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Number 340

March | April 2016

nitrogen+syngas

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The global ammonia market

Nitrogen project listing

Feedstock purification

Urea formaldehyde manufacture

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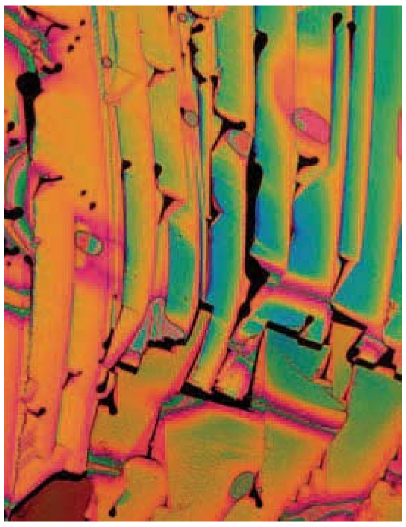

Cover: *Polarised light micrograph of a urea crystal.*
Sidney Moulds/Science Photo Library

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BCInsight

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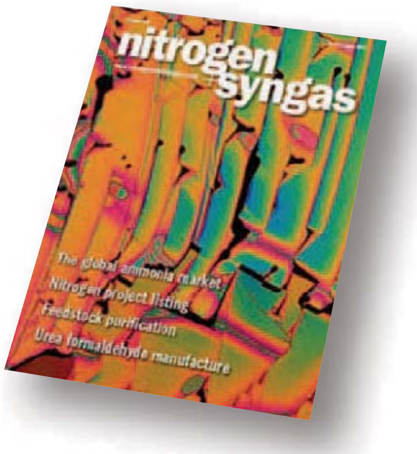
Milestone for US gas signals a period of oversupply



> **Sabine Pass itself was conceived back in 2001 as an LNG import terminal.**

In February Cheniere Energy's Sabine Pass LNG terminal at Port Arthur, on the Texas-Louisana state line, began loading its first spot cargo of liquefied natural gas (LNG) for export. The cargo was reportedly destined for Petrobras; Brazil needs gas to cover for low hydroelectric power production from reservoirs depleted by drought as it moves into summer in the southern hemisphere. But, more importantly, the cargo also marked a new milestone – the first export of US shale gas.

The transformation of the US from a gas importer into a gas exporter marks one of the biggest shifts in the international gas industry over the past couple of decades. It seems strange to recall now, but I can vividly remember how – as recently as a decade ago – the US was going to be the great promised land for LNG exporters, with steadily growing gas demand for power generation and falling production, and the country was forecast to be importing tens of millions of tonnes of LNG by 2020. Indeed, Sabine Pass itself was conceived back in 2001 as an LNG import terminal, and there were dozens of other similar proposals in the early years of the 21st century. But by the time Sabine Pass opened as an LNG import terminal in 2008, the ground was already beginning to shift as shale started impacting on the domestic US market, and within two years Cheniere had applied for a permit to convert its import terminal into an export terminal. Now it is the export projects which have proliferated, and within the next few years new terminals are due to come on-stream at Freeport, Cove Point, Cameron and Lake Charles, with – including Sabine Pass – a combined capacity of 55 million t/a by 2030, and another second wave still in the permitting process could nearly double that. The US is set to join Australia and Qatar as one of the largest LNG exporters in the world.

The advent of the export of US shale gas has come at a tough time for gas producers and marketers; a couple of years ago LNG could be sold in Japan for $17.00/MMBtu, and everyone wanted to be in on the market. Today the price is $8.25/MMBtu, and as well as new US capacity there are now new LNG trains coming on stream in Australia, Russia, Malaysia and Indonesia. Australia alone has 50 million t/a of new capacity slated over the next few years, and the global total could be as much as 100 million t/a of extra supply in five years' time. However, with domestic US gas prices down to $1.80/MMBtu at the Henry Hub, US gas producers are becoming desperate to reach foreign markets, and are willing to take what prices they can get.

The question is where all of this LNG will go. Japan has been something of a lifeline for the LNG industry, as its shutdown of almost all of the country's nuclear capacity post-Fukushima has led to a massive increase in its imports of LNG, and Europe seems set to become a big customer for US LNG as it tries to wean itself off Russian gas imports, but, as with many other commodities, a lot of new LNG capacity was predicated on increasing demand from China, which currently does not look set to materialise any time soon. The world seems set to face a prolonged period of LNG oversupply, with knock-on effects into gas markets all around the world. This in turn is sure to feed through into base prices for ammonia and urea, and margins are unlikely to be as bounteous as they have been in recent years. ■

Richard Hands, Editor

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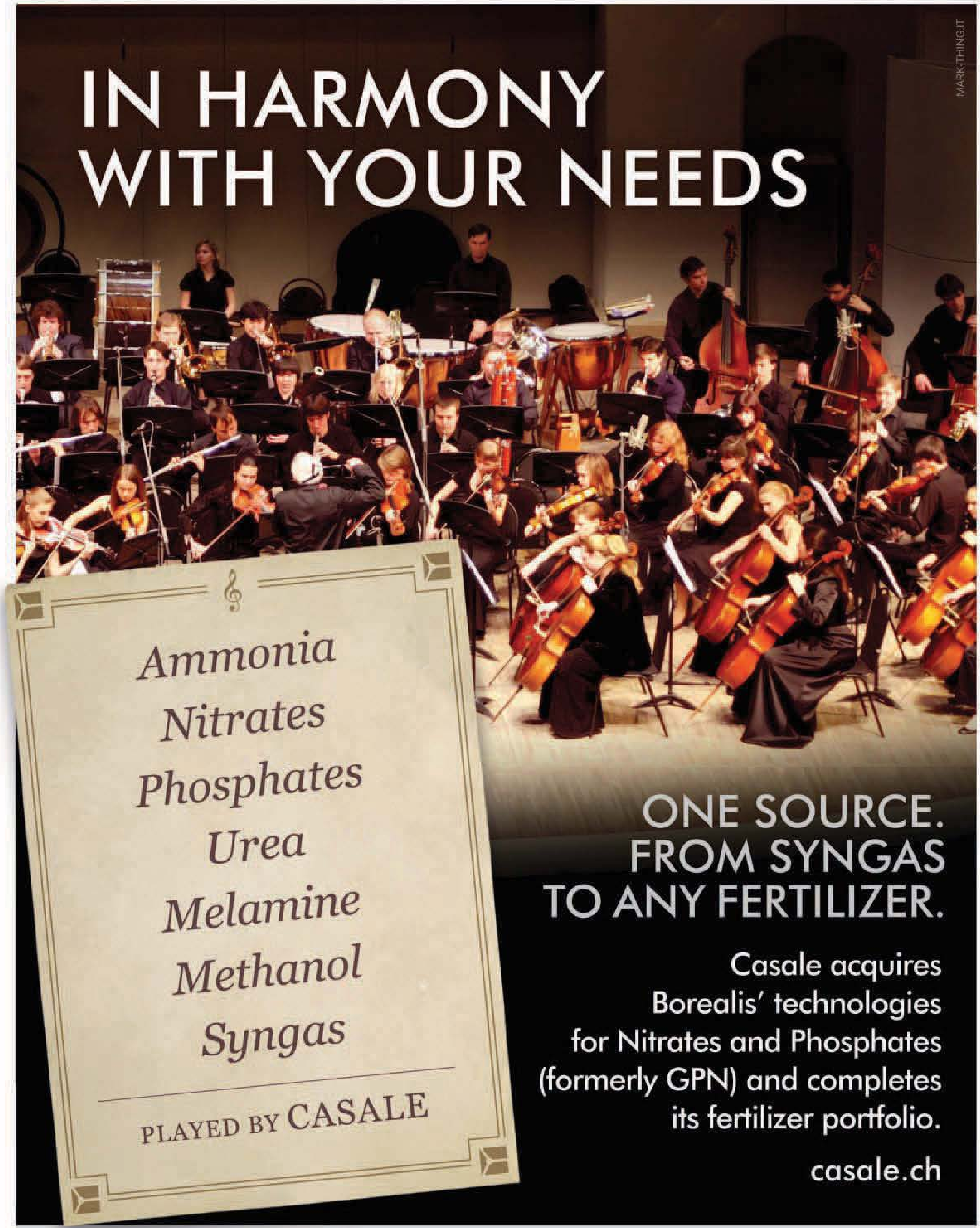

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Price trends

MARKET INSIGHT

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

NITROGEN

The nitrogen market was once again dominated by tumbling prices during the opening months of 2016, amid the effects of the chronic market oversupply that was so prevalent through 2015. Downward price movements were further compounded by sluggish demand across Europe – with the exception of Turkey – and Latin America, while major urea buyer India was not expected to come back to the market until at least April. The result was that in January 2016 the key nitrogen price benchmarks fell to five year lows, with the urea market in particular feeling the strain of weak market sentiment and buyers holding off in anticipation of further price cuts.

However, in the short-term there are demand prospects on the horizon from some key importing regions. In the ammonia market, the supply/demand balance tightened in late February and early March due to emerging seasonal demand in the US and Europe and the prospect of tight supply from the traditional export regions in March (namely Trinidad and the MENA region). In the US, demand has been firm amid spring plantings and buying is expected to continue in the coming month as application levels increase once better weather allows farmers to begin fieldwork.

The urea market has experienced a far looser supply/demand balance than ammonia but is also expected to see the onset of demand in the coming months which will support prices somewhat. Seasonal demand is expected to support market sentiment until May and June. Indian urea stocks were comfortable in February but there has been speculation that the next sizeable purchase tender will be issued in April in line with the country's new financial year. European urea demand is also expected to pick up towards the end of Q1 ahead of the spring application season, while the US is likely to see some of the strongest demand prospects in the coming months in light of its tight domestic supply.

These seasonal demand developments will no doubt limit the extent to which the current supply-driven environment puts downward pressure on prices, but what about the longer-term prospects for nitrogen fertilizer demand?

The global nitrogen market hosts a wide range of under and overindulgent practices, that is, countries that do not apply enough nitrogen fertilizer to produce optimum yields, and those that apply too much, leading to nutrient losses and environmental impacts. China is the most striking example of the issues that can arise when nitrogen is subsidised as a means of enabling food self-sufficiency. Chinese nitrogen fertilizer application has now grown to such an extent that the government recently announced a policy to limit growth to 1% per year between 2015 and 2020 in a bid to prevent further environmental impacts of nitrogen pollution.

Generally speaking, nitrogen is the most "recession-proof" of the fertilizer products; it forms the building blocks of plant growth and is an essential requirement to produce crop yield. Even in periods of low crop prices and economic uncertainty, nitrogen application remains a crucial investment for farmers to secure yields. This is in contrast with the other macronutrients such as phosphates and potash, where there is often decreased application in years when agricultural budgets may be tighter, as farmers choose to skip a year in favour of securing a margin on their crop production.

In the meantime, the nitrogen market remains overwhelmingly supply driven and in the short-term capacity additions show little signs of slowing. In the last month alone, several nitrogen capacity investment projects reached milestones in their construction process, such as the Mendeleevsk plant, which was commissioned in Russia in February, KBR's confirmation that work was restarting on the Kima plant in Egypt, and Petronas' SAMUR project in Malaysia reaching 90% completion, to name but a few. Furthermore there is the addition of significant new North American nitrogen capacity in 2016 and 2017, which will have consequences far beyond reducing the region's import dependency.

In the longer term, however, it is important to remember that the prevailing nitrogen market price weakness is part of a larger cyclical price downturn in energy and nitrogen commodity markets which naturally follow the cyclical upturn we saw in the mid-2000s. As we commented in the last issue of *Nitrogen+Syngas*, with the amount of spare capacity in the market today, and continuing downward pressure on nitrogen prices, overall profits decrease. What's also interesting though is that with weaker energy prices, the profitability gap between high and low cost producers is much smaller. This tends to provide a significant disincentive to building new capacity. So in the next few years we would expect to see interest in building greenfield plants diminishing. By the end of 2016 the picture is likely to reveal capacity growing more slowly than demand, implying that utilisation rates will creep up. ■

Table 1: **Price indications**

Cash equivalent	**mid-Jan**	**mid-Nov**	**mid-Sept**	**mid-July**
Ammonia ($/t)				
f.o.b. Caribbean	270	360	405	420
f.o.b. Arab Gulf	308-323	390-425	420-450	380-400
c.fr N.W. Europe	348	410-445	474	455-480
c.fr India	368	440-470	463	420-455
Urea ($/t)				
f.o.b. bulk Black Sea	220	258-263	255	262-273
f.o.b. bulk Arab Gulf*	201-211	255-263	266	268-278
f.o.b. bulk Caribbean (granular)	221	250-255	264	280
f.o.b. bagged China	222	252-255	263	282-287
DAP ($/t)				
f.o.b. bulk US Gulf	395	410	458	470
UAN (€/tonne)				
f.o.t. ex-tank Rouen, 30%N	175	183-186	186	195-197

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

Source: Fertilizer Week

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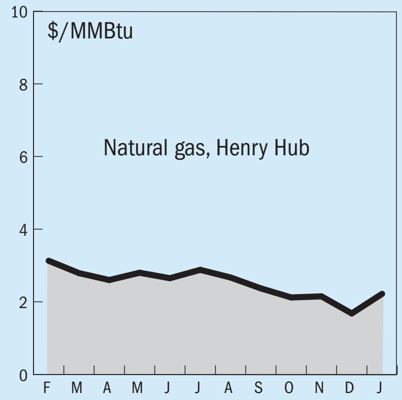
END OF MONTH SPOT PRICES

natural gas



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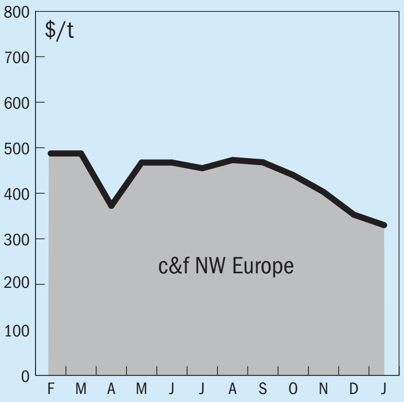
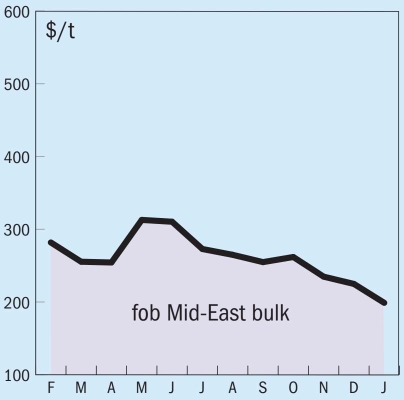
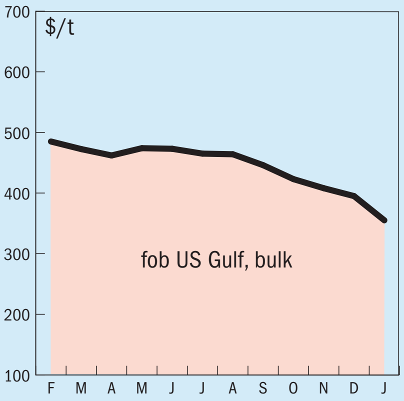


urea



diammonium phosphate



MARKET INSIGHT

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

METHANOL

In the US, the February contract natural gas reference price was settled at $1.90/MMBtu for Texas, and $2.30/MMBtu for Louisiana, with Texas down $0.31/MMBtu from January as spring weather decreases the heating load. Overall methanol demand has remained flat-to-slightly-up this week with demand expected to pick up through March. Warmer weather has spread through the southern states leading to an increase in construction activity. There have been some cancellations on the supply side; Valero say they will not move forward with a 1.6 million t/a unit at St. Charles, LA, while Northwest Innovations Works say that the company is suspending work at their Tacoma site in response to public concern and opposition. This news is not surprising given current price levels in the US Gulf which have been hovering around the $150/t level for 2016 with the only saving being that natural gas prices have been so low. These cancellations/delays are likely just the first of many.

North American operating rates remain around 85%. Venezuelan units continue to run at an estimated 65% of capacity and the last Chilean unit at 40% of nameplate capacity. The US spot market was trading at $0.435-0.45/gallon f.o.b. Houston in thin trading. No April deals were identified, though ideas are heard at similar levels to those for March. The official posted reference prices are $0.75/gallon for Methanex and $0.65/gal for Southern Chemical, both a rollover from February.

