea-ammonium nitrate (UAN) solutions technologies, as well as granulation technology. The move followed on from Casale’s acquisition of Czech plant engineering and construction firm Chemoproject Nitrogen in 2014, and Casale’s earlier purchase from Borealis of their melamine technology in 2013.

The flurry of acquisitions from Casale turns them into a much broader-based company from their traditional focus upon ammonia, urea and methanol, and gives them more opportunities for technology integration, as between urea and melamine production, for example. Adding plant engineering capability also makes them a much more one-on-one competitor to a company like the recently re-branded thyssenkrupp Industrial Solutions, which as well as having plant construction and engineering expertise operates the old Uhde portfolio of technologies, including hydrogen, ammonia, urea, nitric acid, ammonium nitrate, CAN and UAN processes.

Indeed, KBR’s purchase of Weatherly can be seen as a counter to this, adding nitric acid and ammonium nitrate to their ammonia and gasification licensing, and producing three companies – Casale, KBR and thyssenkrupp IS – which can build a complete nitrogen complex, from feed through ammonia production to downstream nitric acid and AN production. It is interesting to consider where this puts Haldor Topsoe, Stamicarbon, Saipem and Toyo, and whether further consolidation is possible.

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# Nitrogen+Syngas index 2015

A complete listing of all articles and news items that appeared in *Nitrogen+Syngas* magazine during 2015.



Changes in the global market for urea, *Nitrogen+Syngas* 335, May/June 2015.

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	Downer sues Tecnicas Reunidas over Burrup plant	Nov/Dec	10
	Leigh Creek to develop UCG, looking at AN	Nov/Dec	11
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	Chambal Fertilizer shuts down urea line at Kota	Mar/Apr	11
	CIL signs up for coal gasification plant	Jan/Feb	12
	Direct farm subsidy scheme proves a headache	Sep/Oct	11
	Environmental clearance deferred on Shyam	Nov/Dec	10
	FACT re-starts due to lower gas prices	Mar/Apr	11
	Gas prices fall for fertilizer producers	Nov/Dec	10
	Gas prices to be unified from July	Jul/Aug	10
	Government leaning towards coal-based production	May/June	11
	Government to set uniform gas prices for urea	May/June	11
	Government relents on naphtha-based plants	Jul/Aug	10
	GSFC looking at urea, caprolactam	Sep/Oct	11
	Land allocated for new urea plant	Mar/Apr	11
<b>Indonesia</b>	Mitsubishi in sales joint venture	Nov/Dec	10
	Move to recommission Sindri plant	Sep/Oct	11
<b>Iran</b>	Tata Chemicals considering \$1 billion fert sale	Sep/Oct	11
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	Iran sets gas price for Indian urea plant	Nov/Dec	12
	Plans for petrochemical complex dusted off	Jul/Aug	13
	Plans for urea joint venture resurface	May/June	14
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	ICL to develop potassium nitrate capacity	May/June	12
<b>Japan</b>	New research centre for plant nutrition	Jul/Aug	12
	New catalyst could boost ammonia production	May/June	12
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	Korean investment in Malaysian AN plant	Sep/Oct	12
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<b>Netherlands</b>	Fire shuts OCI's Geleen facility	Nov/Dec	11
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	New urea granulation plant for Yara	Sep/Oct	11

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<b>Qatar</b>	Qafco partners GE for downstream services	Mar/Apr	12
<b>Russia</b>	Contract signed for new ammonia plant	Jul/Aug	10
	Eurochem signs MoU to build five new urea plants	May/June	10
	EZOCM looking to expand outside CIS	Mar/Apr	12
	Uralchem to increase capacity at Perm	Mar/Apr	12
<b>Saudi Arabia</b>	Ma'aden returns to production, Saftco V delayed	May/June	12
	Saftco V achieves commercial production	Sep/Oct	12
<b>Slovakia</b>	Alfa Laval to supply ammonia WHB	Nov/Dec	12
	New fertilizer plant in Slovakia	Mar/Apr	14
<b>Spain</b>	Nitric acid explosion causes vapour cloud over town	Mar/Apr	12
<b>Sweden</b>	Chemprojekt to build new nitric acid plant for Yara	Mar/Apr	12
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<b>Switzerland</b>	Casale adds key fertilizer technologies to portfolio	Jan/Feb	10
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<b>USA</b>	Agriculture may emit more N <sub>2</sub> O than believed	Sep/Oct	10
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	Alaska looks to tax credits to restart Nikiski	Mar/Apr	10
	Alaskan gas bill passes state House of Representatives	May/June	11
	Ammonia leak hospitalises 16	Jul/Aug	12
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	Borealis and Agrifos to set up ammonia plant	Jul/Aug	10
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	CF and OCI in \$8 billion merger	Sep/Oct	10
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	CVR buys Rentech Nitrogen	Sep/Oct	10
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	Ground broken on new Yara/BASF ammonia plant	Sep/Oct	10
	KBR awarded ammonia plant EPC contract	Mar/Apr	10
	KBR to revamp Mosaic ammonia plant	Jan/Feb	11
	Koch expansion moving ahead	Mar/Apr	10
	Lawsuit alleges precious metal price fixing	Jan/Feb	11
	LSB faces rising costs for nitrogen plants	Sep/Oct	10
	Reliable approach to reformer flue gas tunnels	Jan/Feb	11
	Sabin Metal marks 70th anniversary	Jan/Feb	10
	Solex to install urea cooler at Dakota Gasification	Mar/Apr	10
	Tecnimont to build new ammonia-urea plant	Jan/Feb	10
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<b>Uzbekistan</b>	Contracts awarded for ammonia-urea plant	Sep/Oct	12
<b>Vietnam</b>	Ammonia expansion for PetroVietnam	Jul/Aug	13
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	AzMeCo starts receiving Russian gas	Nov/Dec	16
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<b>Canada</b>	Atlantic Hydrogen files for bankruptcy	Nov/Dec	16
	Investment decision by year end on MTG plant	Mar/Apr	18
<b>Chile</b>	Gas supply agreed for Methanex methanol plant	Nov/Dec	16
<b>China</b>	Coal to SNG project complete by 2018	Jul/Aug	14
	Collaboration on fluid bed MTG process	May/Jun	16
	Continuing growth for coal to olefins	Nov/Dec	14
	Contracts for SNG expansion	Mar/Apr	19
	Ethylene glycol from syngas	Jul/Aug	14
	First reference for Clariant sour gas shift catalyst	Nov/Dec	14
	NDRC approves nine CTO plants	Jul/Aug	14
	New guidelines for coal gasification projects	Sep/Oct	14
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	Ordos SNG project receives water resource approval	May/Jun	18
	SinoCoking to sell hydrogen to Shenna	Mar/Apr	19
	Successful outing for novel PDH technology	May/Jun	18
	SES highlights gasification progress	Nov/Dec	15
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	UOP licenses MTO technology	Sep/Oct	14
<b>Egypt</b>	BP signs off on \$12 billion gas development	May/Jun	16
	Massive gas find off Egypt	Nov/Dec	15
	MoU signed for formaldehyde plant	Mar/Apr	19
<b>Germany</b>	Linde commissions new syngas research plant	Nov/Dec	14
<b>Iceland</b>	Chinese money for renewable methanol plant	Sep/Oct	14
	CRI to promote power to methanol technology	May/Jun	19
	Renewable methanol plant to triple production	Mar/Apr	18
<b>India</b>	Jindal Steel will build more coal to gas plants	Mar/Apr	18
	SES and Simon India extend joint marketing	May/Jun	18
<b>Iran</b>	Ten million tonnes of methanol waiting in the wings	Sep/Oct	16
<b>Japan</b>	Hydrogen fuel push for Olympics	Mar/Apr	18
<b>Netherlands</b>	OCI to buy BioMCN	Jul/Aug	14
<b>Nigeria</b>	ExxonMobil to up gas production	Sep/Oct	16
<b>Qatar</b>	Qatar launches CO <sub>2</sub> recovery project	Mar/Apr	18
	Qatar Kentz wins EPC contract for Pearl GTL project	Jan/Feb	15

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<b>Singapore</b>	MI working to reduce methanol deaths	Jul/Aug	17
<b>South Africa</b>	Fuel cell power for Impala	May/Jun	19
<b>Sudan</b>	Agreement signed for GTL plant	Nov/Dec	15
<b>Sweden</b>	Biogas demonstrator plant comes on stream	Jan/Feb	15
	Linde to build hydrogen fuel station	Mar/Apr	18
<b>Thailand</b>	Update on waste gasification project	Jan/Feb	14
<b>Trinidad &amp; Tobago</b>	New gas expected by 2020	Nov/Dec	15
	New methanol plant for Trinidad	May/Jun	18
<b>Turkmenistan</b>	GTL plant for Turkmenistan	Nov/Dec	16
<b>UAE</b>	Dubai to run municipal vehicles on biodiesel	Mar/Apr	18
<b>UK</b>	Construction nearing completion on waste plant	Jul/Aug	17
	Johnson Matthey sells Research Chemicals	Nov/Dec	16
	Moratorium on unconventional gas may half UCG	Sep/Oct	17
	Oil and gas discovery in southern England	May/Jun	16
	Shell agrees terms for BG takeover	May/Jun	16
<b>USA</b>	Air Liquide to build O <sub>2</sub> plant for Yuhuang	Mar/Apr	16
	Air Liquide to engineer methanol complex	Sep/Oct	16
	Another MTG plant for Louisiana?	Jan/Feb	14
	BASF selects Texas for MTP complex in US	May/Jun	16
	Biomass to urea plant proposal	Mar/Apr	16
	Celanese considering second methanol plant	May/Jun	16
	End of the line for coal gasification plant	Mar/Apr	16
	Ground broken on GTL plant	Jul/Aug	16
	Ground broken on new methanol plant	Nov/Dec	15
	GTL from shale	Jul/Aug	16
	HEC gets six month stay of execution	Sep/Oct	16
	Linde expands production at LaPorte	Jul/Aug	16
	LNG exports approved by DoE	Jul/Aug	16
	Methanex starts up first Geismar plant	Mar/Apr	16
	Methanol expansion project underway	Mar/Apr	16
	Methanol production begins at Pampa	Jul/Aug	16
	MTG licensing tie-up	Jul/Aug	14
	NWI looking at low emission reforming	Sep/Oct	16
	OCI suffers unplanned outages	Jan/Feb	14
	Sasol abandons GTL project	Mar/Apr	16
	Velocys settles IP dispute	Nov/Dec	16
<b>World</b>	World hydrogen market to reach 325bcm by 2020	May/Jun	19

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# Reformer performance and tube life management

The steam reformer is widely accepted as being the most complex and energy intensive part of any ammonia, methanol or hydrogen plant, and as such, it is important to ensure that it is operating and maintained under the best possible conditions. This will enable the operators to produce the maximum achievable levels of product, extend reformer tube lives and increase energy efficiency.

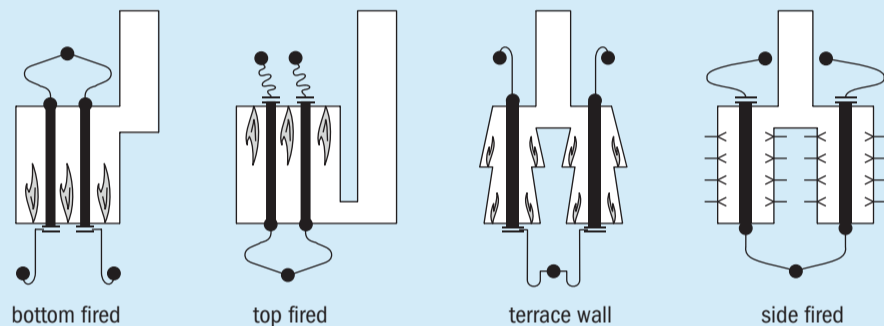
**C**atalytic steam reforming of hydrocarbons in tubular reformers is the most common process for production of synthesis gas. The reforming reactions are highly endothermic, and the heat provided by combustion of fuel gas in a furnace box is transferred to the catalyst tubes mainly by radiation.

Tubular steam reformers are divided into four categories depending on the location of the burners (Fig. 1).

Depending on the design, the furnace box may contain a single row of tubes or multiple rows. Catalyst tubes in top-fired and bottom-fired reformers are arranged in several parallel rows with the burners located between the tube rows either in the top or the bottom of the furnace box. Tubes in side-fired and terrace wall reformers are arranged in single rows between two opposing furnace walls. Accurate temperature measurement is difficult in any reformer configuration, but is particularly so when the tubes are arranged in multiple parallel rows.

Catalyst tubes continuously degrade due to the harsh environment of the furnace box from the moment they are commissioned. The upper limit of tube wall temperature is primarily governed by the tube design temperature. If the temperature is too high, there is a high risk of premature creep damage and coke formation, resulting in tube failure and process flow restriction. Continual operation at temperatures just 20 degrees higher than the design temperature will cut the tube lifetime in half<sup>1</sup>. On the other hand, too-low temperatures lead to decreased production output and profit losses. Operating variables that determine the optimal tube

Fig 1: Reformer furnace configurations



Source: Topsoe

skin temperature include fuel consumption, steam-to-carbon ratio, and feed gas rate. In general, for a plant to optimise tube lifetimes and production, operating temperatures should be kept as close as possible to the design temperature.

Accurate TWT monitoring is crucial to maintain operation near design temperatures. Common temperature measurement methods include imagery, direct contact, infrared thermometry, and calibrated eyeball. The factors that determine which of the available methods should be used include accuracy, cost, reproducibility, feasibility and simplicity. One simple option is a thermocouple, welded on the tube surface or embedded in the tube wall. These can provide accurate, continuous measurements, are low in cost and are simple to use. However, the lifetime of thermocouples is short under the harsh conditions of the reformer furnace, and the method of attachment to the catalyst tubes often compromises the inherent accuracy.

The performance of the primary reformer is of crucial importance to plant

efficiency and production because of the reformer's role as the single largest consumer of energy. It is therefore extremely valuable to conduct regular reformer assessments that result in solutions aimed at optimising parameters for peak reformer performance.

## Topsoe reformer management

Topsoe has recently improved upon its already extensive repertoire of services and tools used for assessment and optimisation<sup>2</sup>. A primary focus of the improvements is on tube wall temperatures (TWT) due to the lack of precision when using conventional methods for measuring TWT. Results from extensive infrared pyrometer studies reveal why and when to use different pyrometer types, and this knowledge will help producers optimise their steam reformers by maintaining operation closer to design temperatures. Topsoe is also introducing a system for advanced reformer surveillance, which provides additional temperature data via continuous

monitoring. With these advances in steam reformer services, Topsoe can better help customers adhere to design limits, identify bottlenecks, save energy, increase tube lifetime, and optimise operations.

### Optical infrared pyrometry

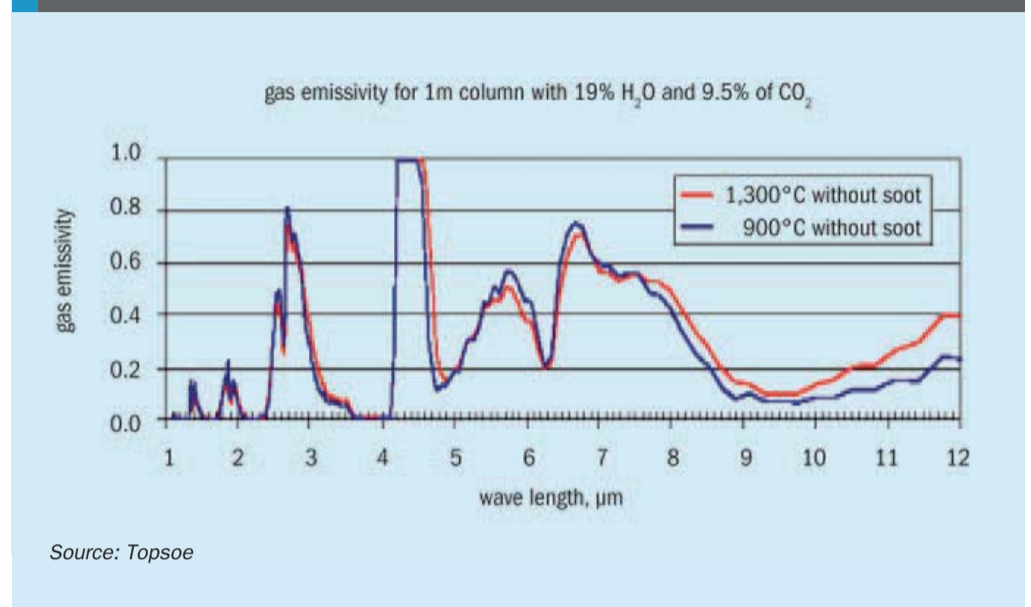
Currently, the technique most commonly employed in the industry is based on IR pyrometry. The pyrometers are optical remote-sensing devices that detect the thermal radiation emitted by a target object. Benefits of this method include cost-effectiveness, relative ease of use, speed, and reproducible results. The disadvantages include frequent improper use and multiple sources of error.

### Sources of error

One of the largest sources of error in optical IR pyrometry is derived from the uncertainty of the tube wall emissivity. Emissivity ( $\epsilon$ ) is by definition a number between 0 and 1, with 1 indicating a perfect emitter or backbody, and 0 a perfect reflector. All real objects emit only a fraction of the radiation of a blackbody. Objects with an emissivity less than 1 are partial reflectors of radiation, which means that radiation impinging on the tube from the surroundings will also contribute to the signal received by the pyrometer. The surroundings include refractory walls, floor, ceiling, flames, and other tubes, and are generally not uniform in temperature<sup>1</sup>. Obtaining accurate TWTs therefore requires good knowledge of tube wall emissivity and contributions due to background temperature and reflection.

Most infrared pyrometers measure spectral radiance over a small wavelength range. The optimum wavelength is a balance between lower background and emissivity uncertainties at longer wavelengths and lower TWT measurement uncertainties at shorter wavelengths. Industrial thermometers typically operate in the infrared part of the spectrum. Since the flue gas in reformer furnace boxes also absorb and radiate infrared energy in selective wavebands, it is important to choose narrow pyrometer operating wavelengths that minimise the interference. Figure 2 shows the absorption and emission of flue gas (in the absence of soot), which is dominated in the infrared region by water and carbon dioxide. Given the minimum values at 1.0  $\mu\text{m}$  and 3.9  $\mu\text{m}$ , these are the two most common operating wave bands used for pyrometer measurement of reformer tube

Fig 2: Absorption and radiation of flue gas in the IR range



walls. It should be noted that the presence of soot should be taken into account when performing TWT measurements, since scattering of radiation by soot can also be a source of error.

### Tube wall emissivity

General industry practice recommends an assumed tube wall emissivity of 0.85 for 1.0- $\mu\text{m}$  pyrometers and an assumed emissivity of 0.82 for 3.9- $\mu\text{m}$  pyrometers. However, one factor affecting the actual tube wall emissivity is the condition of the reformer tubes. New, clean reformer tubes have a rough oxide layer that gives them an emissivity as high as 0.9. As they age, reformer tubes shed their rough oxide layer and become smoother on the surface, which results in a lower emissivity. Proximity of reformer tubes to direct flame impingement also lowers the tube wall emissivity due to a faster aging process. If foreign material is deposited on the tube surface, emissivity can be further reduced to as low as 0.6. For example, for reformers situated in desert locations, a lower tube wall emissivity should be assumed.