European spot methanol prices rose slightly. Demand was soft but stable. Zagros in Iran will restart its two lines soon due to improving market conditions, it is understood. In Saudi Arabia, facilities are running normally following a period of turnarounds earlier in the year. The EMethanex unit in Damietta, Egypt, has restarted following repairs to a gas pipeline but this facility is expected to shut down later in 2016 during the peak electricity demand period. Restarts at idled units in the European region will be challenging in the current price environment. Current price levels are not conducive to a restart at this point in time. IHS Chemical expects a decrease in the 2Q 2016 West European Contract Price, due to a decline in energy costs and challenging fundamentals for energy related applications of methanol in China, which significantly affected global methanol pricing over this quarter. However, the contract price will need to be at a level which will keep the European market competitive; a significant portion of Middle East exports have been redirected to higher-priced regions. Based on confirmed trades seen in the market, IHS Chemical post the European spot methanol price for March at €149-152.5/t f.o.b. T2 Rotterdam.

Demand in India is depressed, although the Indian rupee strengthened over the past week. Consumer confidence remains similarly soft for the time being. Methanol cargo prices basis India were assessed up slightly at $185-188/t c.fr T1.

The Chinese market is stable. The price spread of imported material between buyers and seller increased, therefore there were limited deals concluded. Major participants were still looking for price directions. Demand into key derivatives was weaker, though formaldehyde was stable with an average operating rate of 60-70%, with good market sentiment in construction, internal decoration and fibreboard. Domestic supply declined a little on the back of an intensive turnaround of producers in northwest China, leading to the average operating rate falling to 52% overall on nameplate capacity (based on 64 million t/a). Coastal inventory level is stable. Olefins prices are supported by restricted availability and this should keep MTO units operating on economic grounds but technical issues have brought forward a turnaround at Zhejiang Xingxing, which will reduce spot methanol buying from MTO producers.

In Korea, driven by price improvement in major petrochemical products, the market picked up to $220/t, with the market confident of new methanol business, but spot buying remained limited. In Southeast Asia, supply appeared stable with all regional units running well. The amount of spot discussion remains at a low level. Asian spot prices are posted in a narrow range of $205-220/t c.fr. ■

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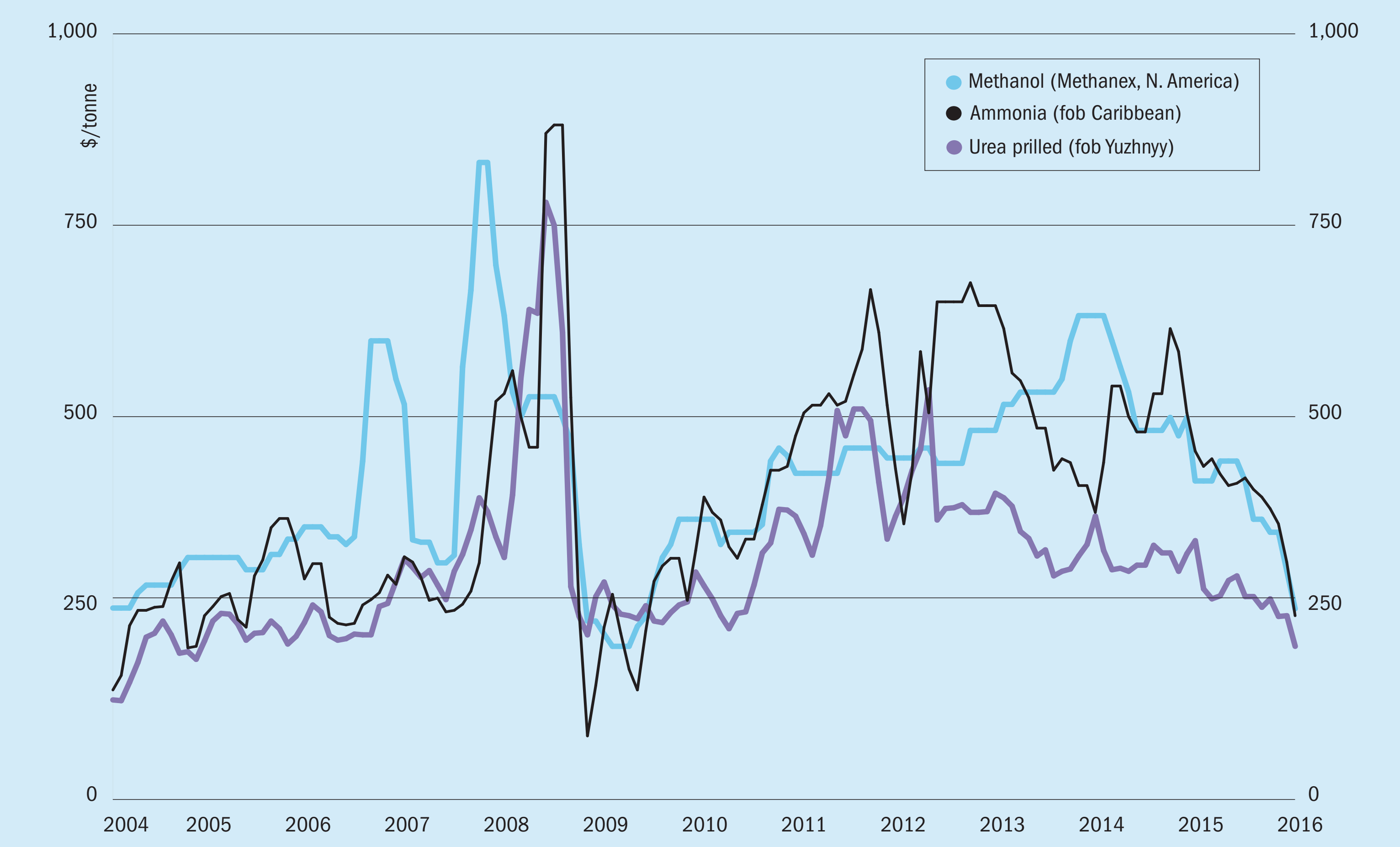
Market outlook

Historical price trends $/tonne



Source: BCInsight

AMMONIA

- The global ammonia supply/demand balance is expected to tighten amid the onset of seasonal buying going in March and April.
- Export-orientated ammonia producers such as Russia have posted improving margins in line with falling energy costs and exchanged rates, boosting cost competitiveness.
- Demand from the US and Europe and the prospect of tight supply from export regions is expected to lead to price increases in the next few months although these are likely to be short-lived as the market remains oversupplied.
- In the US, healthy ammonia demand is expected throughout the spring application season – market sentiment has improved as a result of recent crop forecasts which show there are large acreages dedicated to corn.
- In the short to medium term, new capacity additions are likely to keep a lid on prices.

UREA

- The global urea market saw an uptick, or at least stabilisation, of prices in some regions in February. This uptick came slightly earlier than expected, after prices crashed to a decade low in January, on the back of strong demand in the US and supply outages in North Africa.
- Strong demand from the Northern Hemisphere is expected to support urea prices globally between March and April, as farmers prepare for the spring application season.
- The impact of strong demand in the US on the international market is expected to last through at least March, and European spring demand is expected to kick in soon. Demand outside the US is expected to be quieter for the time being as buyers digest the price increases and wait to step into the market.
- India is not expected to return to the market until late March at the earliest, however offer levels are expected to move up.

METHANOL

- Supply in Northwest China is expected to continue gradually decline due to an intensive period of planned turnarounds in March and April.
- Seasonal construction activity appears to be picking up in China, boosting formaldehyde demand, but DME remains very weak.
- There is no re-start date as yet for the large MTO unit at Zhejiang Xingxing, reducing merchant methanol demand, but olefin markets are supporting other producers.
- Seasonal demand is also picking up in the US. Prices are subdued by low gas prices, which means that methanol availability still remains good in North America.
- European contract methanol prices for Q2 are expected to fall in line with falling feedstock gas costs, but not too far, in order to maintain Middle Eastern import interest when they can get higher sale prices in other regions. ■

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Nitrogen Industry News

CHINA

Topsoe establishes new R&D Centre

A newly established Topsoe R&D subsidiary – Haldor Topsoe Science & Technology (Dalian) Co. Ltd. – has entered a comprehensive collaboration agreement with Dalian Institute for Chemical Physics (DICP). This was celebrated at a ceremony Wednesday, January 27, 2016 at DICP. Under the framework of this collaboration agreement Topsoe will select and fund relevant projects at DICP, and DICP researchers will perform the research on contract for Topsoe. DICP is the leading Chinese catalysis research institute and the world's largest catalysis R&D center. The R&D center is Topsoe's first outside Denmark.

"The relationship between DICP and Topsoe can be traced back to the early 80s, when Topsoe was one of the pioneering foreign companies coming to China. Our relation has grown stronger in the past few years, and we are delighted to be able to lift this long-term relationship to a new level through this formalized partnership. Topsoe is an important player in the commercial application of catalysis and has also contributed very significantly in the R&D field. We look forward to joining forces to make new advances within catalysis," says Professor Can Li, Director of Dalian National Laboratory for Clean Energy at DICP.

"The new R&D Center in China will enable us to establish R&D alliances with Chinese business partners and research institutes. Investments in catalysis research in China are huge and the scientific level is high, so R&D partnerships will have significant importance to our future success in China, both scientifically and commercially," says Jesper Nerlov, Chief Technology Officer, Topsoe.

"A number of selected R&D projects have been kicked off today at the opening ceremony. These projects are within Topsoe's main business areas Chemicals, Refinery and New Business," says Fei Chen, Vice President for New Business R&D, Topsoe.

The R&D center is a significant expansion of Topsoe's engagement in China, where the company already has a regional office in Beijing as well as a newly built catalytic converter plant in Tianjin. ■

AN blamed for Tianjin explosion

The explosions at a warehouse in the port of Tianjin which killed 165 people in August were caused by a fire which spread to an unlicensed ammonium nitrate stockpile, according to the official report by the State Council. The investigative team determined that the fire began among nitrocellulose stocks which had dried out and lost their humidifying agents because of the hot summer weather, and then spread to the nearby illegal stores of up to 800 tonnes of ammonium nitrate, leading to two huge explosions. Twenty-five officials will face prosecution over the blasts on suspicion of dereliction of duty, abuse of power and bribery. The dead included 99 firefighters and 11 police officers responding to the emergency, who were unaware that AN was being stored. A further 798 people were injured.

Investigators have previously ruled that regulators failed to enforce safety rules, including keeping hazardous materials at the Ruihai International Logistics warehouses a proper distance away from residences. Several dozen people, including warehouse executives and local government officials, have already been arrested. Ruihai falsely claimed that it would store ordinary goods when it applied for planning approval, and colluded with corrupt officials to approve the construction of its facilities, according to Chinese authorities, and after it began operating the warehouses, it stored hazardous materials without a license, stockpiled prohibited materials, and stored chemicals in excessive amounts. About 10 government agencies could have detected the illegal storage of ammonium nitrate, but none did, the report concludes.

SWITZERLAND

ChemChina to buy Syngenta

The China National Chemical Equipment Corporation (ChemChina) has agreed to buy Swiss-based agrochemicals giant Syngenta in a deal worth $43 billion. This will be the largest acquisition of an overseas company by a Chinese one, and second only to the Dow-DuPont merger in terms of deals in the chemical sector. Syngenta's directors unanimously support the deal, and have recommended it to shareholders. The plans would see a new 10-strong board of directors, including four of the current board, overseen by ChemChina chairman Ren Jianxin. Syngenta will, however, keep its existing management structure and headquarters in Basel, Switzerland.

The acquisition of an agrochemicals business is likely motivated by China's desire to ensure food security, amid concerns that agricultural productivity is not keeping pace with population growth. Its purchase of Syngenta – a producer of genetically modified (GM) seeds – also hints that China is becoming more willing to embrace GM technology. The country has been reluctant to adopt GM because of public opposition, and while GMOs are used for animal feed, they are not yet grown as food crops.

AUSTRALIA

Orica expansion approved

The New South Wales Department of Planning has approved Orica's proposal to increase ammonia production at its Kooragang Island facility. The approved application will allow an increase in ammonia output at the site from 360,000 t/a to 385,000 t/a. The increase requires no extra works; after commissioning of the plant, Orica found it could run the plant continuously at a rate of 1,050 t/d of ammonia for 18 months between shutdowns. There will be no increase in production of nitric acid and ammonium nitrate, which will remain at current levels; surplus ammonia that is not used on site will be shipped to Orica's Gladstone facility in Queensland.

Investigation into ammonia leak

Eight contract workers were hospitalised by an ammonia leak at the Yara Pilbara plant in Western Australia in early February, although it is understood that none were seriously injured. Two inspectors have been sent by the Australian Department of Mines and Petroleum to conduct an investigation into the circumstances of the leak.

UNITED STATES

CSB report on West calls for tighter standards

In its report on the April 2013 explosion at the West Fertilizer facility in Texas which killed 15 people and damaged 150 build-

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ings, the US Chemical Safety Board (CSB) said that the Occupational Safety and Health Administration (OSHA) and Environmental Protection Agency (EPA) both need to tighten their standards on ammonium nitrate. The explosion resulted from a fire which caused 40-60 tonnes of fertilizer grade ammonium nitrate (FGAN) to detonate. Twelve emergency responders and three members of the public were killed.

"OSHA efforts to oversee facilities that store and handle FGAN fell short at the time of the incident," the CSB said. The report says that OSHA's Explosives and Blasting Agents standard "was not very well known among those in the fertilizer industry, likely due in part to the fact that application of the section was unclear and (it) had rarely been used previously to cite fertilizer facilities". It also criticised OSHA for leaving AN off its list of hazardous chemicals whose handling triggers the Process Safety Management standard.

The Agricultural Retailers Association (ARA) released a statement saying it was pleased the CSB recommended that OSHA either implement Process Safety Management on FGAN or update its storage and handling standards. "ARA has ardently advocated for the latter. Applying PSM to retail facilities is inappropriate, inefficient and unnecessarily burdensome." The ARA points out that PSM was designed for manufacturing facilities, while West was a retail/storage site.

As far as the EPA goes, the CSB said that West was covered under the EPA's Risk Management Program rule for its anhydrous ammonia tank (which performed as designed and remained intact throughout the incident), but not for its storage of FGAN, and argued that if EPA had included GFAN under the Risk Management Program rule, West Fertilizers would have been required to apply it for its storage of FGAN and perhaps could have reduced the risk of a catastrophic accident.

Foster Wheeler to manage ammonia plant construction

Amec Foster Wheeler said it has been awarded a contract to provide project development support and design engineering for an integrated 80,000 t/a anhydrous ammonia production plant and electric power cogeneration facility, through a new agreement with Grannus, LLC, a US-based clean technology and project development company. The plant is situated in Kern County, California.

According to Grannus, the medium-scale fertilizer plant is designed to be smaller and sited closer to the end user, and to require a smaller overall investment than today's world-scale fertilizer plants. Amec Foster Wheeler's scope of work includes engineering design and definition work utilising key process technology and equipment suppliers, as well as execution planning activities for the facility. Work has begun on the project and the plant is expected to be operational by Q4 2017. The cost of the contract was not disclosed.

"This award confirms our combined technical expertise in process engineering and integration, and our ability to work on this landmark project. We are delighted to continue building our relationship with Grannus to support them on this important project," said Simon Naylor, Grannus Group President Americas.

TKIS to build new fertilizer plant

Midwest Fertilizer Company LLC has contracted thyssenkrupp Industrial Solutions to design and build a its new nitrogen fertilizer plant in Posey County, Indiana at a cost of $2.0 billion, with financial close expected in mid-2016. The contract covers engineering, procurement, construction (EPC) and related services. Construction will start after the financial close, and the facility is expected to be fully operational in 2020. The complex, which is being developed by Pakistan's Fatima Group, will produce 730,000 t/a of ammonia, 730,000 t/a of urea, 1.4 million t/a of UAN solutions, as well as diesel exhaust fluid (DEF). Midwest Fertilizer has secured a $1.26 billion tax free loan for the plant, facilitated by Posey County, Indiana under the Heartland Disaster Tax Relief Act of 2008. The remaining funds will be comprised of equity, tax increment-financing bonds, and some term debt.

"thyssenkrupp is a world leader in fertilizer plant technology," said Mike Chorlton, president and CEO, Midwest Fertilizer Company. "This partnership allows us to move forward with the next phases of this world-class project in southwest Indiana."

EQUATORIAL GUINEA

EPC contract awarded for urea complex

Riaba Fertilizers Ltd has awarded the EPC contract for a new petrochemicals complex to a Chinese consortium led by East China Engineering Science and Technology Co. Ltd (ECEC). The complex, at Riaba on Bioko Island will use offshore gas reserves supplied by Noble Energy and partners Gunvor, Atlas Petroleum International and Glencore, to feed an ammonia-urea complex with a capacity of 1.5 million t/a. A pre-front end engineering design (FEED) has been completed by Worley Parsons. Ground breaking is expected at the end of March, with completion in 32 months. A ground-breaking ceremony expected by late March.

BRUNEI

India considering Brunei joint venture

India's vice president Hamid Ansari indicated that India is looking at the possibility of using Brunei's gas locally for setting up a joint venture fertiliser plant in the country along the lines of the Oman-India Fertilizer Company (Omifco). The comments came during a five-day visit to Brunei and Thailand.

Brunei is the fourth largest producer of LNG in the world, and among the largest gas exporters with a 9% share of the export market. More than 90% of Brunei's gas production goes to feed LNG production for exports, most of it going to South Korea.

UZBEKISTAN

Casale to modernise nitric acid plant

Navoiazot has contracted Swiss-based Casale to modernise the form's nitric acid plant. The €177 million contract, awarded on a turn-key basis, will be Casale's first nitric acid contract award since buying the former GPN technologies from Borealis last year. Production will increase from 350,000 t/a to 500,000 t/a. The project will be financed from Navoiazot's own resources, as well as a loan from the Fund for Reconstruction and Development of Uzbekistan, loans from foreign banks, and a loan from Casale.

MALAYSIA

Ammonia to form part of phosphate complex

A power supply agreement has been signed for the new Malaysian Phosphate Additives (Sarawak) phosphate complex in Samalaju Industrial Park, Bintulu. The complex, which is expected to be commissioned in 2018, will be Southeast Asia's largest integrated phosphate complex, comprising nine different which that will produce 500,000 t/a of food, fertiliser and halal feed phosphates, 900,000 t/a of coke and 100,000 t/a of ammonia.

MPAS is a joint venture between Malaysian Phosphate Additives and Cahya Mata Sarawak, each with a 40% stake. The remaining 20% is held by Arif Enigma Sdn Bhd, an associate of Tradewinds Plantations group.

GERMANY

Pumps for extreme operations

At the Nitrogen+Sygnas conference in Berlin, Friatec AG subsidiary Rheinhütte Pumpen will showcase its range of centrifugal pumps for conveying corrosive and critical media such as nitric acid. Nitric acid presents particular challenges in fertiliser production as regards the choice of pump materials; low-molybdenum stainless steels with high nickel content have proven to be effective, and Rheinhütte pumps meet these requirements by using casting material 1.4306S, both for vertical and horizontal designs. If a higher resistance to corrosion from nitric acid is required, for example in case of high temperature applications, silicon-alloyed special materials are available, while for heavily abrasive media, highly wear-resistant ferritic stainless steels can be used. At moderate temperatures, high-quality plastic pumps at the periphery of the system are a useful complement to metallic pumps for all types of acids and alkalis.

Pumps set up vertically offer a significant advantage concerning the design of the shaft seal, in particular for especially hot or aggressive media. Due to the positioning of the shaft passage above the tank, simple seals (including no-emission types) are possible. The vertical setup also has safety-related benefits, since there is no need for a tank passage beneath the surface of the medium. With the vertical pump of type RCEV, an immersion depth of up to 2,000 mm is possible. Owing to the specially designed hydraulics and the absence of submerged slide bearings, safe dry running can be achieved. The RCEV pump is used in case of erosive solids in the medium or if handling concentrated ammonium nitrate solutions and melts.

The submersible vertical pump of type GVSO (type VS4 according to ISO 13709) safely conveys heavy media such as highly-concentrated alkalis, ammonium nitrate solutions and melts. On account of its special vertical and partially multi-stage hydraulics, as well as its small fitting dimensions, the GSVO can especially be used when there is limited space.

For fluids that are difficult to pump, leakage-free sealing is required for horizontal conveyance. The RN (ISO 5199/22858) and RCE (ISO 5199) pump series can be configured according to the specific application. For applications involving highly concentrated, hot nitric acid, magnetic coupling pumps of type RMKN are an appropriate choice. With the special Hoko-HNO_3 design, all components (including the spacer can) are made of a high silicon-alloyed casting material.

UNITED KINGDOM

Treatment for wastewater streams

A 1,000 t/d urea plant generates approximately 500 m^3 of wastewater per day which contains large amounts of contaminants, particularly ammonia. Reducing wastewater volumes is a key factor in minimizing the environmental impact of the production of fertilisers. UK-based Suez Advanced Solutions has developed what it calls Electrochemical Technology (ECT) for treatment of waste water and odours using the application of an electric current between an anode and a cathode which generates reactions that destabilise pollutants and converts them into more biodegradable compounds. It is divided into three main reactors, which are all compact and modular, and which can be expanded according to production needs. The company says that ECT can remove up to 95% of contaminants common to fertiliser manufacturing processes, such as ammonia, nitrates, phosphoric acids and solvents. It has also been shown to eliminate a range of contaminants where other methods have failed.

Suez also offers an advanced bio-filtration technique to remove odours from the fertilizer manufacturing industry, composed of a non-biodegradable bio-medium inoculated with selected microorganisms of natural origin, which enable a more efficient treatment specific to each effluent. In addition to common odour issues like H_2S, ammonia and mercaptans, it can also remove other soluble odorous components such as thioether, aldehydes and terpenes with 95% efficiency.


PHOTO: HAARETZ

The Haifa ammonia tank.

EGYPT

KBR re-starts work at Kima

KBR says that it has been awarded a new contract for proprietary equipment and has restarted work under the previous contract awarded by Tecnimont for the license and basic engineering design of a 1,200 t/d ammonia plant which is being built for Egypt Chemical Industries Holding (Kima) at Aswan. The plant is being built on a fast-track basis and will support regional development plans in Aswan and Egypt's drive to build modern fertilizer complexes.

ISRAEL

Threat to ammonia tank

The ongoing controversy over an ammonia tank at the northern Israeli port of Haifa gained a little more momentum in January when Hezbollah leader Hassan Nasrallah, in a televised address from his bunker in Lebanon, threatened to hit the 14,000 tonne ammonia tank with a missile, claiming that the effect would be equivalent to a "nuclear bomb". The ammonia tank is used to store imports which are used by Israel Chemicals Ltd (ICL) for downstream fertilizer production, and there has been a long campaign to move it, which appeared to bear fruit in 2013 when the cabinet agreed in principle to build an ammonia plant in the southern Negev region and close the Haifa tank by 2016. ICL since then has been looking at various downstream options, including potassium nitrate production, and there is also a question of a gas feed to any new ammonia plant and the pace of development of Israel's offshore gas reserves. In the meantime the Haifa tank continues to be used, and the municipality of Haifa in an ironic press statement thanked Mr Nasrallah for raising the issue, hoping that it would prompt government action. ■

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UNITED STATES

NWIW "pauses" methanol plant application

China's Northwest Innovation Works (NWIW) has suspended its application for an environmental permit to build a \$3.4 billion, 20,000 t/d methanol complex at the port of Tacoma. NWIW said that it had been "surprised by the tone and substance" of local opposition to the project, and that in response it had elected to pause the State Environmental Policy Act (SEPA) environmental review process for the complex. NWIW said it will spend the next several months engaging in further dialogue with the local community to share more details about the proposed project, discuss their environmental and safety procedures and listen to people's concerns.

NWIW is also planning to build two other mega-methanol plants in the northwestern states of the US, for export to China to feed methanol to olefins production, each of 10,000 t/d, at Port St Helens, Oregon and Port Kalama, Washington State. ■

Carbon dioxide-to-methanol process

US researchers say that they have made a breakthrough in catalytic conversion of carbon dioxide to methanol. The researchers at the University of Southern California (USC) are led by Nobel laureate and methanol advocate professor George Olah and Dr G. K. Surya Prakash, who also advise Iceland's Carbon Recycling International, which converts CO_2 to 4,000 t/a of methanol using renewable hydrogen. The current breakthrough, according to USC, uses milder conditions than existing carbon dioxide-to-methanol processes, using a soluble catalyst that encourages hydrogen and carbon dioxide to react more readily. USC has also been looking at potential absorbents for capturing carbon dioxide directly from air, where it is present at only 400 ppm levels, and that inspired them to consider a hybrid approach using amine absorbers which are soluble in the same solvents as ruthenium catalysts. Currently, however, the process exists only at laboratory stage, and some experts in the field have expressed doubts that the process could be successfully scaled up to industrial levels.

Topsoe to license new methanol plant

Haldor Topsoe has been selected to deliver its licensed process technology, basic engineering, catalysts and services for as new greenfield methanol plant in the US. The new 1.3 million t/a facility will be sited in Clear Lake, Texas, and is being developed by Fairway Methanol LLC, a 50/50 joint venture between Celanese Corporation and Mitsui & Co. Ltd. The methanol plant will use Topsoe's two-step reforming technology, including an autothermal reformer and a proprietary waste heat boiler design. It will also feature Topsoe selective catalytic reduction and CATOX technologies to keep emissions of NOx and volatile organic compounds in compliance with US environmental regulations.

"We are very pleased that Fairway Methanol LLC has chosen Topsoe's technology for this important project and we are confident that they will see clear, long-term benefits from this industry-leading solution," says Per Bakkerud, group vice president of Topsoe. Anders N. Olsen, CEO of Haldor Topsoe, Inc., added: "We see this as a landmark reference to showcase our methanol technology in the North American market. We expect more companies will be inspired by Fairway Methanol LLC and realize the value of a strong technology partner with a significant local presence to bring their projects to fruition."

Ground broken on new methanol plant

G2X Energy held a ground breaking ceremony in January to celebrate the beginning of Phase 1 construction on their new world-scale methanol facility at Lake Charles, Louisiana. The Big Lake Fuels methanol plant will produce 1.4 million t/a of commercial grade methanol, and there are plans to add downstream methanol to gasoline capability in the future.

"We are extremely pleased to be moving forward in such a significant way on this project. All state and federal permits for construction have been obtained, staffing of key management is underway and our site preparation has begun," stated Tim Vail, President and CEO of G2X Energy. "G2X, and our partners Methanol Holdings Trinidad Limited and Southern Chemical Corporation, are very appreciative of the efforts of the local, state and federal elected officials who, through their efforts, have allowed us to each this point. We feel this facility will be a large part of the revitalization of the methanol industry in Southwestern Louisiana.".

Port of Lake Charles Executive Director Bill Rase added; "The Port of Lake Charles would like to congratulate G2X Energy on the ground breaking of their new, world-class, methanol facility. We have been a proud partner with G2X in reaching this milestone and are excited about the jobs and economic benefits this project will bring to the Port of Lake Charles and the community as a whole. Being the 11th busiest port in the nation, we are constantly striving to partner with professional, dedicated companies like G2X and truly believe they will add greatly to the synergy of industries we have built along the Calcasieu Ship Channel."

G2X will license Johnson Matthey technology for the new plant, which will be completed in 2019.

"We are delighted to work with our partners G2X Energy to deliver a natural-gas-based methanol facility that is world class," said Karen Oliver, Managing Director Johnson Matthey Process Technologies' Chemicals Business. "We will ensure our long experience of excellence in methanol process technologies, with our priority on safety, will support G2X Energy in their commitment to converting abundant natural gas to higher-value fuels. We have worked extensively with the Proman Group in the past, and the Big Lake Fuels Methanol Plant will continue to build on our long-standing relationship."

Landfill GTL plant agreement

UK-based Velocys says that it has agreed with Ventech Engineers to install process modules and piping for ENVIA's Oklahoma City landfill gas-to-liquid (GTL) plant. The contract marks the final major step in the construction of the plant, which will convert natural gas from landfill to syngas and then downstream clean diesel, kerosene, naphtha and waxes using the Fischer-Tropsch process. The construction of the modular process units by Ventech Engineers is reportedly

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complete. The manufacture of Velocys' full-scale reactors and the initial catalyst charge to be used at the plant were completed in 2015. Mechanical completion of the GTL plant, which will be adjacent to Waste Management's East Oak landfill, is expected in mid-2016.

Velocys has also made available to ENVIA Energy up to $12 million of further funding as part of a stakeholder capital contribution, following which Velocys expects that this commercial reference plant will be fully funded through construction, completion and operation. ENVIA Energy is a joint venture between Waste Management, Ventech Engineers, and Velocys, formed in March 2014 to produce renewable fuels and chemicals from biogas and natural gas, using GTL technology.

David Pummell, CEO of Velocys, said: 'ENVIA's Oklahoma City project is continuing to meet key milestones. The additional funding Velocys has released demonstrates both the Company's commitment to the project, which will provide an important commercial reference plant for the use of Velocys' technology, and the confidence we have in its success.'

Refineries turn to over the fence hydrogen

The amount of hydrogen sourced by refineries from industrial gas producers jumped by 135% between 2008 and 2014, according to the US Energy Information Administration. During that same period, in-house hydrogen production by refineries grew by less than 1%, it said. The report attributed the shift to growing demand for diesel and tighter sulphur regulations.

IRAN

Topsoe to license methanol technology

Haldor Topsoe says that it has secured a contract from Iran's Badr-e-Shargh Petrochemical Complex to provide licenses, engineering, proprietary equipment, materials and catalyst for a new methanol plant to be located in Chabahar, as part of a new industrial zone in the town. Topsoe says that it has also decided to open permanent offices in Tehran, in the expectation of the opening up of the country's refining and petrochemical industry following the lifting of UN sanctions. Topsoe managing director Jens Ole Madsen will lead the company's Tehran branch.

Topsoe EMEA region and chemical business unit executive vice-president Per Bakkerud said: "I have no doubt in my mind that Iran is on the doorstep to a period of great progress. The business culture, the high level of education and of course the abundant natural resources is more or less a guarantee for blooming business in the petrochemical and refining industry. And we are eager to resume the great working relationship with Iran and the companies here that we have enjoyed for many years. I'm glad that we can now look towards future successes."

CHINA

New ASU for methanol production

Praxair says that it has successfully started up a large air separation plant in northern China for the Yankuang Guohong Chemical Co., Ltd facility in Zoucheng City. Yankuang Guohong Chemical Co., Ltd is a subsidiary of Yankuang Group. Through a long-term contract, Praxair's 3,000 t/d plant will supply on-site oxygen and nitrogen to Yankuang and replace the existing customer-owned air separation units (ASUs). The gases will be used in the coal gasification process for the production of methanol and downstream chemicals.

"Praxair's world-class air separation plant is helping Yankuang achieve energy efficiency while providing enhanced safety and reliability performance," said Will Li, president of Praxair China. "The success of this project is due to the strong cooperation between our companies, and we look forward to continuing a long and successful commercial relationship."

PAKISTAN

Progress on UCG project

Pakistan's Minister for Planning and Development Ahsan Iqbal has said that development of the Thar underground coal gasification project remains "the top priority" of the government. He was speaking at a progress review of the project at Thar. Syngas purification plants, eight power generators and horizontal directional drilling machinery have all so far been installed, and initial power production of 5MW began in May last year. Eight more generators and other facilities will be installed in the near future. Project director Dr Samar Mubarak Mand indicated that 322 million scf/d of syngas could be produced from the extensive lignite deposits. The minister has asked for a commercial feasibility study to be prepared, and said that private or public investment could be made available for the project if it was found commercially viable. ■

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People

The Iowa Fertilizer Company (IFCo) has announces that **Larry Holley** will join its senior leadership team as president. He will manage IFCo's project development, as well as oversee the approximately 200 full-time employees and 2,000 construction workers who are building the $1.9 billion plant, according to the company.

"Larry Holley has more than 40 years in the heavy industrial sector, serving in executive level positions across several different complex industries – most notably nitrogen fertilizer production and project management – and we are excited he will bring that wealth of experience to Iowa Fertilizer Company," said Michael Bennett, independent non-executive director and chairman of OCI NV, the parent company of Iowa Fertilizer Company. "As the president of Iowa Fertilizer Company, Larry will lead our team in delivering a timely, reliable, high-quality, and domestic source of fertilizer to farmers throughout the United States."