New findings from a recent study show that actual tube wall emissivities also depend on a combination of operating temperature and wavelength. The study was conducted by Topsoe and the Technical University of Denmark on tubes at typical operating temperatures. Not only were actual tube emissivities found to vary as a function of temperature, they were also found to vary as a function of wavelength at different temperatures. It can be concluded from the observations that use of optical IR pyrometers should be accompa-

nied by very careful consideration of tube wall emissivity assumptions.

### Uncertainties

It is important to have a sufficient understanding of measurement uncertainties involved in reformer temperature determination in order to minimise inaccuracies. As described by Saunders<sup>1</sup>, there are three components of uncertainty. One is the uncertainty of the tube measurement itself, which is due to the intrinsic accuracy of the pyrometer and fluctuations in the furnace conditions. A second uncertainty is that of the emissivity. The value of this uncertainty is usually 0.05 at best, but it can be reduced by measuring a good set of tube samples. The third uncertainty component is the uncertainty in background temperature measurement and is generally the most difficult to assess. Results of Saunders's uncertainty analysis reveal that the dominating uncertainty component depends on the actual operating conditions. For example, given an effective background temperature of approximately 880°C and low TWT, the largest uncertainty is from the emissivity component. For the same background temperature and high TWT, the TWT measurement uncertainty dominates. Performing an uncertainty analysis is therefore useful in determining which uncertainty component should receive the most attention in order to improve accuracy.

### Comparison of 1.0- $\mu\text{m}$ and 3.9- $\mu\text{m}$ pyrometers

The 1.0- $\mu\text{m}$  pyrometer has been used more extensively in reformers, with the

3.9- $\mu\text{m}$  pyrometer only being introduced to the industry in the 1990s. The increased interest in the longer wavelength is partly due to its lower sensitivity to uncertainties in background reformer temperature and in tube wall emissivity. It is important to note that the sensitivity of the 3.9- $\mu\text{m}$  pyrometer is lower only in the case where background temperature is higher than the target (tube wall) temperature, which is most often the case in reformers. However, when the background temperature is lower than the target temperature, the opposite is true, i.e., the sensitivity of the 1.0- $\mu\text{m}$  pyrometer is lower.

The 3.9- $\mu\text{m}$  pyrometer may be less sensitive to uncertainties, but it has also been observed to give less accurate TWT measurements. In one study, Topsoe inserted a target object between the tubes of an operating side-fired reformer and found that the 3.9- $\mu\text{m}$  pyrometer consistently measured higher temperatures after correction than thermocouples placed in the object. The 3.9- $\mu\text{m}$  pyrometer measurements were also found to be higher than predicted by reformer simulations. Reformer simulations, such as the proprietary simulation programs developed by Topsoe, provide optimum operating temperatures using detailed equipment information and actual operating data. As part of the simulation, advanced process modeling balances the reactivity in the catalyst tubes and heat flux along the tubes and finds the correct heat balance between the radiant furnace box and convection section. Further details of Topsoe's simulation programs are provided in Jensen et al<sup>3</sup>.

In contrast to 3.9- $\mu\text{m}$  pyrometers, 1.0- $\mu\text{m}$  pyrometers have been found to measure temperatures in agreement with thermocouples in the same target object, and the TWT measurements were consistent with predictions from reformer simulations. However, the high sensitivity of the 1.0- $\mu\text{m}$  pyrometer makes it less straightforward to use. In the absence of thorough training, the 3.9- $\mu\text{m}$  pyrometer with its lower sensitivity should be used. The 3.9- $\mu\text{m}$  pyrometer is also the more conservative choice, since the higher temperature readings generally lead to adjustments that do not exceed design limits.

#### Recommended measurement procedure

Proper user training is crucial in order to obtain the most accurate and precise measurements using optical IR pyrometers, particularly the 1.0- $\mu\text{m}$  pyrometer. Users

can receive training through Topsoe Academy™, which offers a series of courses that features expert technical knowledge aimed at improving plant efficiency and reliability. Below are some of the most important considerations that should be employed when performing IR pyrometer measurements.

#### Preconditions

Prior to measurement, the user must ensure that the plant is in stable operation and that sufficient draft is present to prevent overpressure in the furnace box during measurement. The user should also ensure that overpressure protection of the furnace box is in place.

#### Cooling rate

The vacuum inside the furnace box will cause cold air to rush into the furnace box when the peephole door is opened, which will cool the catalyst tubes. Evaluation of the cooling rate requires repeated TWT measurement of the same tube while the door is kept open. For example, measurements are taken every five seconds for the first 15 seconds, followed by measurements every ten seconds up to one minute. The peephole door must be kept closed for several minutes before this procedure can be repeated with the same or different tube. Since the tubes may experience significantly varying cooling rates, it is recommended to perform these measurements on two additional peepholes located at opposite ends of the reformer. The cooling rate is faster for tubes closer to the peephole doors but after about 120 seconds TWT does not continue to drop. In general, the cooling rate is low enough to allow a maximum open peephole time of three seconds.

#### Reproducibility

The reproducibility of the TWT measurements must be verified by consecutively measuring selected tubes five times, with at least one in front of and one away from the peephole. The peephole must be closed for a minimum of 30 seconds between measurements in order to avoid the cooling influence of outside air. A similar procedure can be used to verify the reproducibility of furnace wall temperature measurements. The deviation should generally be less than 2-3°C.

#### Normal horizontal measurements

The temperature measurements are performed in three steps:

- the tube wall temperatures are measured through all the side peepholes;
- the furnace wall temperatures are measured between the tubes from the opposite side;
- the appropriate correction due to furnace wall reflection is calculated and applied.

The pyrometer emissivity should be set to 1.0, continuous measurement mode should be selected, and the focus lens should be adjusted with respect to the distance to the tubes.

Vertical measurements can also be performed through end wall peepholes on all levels. Scanning tubes vertically identifies the location of maximum TWT. Maximum TWT, normally located at the peephole level, can be affected by variations in catalyst activity or the firing profile.

#### Gold cup pyrometry

Another trusted method of TWT measurement used in the industry is the gold cup pyrometer. With this instrument, the target object is surrounded by a gold plated hemisphere (gold cup) and effectively acts as a blackbody. Errors due to background contribution and inaccurate emissivity assumptions are eliminated, and the radiation measured by the gold cup pyrometer can be directly converted to the true temperature of the object. Due to its accuracy, the gold cup pyrometer is often used as a reference standard. A disadvantage of the gold cup pyrometer is its heavy and cumbersome weight. It also has a limit to the distance that it can be inserted into the furnace. Use is otherwise relatively simple, and minimal training is needed. This method is recommended as a supplement to regular and frequent measurements using optical IR pyrometers, and it is offered as part of Topsoe's steam reformer assessment/optimisation services. Experts at Topsoe are well trained at providing the most accurate TWT measurements possible using a variety of methods and can use the gold cup to assess the effectiveness of other methods typically used at a plant.

In 2015, Topsoe performed a comparison study between gold cup measurements and measurements using optical IR pyrometers. The study was conducted on two side-fired reformers at separate plants that experience similar environmental conditions. All tubes were cleaned shortly before measurement. Results showed that



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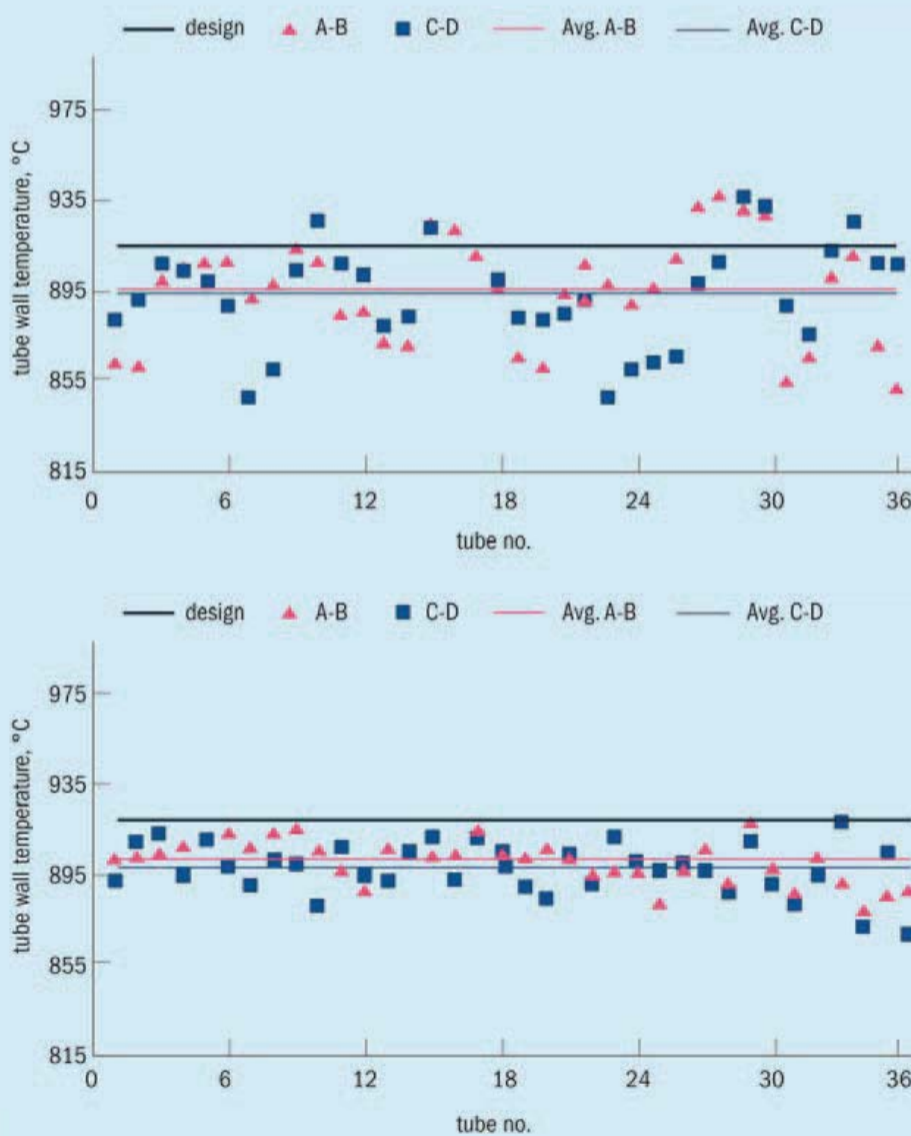
Table 1: Levels of Topsoe steam reformer assessment/optimisation service

Scope of service levels	1	2	3	4
Visual inspection of reformer tubes and firing	✓	✓	✓	✓
IR pyrometer TWT measurements	✓	✓	✓	✓
Comparison of customer and Topsoe IR pyrometer readings	✓	✓	✓	✓
Flue gas composition and furnace draft measurements		✓	✓	✓
Data collection, reconciliation and simulation		✓	✓	✓
Extended data collection and advanced simulation			✓	✓
Fuel/combustion air header/burner pressure measurements			✓	✓
Reformer trimming and optimisation*			✓	✓
Flue gas oxygen analysis in convection section*			✓	✓
Modelling, evaluation and optimisation of convection section*			✓	✓
Gold cup TWT measurements, vertical and horizontal profile				✓

\*Optional

Source: Johnson Matthey

Fig 3: TWTs before and after reformer furnace improvements



Source: Topsoe

the 3.9-micron pyrometer measured consistently higher than the 1.0-micron pyrometer. They also showed that measurements from both of the optical pyrometers, after correction and the appropriate assumptions for tube wall emissivity, were typically higher than measurements from the gold cup pyrometer.

Differences between the gold cup measurements and the corrected measurements from optical IR TWT measurements were in large part attributed to inaccurate tube wall emissivity assumptions. A more accurate tube wall emissivity can be determined by adjusting it such that the corrected optical IR measurements match those of the gold cup.

The conclusion that assumed emissivities may often be too high is supported by a recent research project conducted between Topsoe and the Technical University of Denmark. Furthermore, findings from the joint project are in agreement with the distribution curve of adjusted emissivities obtained using the 1.0- $\mu\text{m}$  pyrometer in this study. On the other hand, the distribution curve of adjusted emissivities for the 3.9- $\mu\text{m}$  pyrometer is not in agreement with the literature and the joint project, and this approach of reconciling emissivities may therefore not be recommended for the correction of 3.9- $\mu\text{m}$  pyrometer measurements.

**Topsoe Furnace Manager**

Other developments for improved reformer management include on-line measurement of TWT using a new Topsoe monitoring system called the Topsoe Furnace Manager (TFM), which is a permanent installation of an array of image collectors. The significant advantage of the TFM over hand-held methods is its remotely accessible and continuous real-time data of the furnace interior around the clock. Data acquisition does not require any opening of peephole doors, and operators can balance the reformer firing and respond to an alarm without direct interaction with the furnace box. This represents a major improvement in reformer performance and personnel safety.

Use of the TFM is straightforward and does not require extensive training. On-line flame and TWT monitoring results are presented via universally understood images and data for the entire lifetime of the furnace. Remote access to the data and images also means that off-site experts can participate in troubleshooting and furnace optimisation, engaging the entire

furnace support organisation. In addition, logs of historical data are kept available, which easily provide benchmarks for training purposes and turnaround reference.

The many capabilities of the TFM make it very effective in helping plants avoid unwanted incidents. It acts as a safeguard against furnace overheating and works in parallel with existing safeguards, such as a BMS. It provides information facilitating process safety management and reliability, availability, and maintainability (RAM) evaluations, which leads to better knowledge of mechanical integrity, compliance audits, failure rates, failure modes, time-to-repair, and costs. Clients have for example found the TFM to be particularly valuable in maintaining safe and optimal reformer operation with older end-of-life catalyst tubes. The TFM has also helped a plant continue operation with a small process leak in the furnace box for over two years. Finally, the reliability of the TFM has also been demonstrated by a case in which after 45,000 hours of operation, all catalyst tube creep measurements were less than 1%.

The TFM is commercially proven in both side-fired and top-fired reformers. Its economic benefits include reduction of fuel consumption and efficiency improvements that could amount to a few hundred thousand US dollars a year and a similar amount in savings resulting from improvements in personnel productivity.

### Topsoe steam reformer assessment/optimisation

Tube wall temperature measurement is only one of the many steam reformer services that Topsoe experts can provide through the company's four-level steam reformer assessment/optimisation service. The general scope of each service level is shown in Table 1. The aim is to help plants adhere to design limits, identify bottlenecks, save in energy and resources, increase tube lifetime, and optimise normal operations. The services are customised to each plant's needs, and results are used to provide specific recommendations. Topsoe uses the detailed insight into the reformer's performance to maximise its efficiency, stability and throughput. Figure 3 shows an example of TWT measurements before and after Topsoe improvements to the furnace. The top graph indicates high and fluctuating TWTs, while the bottom graph shows more consistent TWTs, which are necessary for optimal performance.

### Johnson Matthey improving reformer operation

For a reformer to achieve maximum production in the most energy efficient manner, extending the reformer tube life and reducing trips and failures is something that needs consideration at all stages from the moment the catalyst is chosen, through its loading, start-up, operation and maintenance of the reformer. The right loading techniques, operation and process optimisation of catalysts can give world-beating performance, however they have to be considered as a whole and not in isolation.

In the following sections Johnson Matthey focuses on reformer operation prior to start up, during transient conditions such as start-up, steady state operation and even during a turnaround with the aim of highlighting best practice to improve the reformer's performance and increase reformer tube lives<sup>4-7</sup>.

#### Prior to start-up

Good reformer operation can be influenced even before the plant start-up and depends upon three parameters: the correct type of catalyst, an even catalyst loading and good quality reformer tubes.

It is important to determine in advance the correct catalyst combination for the process feed gas to the reformer. Special care must be taken to ensure that all components are considered in order to prevent future operational problems such as catalyst poisoning resulting in premature short lives. One example of this is alkalisated formulations such as KATALCO<sub>JM</sub><sup>™</sup> 25-series in which low levels of potash are added to the catalyst. This is incorporated into the

Fig 4: CATACEL<sub>JM</sub> SSR catalyst



PHOTO: JOHNSON MATTHEY

calcium aluminate support structure in a way that slowly hydrolyses, releasing alkali at the concentration required to ensure continuous carbon-free operation without loss of activity.

Another potential catalyst option would be to use the new Johnson Matthey CATACEL<sub>JM</sub> SSR<sup>™</sup> which is a coated catalyst on a formed structure as shown in Fig. 4. Individual units are stacked on top of each other around a central axle assembly for charging the reformer tube. The structure is designed to direct the gas against the tube wall which results in a significant performance benefit compared to pellets. This benefit can be used to reduce tube wall temperatures, and as a consequence to increase production in the reformer.

As well as selecting the correct catalyst combination, the right loading technique is also essential. The loading must ensure that an equal amount of catalyst is dense loaded into each tube, hence ensuring an even flow of process gas, and therefore

Fig 5: Effect of tube loading on tube temperature variation

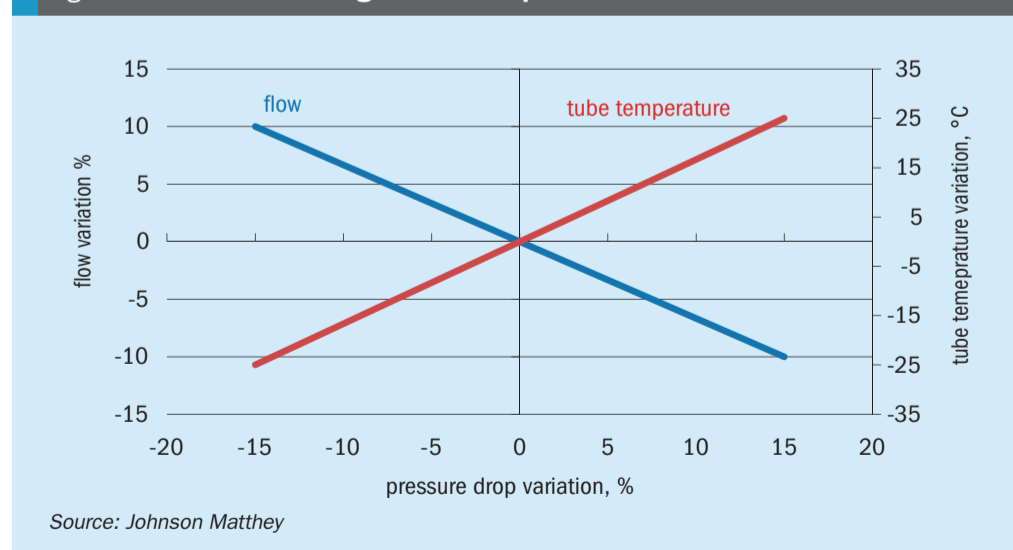
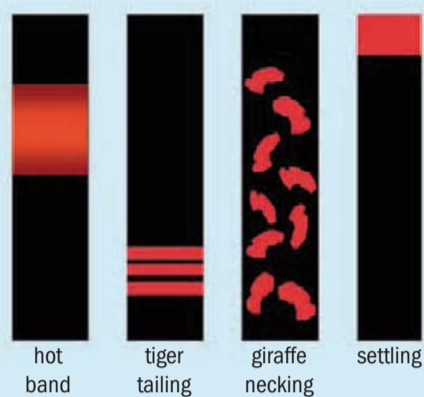


Fig 6: Typical hot spots that can occur in a reformer tube if packed incorrectly



Source: Johnson Matthey

Fig 7: Reformer tube defect post fabrication

PHOTO: JOHNSON MATTHEY



allowing a similar cooling effect due to the endothermic reaction during operation.

Firstly any variance in catalyst loading means that the reformer is starting its operating life with potentially hotter areas that need protection during operation otherwise they will lead to premature tube failure, differences in catalyst pressure drop between tubes will result in differences in flow between tubes and this in turn will result in difference in tube wall temperature – the results for a typical furnace are shown in Fig. 5.

Secondly, packing with a given tube must be uniform with no voids or bridging because if there is no catalyst present this will create areas where there is no reaction to remove heat from the tubes and will result in localised “hot spots” on the reformer tubes. Care must also be taken to allow for expansion and sufficient catalyst must be installed so that catalyst does not settle down so as to expose empty space at the top of the reformer tube. The image in Fig. 6 shows these issues could appear as hot sections of tube within the reformer.

To mitigate loading related issues in recent years it has become standard practice to use UNIDENSE™ (a trademark of UNIDENSE Technology GmbH) as the standard loading method for pelleted

catalysts. CATACEL<sub>JM</sub> SSR uses a specialist loading method for the structured catalyst, and in view of the unique structure, any risks from poor catalyst packing and settling over time are eliminated.

In addition to correct catalyst selection and loading method, it is also important to ensure that any new reformer tubes are free from fabrication defects. A large change in reformer tube internal diameter due to a fabrication defect reducing the wall thickness can have serious consequence on the tube life and therefore plant availability. Figure 7 shows a tube that was found to have a boring defect during fabrication.

A baseline inspection of the tube internal diameter before the tubes are installed in the reformer, (or at the tube fabricators workshop), can determine any defects and hence allow replacement of the tube before installation.

#### During start-up

It is well known that large-scale steam reformers in syngas plants are vulnerable to over firing, especially during plant transient conditions. This vulnerability is due to the fact that external temperature instrumentation lags during normal operation, and that human monitoring cannot be conducted on a sufficiently frequent basis. The consequence is that, over the past decades, many of the catastrophic reformer failures documented have occurred during the most common transient operation – start-ups.

It is therefore essential that regular and frequent visual inspections of the reformer tubes are made during critical periods associated with transients as most reformer instrumentation is designed for monitoring and control of operation during steady state operation.

Case study 1 describes a failure during a start-up on a large plant and illustrates the damage to the reformer that can occur in a relatively short time period.

#### Case study 1: Top-fired reformer start-up incident

This case study is based on the actual experience of an operator of a large modern top-fired reformer. They suffered from significant tube failures during a plant start-up, resulting in losses running to US\$ millions in terms of lost profits and downtime. This catastrophic failure was caused by over firing during start-up and was the result of a number of coincident factors.

At the time of the incident, the site had steam shortages and this led to pressure

to conserve steam. In addition to this, the plant was under pressure to avoid a shut-down if at all possible due to low product stocks. The burners on their reformer usually received fuel from two different sources and these were mixed. One of the sources was of low calorific value, and the other a much higher calorific value. At the time of incident, the plant was unexpectedly receiving all of its fuel from the high calorific value source.

During the previous two years the operators had seen many start-ups and shutdowns for a variety of different reasons, and were therefore relatively familiar with the procedures. The plant tripped on loss of feedstock to reformer as a result of a valve failure.

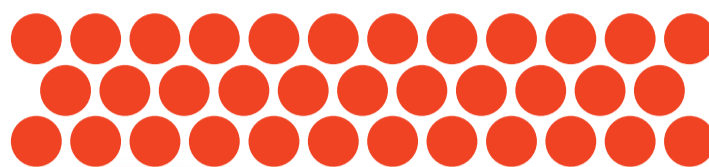
In their efforts to bring the plant back on line quickly, the feedstock to the reformer was not isolated adequately by a valve and the set point on the reformed gas pressure was not reduced. In addition to this steam introduced for the plant restart was at a reduced rate and all of the burners were lit (a deviation from the written procedure). According to the instruments in the control room, the reformer tube pressure remained at normal operating conditions of 16 barg. The reformer tube exit temperature also “looked normal” and throughout the incident never exceeded 700°C.

However, since all the burners were lit, there was nearly three times as much fuel going to burners than there should have been. In addition to this, the high calorific value fuel added an extra 15% heat release. When the first tubes ruptured, the radiant box high pressure alarm activated, but the plant did not trip as the high pressure trip was by-passed. The oxygen level in the flue gas dropped to zero and flames were seen coming from the reformer peep holes. Visual inspection of the reformer then revealed “white hot furnace and tubes peeling open” and the emergency shutdown was the activated.

The entire set of reformer tubes had failed, Fig. 8, and the incident from start to finish took less than 30 minutes.

One method which would prevent such an event is to use online temperature measurement, such as a relatively new in-tube temperature measuring technique, called the CatTracker™ (a trademark of Daily Instruments). CatTracker can be installed in steam reformers using a method patented by Johnson Matthey. The CatTracker is a multipoint thermocouple that is installed in the centre of the reformer tube amongst the catalyst and has been shown to have the following benefits:

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Fig 8: Case Study 1: Result of reformer over-firing

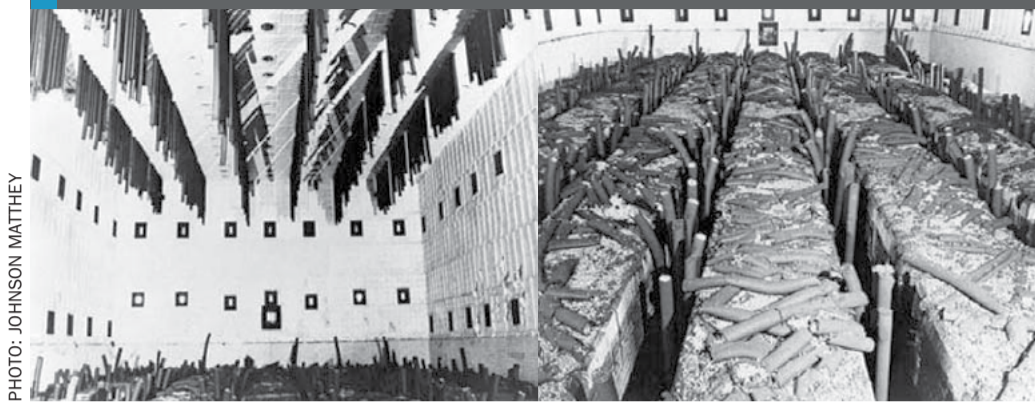
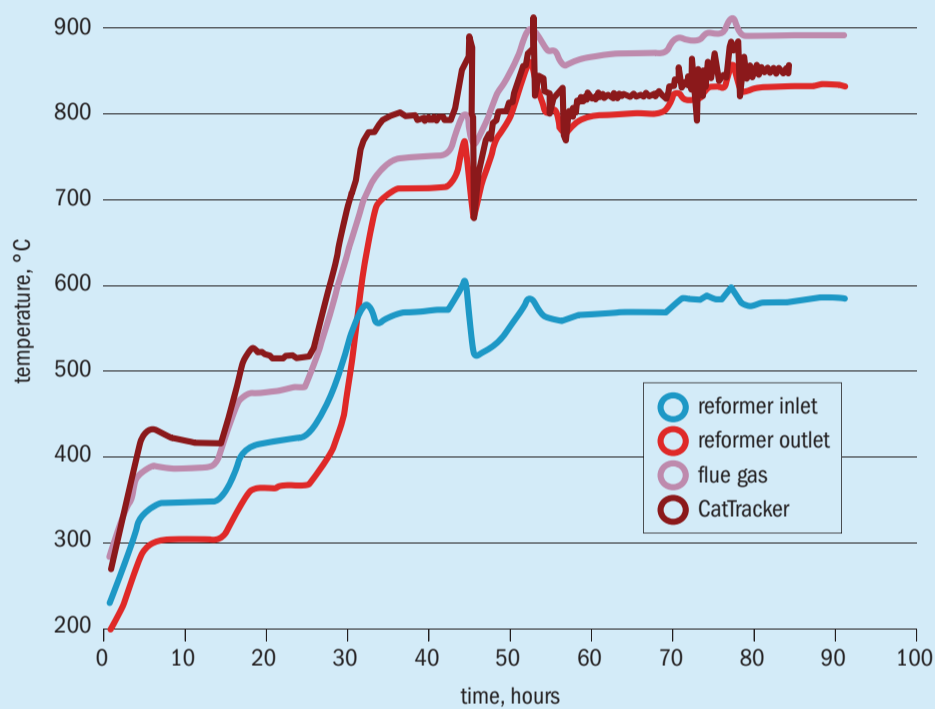


PHOTO: JOHNSON MATTHEY

Fig 9: Reformer temperatures during start-up



Source: Johnson Matthey

- In-line monitoring (as shown in Fig. 9) has shown that the CatTracker temperature probe can be used as the leading indicator to the temperature in the reformer tubes. This provides continuous monitoring of the in-tube temperature profile, with the profile available in real time through the plant DCS.
- Interlocks or trips, based on process gas temperatures within the catalyst tubes, preventing potential injury to plant personnel and costly reformer damage.
- Tube temperature profile measurement leading to more accurate prediction of the operating proximity of the catalyst to the carbon formation zone, thus allowing operation whilst avoiding carbon formation.
- Operation of the reformer at lower steam to carbon ratios without the risk of carbon formation.
- Improved understanding of catalyst parameters for an enhanced understanding of catalyst performance and change out scheduling.
- Monitoring real reformer tube outlet temperature to determine actual pigtail temperatures leading to a reduction in pigtail failures.
- Early detection of catalyst poisoning.

### Steady state operation

Once the plant is operating in steady state, it remains important to continue monitoring and balancing the reformer to ensure the best efficiency in terms of production and maximum tube life.

Process operators can do this with a variety of temperature measuring techniques that are currently available. These are the optical pyrometer, the gold cup

pyrometer and the reformer imager.

The optical pyrometer is an instrument which most operators own and which has been proven over decades to provide good results when compensated for background radiation. However, using it is a challenging and time consuming exercise; often the first few tubes in each row are not visible from the view port and it is also difficult to discern the individual tubes towards the centre of the furnace. As a result, operators often record only the maximum temperatures measured during a survey, rather than all the tube temperatures giving a limited view of furnace uniformity.

A gold cup pyrometer is an accurate measurement of tube wall temperatures when required. It is a direct contact pyrometer and therefore no correction is required for background radiation. However the gold cup pyrometer too, has its limitations. The method requires the gold cup to physically touch each tube; therefore measurement is limited to only a small number of tubes in each row for a multi-row furnace.

Johnson Matthey has another technique known as the Reformer Imager which is recognised as “best in class” for measuring reformer tube wall temperatures. It operates in the infra-red spectrum and captures significantly more information than the two techniques described above. The imager has a lens which allows a wide viewing angle therefore temperature readings are available for parts of the tubes that cannot be seen by the naked eye, e.g. the tops and bottoms of the tubes and tubes closer to the walls. The videos are recorded directly to a laptop and can be taken away for further analysis and can be used as reference to compare reformer performance over a given time period.

Any or all of these techniques can be provided by Johnson Matthey in the form of a reformer survey or used for reformer balancing on a customer plant. The reformer survey goes beyond straightforward TWT measurement and incorporates temperature correction and process engineering simulations to characterise a reformer performance, benchmarking against similar reformers and troubleshooting operational problems. This type of survey allows the operations team to make changes to the reformer balancing to improve reformer performance, often delivering significant value to the customer and improved plant production and efficiency. Johnson Matthey can also assist on site with reformer balancing to either increase production,

Fig 10: View from peephole (left) and reformer hot spots (right)

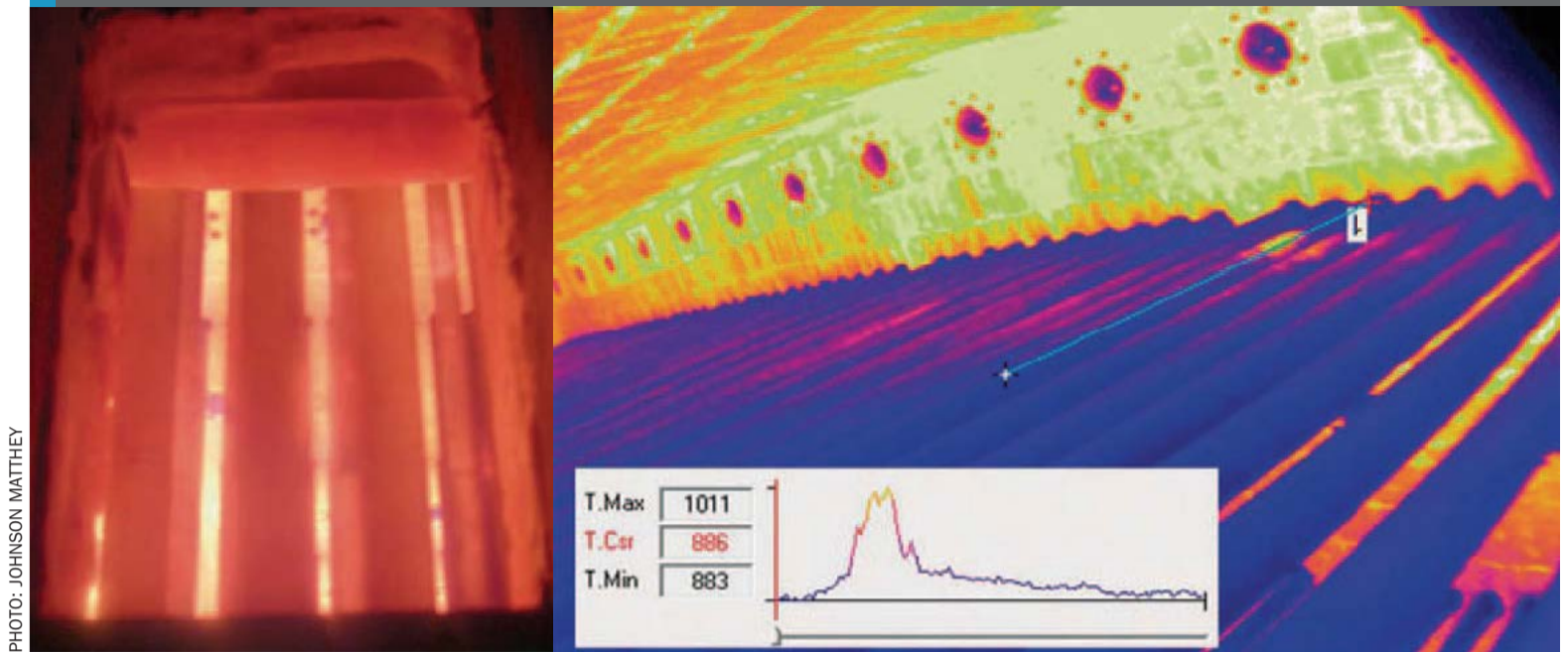


PHOTO: JOHNSON MATTHEY

reduce maximum tube wall temperatures, improve energy efficiency or a combination of all three.

Case study 2 describes how a reformer survey with the reformer imager was used to diagnose and solve a plant problem that was previously not understood while case study 3 details how these techniques can be used for balancing the reformer.

**Case study 2 – Reformer analysis and solution development**

A top fired reformer on a methanol plant which had a competitor’s steam reforming catalyst installed, suffered from hot spots and hot bands on the tubes in its first three years of operation. Within a few months of operation of its second charge, two tubes had ruptured and additional hot tubes and hot spots developed after every plant trip. To manage the furnace, the operator trimmed the fuel to the burners in the problem areas of the reformer. Conventional temperature measuring devices such as an optical pyrometer were unable to measure the temperature of all tubes. Consequently, it was impossible to determine if the problems were caused by poor catalyst packing, carbon formation or poisoning.

The operator sought Johnson Matthey’s help with root cause analysis. Johnson Matthey engineers conducted a reformer survey using the reformer imager, temperature measurements were taken quickly and easily in all parts of the reformer. This identified hotspots on tubes that were hidden from the peephole view and also hot spots that were previously invisible due to flame interference (see Fig. 10).

Table 2: Summary of production increase

Parameter	Initial	Final
Reformer exit temperature, °C	800.6	798.9
Primary reformer methane slip, %	11.12	11.03
Production, t/d	1,633	1,637
Process gas flow, kg/h	31,047	31,059
Natural gas fuel flow, kg/h	2,869	2,850

Source: Johnson Matthey

With thousands of data points available, the exact location of the hotter tubes and hot spots could be pinpointed. With these data, Johnson Matthey’s experts determined the most likely cause of the problems was carbon formation as all of the hotspots were localised and well delineated. Hot tubes were consistently found at the end of rows, suggesting additional heat reflection from the refractory walls.

As a result of the survey, Johnson Matthey’s engineers made short and long term recommendations including amendments to the plant start-up procedures. The catalyst was also replaced with a Johnson Matthey catalyst that would not form carbon during operation.

**Case study 3 – Reformer balancing**

A reformer survey was conducted at a customer site to evaluate the performance of the reformer and catalyst. The general condition of the reformer was satisfactory, but there was a wide tube wall temperature spread. At the time, there were six burners out of service and seven burners throttled.

The survey involved:

- The use of a reformer imager to capture images which were used to determine the temperatures of the reformer tubes
- Visual observation of the condition of the tubes, refractory and burners
- Recording of burner valve positions
- Collection of process data to model the performance of the primary reformer.

The survey indicated that the outer rows were hotter than the inner rows so the fuel header pressures were reduced on the outer rows in an effort to lower those temperatures. Following these adjustments, several further tube wall temperature surveys were done to assess the conditions of the reformer.

A summary of the process changes effected by the adjustments is shown in Table 2.

As a result of improving the temperature distribution within the reformer, fuel usage and methane slip at the exit of the primary reformer were both reduced,

leading to improved energy efficiency and a modest increase in ammonia production.

### Turnaround activities

Good reformer monitoring does not end when the plant is shut down for a turnaround. Whether the catalyst is replaced or not, the tubes should be physically measured for creep growth during the last production run to back up the results of the temperature monitoring in terms of whether any tubes need replacement. If the catalyst is being changed out, it is always recommended to conduct an internal inspection of the tubes using a technique such as the LOTIS™ inspection to determine the diametrical growth of the tubes. If the catalyst is not being removed, the MANTIS™ inspection technique can be utilised (LOTIS and MANTIS are trademarks of Quest Integrity Group LLC).