From 1974 to 2005, Holley worked for Mississippi Chemical Company, rising to the position of chief operating officer, where he directed all elements of the company's manufacturing and business operations. He was responsible for a variety of technical and management functions, including development support and the early operation of a world-scale KBR ammonia project in Trinidad, as well as major turnarounds across its nitrogen fertilizer portfolio. Since 2005, Holley has filled a number of strategic and management roles including president and CEO of the CBM Group, LLC, and president and general manager of Noranda Alumina, LLC and Noranda Bauxite. He replaces **Shawn Rana**, who recently left the company for family reasons.

"IFCo made significant progress during Shawn's tenure and the company wishes him and his family the best," Bennett said. "There is tremendous customer demand for a reliable, stable, and domestic source of nitrogen fertilizer and we are excited to be able to deliver that product for farmers here in Iowa and around the country.".

Clariant has announced the appointment of **Britta Fuenfstueck** as a new member of its executive committee, effective from April 1st, 2016.

"We are very pleased that Britta Fuenfstueck will join Clariant. With her extensive international experience as a senior executive in the technology industry, she will be a strong addition to our management team and will bring valuable insights and perspectives," said Hariolf Kottmann, CEO. "I look forward to Britta Fuenfstueck's contributions toward the long-term development of Clariant and her support for our top priority: to generate value for our customers, our employees, our company, and our shareholders."

Fuenfstueck was previously CEO of the Clinical Products Division at Siemens Healthcare, a position she has held since 2013. During her career with Siemens, she also held various leadership positions, including CEO of the Molecular Imaging Business Unit, Vice President, Strategy and M&A for the Healthcare Sector, as well as Director, Product Portfolio and Planning, Marketing and Sales, for Siemens' Magnetic Resonance Business Unit. Shestudied engineering physics at the Johannes Kepler University in Linz, Austria.

PClariant has also added **Eveline Saupper**, **Claudia Suessmuth Dyckerhoff** and **Peter Steiner** to its Board of Directors, subject to the approval of the Annual General Meeting of Clariant Ltd taking place on April 21st. At the same time, **Peter Isler**, a board member since 2004, has reached the statutory maximum age for board members of 70 and will therefore resign.

Rudolf Wehrli, Chairman of the Board said: "I'd like to sincerely thank Peter Isler for his valuable contribution during all the years, especially for his achievements as Chairman and longstanding member of the Audit Committee, for his contribution as a member of the Chairman's Committee as well as lately the Compensation Committee." ■

Calendar 2016

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

23-24
International Fertilizer Society Meeting,
BUDAPEST, Hungary
Contact: International Fertiliser Society,
PO Box 12220, Colchester,
CO1 9PR, United Kingdom
Tel: +44 1206 851819
Email: secretary@fertiliser-society.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

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

OCTOBER

25-27
IFA Production and International Trade and
IFA Crossroads Conferences, SINGAPORE
Contact: IFA Conference Service,
28 rue Marbeuf, 75008 Paris, France
Tel: +33 1 53 93 05 00
Email: ifa@fertilizer.org

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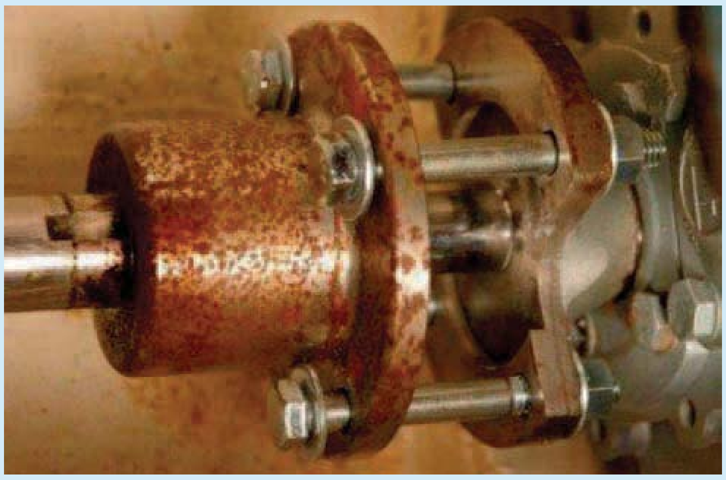
Plant Manager+

Problem No. 35 Urea plant preservation

Preservation covers the method of protection and preventative maintenance carried out on equipment and systems before and after they are put into use. In power plants, fertilizer units and refineries, expensive equipment like turbines, chillers, power generators, boilers, condensers, panels etc. begin to rust and corrode when in storage or during shutdown or even when in operation due to the presence of excess moisture in the air. Proper downtime procedures are extremely important. In this round table discussion, preservation and its importance is discussed by urea plant experts.



Above left: Failure due to carbon build up.
Above right: Corrosion on the HP flange in the urea plant.

Mr Nauman Talat of Pakarab Arab Fertilizer in Pakistan starts the round table discussion: We are planning to preserve our urea plant for a period of six months to one year using air. Please share your experiences in this regard.

Mr Easa Norozipour of Khorasan Petrochemical Company in Iran replies: As you know, special materials of construction are used for the equipment in urea plants. My recommendations for long term preservation of the plant are, if possible:

- wash the equipment well with demi-water;
- purge and dry with nitrogen;
- use nitrogen blanketing with positive pressure.

Mr Mark Brouwer of UreaKnowHow.com from The Netherlands offers his advice: In addition, take care that no salts (chlorides, nitrates, carbonates) can enter the tube side, shell side and the area between the loose liner and the pressure bearing carbon steel, so also plug the leak detection holes. Control a positive pressure with a manometer.

Mr Assadollah Rastegarpour of NEBCO from Iran shares his experiences: There is always a risk of accumulation of carbamate and urea in dead points, low points, around ejectors nozzles, and especially in the HP stripper. Ensure that these are flushed with boiler feed water and take samples from the drains to check for traces of urea and carbamate. If you decide to pressurise the system, blank it with a blind flange. I recommend blanking all control valves in the outlet of the reactor and HP stripper. In order to reduce the risk of control valves seizing up, take important and special valves to the shop for checking and to store them in good condition.

Mr Shiraz Chandio of Engro Fertilizer from Pakistan joins the discussion: We are also facing a similar situation at our plant due to gas shortages nowadays. After complete and thorough flushing of the vessels and piping, we preserve all the vessels and piping with a nitrogen blanket with positive pressure and continuous flow (where pressure blanket is not possible). We have also preserved the cooling water system after a shock dosing of chemicals as recommended by our process guys. Steam headers are also preserved with nitrogen.

Mr Majid Mohammadian of PIDEMCO from Iran shares his experiences: Take care to preserve the rotary equipment too. My suggestion is that if you have power and cooling water (small requirements), keep lube oil systems for the CO_2 compressor, ammonia and carbamate pumps in operation or on a regular basis and, for other rotary equipment, turn their shaft on a regular basis.

Easa adds: If you are planning to preserve the plant over the winter take care that equipment doesn't freeze, especially rotary devices. Any residual water inside devices can freeze and cause a rupture. We experienced this in our plant during the winter season (-20°C), even though we purged the equipment with nitrogen some items were forgotten.

Mark offers some more recommendations: Below are some recommendations when operating a urea plant at low loads, different loads, with many shutdowns and with a long shutdown:

Control/limit temperature changes during plant start-up/shutdowns and for frequent load variations. During these changes big stresses are put on the liners.

Operating at low plant loads can be critical for the HP stripper, HPCC and HP scrubber in cases where gas and liquid at the inlet of these HX are not well distributed. This is critical to avoid higher corrosion rates.

In the HP stripper each tube needs a minimum amount of liquid. When the liquid dividers are older, the liquid holes become bigger and the liquid level at low loads can become too low to assure that each tube receives the minimum amount of liquid. In the HPCC and HP scrubber a gas cushion should be present below the gas distributor at the inlet.

Changes in plant operating and monitoring procedures for prolonged low load operation: Take daily nickel analysis in the urea melt and also establish a baseline nickel level during normal operation, so you know when the nickel content become too high. Carrying out more frequent inspections would also seem to be a good idea.

Preservation of equipment's during prolonged shutdowns: In general pay attention to chlorides and nitrates. Ensure that they cannot enter the equipment and/or the leak detection system.

An increased risk of corrosion due to frequent and prolonged shutdowns is especially valid for equipment in a marine atmosphere.

As a result of "breathing" due to cyclic temperature changes infiltration of chloride containing vapour is likely to occur. These chlorides may cause stress corrosion cracking in austenitic stainless steels and pitting/crater type attack in carbon steel equipment. To avoid the ingress of chloride containing vapour, the equipment should be made inert with nitrogen (0.3-0.5 bar gauge). Avoiding the ingress of chloride containing moisture is of utmost importance for the shell side of HP heat exchangers like the HPCC, stripper, HP scrubber with austenitic stainless steel tubes in carbon steel tube sheets. Chloride containing moisture in the gaps (crevices) of the tube to tube sheets cannot be removed by flushing with chloride free condensate or demi water.

Special attention is required for the increased risk of CUI (corrosion under insulation) due to frequent and prolonged shutdowns. Cyclic temperature changes are most detrimental with respect to CUI and need specific preventive measures like painting and maintenance at the (aluminum) insulation cover sheeting to avoid ingress of moisture. It has to be emphasised that the increased risk of atmospheric corrosion should not be ignored.

Mr Nasir Hussain of Pak Arab Fertilizers Multan from Pakistan describes the planning for their urea plant: We are planning to preserve stainless steel equipment and piping with instrument air and use nitrogen for carbon steel material.

Mr Ali Salman Bokhari of Pak-American Fertilizers in Pakistan asks a question: Please explain the significance of providing a gas cushion in the HPCC and scrubber below the gas distributors at the inlet. What measures should be taken for preservation of control valves and other instrument items?

Mark replies: This is only valid for low plant load conditions to assure a good gas/liquid mixing before condensation of the gas. Otherwise condensation corrosion can occur with 316L UG material. ■

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.

Changing patterns of ammonia trade

Increasing domestic US nitrogen production and faltering gas supplies in some key producing regions are shifting the balance of ammonia trade flows, while new ammonium phosphate capacity in Morocco is also set to see increased need for ammonia imports.

Above: Ammonia storage tanks at the port of Tampa.

The global ammonia industry totaled around 230 million t/a of capacity in 2015. Total production from this reached 174 million t/a of ammonia that year, or around 76% of nameplate capacity, although old and unproductive capacity in China distorts this figure slightly. The amount of ammonia that actually crosses borders however is a much smaller proportion of this – 17.7 million t/a in 2015, or about 10% of the total. Risks and costs involved in transporting ammonia, which must be carried as a refrigerated liquid and escape of which may prove fatal, makes for a preference for captive use for ammonia, especially in the production of urea.

Although the number is small as a proportion of the total nitrogen market, there are nevertheless a considerable number of downstream producers who rely upon imported ammonia for their production, and this gives producers in advantaged locations, where gas costs are low, opportunity to sell ammonia onto the merchant market. Figures 1 and 2 show the major exporters and importers in 2014. Major net consumers are led by the United States and India, with Japan and various European countries also large buyers. Morocco also appears high up in the list, and the US, India and Morocco are primarily importing ammonia for ammonium phosphate production. In Europe it is mainly nitric acid and ammonium nitrate manufacture, and Europe, Taiwan and Japan use ammonia for downstream industrial or 'technical' uses.

The exporters, conversely, are led by Trinidad, which has nearly 20% of the export market, mainly supplying the US, and the FSU and Middle East are also large suppliers, with Algeria a supplier to

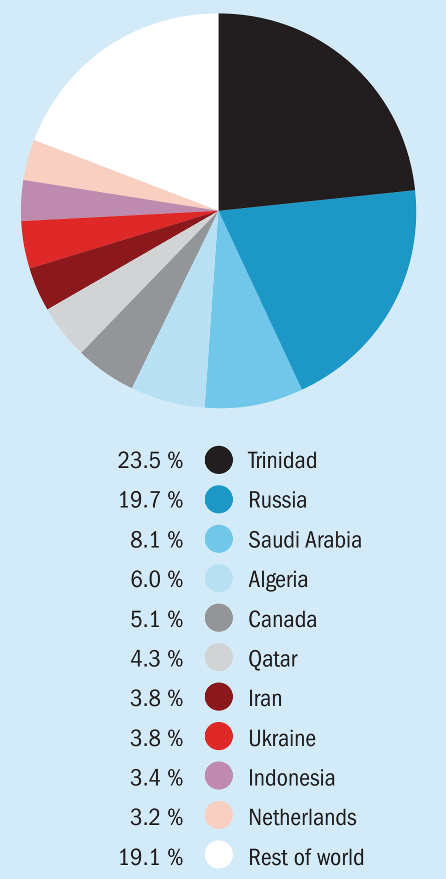
Europe and Morocco and Canada mainly to the US. However, these bald figures mask some significant changes which are occurring in the world ammonia market, due to a variety of factors, and patterns of trade are set to change over the coming years.

The expense of transporting ammonia as compared to urea means that while the urea market is relatively global, ammonia markets tend to be more regional, and in particular cluster around two 'hemispherical' markets – west of Suez and east of Suez. West of Suez the Atlantic ammonia trade sees product from the Black Sea and North Africa travelling to Europe and the US, and in the east from the Arabian Gulf to India and southeast Asia.

North America

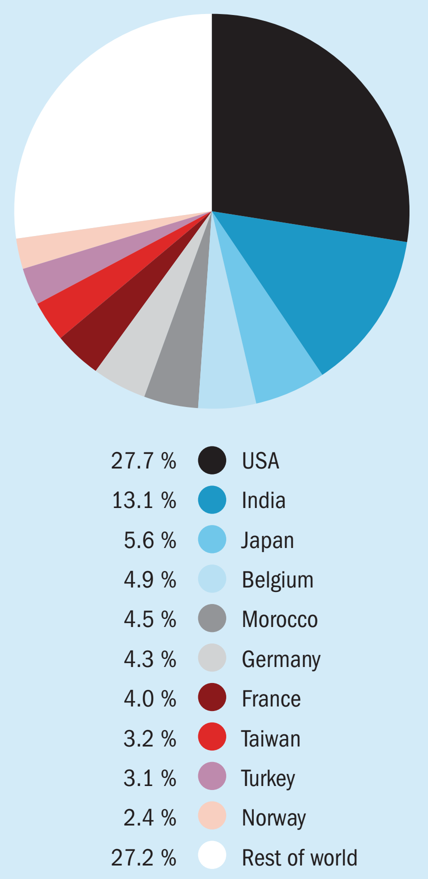
While it has been extensively discussed, the changing North American gas market is causing the largest disruption to ammonia markets at present. Trinidad's ammonia industry had developed predicated on supplying the US market as high gas prices in the US forced the shutdown of domestic ammonia capacity. Now Trinidad faces production curtailments due to gas shortages, and gas prices on the island have not been high enough to encourage the development of new gas reserves at the same time that US gas production has blossomed – now to the point where the US is beginning to export natural gas as LNG. Gas prices in the US are now equivalent to or lower than those in Trinidad, and producers are sited much closer to end use markets. The result has been a wave of new capacity building and plant re-starts. Some fertilizer producers who had idled ammonia plants and taken to buying on the open market have re-started old plants or built new ones. PCS restarted a 500,000 t/a plant at Geismar, Louisiana, and IncitecPivot will open a new 800,000 t/a plant at Waggaman, Louisiana this year to feed explosives production. Simplot and Mosaic also have new capacity, and new merchant ammonia capacity opened in the US at Beaumont. Yara is developing a 750,000 t/a ammonia plant at Freeport, Texas in conjunction with BASF to run downstream fertilizer and industrial production, and there are numerous debottlenecking projects which are also increasing capacity incrementally.

Fig 1: **Top 10 ammonia exporting countries, 2014**



Source: IFA

Fig 2: **Top 10 ammonia importing countries 2014**



Source: IFA

Canada is seeing a little of the same effect, as shale affects gas production there, as is Mexico, where Pemex is looking to re-start two of its idled ammonia plants, and overall, between 2012 and 2018, North American ammonia capacity will have risen from 16 million t/a to 25 million t/a, some of it integrated into downstream urea and UAN production, but some of it substituting for imports.

Trinidad, which effectively forms part of the North American market, is going to face much tougher going in selling into the US over the coming years, and will likely have to look towards Europe and Latin America as an outlet for its production. Gas curtailments have also reduced output by about 10% in 2015.

CIS

Countries of the CIS are also seeing changing patterns of production. Russia and some of the countries of central Asia have access to gas at relatively cheap costs and this has become one of the major regions of the world for new nitrogen capacity development, including some standalone ammonia capacity. Acron has its new 500,000 t/a Ammonia-4 plant commissioning at Veliky Novgorod, EuroChem is building a 960,000 t/a ammonia plant at Kingisepp on the Baltic coast and its new Nevinnomyssk plant in the Caucasus will have excess ammonia capacity over and above that required for urea production. There is also excess ammonia coming from JSC Ammoni in Tatarstan and Minudobreniya. Uzbekistan is also building a plant at Navoi with 1,000 t/d of excess ammonia capacity, and there has been talk about merchant ammonia capacity in Russia's far east as well.

Russia has long been one of the largest exporters of ammonia, from both the Black Sea and the Baltic. In the Black Sea it was traditionally rivaled by Ukraine, but a major change on the export side has been the decline of the Ukraine as a source of ammonia. Ukraine exported 1.5 million t/a in 2012 and 2013, but in 2014 and 2015 this dropped to 700,000 t/a. Disruption caused by the conflict in the east of the country and continuing wrangles over natural gas supplies continue to crimp output.

East Asia

China has not historically been a major importer or exporter of ammonia, preferring to trade urea instead. China's ammonia

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Ammonium phosphates

While much ammonia capacity is associated with downstream urea production, the same is not necessarily the case with ammonium phosphates. Phosphate production clusters preferentially by centres of phosphate mining, which do not always correlate with places with sufficient natural gas (or in the case of China, coal) supply to generate ammonia domestically. For this reason, several large ammonia importers do so in order to feed ammonium phosphate production. Thus although ammonium phosphates represent only 6% of all ammonia demand, they represent a much greater proportion of traded ammonia. Foremost among the major importers for MAP/DAP are the US and India, which between them accounted for 40% of ammonia imports in 2014.

Here, though, there is a change in the patterns of production. Indian ammonium phosphate production peaked in 2004 at 5.2 million t/a, and since then has declined to around 3.5 million t/a, out of installed capacity of 7.3 million t/a. As noted above, Indian subsidy policy has tended to make DAP more expensive than urea, discouraging its use at a time when prices for imported ammonia have been high. Likewise there has been something of a shift back towards single superphosphate (SSP) production and consumption rather than DAP, and this has tended to reduce import demand for ammonia.

In the US, the fall has been even more dramatic. Over the course of a decade, US DAP production fell from 10.4 million t/a in 2014 to 4.5 million t/a in 2014. Producers such as Mississippi Phosphates and PotashCorp have shuttered production due to high costs, diminishing ore grades and the difficulty in getting permits for new mines, and this too has cut into imports of ammonia.

New DAP production has come overwhelmingly in two regions – firstly China, where the ammonia is produced from coal-based feedstock, and which as we noted above does not play a major part in global ammonia trade, and secondly in the Middle East and North Africa. Jordan has expanded its phosphate production, but the largest slices of new capacity have been in Saudi Arabia, which is seeking to broaden its economy away from oil and petrochemicals, and Morocco, the largest holder of phosphate reserves, which is seeking to capture more of the value chain via its relatively low production cost phosphate, and move from phosphate mining and supply of concentrates towards downstream complex phosphate production. Morocco's state phosphate monopoly OCP is now in the middle of a major expansion of its domestic MAP/DAP capacity, adding four new MAP/DAP complexes between 2015-17 and another six to 2022. This will add 10 million t/a in terms of MAP?DAP production, and will require 1.9 million t/a of additional ammonia, none of which can be produced domestically.

Saudi Arabia is using natural gas gathered from oil production to generate its own ammonia capacity for its new phosphate complexes, Ma'aden at Ras al Khair on the Red Sea coast north of Jubail, and Waad al Shamal in the northwest of the country near the Jordanian border. Indeed, Saudi Arabia's ammonia output has fallen because of the completion of the Safco V urea plant, which has absorbed 600,000 t/a of ammonia that was previously exported. ■

exports were nil in 2014, and its imports around 200,000 tonnes. Chinese self-sufficiency in urea has led to the country becoming a major net exporter, but it has never really concerned itself with ammonia export. Ammonia imports into East Asia are mainly to Taiwan and Japan, where they are used in industrial processes. Indonesia is a net exporter, and is currently in a capacity replacement programme for older, less efficient plants, some of them built in parts of the country where gas is no longer available.

India

India imported 2.2 million t/a of ammonia in 2015, slightly down on 2014 as phosphate production was cut back slightly. India's ammonia imports are very much driven by production of ammonium phosphates, and phosphates have suffered slightly in recent years form the nutrient based subsidy scheme which has not included urea, allowing an over-concentration on application of urea at the expense of other fertilizers (DAP demand fell by 3 million t/a over the past five years). India has ambitious plans to develop new ammonia/urea capacity to try and reduce its dependence on imports, but aside from some plants based on coal and coalbed methane, it is hard to see where the feedstock to run the plants will come from.

Technical nitrogen

As well as fertilizer manufacture, ammonia is used in a number of downstream processes, including the manufacture of hexamethylene diamine (HDMA) as an intermediate in nylon production, acrylonitrile for fibres and plastics, caprolactam again for nylon, isocyanates for polyurethanes and hydrazine, as well as in metal treatment operations, industrial refrigeration etc. Technical demand for ammonia is concentrated in industrialised regions, mainly East Asia, Western Europe and North America, and high gas prices and lack of gas availability in Europe, Japan, South Korea and Taiwan have encouraged ammonia demand in those regions. New industrial demand has come particularly in China, where caprolactam for fibre manufacture has boosted demand, but again Chinese domestic ammonia production has been able to supply this.

Changing trade patterns

Ammonia capacity is likely to grow to 270 million t/a by the end of the decade, but demand is not scheduled to keep pace. Most of the growth is in capacity associated with downstream production, but there is a growth in merchant ammonia production forecast to around 20 million t/a. There have been some closures, such as Ruhr Oel's ammonia plant in Germany and Ube Sakai in Japan, and it remains to be seen how many of Ukraine's plants ever re-open, but in spite of new phosphate-based demand in Morocco, ammonia markets look set to be oversupplied. Gas restrictions in Egypt and Trinidad have ameliorated this to an extent, but growing domestic US capacity is set to displace Trinidadian tonnes into the west of Suez market, at the same time that new capacity comes on-stream in Russia and demand is reduced as Mexican ammonia production restarts. ■

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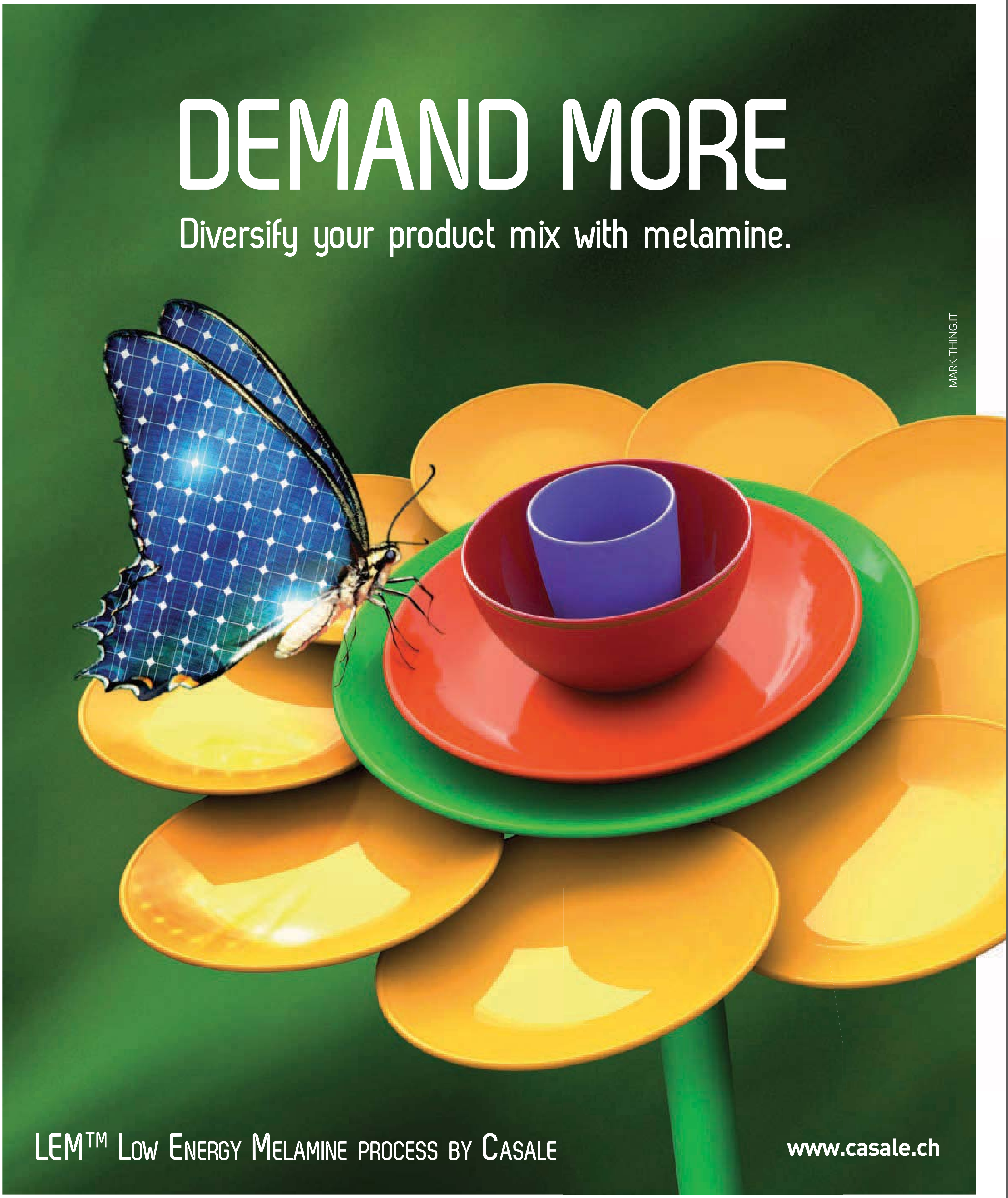

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Nitrogen+Syngas 2016

This year's Nitrogen+Syngas conference returned to Berlin's Estrel Centre from February 29th to March 3rd.

PHOTO: LUCERTOLONE/SHUTTERSTOCK.COM

Nitrogen+Syngas conferences seem to get bigger and bigger. On this, its second visit to Berlin, the conference managed to attract a record 700 delegates and the associated exhibition comprised 70 stands. While the format was largely the usual one – beginning with a workshop (organised by UreaKnowHow.com) and a morning of market papers before shifting into parallel technical sessions, this year saw several round table discussion sessions which gave a more interactive feel to the conference.

Market papers

Patrick Heather of the Oxford Institute for Energy Studies began with a run-down on changing gas markets Europe was facing greater reliance on imports, he said, and while Russia would remain a significant supplier – long term take or pay contracts represent over 110 bcm/year od demand out to 2030 - LNG would take an increasing share of those imports. Shale remains a US phenomenon, but LNG is set to be the big story going forward. Worldwide, LNG's share of gas shipped across borders will increase to 45% out to 2040, and gas traded on a short term rather than contract basis is set to increase from 12% to37% of shipped gas. Via LNG gas markets are globalizing, though there is no global price standard as yet. Still, as hubs progress there is a continuing move from oil indexation to market pricing – continental Europe is now roughly 50-50. A similar move in Asian markets still awaits a hub development, with Singapore possibly in the frame as an LNG trading hub.

Alistair Wallace of CRU reviewed where markets had gone in the past couple of years. High nitrogen prices and China's drive for self-sufficiency had led to overcapacity, especially in Chinese urea and falling nitrogen prices over the past 18 months, from around $315/t to $200/t. New capacity is still coming onstream; 11 million t/a in 2015, 13 million t/a in 2016 and 9 million t/a in 2017, although some of these may slip. China alone will add 20 million t/a of urea capacity from 2012-2020, and another 33 million t/a will come from the US, CIS and Middle East. Urea prices have consequently declined to the cost of the marginal producer – now China. In theory current prices are lower than Chinese marginal costs, although there has been no significant price response so far. China's remnimbi devaluation has helped at the margins, but there is also what he described as "zombie" capacity – plants which operate in spite of losses, often integrated with profitable coal production, supported by cheap loans from state banks and/or where jobs are important. As a result Chinese operating rates remain around 70%. Some rationalization was, however, likely in the medium term, he said. The betting is that the urea market will bottom out around $210/t in 2016 and rise thereafter as oil and coal markets recovered.

CRU's **Johnathan Humphrey** presented a general economic outlook. China continues to grow, albeit at a reduced rate of 6% per year, and is moving towards consumption rather than investment led growth, but there is short term volatility, reflected in the stock market. Meanwhile, currencies have weakened as investors have moved capital out of emerging markets, but these too are still seeing average growth of around 4%, ranging from around 7% in India and SE Asia down to close to zero, but very low in Ukraine and Venezuela. The strong

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dollar has led to lower commodity and energy prices, but geopolitical risks still cloud the picture including: the rise of populist politicians globally; possible ‘Brexit’ from Europe; the continuing conflict in Syria and Iraq; Brazil, where economic woes are compounded by corruption scandals; and rivalries over the South China Sea and North Korea’s unpredictability.

Emerging regions

The prospects for Iran’s nitrogen industry were discussed by a panel comprising **Stephen Zwart** of Stamicarbon, **Harold van der Zande** of OCI, **Ali Ashgar Bazgir** of Tappico and **Mohammed Ali Vedikheil** of Iran’s Phoenix Vision Group. With the rollback of sanctions, the IMF is forecasting GDP growth of 4% in 2016 and 5% by 2020. Iran also has considerable potential for extra domestic demand for fertilizer, with typical application rates of only 18kgN/hectare, compared to 86kgN/ha worldwide, and in 2012 Iran itself did apply 53kgN/ha. The country has imported 11-16 million t/a of cereal grain, and could do with a boost to domestic production. On the supply side, meanwhile, Iran has the largest gas reserves in the world and there are 10 urea plants under construction with a total capacity of 10.8 million t/a, and two – Marvdasht and Pardis – are close to completion, but others are likely to need foreign financing and technical assistance. There was a range of optimism on the prospects for completing all of this new capacity, especially as regards shortages of skilled personnel, but with gas prices around $1.50/MMBtu for feedstock gas, it was argued that Iranian export capacity should be able to find a market, and the government has been keen to develop port infrastructure for export. The likelihood was that 2-4 of these urea plants should be up and running by 2019.

There was also a panel session on the prospects for increased fertilizer demand from Africa. **Mark Brouwer** of UreaKnowHow.com and **Theo Verweerden** and **Prosper Ndayiragije** of DSM noted that there is a potential market of 35 million t/a of nutrients in sub-Saharan Africa, but in order to stimulate it local farmers need things like credit to buy farm inputs, and help with delivery/logistics, training and education. Notore in Nigeria partnered with a local NGO and was able to work via promoters in villagers, in a few years reaching two million small farmers. DSM worked with the International Finance Corp, World Food Programme, Clinton Health Access Initiative and the Rwandan government on its own Project LIFE, aiming to prevent malnutrition in one million children (43% of Rwandan children suffer from stunted growth as a result of poor nutrition in their first two years). Via nine agricultural cooperatives they have reached 9,000 farmers.

Catalyst developments

Haldor Topsoe launched its new *SK-501 Flex*™ high temperature shift catalyst, the world’s first commercial iron-free high shift catalyst, with zero chromium content. Topsoe claims that it can operate at any steam-to-carbon ratio, while its increased activity means ammonia producers can choose to save on feedstock and energy or boost production up to 5% with the same set-up, potentially increasing revenue by $11 million per year for a large-scale ammonia plant, or $57 million over a catalyst lifetime. Industrial trials have shown activity and stability over several years of operation, and significantly exceeds the results with conventional iron-based catalysts. The *SK-501 Flex* formulation is based on zinc oxide and zinc aluminium spinel that has been known to catalyse the water gas shift reaction, but Topsoe researchers say that they are the first to transform this insight into commercial benefits. The iron-free formulation prevents formation of unwanted iron carbides that reduce the catalyst strength of conventional iron-based HTS catalysts. Without chromium and its associated risks, there is no need for extra procedures at start-up. In addition, activation is fast and typically takes place in connection with the start-up of the reforming section, so costly downtime is reduced significantly.