These techniques measure the internal or external diameter of the tubes, hence showing the creep growth during operation. Creep of reformer tubes is expected and tubes have a design lifetime of 100,000 hours if operated consistently well. Periods of operation at temperatures near or over design temperature accelerate creep growth and can lead to premature failures. The physical measurement will determine which tubes have the most diametrical growth and therefore may need replacement during future turnarounds.

If required, a remnant life assessment on the tubes can also be carried out using these diametrical measurements to determine the end life of each tube assuming operation similar to that previously experienced.

### BD Energy Systems tube overheat protection

One of the most significant reformer incidents is overheating catalyst tubes to the point of failure. These events inevitably have a serious impact on the business with significant repair costs and loss of production.

Reformer tubes expand when heated to operational temperatures. The expansion is related to the tube temperature and is sufficiently precise that overheating conditions can be detected in time to avoid damage to the tubes.

Current on line detection of overheating relies on interpretation of process data from which tube temperatures are inferred either by the operator or an algorithm in the control system.

These systems are not fool proof and fault tree studies reveal a number of ways they can fail to prevent overheating. However, by directly measuring the variable of interest, the tube temperature, via tube growth, these loopholes are closed and robust overheat protection is provided.

The Tube Growth Monitor (TGM) technology, which is licensed to BD Energy Systems and is available to non-methanol reformers applications, detects and alarms changes in reformer tube temperature before dangerous levels are reached earlier than current instrumentation. This makes possible an absolute protection against tube overheating incidents as long as the operators act upon the information promptly. The number of temperature monitors and the nature of the very visual display of the temperature indications and alarms from the TGMs cannot be missed or ignored.

Together with a robust protection against overheating, the TGM real time reformer temperature data allows multiple uses of this data to improve reformer performance and tube life management.

### Principle of operation

All metals experience thermal growth due to temperature change that is characterised by a “linear expansion coefficient.” This is the fractional change in length per degree of temperature change from a reference temperature.

Equation (1) shows the relationship between length (L) and temperature (T):

$$\frac{\Delta L}{L} = \alpha_L \Delta T \quad (1)$$

Where  $\alpha_L$  is the linear expansion coefficient which is specific to each metal or, in the case of a reformer furnace, each tube material.

For the purposes of this article, the theoretical relationship is described as above, but it must be noted that there are some practical factors that require attention:

First, in most reformer furnace designs, the tube is heated in such a way that a temperature profile is obtained with the objective of reaching an optimum in terms of the overall extent of chemical reaction. The change in temperature obtained from the expansion using the equation (1) will account for a change in the bulk or average temperature of the tube. This cannot be used to determine the actual temperature of the tube at any given point, though still will give information about the operational

temperature of the tubes in relation to the heat input to the reformer radiant box.

Second, locating the point of highest temperature in a reformer tube length, or the highest temperature tube, using infrared pyrometer equipment is time consuming and potentially difficult. These measurements are also subject to inaccuracies due to the complexity of the reformers configuration and the effects of varying degrees of reflected radiation from higher temperature walls within the reformer fire-box enclosure. Due to the significant effort involved in gathering pyrometer tube temperature data, this data is generally only available once per shift at best. Therefore, the operating conditions of the reformer may change while the field operators are trying to locate the maximum tube temperature within a reformer.

### Current protection weaknesses

Conventional operator supervised overheat protection practices and automated systems using process instrumented inputs have inherent weaknesses and limitations that must be understood.

Reliance upon operator supervision for overheat protection places a tremendous burden upon operators to understand the dynamic behaviour of a reformer furnace during non-steady state operations.

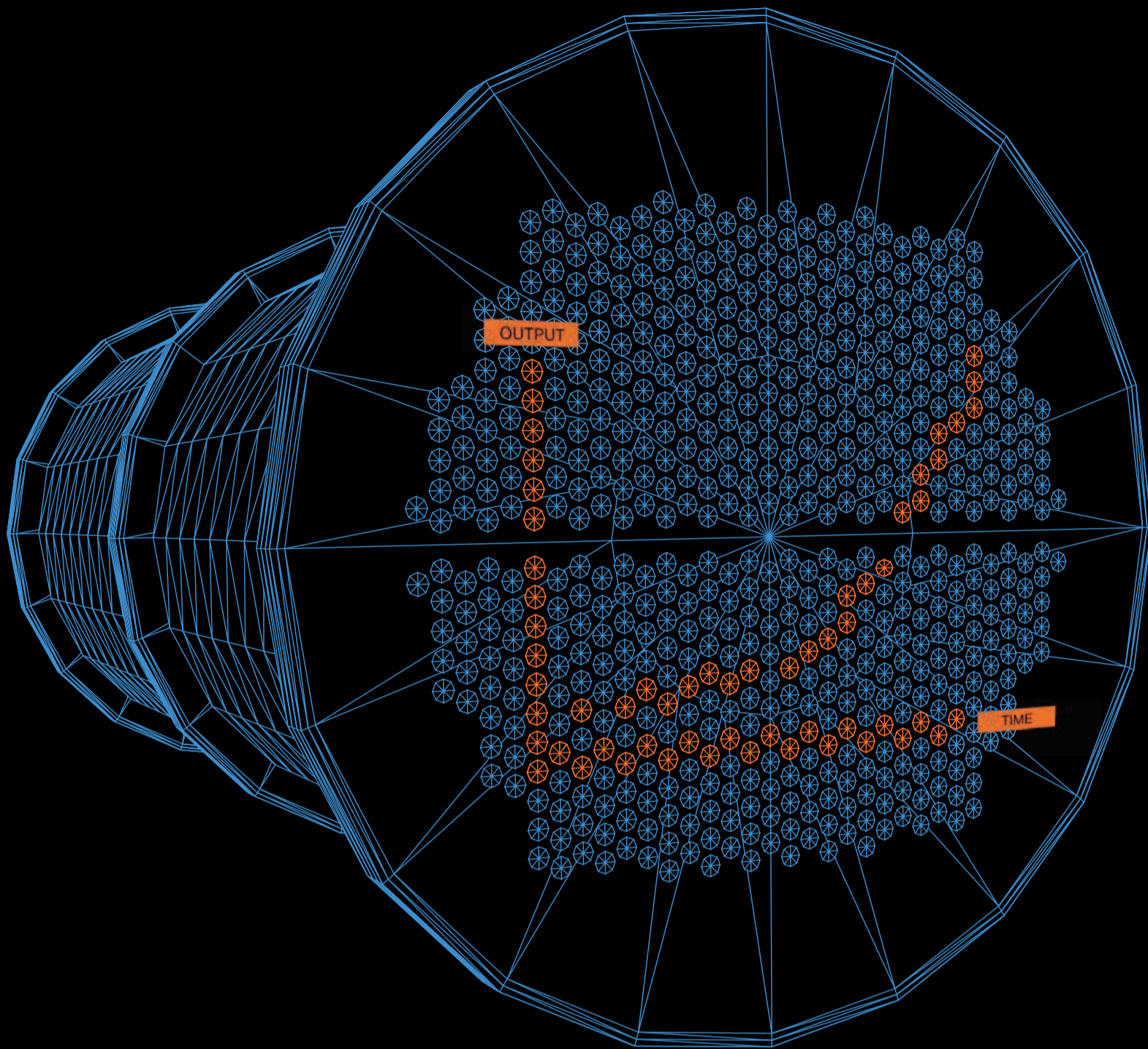
The time considered for action to stop a temperature increase to prevent an overheat event must be enough to allow the temperature “inertia” to dissipate, without over-correction in order to avoid damage to the tubes. There is limited time available for analysing data and making a decision during an overheating incident. This understanding comes only with time and experience but is a critical need to enable operators to make correct decisions quickly and to avoid damage.

To avoid placement of such a heavy burden on plant operators, many plants have adopted some type of automated overheat protection system.

Many conventional automated overheat protection systems are based on use of fuel firing limitations programed into the control system. These firing limitations are based on correlations intended to limit the fuel firing rate based on a number of critical measured process operating parameters. The intent of such correlations is to avoid human error during non-steady state operations. However, a remaining weak conventional automated overheat protection relies upon the proper function of



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multiple instruments that measure those critical process parameters.

Most of the published overheating incidents show root causes related to human behaviour or with a great influence of this factor. Hence, the probability of overheating incidents is always present as long as human action is part of the decision and operation process intended to avoid such an event.

A factor that greatly influences the human behaviour is the plant reliability. The reformer reliability and plant on-stream reliability has been improving over time. This means that there are much longer periods of stable operation time between unsteady state operations such as start-ups, shutdowns and the occurrence of serious problems like reformer trips. As a result, operators deal with unsteady conditions less frequently, making these events somewhat unfamiliar<sup>9</sup>

Another condition to consider is the retention of corporate knowledge that has been built up over many years when there is staff turnover due to retirement, promotion or job relocation<sup>9</sup>.

### Preventing reformer tube failures

Case study fault tree analysis and actual operations experience has shown that the probability of prevention of reformer tube failures related to overheating by reading the tube thermal expansion is "almost certain" for the following cases:

- Exotherm during steam out oxidation of catalyst.
- Incorrect burner light off sequence.
- Incorrect burner shut off sequence.
- Fuel gas header pressure relying on autoramping during rate change resulting in overshoot of temperature.
- Low steam flow or maldistribution of steam flow during steam out.
- High fuel gas header pressure during steam out as control valve manual bypass is open.
- Attempt to introduce feed gas with manual isolation valve closed. Programmed protection system allows increase of fuel firing based on feed control valve % open.
- Incorrect trend graph loaded into automated ramping software for start-up control.

And detection and prevention of overheat with the TGM system would be "probable" for the following two cases:

- Collateral damage from end of life tube failure.

Fig 11: Example of TGM wireless installation in a top fired reformer

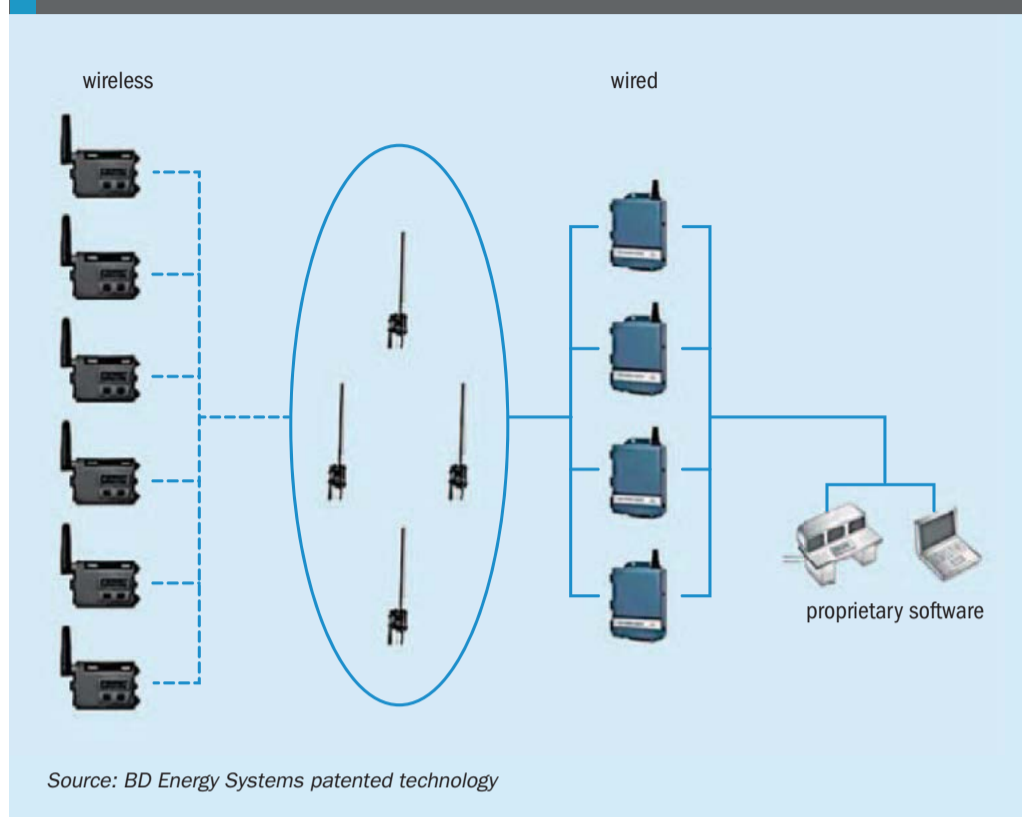
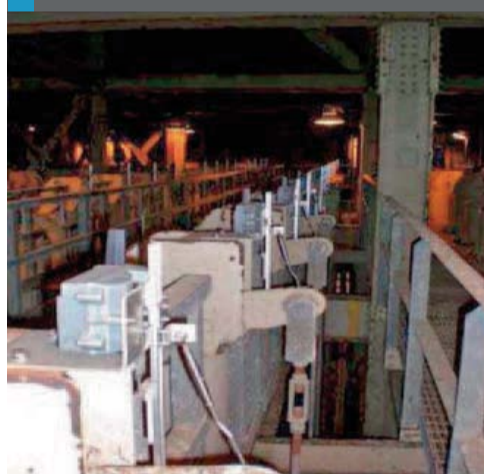


Fig 12: Real TGM field set up in a top fired reformer



- Burner tip failing giving jet flow and local tube impingement.
- There are other failure mechanisms for reformers that will not be prevented by TGM installation, thermal shocking the tubes with a slug of water for instance or a firebox explosion, but fortunately these are much less common.

### Conceptual engineering and real application

The Tube Growth Monitor (TGM) concept uses an instrument that is easy to install and set up.

The TGM system design must consider the location of burners and how the tubes are supported in the radiant box (springs

or counter weight hangers). With this information a rather simple mechanical design can be developed for installation of instruments on each hanger to ensure that they accurately measure the tubes thermal growth.

The system configuration is adaptable to the needs of each furnace operator. It normally requires that a signal of the tube growth is sent to the DCS in order to allow the operators and engineers to have on-line data. Additional trending can be made if the data is available to the plant process historian.

Although it is possible to have a local reading only, this setup is not recommended when the final intent is to protect the tubes from an overheating event as it would require constant attention from an operator to detect changes.

In a top-fired design, the TGMs are normally installed on the tubes spring hanger or counterweight hangers and the signal is sent to the DCS for processing and distribution to other plant systems like data historian and CMMS (Computerised Maintenance Management System), as a standard process data point.

A typical TGM set up for a top fired reformer can be seen in Fig. 11, which shows a series of TGM (battery powered) connected to the reformer spring hangers. The signals are sent wirelessly to a set of four antennas that receive the

Fig 13: Initial condition before the incident

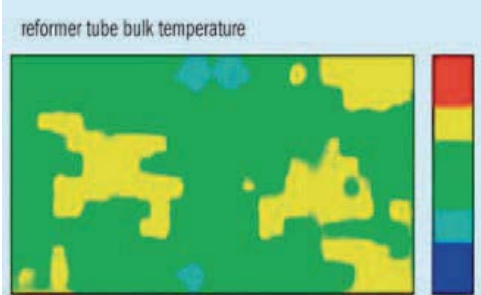


Fig 14: Hot banding condition after the incident

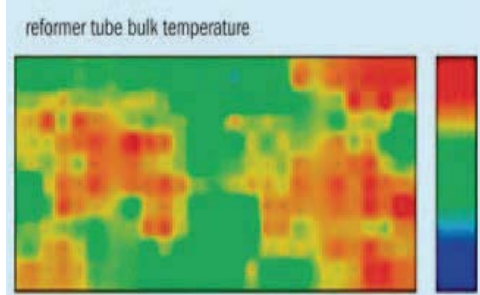
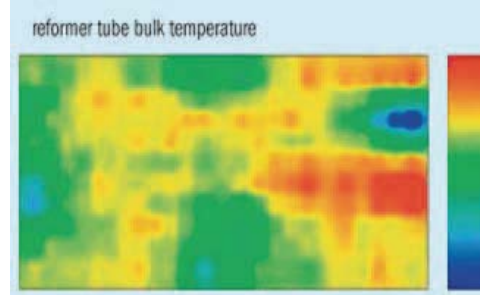


Fig 15: Hot and cold spot areas in the reformer



signal and send it to four gateways that transmit the data to the system (DCS and data historian). The flexibility of the wireless devices allows for easy customisation according to the needs of the reformer operator.

There is also the option of a wired TGM installation, but more work and budget would be required for the wiring and accessories related.

The number of antennas and gateways determines the frequency of the scan rate for each TGM input and therefore the level of reliability and redundancy provided to the data management processing.

For the real application described in this article, wireless communication TGMs were installed on each spring hanger of a top fired reformer, as shown in Fig. 12. Each TGM reads the thermal expansion of four tubes, as that is the spring hanger support configuration.

Finally once the information is contained in the DCS and data historian, the data is presented on proprietary software developed to display a representation of the radiant box plant view. This software is able to present a graphic display of the reformer in plan view showing a gradient of colours to indicate displacements of the TGMs installed on the hangers.

The software also presents statistical information about temperature showing the maximum, minimum, and average at different times as well as standard deviation and rate of change statistics. It also allows for taking images and saving events amongst other features.

In addition, the software allows for remote connection to the data making it possible to monitor the reformer condition at all time and from remote locations.

The data collected can also be exported to an Excel spreadsheet for further analysis and interpretation. This feature facilitates the development of life management strategies for the catalyst tubes.

### Application

This technology is currently installed in two top fired reformers (spring hangers) and one terrace wall reformer (counter weight) with the first installation made in 2012 following the type of configuration shown in Fig. 11. The software developed facilitate the operators' surveillance, and for data acquisition and analysis for engineers.

On one of the top fired reformer installations a total of 200 TGM are mounted on the tube spring hangers as shown in Fig. 12, the signal of each TGM is directed to the DCS and data historian.

There is one display representation for the DCS and another for the data historian.

Since the initial installation of the TGM system, a number of temperature excursions have been observed and damage was successfully averted by operator action. The data gathered, makes it quite clear that TGMs are the first alarms to indicate a temperature excursion.

### Visibility to control room operators

To enable the TGM system to deliver the desired protection requires constant and clear visibility to control room operators. This allows for quick reaction time in case any temperature related problem occurs in the reformer.

Software displays are on 100% of the time and located next to the shift leader position and viewable by everyone in the control room.

The panel DCS view can be called up and is often used by operators.

### Data quality

The TGM data is able to yield 5°C accuracy with 1,050°C of span. As explained this is a bulk or average temperature of the tubes supported by each spring or counter weight.

The scan rate for the data is set by the owner and is a trade-off between

battery life and response time. A one-minute update rate has been found to be a compromise that gives reasonable battery life and good response time and data resolution.

### Case histories

Since the installation of the TGMs in a real application, there have been numerous cases examples that have shown the value of these instruments.

The following cases show just two situations where the TGMs have given valuable information to the operators:

#### Local heating

Many tubes underwent a 'hot banding' episode after an instance of heavier feed gas. A reformer went from the condition shown in Fig. 13 to the condition shown in Fig. 14 over a period of 44 minutes.

With no change in operating parameters, the rise in temperature was detected by the TGMs and the field operator measured 1,000°C with the pyrometer but only over a 1 m length of tube.