Clariant, meanwhile, launched new a low temperature shift (LTS) chloride guard catalyst called *ShiftGuard*™ *200* at the conference. *ShiftGuard 200* effectively adsorbs and retains chlorides so that any downstream main LTS catalyst, such as *ShiftMax*® *217*, is protected. Chloride is a typical catalyst poison in an ammonia or hydrogen plant that leads to an increasing and irreversible reduction of catalyst activity and mechanical stability. This can result in a significant catalyst lifetime reduction. Clariant says that in tests, *ShiftGuard 200* demonstrated “outstanding” chloride removal performance and better mechanical and hydrothemal stability as well as a high added low temperature CO shift activity with low methanol by-product formation. It is completely chromium free and therefore fulfils the EU criteria of a sustainable product by avoidance of critical metals and compliance with REACH regulations.

Co-production

Several papers looked at enhancing a nitrogen plant’s output via co-products. Johnson Matthey’s urea formaldehyde concentrate (UFC) process is detailed elsewhere in this issue, but Shell also discussed its *UreaPlusS* product, whereby micronised sulphur is incorporated into a urea granule. The process, as described by **Harald Franzrahe** of UFT and **Rafael Garcia** of Shell, uses UFT’’s fluid bed granulation technology.

A more radical example of co-production came from **Alberto di Amicis** of Eurotecnica, adding a melamine side-stream to a urea plant. He presented flowsheets for integration into existing urea plants with minimal modification to the urea unit.

Petrokemija in Croatia have modified their plant to produce an ammonium sulphate side-stream. **Nenad Zecevic** said that to contain the corrosive and exothermic sulphuric acid reaction with ammonia a pipe cross reactor was developed which also introduces an additive later required in the AS granulation section.

Plant integration

On a similar theme, improving the integration between ammonia and urea units where possible can lead to cost and energy savings. **Yevgeny Makhynya** of thyssenkrupp Industrial Solutions showed a new process scheme which removed CO_2 from the ammonia synthesis gas using aqueous ammonia at syngas generation pressure. The rich solution is compressed to urea synthesis pressure and then regenerated – the latter is an energy-intensive part of the process but can use heat from the existing steam system, a modification also necessitated by the removal of the CO_2 compressor. The plant uses 2% lower energy after the modifications, with opex benefits of the order of $4.5 million per annum.

Casale’s philosophy for such integration, as described by **Elio Strepparola**, can occur at both a process level, better exploiting the synergies between the different plant units (the addition of a melamine stream was one of the examples cited)

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and no less importantly on an engineering level, impacting on the project execution, improving the interaction between licensor, EPC contractor and end user.

CO_2 removal

Carbon dioxide emissions from plants are coming under increasing scrutiny these days and several schemes for CO_2 removal from process streams were presented. **Syed Ali Raza Sani** explained how Engro Fertilizers in Pakistan had commissioned a 340 t/d low pressure CO_2 recovery system at their ammonia plant, using an amine solvent to take 90% of the CO_2 from reformer flue gas and pass it to the urea unit for additional urea make.

Agrium's Carseland, Alberta plant uses a promoted MDEA solvent to remove CO_2 from ammonia synthesis gas, but was found to be operating unexpectedly close to its capacity limit. The issue turned out to be flow maldistribution and consequent poor absorption performance across the lower 5m of packing. **Ralph Weiland** of Optimised Gas Treating explained how the diagnosis had been made, and the steps taken to ameliorate it.

Finally, **Paul Tawiah** of Shell Canada discussed how his company had implemented a flue gas recycle to a steam reformer furnace in the hydrogen plant of the Shell Scotford Upgrader in Alberta. The recycle was implemented as a result of the installation of a CO_2 capture system via an absorber which removes 1 million t/a of CO_2.

Revamping

Several plant revamping strategies were examined by speakers. **John Pach** of Johnson Matthey reviewed several case studies which had improved production rate and decreased energy consumption, for example by installing a pre-reformer to reduce load on the main reformer and allow better energy integration with the primary reformer. Retubing the reformer with thinner, stronger tubes can allow more catalyst to be installed and increased firing rates, while improved burners can produce more even firing.

Giovanni Genova of Casale and **Filippo Pellicani** of Clariant considered the concept of exergy – the amount of energy in a system available for use. A conventional energy analysis of an ammonia converter highlights chiller and air cooler thermal duty as the main sources of energy loss, whereas exergy analysis shows that more work can be gained from improvements to, for example the separator and syngas compressor. They highlighted this for the case of a switch to an axial-radial ammonia converter. Combined with high activity catalyst (in this case Clariant's *AmoMax 10*), specific energy consumption was lowered by approximately 25% compared to the base case.

Reformer management

Reformers suffer the highest thermal stresses in a syngas plant, and can be a major source of issues. **Dan Barnett** of BD Energy Systems considered overheating in reformers, which can damage tubes and lead to failures, and ways of measuring and ameliorating this, via tube growth monitoring in real time, which can provide an approximation to tube temperature to within 5°C.

As well as absolute temperature issues, temperature cycling during startup and shutdown can lead to creep failure of reformer tubes, a particular issue where gas supply restrictions lead to stop-start operation. **Daniel Drabble** of Quest Integrity showed analytical methods for calculating time to failure for a variety of materials and conditions which can indicate when tube replacement is required.

Haldor Topsoe have developed a reformer monitoring system focusing on tube wall temperatures, as described by **Kim Carlsen**. Measuring the temperature is done with infrared or gold cup pyrometers, but measurements must then be adjusted for tube emissivity.

Turbomachinery

Rotating machinery can be a source of bottlenecks in plants, and the long lifetime and high replacement cost of such equipment can be a barrier to replacement. **Petr Polak** of Siemens Industrial Turbomachinery considered the implications for replacement of steam turbine systems with reference to case studies at Azoty Police in Poland and Dorogobuzh in Russia.

Robert Werner of Alstom Power also highlighted the issue of ageing turbine equipment – often 30 or 40 years old in some plants. His case studies, at Odessa Port Plant in Ukraine, Kuybishev Azot and Kemerovo Azot in Russia also demonstrated the benefits that can be gained through uprating of turbomachinery at such facilities.

Replacement of a syngas compressor at Achema's ammonia plant in Lithuania was the subject for **Aleksandr Liubimov** of Entechmach, which led to an energy saving of 4.5 MW over the original compressor and allowed for an increase in ammonia production at the plant.

Modernisation of syngas compressors was also the topic for **Tomoyuki Nishikawa** of Mitsubishi Heavy Industries, arguing that modernisation by the original equipment manufacturer (OEM) avoids the risks when operating with higher steam pressures than the original design.

The issues that can be caused by operation of old compressors were highlighted by **Alireza Orooji** of the Pardis Petrochemical Company in Iran, who described corrosion of the air compressors at the Pardis ammonia plant, accelerated by the sea-side location.

Finally, **Syed Muhammad Haseeb Bukhari** of Engro in Pakistan shared his company's experiences of operating and maintaining rotating equipment over 50 years, and the company's monitoring and maintenance philosophy and practice.

Energy efficiency

Managing and minimising pressure drop across the ammonia process can reduce power consumption and increase efficiency. Johnson Matthey showed how catalyst designs can reduce pressure drop in key stages of the plant, from feed gas purification to reforming and ammonia production.

A large number of plants from the 60s and 70s are still in operation with energy consumption in the range 8.0-9.5 Gcal/tonne ammonia. **Girish Patel** of KBR presented options for reducing this, from modifications to the primary and secondary reformer to installation of a KRES reforming exchanger, modification of shift converter internals, reducing compression requirements and improving instrumentation/control systems.

Six Sigma is a set of data/process tools for optimising processes developed by Motorola in the 1980s. Engro Fertilizers in Pakistan have applied it to their ammonia-urea unit at Daharki and **Arlan Naseem** explained the rationale and process and the results obtained from the analysis.

Last year Energy Recovery presented their hydraulic turbocharger system – *IsoBoost* – intended to recover and transfer hydraulic energy from process steams. This year's follow up, by **Max Shirazi**, presented a case study from a gas plant in Texas, which was able to save 60% of the energy required to pressurise the amine system for CO_2 recovery, saving 1.4MWh per year in energy consumption.

Nitric acid

Casale has recently acquired both plant constructors Chemoprojekt and the former GPN nitric acid technology license, and **Iacopo Cerea** discussed how these new capabilities had come together with the design and construction of a 685 t/d dual pressure nitric acid plant for Yara in Sweden.

Operation of nitric acid plants without production of brown/orange NOx clouds from the stack during start-up is becoming more and more important, especially in the US. **Johannes Dammeier** of thyssenkrupp Industrial Solutions presented several options for achieving this, using high pressure steam to heat the process gas cooler and then injecting steam upstream of the expander, though this does require a waste heat boiler. Tail gas heating with a burner or electrically can also assist with this.

Unicore launched its MPAC multi combination gauze pack in 2014 for nitric acid production. **Christian Goerens** updated the conference with results from lab and pilot scale testing which show that metal losses can be reduced by up to 20% compared to conventional gauze layouts.

Corrosion is also a major issue in nitric acid plants, and **Daniel Gulberg** of Sandvik Materials Technology explained how bimetallic tubes can give the advantages of zirconium's corrosion resistance without the expense of complete replacement of stainless steel with zirconium steel.

Operational experience

Yara's new Urea7 plant at Sluiskil in the Netherlands has a nameplate capacity of 3,500 t/d. However, the company has performed two load tests to assess the maximum production that can be obtained, as described by **Eelco Mostert** of its constructors, Stamicarbon. The first load test, in 2012, achieved 4,000 t/d, and the second, in 2015, 4,212 t/d for 24 hours.

Saipem/Snamprogetti has developed its own proprietary high efficiency reactor trays for urea plants, called *SuperCups*, and **Ugo Avagliano** of Saipem gave details of two installations, at Borealis Agrolinz in Austria and Fauji Fertilizer Co in Pakistan, which were presented as proof of concept, with a production increase of 4% even with only two reactor trays replaced.

Engro in Pakistan highlighted some incidents from 40 years of operation of their urea plant at Daharki, including several revamps, and a major ammonia release in 1983 that led to a change in procedure and modifications to make any future release much less significant.

Abdul Jabbar of Fatima Fertilizer Co, also in Pakistan, described issues with a prereformer catalyst installation which gave an unexpectedly high pressure drop. Investigation found that the top surface of the catalyst was broken caused by insufficient distance from the inlet gas distributor to the catalyst.

Rizwan Majeed of Fatima, meanwhile, explained an incident from June last year where an ammonia synthesis converter suffered a rapid pressure increase followed by a fire during a plant step-down. The root cause was found to be differential contraction at the converter outlet flange leading to a leak and auto-ignition of the syngas when it met air.

Other incidents discussed during a final plenary incident analysis session included a failure at a Safco plant in Saudi Arabia caused by hydrogen embrittlement of steel, foaming in a CO_2 removal system leading to a runaway methanator reaction and fire incident at Fatima Fertilizers, and a failure of the outlet pipeline of a urea reactor at Abu Qir in Egypt. ■

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Above: EuroChem's Novomoskovsky plant.

Nitrogen project listing 2016

Nitrogen+Syngas' annual nitrogen project listing covers all new ammonia, urea, nitric acid and ammonium nitrate plants currently under construction or development worldwide.

Contractor	Licensor	Company	Location	Product	mt/d	Status	Start-up date
ALGERIA							
n.a.	Casale	Fertial	Arzew	Ammonia	1,000	RE	2017
AZERBAIJAN							
Samsung	Haldor Topsoe	SOCAR	Sumgait	Ammonia	1,200	UC	2017
Samsung	Stamicarbon	SOCAR	Sumgait	Urea	2,000	UC	2017
BANGLADESH							
Uhde India Pvt	UFT	Kafco	Chittagong	Urea	2,100	RE	2017
BELARUS							
thyssenkrupp I.S.	thyssenkrupp I.S.	Grodno Azot	Grodno	Nitric acid	1,200	UC	2016
thyssenkrupp I.S.	thyssenkrupp I.S.	Grodno Azot	Grodno	Ammonium nitrate	3,500	UC	2016
BOLIVIA							
Samsung	KBR	YPFB	Bulo Bulo	Ammonia	1,200	UC	2016
Samsung	TEC	YPFB	Bulo Bulo	Urea	2,100	UC	2016
BRAZIL							
Technip, TEC	Haldor Topsoe	Petrobras	Uberaba	Ammonia	1,500	BE	2017
Tecnimont	KBR	Petrobras	Tres Lagoas	Ammonia	2,200	UC	2018
Tecnimont	Stamicarbon, UFT	Petrobras	Tres Lagoas	Urea	3,600	UC	2018
n.a.	Saipem	Petrobras	Linhares	Urea	2,300	CA	n.a.
CANADA							
Casale	Casale	Koch Nitrogen	Brandon, MN	Urea	700	RE	2016
Casale	Casale	Terra	Courtright, ON	Urea	1,100	RE	2017
n.a.	Saipem	Agrium	Redwater, AL	Urea	2,600	RE	n.a.
Kiewit, Tecnimont	KBR	Iffco	Becanour, QB	Ammonia	2,200	P	On hold
Kiewit, Tecnimont	Stamicarbon	Iffco	Becanour, QB	Ammonia	3,850	P	On hold

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Contractor	Licensor	Company	Location	Product	mt/d	Status	Start-up date
CHINA							
Casale	Casale	Inner Mongolia Manshi	Erdos	Ammonia	1,630	RE	2016
Casale	Casale	Henan Junhua	Zhumadian, Henan	Ammonia	2,000	RE	2016
Casale	Casale	Henan Jinkai	Kaifeng, Henan	Ammonia	2,000	RE	2017
Casale	Casale	Shenua Ningxia	Ningxia	Ammonia	500	RE	2016
CECC	KBR	Inner Linggu Chem Co	Yixing, Jingsu	Ammonia	1,500	UC	2016
n.a.	Stamicarbon	Inner Mongolia Huajin	Panjin	Urea	2,860	BE	2017
n.a.	Saipem	Wulan Coal Group	Wulanhaote	Urea	4,000	UC	n.a.
EGYPT							
Tecnimont	KBR	Kima	Aswan	Ammonia	1,200	UC	2018
Tecnimont	Stamicarbon	Kima	Aswan	Urea	1,575	BE	2018
thyssenkrupp I.S.	thyssenkrupp I.S.	MOPCO	Damietta	Ammonia	2 x 1,200	UC	2016?
thyssenkrupp I.S.	Stamicarbon	MOPCO	Damietta	Urea	2 x 1,925	UC	On hold
FRANCE							
Casale	Casale, UFT	Borealis Chimie	Grandpuits	Urea	850	RE	2016
GABON							
Technip	Haldor Topsoe	Gabon Fertilizer Co	Port Gentil	Ammonia	2,200	DE	On hold
Technip	Saipem, UFT	Gabon Fertilizer Co	Port Gentil	Urea	3,850	DE	On hold
HUNGARY							
n.a.	thyssenkrupp I.S.	Nitrogenmuvek	Petfurdo	Ammonium nitrate	1,550	UC	2016
INDIA							
Casale	Casale	Zuari	Goa	Urea	1,050	RE	2017
Casale	Casale	Iffco	Kalol	Ammonia	1,140	RE	2016
Casale	Stamicarbon	Iffco	Kalol	Urea	1,780	RE	2016
Casale	Casale	Iffco	Phulpur	Ammonia	1,850	RE	2016
Casale	Saipem	Iffco	Phulpur	Urea	2,115	RE	2016
Casale	Casale	Iffco	Phulpur	Ammonia	1,215	RE	2016
Casale	Saipem	Iffco	Phulpur	Urea	1,600	RE	2016
Casale	Casale	Iffco	Aonla	Ammonia	2 x 1,850	RE	2016
Casale	Saipem	Iffco	Aonla	Urea	2 x 1,600	RE	2016
PDIL	KBR	Matix Fert & Chem	Panagarh	Ammonia	2,200	UC	2016
Saipem	Saipem	Matix Fert & Chem	Panagarh	Urea	3,850	UC	2016
n.a.	Saipem	RCFL	Ramagundam	Urea	3,850	DE	n.a.
INDONESIA							
TEC	KBR	PAU	Sulawesi	Ammonia	1,900	UC	2016
TEC, PT Rekayasa	KBR	Pusri	Palembang	Ammonia	2,750	UC	2016
TEC, PT Rekayasa	TEC	Pusri	Palembang	Urea	2,000	UC	2016
n.a.	thyssenkrupp I.S.	Bakri	Kalimantan	Nitric acid	750	DE	On hold
n.a.	thyssenkrupp I.S.	Bakri	Kalimantan	Ammonium nitrate	900	DE	On hold
IRAN							
Hampa	Stamicarbon	Lordegan Petrochemical	Lordegan	Urea	3,250	UC	2017
Hampa	Stamicarbon	Golestan Petrochemical	Golestan	Urea	3,250	DE	On hold
Hampa	Stamicarbon	Zanjan Petrochemical	Zanjan	Urea	3,250	DE	On hold
n.a.	Stamicarbon	Pardis Petrochemical	Pars	Urea	3,250	C	2016
n.a.	Saipem	Hengan Petrochemical	Assaluyeh	Urea	3,250	UC	n.a.

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47 48 49 50 51 52 53 54 55 56 57 58 59 60

Contractor	Licensor	Company	Location	Product	mt/d	Status	Start-up date
IRAQ							
NFC	KBR	NFC	Baiji	Ammonia	1,200	RE	On hold
NFC	Stamicarbon	NFC	Baiji	Urea	2,250	RE	On hold
MALAYSIA							
MHI	Haldor Topsoe	Petronas	Siptang	Ammonia	2,100	UC	2016
MHI	Saipem, UFT	Petronas	Siptang	Urea	3,500	UC	2016
MEXICO							
n.a.	Saipem	Pemex	Pajaritos	Urea	1,500	RE	n.a.
NETHERLANDS							
Tecnimont, Yara	UFT	Yara	Sluiskil	Urea	1,625	RE	2017
NEW ZEALAND							
n.a.	Casale	Ballance Agri-Nutrients	Kapuni	Urea	1,400	RE	2019
NIGERIA							
TEC, Daewoo	KBR	Indorama	Port Harcourt	Ammonia	2,400	UC	2016
TEC, Daewoo	TEC	Indorama	Port Harcourt	Urea	4,000	UC	2016
Saipem	Haldor Topsoe	Dangote Fertilizer Ltd	Agenbode	Ammonia	2 x 2,200	UC	2017
Saipem	Saipem, UFT	Dangote Fertilizer Ltd	Agenbode	Urea	2 x 3,850	UC	2017
ROMANIA							
Chemoprojekt	Casale	Azomures	Targu Mures	Ammonia	2 x 1,050	RE	2016
Chemoprojekt	Stamicarbon	Azomures	Targu Mures	Urea	1,425	RE	2016
RUSSIA							
NIIK	NIIK, Stamicarbon	Minudobrenija Perm	Perm	Urea	1,720	RE	2016
Casale	Casale	EuroChem	Nevinnomyssk	Urea	1,600	RE	2016
Casale	Casale	Togliatti Azot	Togliatti	Urea	2,200	DE	2019
Tecnimont	KBR	EuroChem	Kingisepp	Ammonia	2,700	BE	2018
Tecnimont	KBR	EuroChem	Nevinnomyssk	Ammonia	2,700	BE	n.a.
Tecnimont	Stamicarbon	EuroChem	Nevinnomyssk	Urea	3,500	CA	n.a.
NIIK, MHI	Haldor Topsoe	JSC Ammoniy	Mendeleevsk	Ammonia, Methanol	2,050	C	2015
MHI, NIIK	Saipem, UFT	JSC Ammoni	Mendeleevsk	Urea	2.050	C	2015
n.a.	Stamicarbon	Uralchem	Perm	Urea	770	RE	2019
Saipem	Sapiem, UFT	Baltic Urea Plant	St Petersburg	Urea	3,500	DE	n.a.
MHI, Sojitz	Haldor Topsoe	PhosAgro	Cherepovets	Ammonia	2,200	BE	2017
Chemprojekt	Stamicarbon	PhosAgro	Cherepovets	Urea	1,500	BE	2017
NIIK, Chemoprojekt	Stamicarbon	PhosAgro	Cherepovets	Urea	1,500	RE	2017
SAUDI ARABIA							
Daelim	thyssenkrupp I.S.	Ma'aden	Ras al Khair	Ammonia	3,300	UC	2017
SLOVAKIA							
Chemoprojekt	Haldor Topsoe	Duslo Sala	Sala	Ammonia	1,600	RE	2018
SPAIN							
Casale	Casale, thyssenkrupp I.S.	Fertiberia	Puertollano	Ammonia	600	RE	2016
SWEDEN							
Chemoprojekt	Casale	Yara	Koping	Nitric acid	685	UC	2017
n.a.	n.a.	Yara	Koping	Ammonium nitrate	1,360	RE	2017

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Contractor	Licensor	Company	Location	Product	mt/d	Status	Start-up date
TANZANIA							
Ferrostaal	Haldor Topsoe	Tanzanian Petroleum	Mtwara	Ammonia	2,200	DE	2020
Ferrostaal	n.a.	Tanzanian Petroleum	Mtwara	Urea	4,000	DE	2020
TURKEY							
n.a.	Haldor Topsoe	Eti Bakir	Mardin	Ammonia	300	UC	2017
TURKMENISTAN							
MHI, GAP	Haldor Topsoe	Turkmenkhimiya	Garabogaz	Ammonia	2,000	UC	2018
MHI, GAP	Saipem, UFT	Turkmenkhimiya	Garabogaz	Urea	3,500	UC	2018
UNITED STATES							
thyssenkrupp I.S.	thyssenkrupp I.S.	CF Industries	Donaldsonville, LA	Ammonia	3,300	C	2016
thyssenkrupp I.S.	Stamicarbon, UFT	CF Industries	Donaldsonville, LA	Urea	3,500	C	2016
thyssenkrupp I.S.	thyssenkrupp I.S.	CF Industries	Donaldsonville, LA	Nitric acid	1,515	C	2016
thyssenkrupp I.S.	thyssenkrupp I.S.	CF Industries	Donaldsonville, LA	UAN	4,300	C	2016
thyssenkrupp I.S.	thyssenkrupp I.S.	CF Industries	Port Neal, IA	Ammonia	2,200	UC	2016
thyssenkrupp I.S.	Stamicarbon, UFT	CF Industries	Port Neal, IA	Urea	3,490	UC	2016
OCI Construction	KBR	Iowa Fert Co	Wever, IA	Ammonia	2,000	UC	2016
OCI Construction	Stamicarbon, UFT	Iowa Fert Co	Wever, IA	Urea	2,200	UC	2016
OCI Construction	thyssenkrupp I.S.	Iowa Fert Co	Wever, IA	Nitric acid	1,530	UC	2016
OCI Construction	thyssenkrupp I.S.	Iowa Fert Co	Wever, IA	Ammonium nitrate	1,900	UC	2016
OCI Construction	thyssenkrupp I.S.	Iowa Fert Co	Wever, IA	UAN	4,300	UC	2016
KBR	Stamicarbon, UFT	Agrium	Borger, TX	Urea	1,800	RE	2017
KBR, Casale	KBR, Casale	Agrium	Borger, TX	Ammonia	1,900	RE	2017
IHI	Stamicarbon	Dakota Gasification	Beulah, ND	Urea	1,000	UC	2017
KBR	KBR	Dyno Nobel	Waggaman, LA	Ammonia	2,400	UC	2016
thyssenkrupp I.S.	KBR	Midwest Fertilizer Co	Mt Vernon, IN	Ammonia	2,200	CA	2020
thyssenkrupp I.S.	Stamicarbon	Midwest Fertilizer Co	Mt Vernon, IN	Urea	2,200	CA	2020
thyssenkrupp I.S.	Casale	Midwest Fertilizer Co	Mt Vernon, IN	Nitric acid	1,530	CA	2020
thyssenkrupp I.S.	Stamicarbon	Midwest Fertilizer Co	Mt Vernon, IN	UAN	4,300	CA	2020
Black & Veatch	KBR, Casale	Koch Nitrogen	Enid, OK	Ammonia	1,680	RE	2016
Black & Veatch	Stamicarbon	Koch Nitrogen	Enid, OK	Urea	2,200	UC	2016
Linde	Linde	Simplot Phosphates	Rock Springs, WY	Ammonia	600	UC	2017
Casale	Casale	Rentech Nitrogen	East Dubuque, IL	Ammonia	1,180	RE	2016
SNC Lavalin	Casale	Summit Clean Energy	Pennwell, TX	Ammonia	n.a.	DE	2019?
SNC Lavalin	Saipem	Summit Clean Energy	Pennwell, TX	Urea	2,245	DE	2019?
Casale	Casale	El Dorado Ammonia	El Dorado, AR	Ammonia	1,210	RE	2016
n.a.	Haldor Topsoe	BioNitrogen	Florida	Ammonia	300	DE	2017
Tecnimont	KBR	Cronos Chemical	Tuscola, IL	Ammonia	2,200	CA	2019
Tecnimont	Stamicarbon	Cronos Chemical	Tuscola, IL	Urea	3,850	CA	2019
KBR	KBR	BASF, Yara	Freeport, TX	Ammonia	2,270	UC	2017
n.a.	Stamicarbon	PCS Nitrogen	Geismar, LA	Urea	450	RE	2018
UZBEKISTAN							
MHI	Haldor Topsoe	NavoijAzot	Navoij	Ammonia	2,000	DE	2018
MHI	Saipem, UFT	NavoijAzot	Navoij	Urea	1,750	DE	2018
VIETNAM							
Technip	thyssenkrupp I.S.	PetroVietnam	Phu My	Ammonia	1,600	RE	2017

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Large-scale DME still looking for commercial outlets

Korea Gas Corp (Kogas) is looking to the US and Asia to commercialise its large-scale dimethyl ether (DME) technology, reports **David Hayes**.

California's stricter vehicle exhaust emission regulations could provide a new opportunity for the Korea Gas Corporation (Kogas) to commercialise its natural gas to dimethyl-ether (DME) production process for the first time, as the company continues with global marketing efforts to promote its proprietary technology. Interest in DME in the United States is growing following an announcement by Volvo USA that it will begin selling DME-fuelled heavy duty trucks to comply with tighter vehicle exhaust emission rules in California.

"California's regulations for exhaust gases from heavy vehicles and buses will be stricter. Many people think that in the near future diesel vehicles will not meet these new requirements and that DME will be the only option; so Volvo and other companies are developing DME-fuelled trucks," explained a Kogas source. "Many companies are interested in DME as diesel consumption for trucks and buses in the US is about 3 billion gallons a year - it's very big."

Oberon Fuels of San Diego already has built a pilot plant to supply DME for Volvo to test its new DME-fuelled trucks. Opened in 2013 in Brawley, California, Oberon Fuels' pilot DME plant is designed for small-scale production using natural gas feedstock and biogas from farms and other sources. Oberon Fuels currently offers three standard production units that can be configured to produce 10,000 gallons per day of DME or 11,300 gallons per day of methanol. The company recently has signed an agreement with Ford to supply DME to test DME-fuelled cars that are under development.

Kogas' LNG terminal – the company is a major force in the LNG market.

Large-scale production

Kogas' interest in DME, however, is in building large scale DME plants that use its own one step production technology. The company already has designed a 300,000 t/a DME plant that originally was due to be built in Saudi Arabia to produce DME from natural gas for export to South Korea, and Kogas began marketing its large scale DME technology after the Saudi deal fell through because the Saudis were not able to provide gas supply. Currently the company is looking to develop DME projects in several countries while work is underway designing a smaller DME plant size to meet different market demands.

A number of companies in the United States are understood to be interested in using Kogas' direct natural gas-to-DME technology to build DME plants that use shale gas and other natural gas feed stocks.

According to the source, American fuel companies are interested in building large scale DME plants to produce DME at lower cost than is possible in a small plant. DME pricing is important as diesel prices are low in the United States and vehicle owners will not want to be faced with increased fuel prices when switching to DME. “Some American companies have contacted us – oil companies, methanol companies and some refineries have enquired,” the source said. “They are interested in our 300,000 t/a plant size and bigger, as the United States will need a lot of DME in future.”

Kogas believes its DME process will allow DME to be produced at lower cost by a large plant. In fact, various size DME plants may be needed in future to meet different customers’ needs and so Kogas is working on modifying some technology designed for its large scale plant for use in medium and smaller scale DME plants.

“We thought that a 300,000 t/a plant would be the optimum size but recently some companies want a small DME plant such as 50,000 t/a in some countries,” the source remarked. “They want to avoid risk, so they want to start producing DME with small scale projects. We are upgrading our process; the main reformers and reactors are Kogas-designed. We have catalyst technology for CO_2 separation, so we are developing a new CO_2 separation process for 50,000 t/a and 100,000 t/a plant sizes. We are changing some processes for different scale DME plants.”

Looking to Asia

In addition to the United States, Kogas is promoting its DME technology in other countries that have large natural gas reserves which could be used to produce DME either for local use or export. In Malaysia, for example, Kogas is carrying out a feasibility study with Petronas, the national oil and gas company, to build a 1,000 t/d DME plant in Sabah State in East Malaysia. The proposed plant will supply part of its output to the local market and export the rest to South Korea for use as a low cost fuel additive to blend with LPG initially and then eventually to be used as a diesel fuel substitute.

The feasibility study has been launched following the signing of a memorandum of understanding by Kogas and Petronas to construct a 300,000 t/a DME plant. The study is intended to enable Petronas to compare Kogas’ one-step natural gas-to-DME technology with Toyo Engineering’s methanol dehydration-to-DME process. Kogas initially obtained a license from the Sabah state government to build a DME plant in a local industrial park with Petrosab, the state oil and gas company, which plans to use natural gas produced in the state as feedstock. The feasibility study was launched after Petronas joined the project consortium in 2014. Petronas already owns a large methanol plant on Labuan island located off East Malaysia and wants to look at the feasibility of using natural gas to produce DME compared with methanol.

Natural gas is the more economical of the two feed stocks, according to Kogas, as gas prices are lower than for methanol which Petronas can sell at the market price. “Kogas’ DME process has never been commercialised, so Petronas wants to check as most DME plants in China use the Toyo process using natural gas or other feedstock. They make methanol and then dehydrate to DME,” the source said. “Our Kogas process does not make methanol as an intermediary. We think our process is more simple as it does not make methanol and is more efficient. Toyo’s is a two step process but ours is one step. It’s not only Kogas that has a one step DME process, other companies have a one step process but none has been commercialised.”

In addition to producing DME for export, some of the proposed plant’s output could by supplied for local use in Malaysia. Although the country has large natural gas reserves, Malaysia imports LPG for household and industrial use. One possibility is that DME could be used to blend with LPG and reduce the country’s LPG import requirements.

Elsewhere in Asia, Kogas is looking at opportunities to develop a DME project in Indonesia where talks are understood to be at an early stage. “In Indonesia we want to make DME from coal. They have a lot of low calorific coal and they want to use it for DME as they import LPG and diesel. They want to replace diesel use or mix DME with LPG,” the source said.

Nigeria

Efforts to commercialise Kogas’ DME technology have been underway for seven years since the one step DME process was first planned to be used in a low cost fuel import scheme designed to enable South Korea to import DME for blending with LPG. In 2011 Kogas began planning to develop a DME plant in Nigeria to use associated gas from oil production that is being flared off and which the government is keen to see captured for commercial use. After signing an MOU, Kogas carried out a feasibility study to build a 600,000 t/a consisting of two 300,000 t/a trains that were due to be built concurrently in a single stage creating a DME plant twice the size of the cancelled Saudi Phase 1 scheme. Several South Korean companies have shown interest in joining the project consortium. However, the project has been pending for two years, and the basic engineering design remains to be done, while buyers are sought for the DME. “Nigeria is pending as it’s too far to export DME to South Korea, so we are trying to export DME to some other African countries. Now the project is pending, maybe sometime we can start again,” the source said.