As a result of the information provided by the TGMs, quick action was taken to increase process steam rate for a period of time until the carbon accumulation dissipated and tube temperature returned to normal. By acting quickly, more significant carbon accumulation and catalyst damage was avoided, potential significant overheat of the tubes was avoided, and production loss was limited.

#### Local hot spots detection and reformer instability

The TGMs are also valuable to detect individual tubes in the reformer or regions of tubes in the reformer that flip between hot and cold due to flue gas flow pattern instability. This is a phenomenon sometimes experienced in large-scale down-fired reformer furnaces<sup>10</sup>. Figure 15 shows this condition in a reformer.

## Chiyoda remaining tube life assessment

The life of catalyst tubes operated in steam reforming furnaces is affected by creep damage under high temperature and high stress level conditions and they finally crack. Usually the leakage is avoided by monitoring the remaining tube life. The progress of creep damage relates mainly to the exposed temperature condition of catalyst tubes. The outer surface condition of the tubes, especially colour and roughness, are changed by exposure to high temperature, and therefore it is possible to estimate the temperature based on the observation of outer surface colour and roughness<sup>11</sup>. With this, the remaining life of catalyst tubes can be managed to maintain the safety and the reliability of the steam reformer furnace.

Chiyoda has assessed the remaining life of catalyst tubes since 1971. Two methods are applied to find the remaining life:

- destructive metallurgical examination, including creep rupture testing;
- non destructive testing method consisting of UT attenuation, ET and dimensional measurements (this method, called 'H' Scan, was developed by 'H' Scan International Inc.)

Chiyoda selected three commonly used heat resisting cast alloys that it has supplied for catalyst tube materials which have the properties of improved creep rupture strength and stress relaxation properties: HK40 (25Cr-20Ni-0.4C) an iron base cast alloy supplied by Chiyoda since 1964; IN519 (24Cr-24Ni-Nb-0.3C) an iron base cast alloy, the most common material supplied since 1974; and HP microalloy (25Cr-35Ni-Nb-0.3Ti-0.5C) an iron base cast alloy, available since 1984. In 2015 more than 80% of catalyst tube material supplied by Chiyoda was HP microalloy.

From the result of destructive metallurgical examinations, the relationship between the changes of inner diameter, namely bulging and life consumption of catalyst tube by creep damage was found. The worst bulged area corresponds to the highest temperature portion. The skin temperature distribution of catalyst tube is estimated to measure the hardness along the tube length. The lower hardness indicates the higher tube skin temperature. The remaining life of catalyst tubes is managed to monitor the changes of inner diameter at the highest temperature area of the tube.

The temperature profile and the value of temperature are estimated from the result

Fig 16: Outer surface condition along the tube length for HP-microalloy catalyst tubes



PHOTOS: CHIYODA

of hardness measurement and microstructure observation. These are destructive examination methods and it is better to find the information with a non destructive method. The outer surface conditions, especially colour and roughness of used catalyst tube, relates to the exposed temperature and time and it is possible to estimate the condition or remaining tube life by monitoring the outer surface condition including the period of operation.

Figure 16 provides an example of the outer surface appearance for HP-microalloy catalyst tubes in an actual plant. These photos were taken from the top level of the tunnel located at the floor in the furnace. The outer surface top and upper portion seem to be covered with reddish black scale and the outer surface of the middle to bottom portion was covered with blackish scale.

It is hard to observe the outer surface conditions from the floor level of the furnace. It is better to find the outer surface condition from a right angle.

Furthermore, not only colour and roughness information, but also spectroscopic information for outer surface can be

obtained by using a spectroscopic probe.

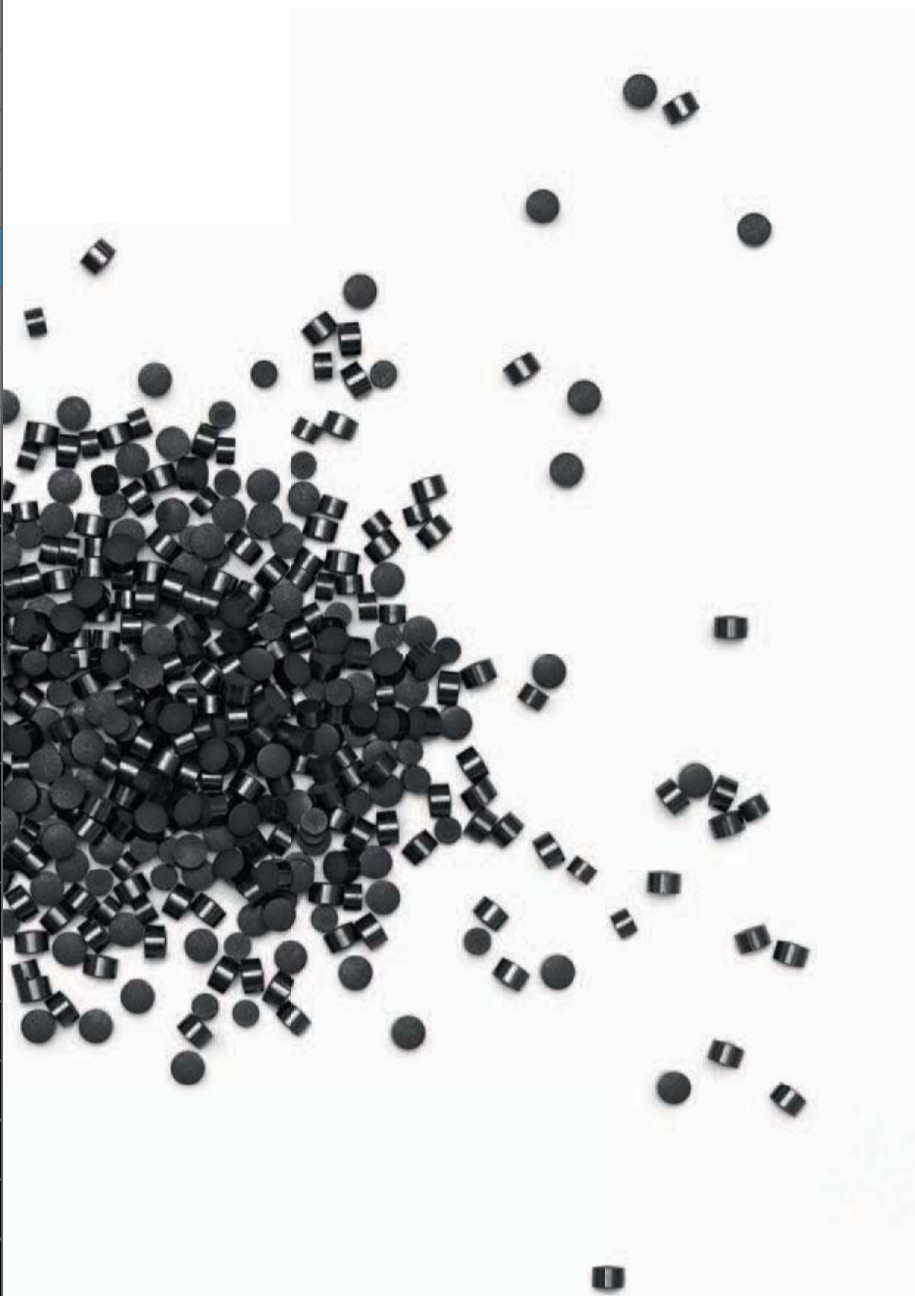
A so-called 'Surface Scanner', consisting of TV cameras with lighting, encoder to measure the distance from the standard point, and an air motor to move along the tube, can be used to observe the outer condition of the catalyst tube automatically. The Surface Scanner can also be combined with H-Scan equipment developed by H-Scan International Inc.

In conclusion, the results of destructive metallurgical examinations and non-destructive inspections for HK40, IN519 and HP-micro catalyst tubes have proven the following:

- The temperature profile of the catalyst tube can be estimated based on the measurement of hardness along the tube length. The lower hardness portion of catalyst tube in the furnace corresponds to the higher temperature portion where the progress of creep damage is faster than other lower temperature portion.
- The temperature of the catalyst tube can be estimated by observing the microstructure compared to the standard microstructure which is prepared under known various temperatures and aging periods. The effect of temperature and time is evaluated with Time, Temperature Parameter (TTP). The Larson Miller Parameter (LMP), a familiar TTP, is applied to HK40, IN5 19 and HP-microalloy materials.
- The outer surface condition, especially colour and roughness, of catalyst tube indicate the tube skin temperature condition. Reddish brown and reddish black colour means low temperature, less than 1000 K, on the other hand blackish colour means high temperature, more than 1000 K, and smooth surface means the higher temperature, more than 11 70 K.
- It is useful to observe the outer surface condition for the whole catalyst tube in the furnace since the distribution of tube skin temperatures for whole tubes, clarifying the higher temperature portion, corresponds to the severe creep damage portion.
- The equipment necessary for observation of the outer surface condition of catalyst tube has been proposed. It was applied to actual catalyst tubes in a steam reformer furnace.
- When areas of blackish surface, especially smooth surfaces, are found, it is necessary for the catalyst tube to be sampled and the remaining life estimated quantitatively by creep rupture testing.

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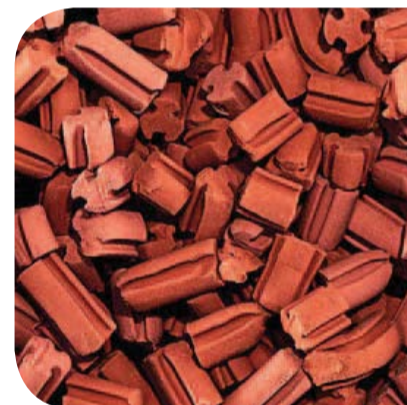
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## Yara life cycle management of reformer tubes

The implementation of a best practice for the life cycle management of reformer tubes is an enterprise-wide goal for Yara International<sup>12</sup>. Yara management recognised that they operated a large number of steam-methane reformers (reformers) globally and the management of these assets was not uniform and could be improved. The number and frequency of tube failures occurring at some of the sites was indicative of the improvement opportunity. More efficient, safe and reliable operation of these assets was viewed as critical to the company's success and reason for the focus on a specific best practice. Quest Integrity was recognised by Yara for their reliability expertise in the ammonia and broader syngas industry, specific service and technology for reformers, and as a provider of world class products and services.

Together, Yara and Quest Integrity developed a project scope and a plan to provide training for operators and reliability groups, implementation of tube temperature management, and reformer performance and reliability surveys of this global set of assets. These best practice program elements of reformer care were implemented for all of the Yara International reformer assets during the time span of mid-2013 to early 2015.

### Overview of Yara reformers

Yara has 12 operating ammonia plants located worldwide. These ammonia plants and reformers vary in process technology, reformer design, capacity, and age as well as production efficiency and reliability. The reformers are a combination of top-fired, side-fired and terrace wall designs. The oldest reformer in the system is the Yara Le Havre, France, reformer commissioned in 1967 and the newest reformer is the Yara Tertre, Belgium, reformer commissioned/rebuilt in 2010 following a firebox explosion. There are a total of 3,180 tubes in these 12 reformers located in eight production complexes around the globe. The oldest tubes were installed in 1977 in Porsgrunn, Norway, and Ferrara, Italy. The average age of the installed reformer tubes is around 20 years.

### Basic elements of Yara catalyst tubes management best practice

Reformer catalyst tube reliability involves the whole production facility organisation (inspection, production, and maintenance)

in order to systematically address all the phases of the tube life-cycle. Yara's approach to the life cycle management of reformer tubes, as detailed in the best practice document<sup>13</sup> Yara BP63 (BP63), involves training of personnel at both an operating and management level to ensure that they have an understanding of materials, common damage mechanisms, and inspection and monitoring techniques. This knowledge is used to evaluate manufacturing or fabrication deficiencies, deterioration or damage in service and the life cycle effects of operation deviations. The goal is to make optimum use of the reformer tubes (plus the pigtailed and headers) over the expected life and avoid untimely and costly premature failures.

Specific steps are defined and accomplished to manage reformer tubes over each operating cycle for the reformer. The operating cycle is defined as the period of time between two consecutive turnarounds, typically four or five years. These asset management steps include an initial survey of the reformer conditions during operation soon after start-up, continuing visual inspection and tube temperature surveys on a regular basis, life assessment of reformer tubes and components prior to a scheduled turnaround, inspection and assessment during turnarounds, and careful observation and execution of start-up and commissioning. The cycle of the activities is then repeated.

### Reformer survey and cost-benefit analysis

In 2013, two years after the development of BP63, Yara performed a survey on all of the reformer assets. From the survey, Yara recognised that the management of these assets was not uniform and could be improved. The catalyst tubes failure history data included in the survey determined that an average of eight tube failures every three years were occurring (not including tube failures due to incidents/trips). It was also clear that the areas with the greatest room for improvement were the temperature monitoring program and the residual life assessment of the catalyst tubes.

During the 2013 survey, a cost-benefit analysis was performed to estimate the potential benefit of a full and uniform implementation of BP63. This analysis was executed according to API 581<sup>14</sup> calculating the tube probability of failure due to creep in the following two conditions:

- full implementation of Yara BP63: "highly effective" inspection, monitoring and assessment program;

- 2013 basis or actual situation: "fairly effective" inspection, monitoring and assessment program.

The result of the analysis showed a significant potential benefit.

Based upon the 2013 reformer survey and the estimated potential benefit, Yara selected Quest Integrity to assist with the global implementation of BP63 through a specialised program of "reformer care" with the ultimate objective to reduce the number and frequency of catalyst tube failures.

### Reformer care

Quest Integrity's Reformer Care includes data analysis, remaining life assessment, and engineering solutions. These services can be applied individually to address a specific issue or may be grouped together to apply to a wide range of integrity issues.

These services offer a unique solution to attain operational safety and reliability goals, enable proactive decision making, eliminate premature harvesting of reformer tubes, improve knowledge of turnaround requirements and reduces costs with proper planning, increase the understanding of reformer operation and limitations, and addresses all reformer systems.

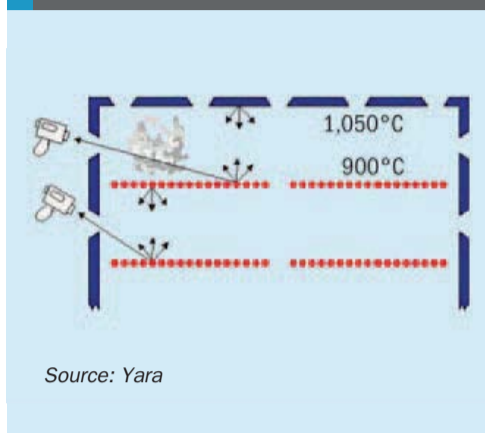
Yara selected a sub-set of these Reformer Care services or program elements to assist with their global implementation of BP63. This set provided Yara with a specialised program of Reformer Care to assist with the strategic management of the 12 reformers.

These key elements included:

- training for operators on visual inspection of reformers including tubes conditions, burner operation, and general condition;
- training for operators on tube temperature measurement practices Implementation of reformer tube temperature correction procedures;
- recommendations for maximum allowable tube metal temperatures;
- reformer performance monitoring to evaluate the current condition in service and identify and recommend performance and reliability strategies for improvement.

These elements were chosen by Yara to close the gaps (against BP63) identified during the 2013 reformer survey, with particular focus on maximum allowable temperature (MAT) determination, temperature monitoring and residual life assessment of the catalyst tubes.

Fig 17: Radiation thermometry errors: effects of tube emissivity, reflected radiation and flue gas



The implementation of the “Reformer Care” services was accomplished according to a schedule developed by Yara. The schedule was based upon Yara’s evaluation of several factors including the turnaround timing of the reformers, history of operational and reliability issues, age of reformer tubes and planned replacements, and availability of site personnel.

The Yara Global Inspection Group had the overall responsibility for coordinating on-site training activities and coordination of information about the reformers to Quest Integrity ahead of the onsite work. Information exchanged included operating and design information, inspection results, and failure and replacement history. This information was used to develop specific training materials for each of the reformers, provide background information for evaluation of the reformers, and to customise the temperature correction software application for each reformer.

Site work and training was completed in 4-5 days per reformer.

### Training

The on-site training for Yara personnel was accomplished in both a classroom and field environment.

Typical training attendance was 10-20 people and a mixture of operation, reliability, and inspection personnel. Quest Integrity senior engineers delivered the training, performed equipment field surveys, demonstrations and practical application of methods presented in the classroom.

The training classroom and field application portions of the work were accomplished in two days and split between reformer performance monitoring and reformer tube temperature measurement and corrections.

### Reformer performance monitoring

The topics covered in the training included background information on reformer reliability management, recognition and troubleshooting of reformer performance issues, reformer tube damage mechanisms and inspection, fitness-for-service and remaining life of reformer tubes, and how to recognise, troubleshoot and prevent common reformer tube failures. This training included normal and abnormal tube conditions, bowed tubes, hot spots, bulges, cold tubes, and conditions due to catalyst damage and poisoning.

Training specific to burners in operation was included to address burner characteristics and design, correct operation of the burner, adjustments, maintenance, and troubleshooting.

Practical training on tube and reformer visual inspection during operator rounds was discussed in the classroom and then practiced in the field by participants using specifically prepared check list

### Reformer tube temperature management

Accurate measurement of reformer tube-skin temperatures is a crucial input to a tube life prediction model (not directly based upon measured creep damage) and is also critical in optimising productivity and ensuring optimum reformer operation. Radiation thermometry is a practicable and reliable method for determining tube-skin temperatures in reformers provided certain measurement corrective practices are used. Radiation thermometry in reformers is prone to a number of errors arising principally from the effects of tube emissivity, reflected radiation and flue gas (Fig. 17). Radiation thermometer readings and thermal images must have corrections applied in order to obtain the true tube-skin temperatures. These corrections depend in a complex way on the geometry of the reformer, the temperature distribution of objects surrounding any individual tube, and the location of that tube in the reformer. The corrections also depend on the operating characteristics of the radiation thermometer or thermal imager used to make the measurements.

The second day of classroom and field training was dedicated to the infrared radiation (IR) temperature measurement theory and techniques for reformer tubes. The classroom training discussed the application of IR pyrometers and imaging cameras for monitoring reformer tube temperatures along with the operator techniques required to obtain consistent temperature

measurements. The classroom training was followed by practical application of the techniques to gain familiarity and a level of proficiency with the IR measurement instruments. Temperatures recorded in the field were then corrected through demonstration and practical experience with Quest Integrity’s CorrectIR™ software application.

### Temperature correction – CorrectIR™

As an element of the Reformer Care implementation, Yara implemented an IR temperature correction program to establish the actual reformer tube metal temperatures. In order to determine the true tube temperatures, corrections must be made to the thermometer readings based on the geometry of the furnace, the emissivity of the tube material, atmospheric effects, and knowledge of the operating characteristics of the thermometer itself. The correction of reformer radiance temperature readings (measured without correction) to corrected temperatures is accomplished using Quest Integrity’s CorrectIR™ software and method. The methodology and software is based upon the work of Dr Peter Saunders. Features of CorrectIR™ include:

- corrects radiance measurements for calibration, size of source effect, flue gas emissions and other instrument and environmental errors;
- calculates the uncertainty associated with individual factors;
- calculates the effective background temperature taking into account rigorous geometry for each tube;
- calculates the tube corrected “true” tube temperature and the total uncertainty.

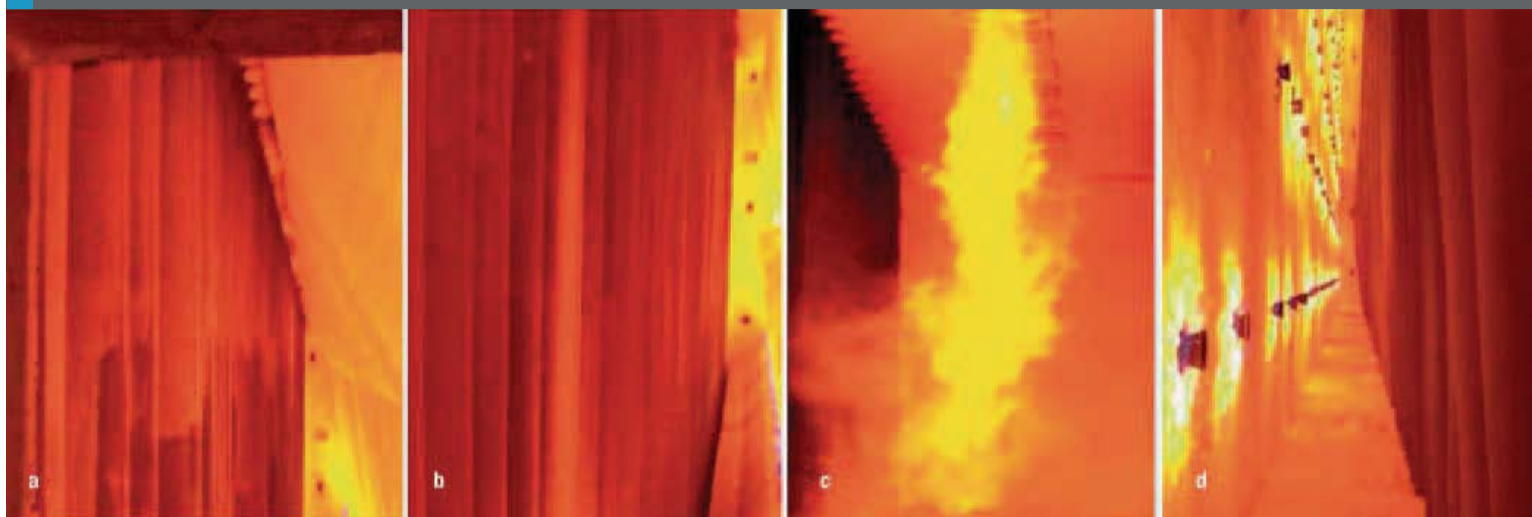
### Baseline reformer tube IR survey and tube wall temperature limits

As an element of Reformer Care, Quest Integrity completed reformer tube temperatures surveys of each of the Yara reformers. These surveys were conducted using IR Imaging Cameras and in some cases IR pyrometers.

The surveys provided Yara with a baseline survey of tube metal temperatures which were corrected using CorrectIR™. The corrected tube wall temperatures (TWT) were lower than the measured radiance temperatures. In many instances when compared to operating temperature limits, these corrected “true” temperatures would allow for increase firing and ammonia production currently.

Quest Integrity also provided recommended TWT limits (maximum allowable temperature, MAT) for each of the Yara

Figs 18 a-d: Examples of issues found during surveys



a) Yara Sluiskil H501 reformer: external scale on upper tubes.

b) Yara Sluiskil H501 reformer: tube appearance as 'hot' due to external scale.

c) Yara Tertre B101 reformer: flame pattern with baking soda test.

d) Yara Ferrara B201 reformer: bowed tubes; uneven tube colour; flame impingement on tubes; tramp air entering from burners out-of-service with air register open.

Source: Yara

reformers. Quest Integrity has developed a unique creep material model for HP alloys that accounts for material aging and creep strain rate with respect to stress, temperature and time. This proprietary creep material model is called LifeQuest™ Reformer.

LifeQuest™ Reformer was used to define reliable and safe tube metal temperature operating limits for the Yara reformers. The main difference between the original design life estimates provided by the tube manufacturer or the furnace designer and the Quest Integrity service life estimate is that for the original design the tubes are assumed to have retained their original mean or lower bound as manufactured creep properties whereas the Quest Integrity model takes into account that the creep properties will progressively degrade. This information was also provided as a creep rupture life curve for each reformer.

The creep rupture life curve for the reformer tubes are used to assess the life impact from changes in operating conditions. With a corrected tube metal temperature (plus uncertainty) value above the safe limit, the creep rupture life could potentially be extremely short resulting in tube failure during the operating period.

Finally, during a reformer shutdown, the actual consumed creep life may be measured and calculated by utilising Quest Integrity's proprietary inspection tools, LOTIS® and MANTIS™ and the Level 3 LifeQuest™ Reformer assessment program.

#### Performance and reliability monitoring

Quest Integrity performed a reformer survey to evaluate each of the Yara reformers. The primary objective of the work was to observe the reformer in operation to assist with balancing the reformer firing and to deliver a reliability strategy to ensure the safe and reliable long term operation of the reformers. The survey and reliability strategy included the following essential elements to achieve optimum performance and reliability of the reformers.

- safety and environmental;
- operating performance;
- reliability issues;
- reformer tube condition;
- reformer mechanical condition;
- burner mechanical condition;
- recommended corrective actions for the reformers.

The issues found during the survey (see examples in Figs. 18 a-d) as well as other industry best practices were addressed by a set of corrective actions. Principled execution towards accomplishing these strategic actions will lead to a higher level of performance and reliability with respect to the reformer.

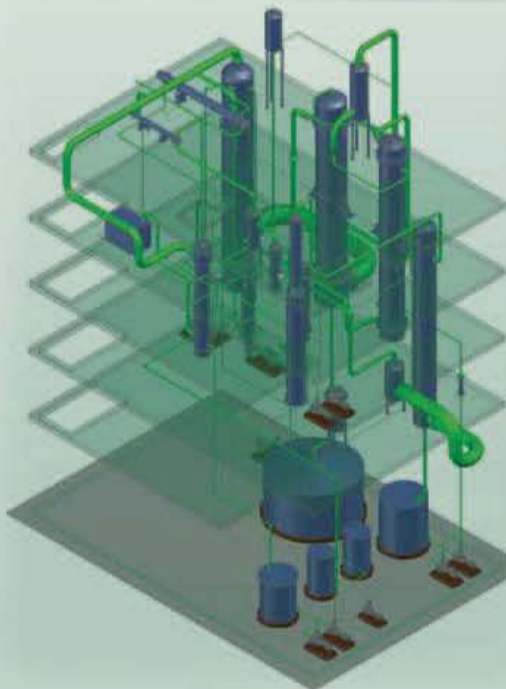
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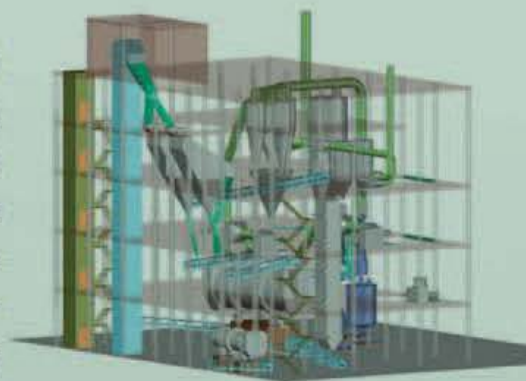
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# Ammonia synthesis catalyst replacement

Conventional loading of ammonia synthesis catalyst by vibration is a very safe method to reach the required bulk density, but is time consuming. Alternative loading methods e.g. the Dense Loading method by thyssenkrupp Industrial Solutions and Showerhead™ loading by Haldor Topsoe provide higher density and faster loading times.

**C**atalyst replacement in an ammonia converter is a difficult and non-routine task during a plant shutdown. Due to the complex nature of the reactor design, the importance of the ammonia converter for the plant performance and that the catalyst replacement is generally on the critical path during an ammonia plant shutdown, there is a high resultant cost risk and a safety risk for the plant owner.

A further difficulty is posed by the fact that the catalyst only needs to be replaced every 10-12 years, and therefore plant operators often lack the relevant experience.

## tkIS full-service package

thyssenkrupp Industrial Solutions (tkIS) makes available its experience and know-how to its customers and offers, in coop-

eration with Johnson Matthey, a full-service package for replacing the catalyst in the ammonia converter. As a general contractor, tkIS not only ensures a competent replacement of the catalyst and a fixed-price catalyst replacement under observance of a set time schedule, but also performance reliability once the plant has been restarted.

The full-service package for execution of the ammonia synthesis catalyst replacement comprises, among other things, project management, supply of the catalyst, refurbishment or replacement of the cartridge, delivery of spare parts, inspection of materials and workmen's services.

The execution concept for the catalyst replacement is reflected in the project organisation chart (see Fig. 1).

The sequence of main activities during a catalyst replacement are:

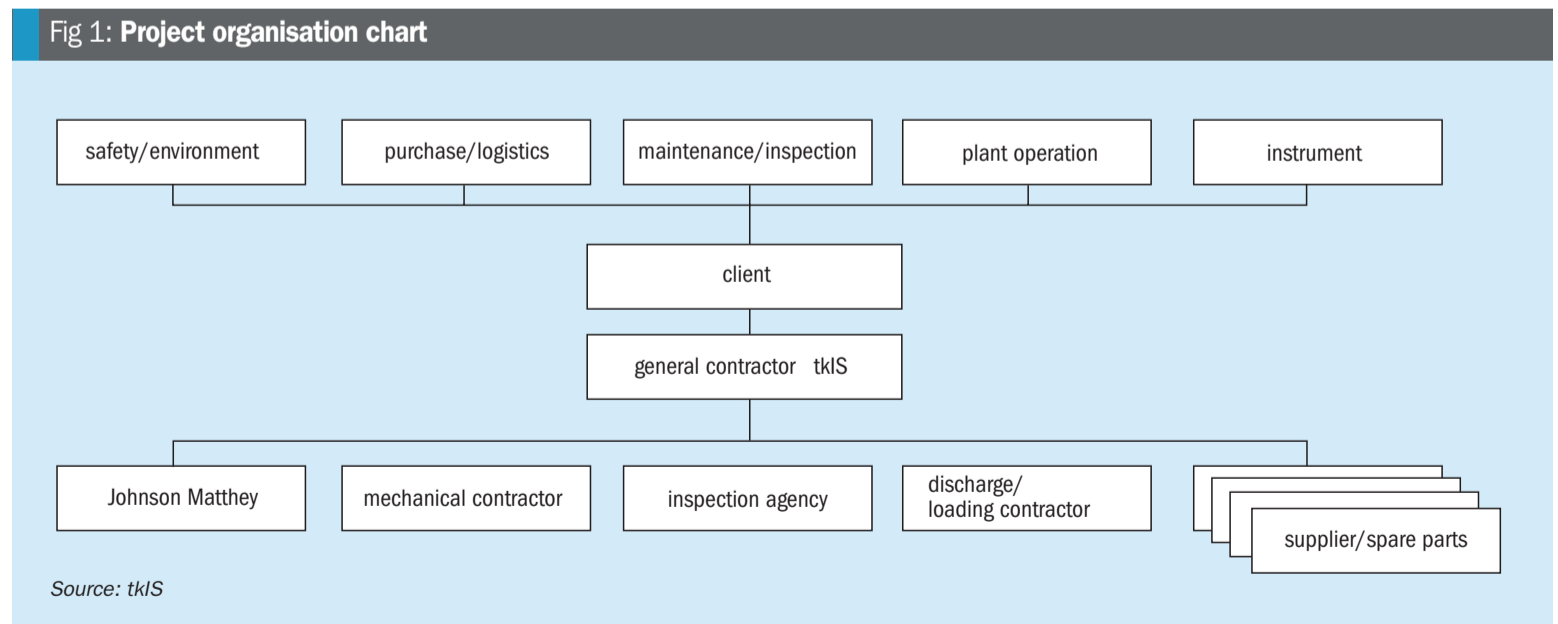
- cooling down;
- nitrogen purging of synthesis loop;
- opening of converter;
- catalyst unloading and disposal;
- inspection and repair works;
- catalyst loading;
- closing of converter / boxing up;
- start up;
- performance test;
- optimisation.

## Loading techniques

Ammonia synthesis catalysts are irregular shaped granules and due to the physical shape some differences in packing density are possible. Therefore any charging method must ensure an even packing density across the converter beds.

Multi-bed converters can be charged using a hopper and tube(s) for filling the catalyst into the catalyst beds. The

Fig 1: Project organisation chart



catalyst should be distributed evenly across the bed when flowing in. The bulk density must be checked regularly as charging proceeds.

Most converters are designed for bulk densities around 2,800 kg/m<sup>3</sup> although it is possible for the charged density to vary from 2,650 to 3,000 kg/m<sup>3</sup> for oxidised catalysts. Bulk densities for pre-reduced catalysts are lower, and a typical target density for pre-reduced catalyst is 2,200 kg/m<sup>3</sup>.

#### Conventional loading by vibration method

The conventional filling method is the catalyst loading by vibration. This method is discontinuous and consists of two steps: The first step is manual filling of catalyst in layers of 30 cm via hoses. The second step is the compacting of the catalyst by using air driven concrete vibrators. The bulk density after manual filling is 2.3 to 2.7 kg/l. Only by vibration the required bulk density of 2.8 kg/l can be reached. The vibration for one layer of the catalyst bed normally takes up to 60 minutes. The vibration method is a very save method to reach the required bulk density, but time consuming. A typical loading rate is only 3 t/h.

As conventional loading is time consuming and the catalyst replacement is often on the time critical path during plant shutdowns, thyssenkrupp Industrial Solutions has developed, in cooperation with Johnson Matthey, a new fast catalyst charging method which has been successfully applied for several Uhde ammonia converters.

#### Catalyst filling by Dense Loading

The tkIS fast catalyst charging method is based on so-called Dense Loading. The advantages of the new loading method are a significantly higher loading rate and, potentially, a higher bulk density. With this technique each particle is given enough time for random fall in the ideal position and void spaces can be avoided. Single distribution of the particles over the whole cross-sectional area of each reactor bed enables a maximum loading rate to be achieved. No compacting by vibration after filling is necessary. Dense Loading is a continuous loading method, either using a static head or a rotating spreader to distribute the catalyst particles over the cross-sectional area of the catalyst bed. By applying the new developed dense loading method bulk densities of more than 2.8 kg/l and loading rates of 15 to 20 t/h can be reached.

Table 1: Duration of main activities during shutdown

Activity	Time required, days
Cooling down, nitrogen purging of the synloop and opening converter	3 – 5
Catalyst unloading	3 – 4
Inspection and repair works	4 – 5
Catalyst loading	4 – 5
Closing of converter	3
Start-up and catalyst activation	2 – 5

Source: tkIS

#### Safety issues

##### Catalyst unloading under nitrogen atmosphere

The catalyst handling has to be performed by a qualified catalyst handling contractor who has the required certificates to work under nitrogen atmosphere. Consequently, the catalyst handling contractor has to work with a breathing protection system/life support system.

Regarding gas analysis equipment the contractor needs to have at least ex-analysis, H<sub>2</sub> analysis and O<sub>2</sub> analysis equipment. At the start of a project, the responsible general contractor has to carry out risk assessments and analyses together with the client and develop safety plans for the project execution team.

##### Safety measures during dumping of catalyst

A safety warden shall be present during dumping of the catalyst. A water hose with a spray nozzle connected to the fire water system shall be ready for use when dumping catalyst.

As the catalyst in the cartridge is active, it is prone to start oxidising as soon as it gets into contact with oxygen. This cannot be avoided. Therefore a safety distance of minimum 5 metres behind and on both sides of the dumping suction truck is required. Water spray after or during dumping of catalyst shall be activated with minimum flow.

Operators handling the catalyst must wear non-inflammable clothing with face protection mask to avoid contact to flash flames during dumping.

##### Other safety precautions

A safety warden shall be present on top of the converter where work is going on.

Catalysts are subject to damage by moisture or liquid water. Precautions must be taken for the unlikely case of rainfall. Either a tent with opening in the top shall

be provided or at least water proof tarpaulins shall be held ready to cover the converters in case of rain.

The thermocouples for temperature monitoring during filling must be functioning and connected to DCS in order to recognize a heating up of the catalyst. Further, a nitrogen hose must be permanently available at converter top during converter opening and catalyst loading.

Operators handling the catalyst must wear non-flammable clothing and oxygen masks when entering the converters.

For mechanical work workers shall wear protective clothing and eye protection during grinding works.

With regard to catalyst dust, eye/face protection, protective gloves, clothing and a dust mask should be worn and dust release to the environment should be avoided.

#### Specific challenges and operating problems

There are a number of specific challenges and operating problems associated with catalyst replacement:

- One challenge for catalyst loading is the limited access to the catalyst beds, especially to the third bed of a 3-bed reactor. The spatial conditions in the ammonia synthesis reactors are restricted due to permanent fittings and internals like the thermowells and there is little space between the catalyst beds.
- Unloading under nitrogen atmosphere to avoid oxidation (fire) of reduced catalyst.
- A catalyst which has experienced many start-up and shutdown operations or has been in operation for more than 15 years is usually compacted in some parts of the catalyst volume such that unloading of the catalyst with vacuum units is hardly possible. The compacted catalyst has to be loosened by using pickaxes or jackhammers. This has to be done under nitrogen atmosphere.

- Working in a confined space with toxic material.
- Control of uniform particle size of catalyst particles to avoid uneven distribution of catalyst or inconsistent bulk density of the catalyst beds.
- Use of pre-reduced catalyst in order to shorten reduction time of catalyst during start-up and in order to reduce likelihood of problems resulting from a flow imbalance. Pre-reduced catalyst is material that has been reduced and then stabilised by re-oxidising the outer surface of the catalyst particle. The catalyst should not be exposed to higher temperatures or sunshine or water (rain) as this may lead to oxidation and self heating of the pre-reduced catalyst.
- Rain shelter required at the top of the converter to avoid wetting of catalyst and cartridge insulation.
- An even packing density across the converter beds must be ensured to gain the maximum benefit from the catalyst. Ammonia synthesis catalysts are irregular shaped granules and due to the physical shape wide differences in packing density are possible. This is undesirable as the gas flow through the bed will be uneven which can create hot spots and/or result in a low converter efficiency. Therefore any charging method must ensure an even packing density across the converter beds.
- In addition to excellent technical expertise also extensive project management skills are required for the success of the catalyst replacement project. A systematic planning of the shutdown is very important, as the project execution

is determined by budget and schedule constraints. Further, the project management has to manage the contractors and specialists like mechanical contractor, catalyst contractor, inspection agency, catalyst supplier as well as clients' departments like safety, purchase/logistics, maintenance/inspection, plant operation and instruments. This results in a high complexity of the task and a professional project management is definitely required.

**Timescale**

The typical duration of the main activities during the shutdown are shown in Table 1.

In summary, the catalyst replacement takes approximately 3-4 weeks.

**Topsoe technical service programme**

Topsoe offers a wide range of services that ensure clients get the full benefit of Topsoe's KM ammonia synthesis catalysts. Users of the KM catalysts gain access to a service platform ranging from advanced test facilities to proprietary kinetic models, combined with 60 years experience in both catalyst and technology related issues.

**Showerhead™ loading method**

Showerhead™ loading is a unique dense loading method developed by Topsoe. It provides a highly uniform and dense loading (Fig. 2), enabling operation at more favourable conditions. This ensures optimal catalyst utilisation, resulting in significantly increased ammonia production. In addition, the Showerhead™ method facilitates a fast and smooth loading,

minimising downtime. Compared to the conventional vibration method, Showerhead™ results in up to 7-8% higher loading density and is 2-3 times faster with no risk of damage to the basket. A significant advantage of higher density loadings using Showerhead™ is an increased production of 150,000 tonnes over a typical catalyst lifetime (Fig. 3).

**Case history**

A large fertilizer producer was looking to improve ammonia loop performance. After consulting with Topsoe, the company decided to revamp the 27-year-old Topsoe S-200 ammonia converter with new Topsoe S-300 internals.

Topsoe recommended its proprietary Showerhead™ loading method to make sure that the plant got the most out of its new basket. Showerhead™ works entirely by the force of gravity, with the fall of catalyst particles into the converter resulting in a higher loading density. To ensure uniform density, the showerhead-like catalyst distributors are in constant motion during the loading. The special Showerhead™ loading equipment was delivered by a team of Topsoe specialists, who provided guidance and assistance during the process.

When the loading was finished, management could see that the Showerhead™ loading method had increased the filling density by more than 5% over the previous loading, where a traditional vibrating-rod method had been used. What's more, the loading team managed to load the reactor at a rate of more than 2 m<sup>3</sup> of catalyst per hour, twice as fast as they had done previously with vibrating rod.

Fig 2: Showerhead™ loading gives higher ammonia catalyst density

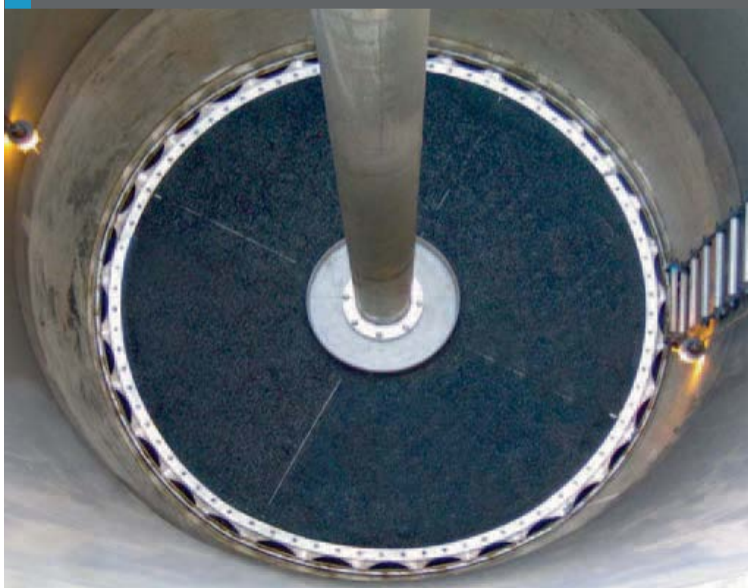
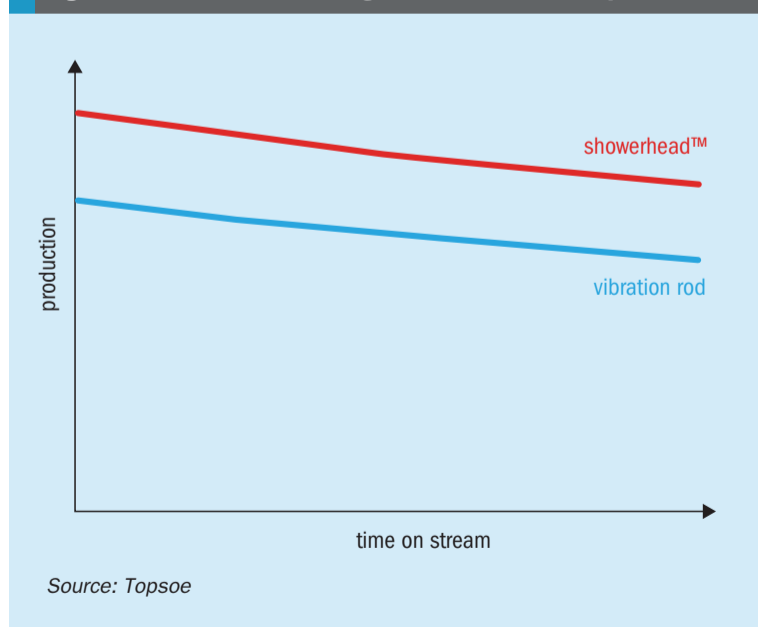


PHOTO: TOPSOE

Fig 3: Showerhead™ loading leads to increased production



Source: Topsoe

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# Inspection of HP urea equipment at KPIC

Plant corrosion inspections are a valuable tool to assess the condition of a urea plant. Remaining lifetimes of tubes, lining and equipment can be determined if a history of measurements and operation are available. In this article, **Hasan Akbari** of KPIC reports on the inspection results of the main high pressure equipment items in the synthesis section of the urea plant at KPIC which have been in service for eight years and were opened for the first time during a recent downtime.

Urea plants operate at high temperature (170-200°C) and pressure (140-150 bar) and produce an intermediate product (ammonium carbamate solution), which is extremely corrosive to materials. The high pressure equipment items in the urea plant are typically multilayer vessels. The inner core (liner) is a protective layer that is usually fairly thick (8-12 mm) to provide sufficient lifetime against process fluid corrosion. Other layers are carbon steel that are fitted around the inner core. Because of the highly corrosive fluid in the synthesis section of the urea plant, inspection of the internal lining is very important. Some reports about inspection and repair of high pressure equipment of urea plants are listed here. Juneja et al<sup>1</sup> introduces various corrosion mechanisms present during urea manufacturing and materials of construction used worldwide for equipment and piping of urea plants. *Nitrogen+Syngas* magazine<sup>2</sup> reviews the key considerations when relining a urea reactor that has been subject to corrosion over time. Gevers et al<sup>3</sup> reports on investment and experiences in a revamp project to ensure process plant designs. Eijkenboom et al<sup>4</sup> presents Safurex material and also inspection results of urea high pressure equipment that has been fabricated from Safurex. Idrees<sup>5</sup> highlights the issues faced in identifying a urea stripper leakage during plant operation without taking the stripper out of service and then rectifying the leakage. Baretelli et al<sup>6</sup> introduces the results of using zirco-

nium tubes in a urea stripper. Fateh et al<sup>7</sup> shares an experience during leak testing of a urea stripper and also recommends possible measures to tackle these problems in order to achieve improved testing effectiveness. Mingda Song et al<sup>8</sup> presents the results of a failure analysis on a ruptured urea reactor. Shugen Xu et al<sup>9</sup> analyses the disadvantage of the original urea reactor design and proposes a new circumferential weld structure for the layered urea reactor.

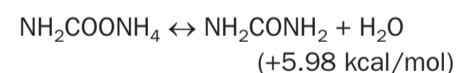
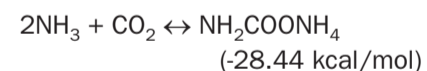
## Urea synthesis section at KPIC

Kermanshah Petrochemical Industrial Company (KPIC) owns and operates one ammonia and urea plant in Iran. The main high pressure equipment items in the synthesis section of the urea plant at KPIC includes: the urea reactor, stripper, pool condenser and off-gas scrubber. The urea reactor and pool condenser are both multilayer vessels while the stripper and scrubber have a solid wall. These equipment items have an internal lining to protect the shell against highly corrosive carbamate solution. Protective linings are weld-overlay cladding or loose lining. Multilayer vessels are built up by wrapping a series of sheets over a core tube. The construction involves the use of several layers of material, usually for the purpose of quality control and optimum properties. Multilayer construction is used for higher pressure. It provides inbuilt safety, utilises material economically and no stress relief is

required. For corrosive applications the inner liner is made of special material and is not considered for strength criteria. The outer load-bearing shells can be made of high tensile low carbon alloys. A solid wall vessel consists of a single cylindrical shell, with closed ends. Due to the high internal pressure and thickness of the wall, the shell is considered as a thick cylinder<sup>10</sup>.

## Process description

The urea plant includes the urea melt unit and granulation unit and has been designed based on the process design package provided by Stamicarbon and Hydro Fertilizer Technology (HFT). Urea is produced in two steps by reacting liquid NH<sub>3</sub> and gaseous CO<sub>2</sub> according to the following equations:



Both reactions take place in a pressure range of 135 to 145 bar at a temperature between 170°C and 185°C. The conversion of NH<sub>3</sub> and CO<sub>2</sub> into ammonium carbamate is fast and exothermic. The conversion of ammonium carbamate into urea and H<sub>2</sub>O is slow and endothermic. Fig. 1 shows a process flow diagram of the urea synthesis section. NH<sub>3</sub> with carbamate from the HP scrubber and CO<sub>2</sub> with the off-gas from the HP stripper, are introduced into the pool

Fig 1: Process flow diagram of urea synthesis section

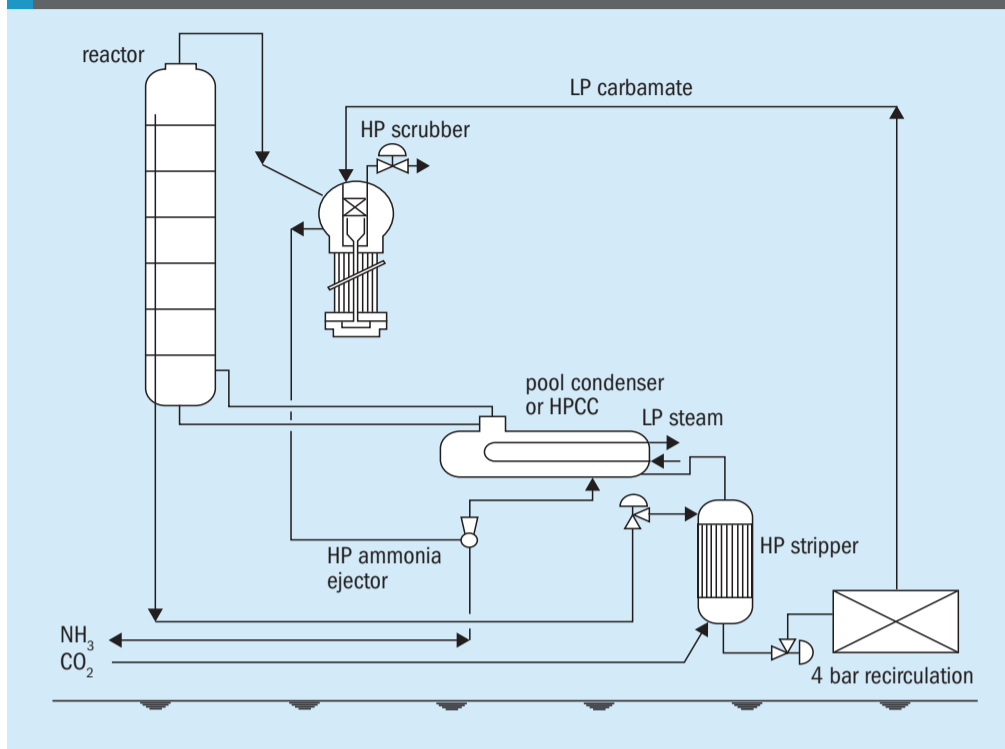


Table 1: Chemical composition of BC.01 and BC.05

Material	C (wt-%)	Cr (wt-%)	Ni (wt-%)	Mo (wt-%)	Mn (wt-%)
BC.01	max 0.045	min 17	min 14	2.2-3	min 3
BC.05	max 0.04	min 24	min 21	1.9-2.7	min 3

condenser, which is a special design liquid submerged U-tube type heat exchanger. The greater part of this gas condenses and forms carbamate. Part of this carbamate is converted to urea in the pool condenser, the remainder is discharged to the reactor. Heat of condensation is used to generate LP steam in the pool condenser at a pressure of about 4.5 bar. The pool of liquid in the pool condenser provides sufficient time to allow urea formation to take place by dehydration of ammonium carbamate into urea and water. The formed urea, the remainder of the carbamate, and the non-converted NH<sub>3</sub> and CO<sub>2</sub> (gaseous) are subsequently introduced into the bottom of the urea reactor where the carbamate is further converted into urea and water. The reactor volume allows sufficient residence time for the reaction to approach equilibrium. Heat required for conversion of carbamate into urea is supplied by additional condensation of NH<sub>3</sub> and CO<sub>2</sub>. Reactor effluent flows through the downcomer of the reactor to the stripper. Reactor overhead gas is sent to the scrubber. The carbamate solution from the scrubber flows to the HP ejector. The ammonia feed pressure induces sufficient head

in the HP ejector to convey the carbamate solution from the scrubber to the pool condenser. Reactor effluent, containing about 34 wt-% urea is distributed over the tubes of the stripper. This is a falling film type shell and tube heat exchanger where the reactor effluent is contacted countercurrently with CO<sub>2</sub>, causing the ammonia partial pressure to decrease and carbamate to decompose. The heat required for this process is supplied by condensation of saturated 21 bar steam around the tubes of stripper. Urea-carbamate solution from the stripper bottom is discharged to the recirculation section while gas from the top is sent to the pool condenser. Oxygen is needed to maintain a passive layer on the stainless steels in the synthesis section. The dehydrogenated CO<sub>2</sub>, containing a minimum of 0.6 vol-% oxygen for passivation, is introduced into the bottom part of the stripper<sup>11</sup>.

### Materials of construction in urea plants

The material of construction essentially affects the reliability, operability and maintainability of the urea plant. When

selecting a material of construction, a combination of material factors (e.g. corrosion and erosion resistance, mechanical property, weldability and cost) and environmental factors (e.g. operating temperature, dissolved oxygen and fluid composition) should be considered. Candidate materials for the aggressive environments in a urea plant include zirconium, titanium, duplex stainless steel and stainless steel. Because of the susceptibility to erosion and difficulty of welding, titanium has been gradually taken over by stainless steel. Stainless steels in a corrosive environment like ammonium carbamate owe their corrosion resistance to the presence of a protective oxide layer on the surface. As long as this layer is intact, the metal corrodes at a very low rate. Stainless steels exposed to carbamate solution can be kept in a passivation state by adding a sufficient amount of oxygen. If the oxygen content drops below a certain limit, active corrosion starts. 316L-UG (BC.01) has been used for a long time in urea plants because of its excellent weldability, fair corrosion resistance and relatively low cost. Several failure modes and the requirement of a relatively large amount of passivation air by 316L-UG in synthesis and recycle sections restricts its operability. Type 25Cr-22Ni-2Mo SS (BC.05) is being used due to its better corrosion resistance than 316L-UG and excellent weldability. These material types are susceptible to chloride stress corrosion cracking.

Table 1 shows the typical chemical compositions of BC.01 and BC.05. Duplex stainless steel has excellent corrosion resistance in both weld metal and heat affected zones regions. These possess better resistance to stress corrosion cracking and hence can also be used in chloride environments. In carbamate solutions with less oxygen, duplex materials like Safurex have proven to be more corrosion resistant than much more costly materials<sup>1</sup>. In the laboratory the corrosion resistant properties for a duplex stainless steel (Safurex) was compared with 25Cr-22Ni-2Mo as well as 316L-UG material in an oxygen free carbamate solution at prevailing extreme synthesis conditions. Under these conditions it appeared that the Safurex material was not affected, while austenitic stainless steel material was severely actively corroded and BC.01 even disappeared (see Fig. 2).

Fig 2: Zero oxygen corrosion test in autoclave<sup>4</sup>



Fig 3: General view of urea reactor<sup>12</sup>

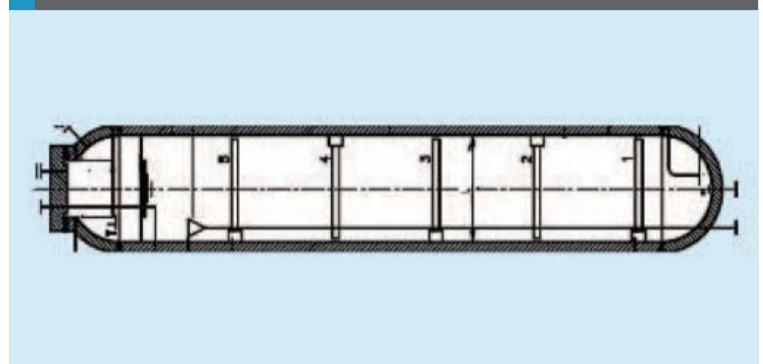


Table 2: Material specifications of reactor components

Shell and manhole loose lining	Overlay welding of heads	Sieve trays, supports and downcomer	Internal nut and bolt
BC.01	BC.05	BC.01	BC.05

### Corrosion mechanisms in the urea synthesis section

Due to the high operating temperature and pressure, as well as the corrosive nature of ammonium carbamate, equipment and piping materials degrade due to corrosion in urea plants.

#### Active corrosion

The corrosion rates of stainless steels exposed to carbamate at relatively high temperatures can be controlled by adding a sufficient amount of oxygen. If the oxygen content drops below a certain limit, active corrosion starts and the corrosion rate becomes extremely high.

#### Erosion/erosion-corrosion

Erosion-corrosion is the acceleration or increase in the rate of deterioration or attack on the metal because of mechanical wear or abrasive contribution in combination with corrosion.

#### Condensation corrosion

As long as the protective oxide layer on the surface of stainless steel is intact, the metal corrodes at a very low rate. Passive corrosion rates in liquid phases are generally between 0.01 and 0.1 mm/year. In gas phases where mixtures of ammonia, carbon dioxide and water vapour can condense to form carbamate solutions, passive corrosion rates can increase to 0.2 mm/year. The condensing effect results in the oxygen becoming too low and accelerated corrosion occur. From the point of view of corrosion prevention, the condensation of NH<sub>3</sub>, CO<sub>2</sub> and H<sub>2</sub>O gas mix-

ture to carbamate solution warrants extra attention. Due to the low amount of oxygen present, a more corrosive condensate is initially formed on condensation. Passivation is believed to take place via a metal ion redox system with oxygen, which is missing in freshly formed condensate. This accounts for the severe corrosion sometimes observed at areas with insufficient or bad insulation, or on so-called “cold spots” in the channels of high pressure equipment and gas lines fabricated from 316L-UG. Such corrosion can be prevented by adequate insulation and tracing.

#### Inter-granular corrosion

In austenitic stainless steels, the cause of inter-granular attack is the precipitation of chromium carbide (Cr<sub>23</sub>C<sub>6</sub>) at grain boundaries. At these locations, chromium rich precipitates are surrounded by metal that is depleted in chromium which is more rapidly attacked than metal surfaces that are not depleted in chromium. In urea plants, this corrosion is caused by the highly oxidising action of oxygen containing urea carbamate solution, a low NH<sub>3</sub>/CO<sub>2</sub> ratio and segregation of impurities in a sensitised stainless steel<sup>1</sup>.

### Result Inspection of synthesis high pressure equipment

#### Urea reactor specifications

The urea reactor at KPIC is multilayer vessel with five sieve trays that is designed and manufactured by Hitachi Zosen Mechanical Corporation. The carbon steel wall has four layers and the internal lining

has a thickness of 8 mm (Fig. 3). The corrosion allowance of the stainless steel lining is 2 mm. The design pressure is 160 bar and the design temperature is 205°C. The shell and head of the reactor have respectively loose and overlay welding lining. Table 2 shows the material specifications of reactor components.

The most common form of corrosion found in urea reactors is general attack. This attack leads to gradual thinning of the corrosion resistant surface over an extended time. Sometimes, however, the overall corrosion attack is accelerated by condensation corrosion, which appears as a jigsaw-like pattern of oxide scale. Internal inspection of the urea reactor at KPIC consists of visual inspection and thickness measurement of internal components by ultrasonic test.

#### Inspection results

##### Inspection of lining

The thickness measurement of the stainless lining showed that the lining surface has uniform corrosion of about 0.5-1 mm. The top sections showed greater loss of thickness and a maximum corrosion rate of 0.125 mm/year. The welding material of the lining is 25Cr.22Ni.2Mo and visual inspection showed that the welding seams were in good condition. Special attention was given to the gas phase and also to the transition between the liquid and gas phase. Fig. 4 shows an image of the lining and weld seams.

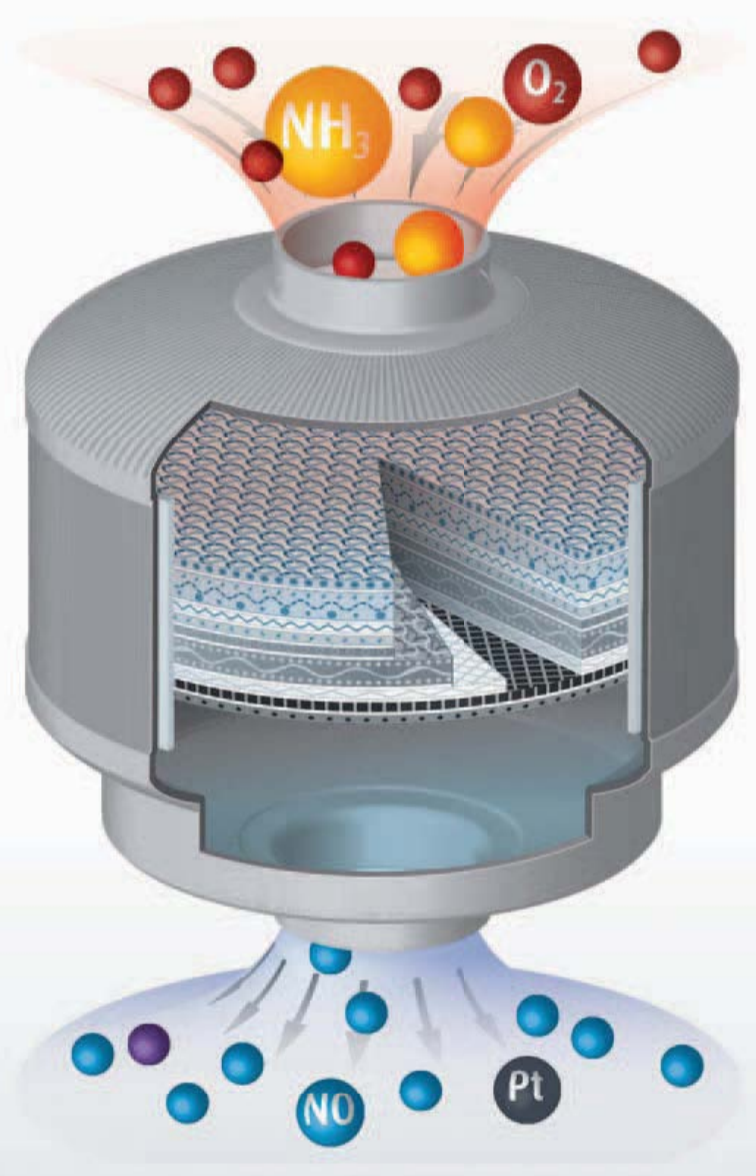
Fig 4: Lining and welding seams of reactor





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**Inspection of sieve trays, supports and downcomer**

Visual inspection and thickness measurement of trays and their supports showed that the thickness has been reduced by between 0.7 and 2.5 mm and subsequently the diameter of the holes have increased by between 0.5 and 3 mm. The maximum corrosion rate was 0.294 mm/year at tray no.s 4 and 5. Visual inspection also showed that all of the nuts and bolts which connect the tray sections were intact and showed no signs of corrosion.

No corrosion was observed at the lug supports and stud bolts of the trays. The thickness measurement of the downcomer also revealed uniform corrosion. The thickness of the downcomer wall is 5 mm and inspection revealed that maximum reduction of wall thickness was 2.25 mm at its top section.

**Inspection of heads and manhole**

The overlay welding of the heads was smooth and no corrosion was observed on the welds, but the lining material of the manhole 316L-UG (BC.01) was corroded. The internal surface of the manhole and head were covered with coloured scale. Figure 5 shows the internal surface of the manhole and the corrosion of the protection layer.

**Radioactive source nozzle inspection**

The urea reactor uses a radioactive source for level measurement. Before entrance to the reactor, the radioactive source was removed and its nozzle pulled out and checked. The material of the nozzle is 316L-UG (BC.01) and its dimensions are 0.D21.3x3.73. The nozzle has three guide plates (thickness = 5 mm) that are welded to the nozzle. Visual inspection showed that the outer diameter of the end cap, a region of the top section and guide plates of the nozzle have severe corrosion. The diameter of the end cap has been

reduced to approximately 18.7 mm (Fig. 6) and the maximum thickness reduction of the guide plates was 2.5 mm in the bottom section. Since the guide plates have been welded to the nozzle, thickness measurement of overall length was not possible, therefore, for safety reasons, the corroded end cap and top section of the nozzle were cut and replaced with new materials 316L-UG (BC.01).



**Stripper inspection**

The stripper was designed and fabricated by SBN in Austria. The material of the tubes, overlay welding of channels and all internal components is 25Cr.22Ni.2Mo (BC.05). The stripper tubes are provided with liquid dividers.

Process conditions are most severe in the stripper, especially at the top of the tubes due to the relatively low oxygen partial pressure and high tube wall temperature. If a liquid divider is not properly seated, a higher corrosion rate may occur due to flooding in the tube.

Visual inspections and eddy current tests of the tubes were performed during the downtime. The liquid distribution system was tested by air. Results of all inspections are explained below.

**Top channel**

Overall, the top channel was found to be in excellent condition. The overlay lining of the channel, nozzles and flange face were smooth and there was no sign of corrosion. The surface of the lining was covered by a thin layer of scale. Visual

inspection also showed a large amount of scale around the tube to tube sheet connections (Fig. 7).

Table 3 shows the chemical analysis of the scale. These deposits probably have two sources. Firstly, they are the product of corrosion of BC.01 (316L-UG) material in the urea synthesis loop, for example internal corrosion of the urea reactor components and the pool condenser lining, and secondly they are from the ammonia converter catalyst. About eight months before the overhaul, the capacity of the ammonia synthesis section decreased, because of a failure at the distributor grids of the ammonia converter. The gap between the distributor grids increased and led to the escape of catalysts into the outlet flow. The catalyst material is iron and chemical analysis of the scale showed a high percentage of iron oxide.

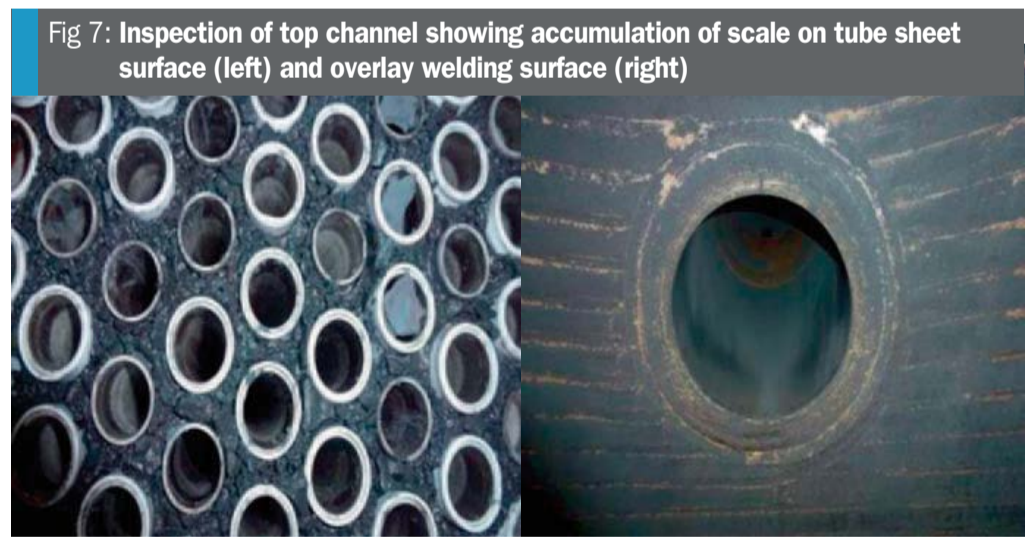


Table 3: Chemical analysis of scales on top tube sheet

Fe <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	NiO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MnO
79%	4%	5%	0.25%	0.3%	0.4%

After removing the scale, the tube to tube sheet welding connection, tube ends and knuckle radius between the tube sheet and liner were checked. No defects or corrosion were observed. The top cover of the exchanger was also inspected visually and no corrosion was found on the liner. The flange face was also acceptable.

#### Bottom channel

The overlay welding, tube ends and their cross sections, knuckle radius between the tube sheet and overlay welding lining, surface of bottom cover and flange face all looked smooth and no corrosion was observed.

#### Eddy current test of tubes

Eddy current testing (ECT) is the most common tube inspection technique. It is based on measuring changes in probe impedance as the probe passes over the defects. The defect depth is estimated by comparing the ECT signal with the signals obtained from machined calibration defects. A calibration tube with defined defects is made out of the same material being inspected.

Analysis of output data from ECT testing showed that all tubes have a corrosion rate of less than 0.07 mm/year and 99.9% of tubes are in very good conditions. The equipment probe did not pass through two tubes.

#### Inspection of liquid dividers

Uniform distribution of the process fluid over the tubes is essential for proper functioning of the stripper. Non-uniform distribution decreases the stripper efficiency and may result in corrosion. The air test is a reliable method of checking the liquid distribution system for proper functioning. The test measures the pressure drop over each liquid divider when a constant air or nitrogen flow passes through. The test is passed when the pressure drop across each liquid divider is within a certain range of the average pressure drop. Figure 8 shows a testing device for liquid dividers. An air test performed at 1 bar (gauge) pressure and normal pressure drop across the liquid divider measured 6 mm H<sub>2</sub>O. Only ten liquid dividers were rejected after the test because their hole diameters had been increased. After cleaning the scale from around the gas tubes, all PTFE bushes were replaced and damaged liquid dividers were substituted with new ones. A leak test was performed before installation of the top and bottom covers of the

Fig 8: Air test device (left) and leak test of tubes by water level (right)



Fig 9: Excellent condition of tubes in pool condenser



exchanger. There was typical discoloration between the lining plates made of BC.01 and BC.05. The different colour is normal. Liner wall thickness measurements were taken at random positions with an ultrasonic probe. Results showed that there was no corrosion on BC.05 linings, but the maximum corrosion rate of lining plates made of BC.01 was approximately 0.06 mm/year.

#### Eddy current test of tube

Eddy current tube testing (ECT) was executed via the low pressure channel of the pool condenser. Because the fluid entering the tube side is steam condensate, there was some scaling in the tubes and so the tubes were first cleaned by water jet and after drying them the eddy current test began (Fig. 10).

The eddy current test showed that the maximum corrosion rate of the tubes was less than 0.08 mm/year. In 4.7% of the tubes no signal was observed due to internal scale and in four tubes some discontinuities were observed. Also, due to the accumulation of hard scale, the probe did not pass through 19 tubes.

exchanger. The purpose of this test is to check for tightness between tube ends and liquid dividers. Water is supplied to the top channel of the stripper to a level of about 30 mm above the tube sheet. If no droplets appear at the bottom ends of the stripper tubes within half an hour of establishing the water level the test is passed. If droplets do appear a possible cause is that the PTFE bushing was damaged during assembly. Burrs or dirt at the tube ends and deviations in dimensions in the sealing parts must be eliminated. Fortunately, there were no leakages and the top and bottom cover were installed.

#### Inspection of pool condenser

The pool condenser is a multilayer vessel with a protective lining. The pool condenser was designed and fabricated by SBN in Austria. The material for the tube bundle assembly is BC.05. Apart from one section of the shell where the lining material is BC.05, the material of the other linings is BC.01 and the lining thickness is 8 mm.

Visual inspection showed that the tube bundle and gas distributor were in excel-

#### Inspection of scrubber

The scrubber was designed and fabricated by SBN in Austria with material of construction as shown in Table 4. The scrubber has three parts: a blanketing sphere, through which the reactor gases pass; the heat exchanger part equipped with a downcomer, through which condensation heat is removed by conditioned cooling water via the in circulation water cooler for the HP scrubber; and the scrubbing part with a

packed bed, in which the remaining gases are scrubbed with carbamate solution from the LP recirculation section. Due to some problems during the downtime, only nozzle N15 (Fig. 11) was opened to perform an inspection of the overlay welding. No corrosion was observed on the lining surface and the interior surfaces were in good condition.

### Conclusions

The Inspection results of the high pressure equipment at KPIC indicated that there was severe corrosion in the urea reactor compared to other vessels. Although the amount of oxygen feed was always in accordance to the Stamicarbon recommended figure, the corrosion rate of the internal components such as trays and downcomer had exceeded the acceptance limit. Overall, components with 316L-UG (BC.01) material that are in contact with carbamate solution in all directions, showed more corrosion. For example, the thickness of the trays (especially at the top), downcomer and guide plates of the radioactive nozzle decreased to approximately 50% of their initial value while all nuts and bolts, lugs and stud bolts which were made of the more resistant material (BC.05) remained intact. These observations confirm that using materials such as BC.05 or Safurex with higher corrosion resistance than BC.01 material is the best choice for important parts of the urea reactor. Corrosion of the end cap and top section of the radioactive nozzle and also safety problems, led to these sections being cut and replaced with new ones. Because of these results, it was decided that the sieve trays and their support, downcomer and radioactive nozzle be replaced with new ones at the next downtime using a material with better corrosion resistance (BC.05 or Safurex). The stripper, pool condenser and spherical section of the scrubber were in good condition and the corrosion rates of their components were close to zero where BC.05 material has been used and even in places with BC.01 material (e.g. pool condenser lining) corrosion rates were in an acceptable range.

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Table 4: Material specifications of scrubber

Stainless steel lining	Tubes	Nozzle pipes	Pall ring	Others
BC.05	BC.01	BC.05	BC.09	BC.05

Fig 10: Low pressure channel of pool condenser and tube condition

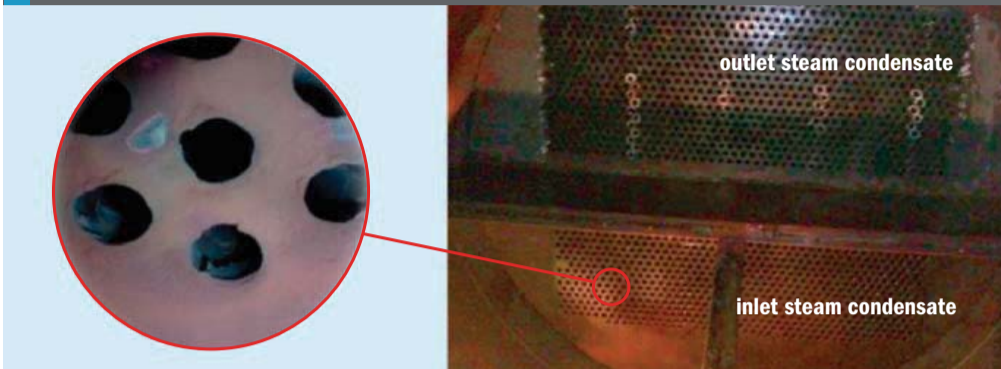
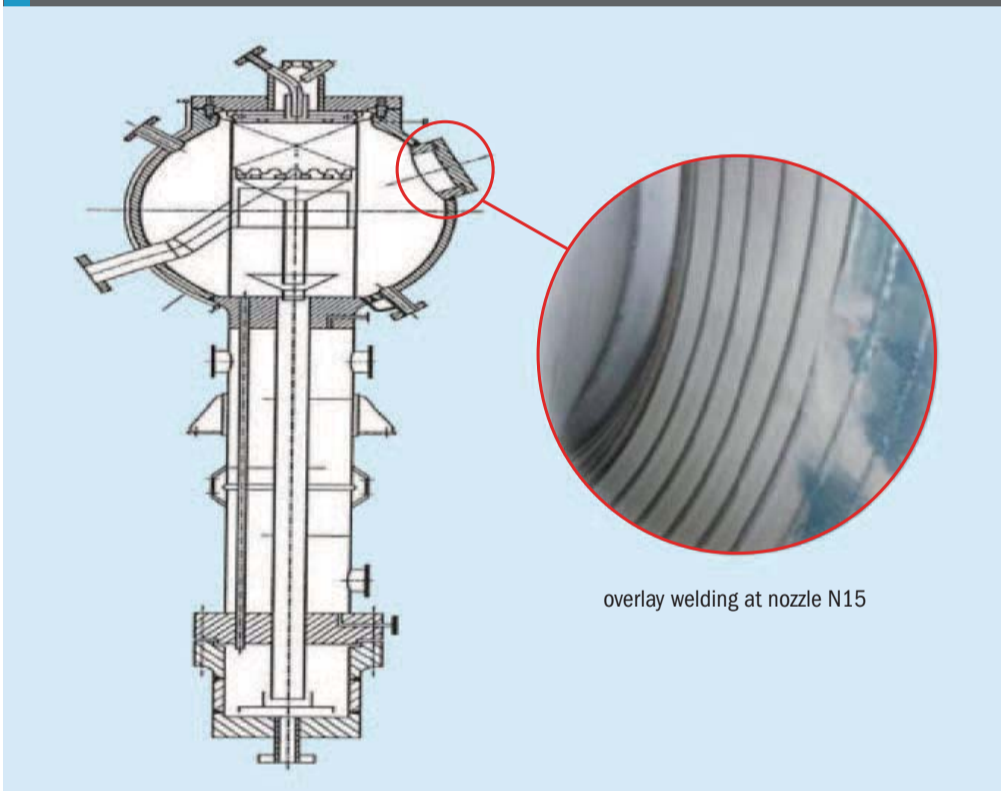


Fig 11: Schematic view of scrubber



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