Floating DME

Meanwhile, Kogas has been working on other longer term opportunities to commercialise its DME technology, including the construction of a floating DME production unit. The idea is similar to a floating LNG production and storage unit that can be moved to different locations as required. Among the advantages a floating production unit would offer is that it can be moved to different gas fields to produce DME when the gas reserves in an individual field are too small for it to be economically viable to build a conventional land-based DME process plant.

“Daewoo Shipbuilding has shipbuilding technology so we worked together. We carried out a preliminary Front End Engineering Design (FEED) for a floating DME production and storage unit as we have a land-based technical design,” the source said. “Right now there is no DME floating production and storage unit market but maybe after some years when DME use grows enormously we can use that engineering package as we have already developed it.”

According to Daewoo Shipbuilding’s engineering team a large plant of about 1 million t/a in capacity is required if a floating DME production unit that is financially viable is to be constructed. Among marine design requirements, the DME production plant along with the onboard power plant, natural gas feedstock storage tank and DME storage tank all need to be built on top of the floating unit which is effectively a customised ship. Only a large capacity floating DME unit would be economically viable to build and operate as a small capacity floating DME production unit would not produce sufficient DME to be cost effective. ■

Tailored feedstock purification solutions

In synthesis gas plants, an efficient feed purification section is crucial for protecting downstream catalysts from potential sulphur poisoning which has costly consequences. With the diversity of feedstocks being used in the industry today, the gas compositions, sulphur species and concentrations show large variations. Thus, more emphasis is required on the feed purification section to ensure proper desulphurisation and the loading scheme of the feed purification section must be tailor-made to meet the demands of individual plants.

Purification is a necessary requirement in many chemical processes so that we can safely and reliably convert our feedstock to salable products. Our demand for increased reliability and longer on-line time means that the expected level of performance we demand of our purification solutions has increased.

The feedstocks we use to make syngas based chemicals such as ammonia and methanol (usually natural gas, but also naphtha or others) have changed and will undoubtedly change again. This change will vary regionally as different feedstocks become available and technologies develop to enable us to safely and sustainably extract the hydrocarbons required for chemical production.

In the last 50 years coal, oil, naphtha, LPG and natural gas have all been used as feedstocks. Now, with the development of shale gas, there is a supply of fossil fuel resources for decades to come.

Feed gas purification for synthesis gas plants is an essential process step which ensures smooth and trouble-free operation of the plant. Purification removes unwanted species from the feedstock that might act as catalyst poisons, leading to deactivation of the catalyst and lower levels of production. While the biggest cause of premature catalyst failure is due to process upsets, one of the biggest barriers to not extending catalyst lives to their absolute maximum is catalyst poisoning.

Table 1: **Typical poisons in hydrocarbon feedstocks**

Poison	Form
Sulphur	Organic sulphur:
	Mercaptans
	Organic sulphides
	Organic disulphides
	Thiophenes
	Hydrogen sulphide (H_2S)
	Carbonyl sulphide (COS)
Chlorine	Organic chlorides
	Hydrogen chloride (HCl)
Arsenic	Arsine
	Tri methyl arsine
Mercury	Mercury

Source: Johnson Matthey

The cost of not enabling catalysts to provide the optimal performance over the whole campaign is hard to measure. Whether you use the cost of an unplanned shutdown alone or include additional lost-opportunity cost, the value of catalysts and absorbents that reliably do what the supplier states, over the whole campaign period can be worth millions of dollars to the operator.

The typical poisons encountered in a hydrocarbon feedstock are shown in Table 1. The geographical location and physical properties of the feedstock both have an effect on the type and level of impurities.

Catalyst poisons

Sulphur

Probably every ammonia or syngas plant is equipped with a desulphurisation unit, removing sulphur compounds from the feedstock. Sulphur removal is essential because it would otherwise lower the activity of the primary reformer catalyst which leads to less production and can lead to damage of the reformer by overheating.

Sulphur components are usually contained in natural gas or naphtha in the range of a few ppm to a few percent. They must be reduced to a level of approx. 0.1 ppm (v) prior to admission of the gas to the primary reformer. Sulphur is present in the feedstock in the form of hydrogen sulphide (H_2S) and organic sulphur (e.g. thiols, also called mercaptans, R-SH), and carbonyl sulphide (COS). Often, the bulk of sulphur is removed from natural gas during its production and only a small quantity remains in the gas sent to the consumers.

In most processes hydrogen sulphide is removed from the gas by passing it over a bed of zinc oxide pellets (ZnO) at a temperature above 350°C in the desulphurisation reactor. H_2S reacts with the ZnO to form H_2O (vapour) and ZnS (solid). The zinc oxide is often referred to as a catalyst as it converts H_2S into another compound, and as the vessel arrangement is similar to other catalyst-filled reactors in an ammonia plant (e.g. CO shift), but it is not a catalyst since it is consumed by the reaction. Prior to conversion of all ZnO

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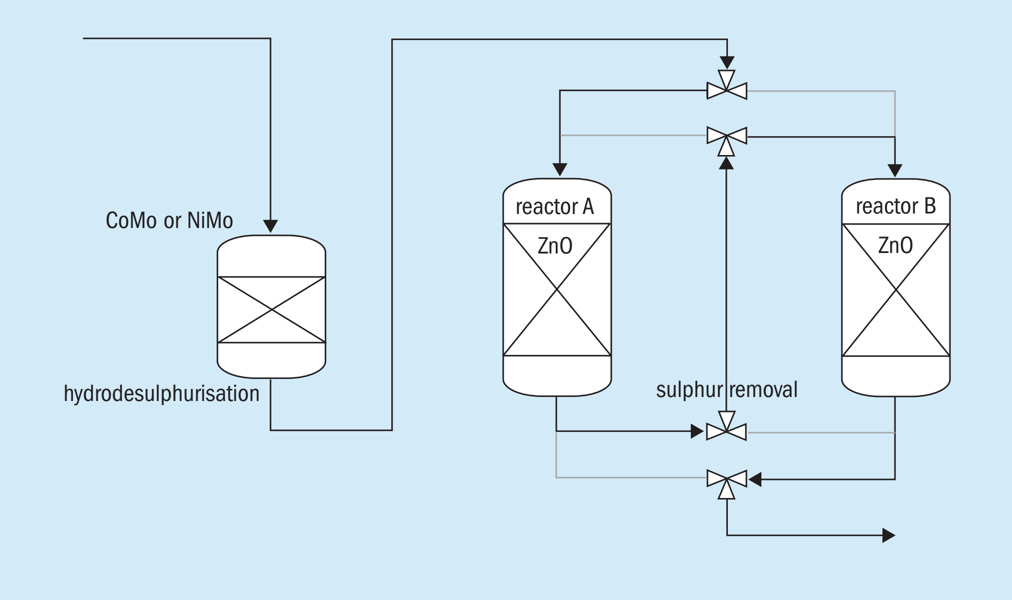
Fig 1: **Lead lag desulphurisation**



to ZnS the vessel must be emptied and re-filled with fresh ZnO. Catalyst suppliers take back the used ZnO/ZnS for processing. (Alternative materials are available operating at lower temperatures).

Sulphur components other than H_2S first have to be converted into H_2S in order to bring them into a form which can be handled by the desulphurisation reactor. This is done by the so-called hydrogenation catalyst, installed directly upstream of the desulphurisation reactor, sometimes in the same vessel. Its active components are molybdenum (Mo) and either cobalt (Co) or nickel (Ni). Over this reactor organic sulphur reacts according to the hydrogenation reaction:

$$R\text{-}SH + ½ H_2 \rightarrow H_2S + R\text{-}H$$

The produced H_2S is removed from the gas in the downstream desulphurisation reactor, while the organic compound R-H passes on and is being decomposed in the primary reformer.

In order to enable the hydrogenation reaction, a small quantity of hydrogen must be available. This is usually achieved by recycling a small stream of synthesis gas from further downstream (e.g. downstream of the methanation unit of an ammonia plant) to the hydrogenation reactor inlet.

Hydrogenation works both with CoMo or NiMo catalysts. In case where the feed contains CO and/or CO_2 in significant quantities, NiMo is the better choice.

Different layouts for hydrogenation and desulphurisation are possible. Their selection depends on the sulphur quantity expected in the feedstock and the desired operating period between shutdowns.

The simplest design is to install hydrogenation catalyst and ZnO bed in a single vessel one over the other with gas flow from top to bottom and ZnO bed at the bottom. Using this design the ZnO bed must be sized to accept all sulphur reaching the plant between two scheduled shutdowns.

In cases where there is a high amount of sulphur present and/or a long running time desired, the ZnO bed and the vessel would become very large. In order to avoid this, the system can be split into three vessels. The first and usually the smallest vessel contains the hydrogenation catalyst followed by two ZnO-filled reactors, A and B (Fig. 1). By using a specific arrangement of piping and valves, the gas can flow through them in sequence A to B, or B to A, or only A, or only B. In this so-called lead/lag arrangement the upstream reactor is active first (say reactor A) and picks up H_2S. As soon as any sulphur breakthrough is detected from reactor A it can be isolated, keeping only B in operation. Then, while the plant remains in operation, the ZnO charge in A can be replaced. After that, the sequence is reversed, the gas flows first through B, then to A, ensuring lowest H_2S at the outlet by the fresh charge in A. The hydrogenation catalyst usually survives much longer than the ZnO. The installation cost for vessels and piping is higher than for the one-vessel solution but it gives the plant owner the flexibility to continue operation even if sulphur levels rise unexpectedly.

For both designs, the recommended design criteria for the hydrogenation and the desulphurisation beds are as follows:

- The hydrogenation catalyst (CoMo or NiMo) should be designed in such a way to convert the highest expected amount of organic sulphur to H_2S. This is essential to ensure no organic sulphur slips through.
- The ZnO bed can be designed to convert the normal or average expected amount of sulphur. If for short time a higher amount of sulphur arrives at the plant (e.g. due to a disturbance in the gas company's upstream treatment unit) it will not endanger the integrity of the plant but will shorten the running time of the ZnO bed which will require earlier replacement. It is not necessary to design the ZnO bed for the maximum expected sulphur amount. This difference is often important as there are sometimes natural specifications found, stating for total sulphur: normal 20 pppm (v), maximum 600 ppm (v).

The ZnO bed solution is not the most economic process at very high sulphur levels. For high quantities of sulphur, scrubbing upstream of the syngas plant might be the better solution. H_2S can be removed from natural gas by absorption into an aqueous solution of amines or other solvents. There are special solvents available for absorption of organic sulphur compounds as well. The process with absorber and desorber is similar to the well-known processes for CO_2 removal from syngas. Such a scrubbing unit has been installed by thyssenkrupp Industrial Solutions in the QAFCO 3 and 4 ammonia plants in Qatar. The acid gas stream produced from the desorber can be burnt in a flare (which is not very friendly to the environment) or it can be used for sulphur or sulphuric acid production.

At high sulphur concentrations it might become necessary not only to remove sulphur from the feed gas but also from reformer or boiler fuel as otherwise the SO_2 level in the flue gas becomes too high and can cause corrosion.

Chlorine

Natural gas sometimes contains chlorine compounds (e.g. hydrogen chloride, HCl) which are poisons to the reformer and other catalysts, especially those containing copper. Usually they are present in much smaller quantities than sulphur. Organic chlorine compounds are converted in the hydrogenation reactor to hydrogen chloride, similar to the conversion of organic sulphur. Hydrogen chloride can be removed by a dedicated material installed in between the hydrogenation catalyst and the ZnO bed.

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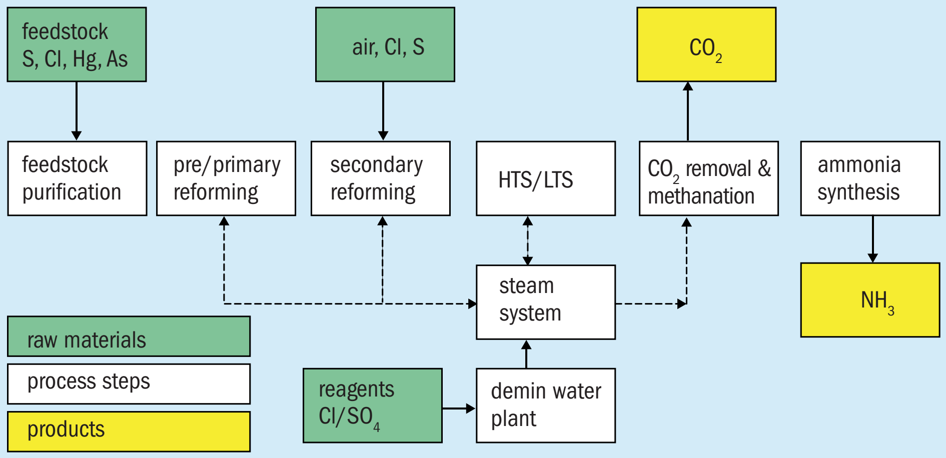
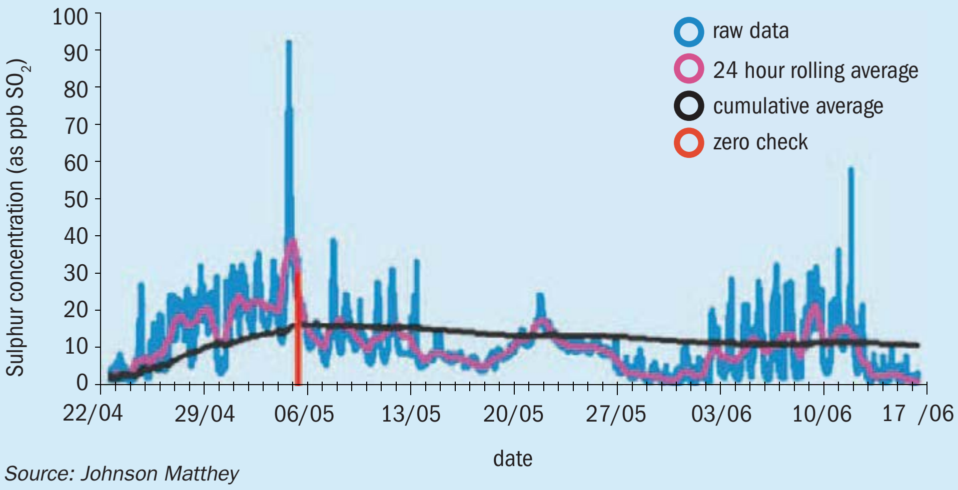
Fig 2: **Simplified ammonia flowsheet**



Source: Johnson Matthey

Fig 3: **Atmospheric monitoring of sulphur levels close to an ammonia plant**



Source: Johnson Matthey

Arsenic

Natural gas sometimes contains arsenic, which acts as a catalyst poison. It can be removed by a dedicated adsorbents installed upstream of the process.

Mercury

Mercury is sometimes present in natural gas in very small quantities, e.g. nanograms per cubic metre. It does not appear to act as a catalyst poison but it passes through the process as vapour and has been found, trapped by condensation, in low temperature vessels like the separator in the ammonia synthesis section. Mercury is suspected to lead to embrittlement of materials of construction thus endangering the mechanical integrity of the plant. Therefore mercury should be removed from the feed upstream of the process plant. Special adsorbents for this purpose are available. Such a mercury removal reactor, filled with Johnson Matthey's PURASPEC$_{JM}$™ absorbents, was part of thyssenkrupp's Sorfert project in Algeria.

Adverse affect of helium

Helium is a component that is sometimes present in natural gas. Although it does not take part in any of the chemical reactions and does not affect any catalyst of a syngas plant, it can lead to lower production rates as it can accumulate in the ammonia synthesis loop, building up a higher inert level and lowering the partial pressure of the reacting components.

Helium, like argon or other noble gases (coming from process air or natural gas), passes through the ammonia process and reaches the ammonia synthesis loop. All inert gases ending up there are removed by the purge gas stream. This purge gas stream usually undergoes a treatment in order to recover valuable hydrogen from it which is being recycled back to the process. In this hydrogen recovery unit, helium behaves differently to argon and methane which are the largest fractions of the inert components. Both in cryogenic and membrane hydrogen recovery units, most of the helium follows the same route as the hydrogen, meaning it is recycled to the synthesis loop, instead of being sorted out such as argon.

If the helium content in the natural gas feed is so high, that it can affect the plant's performance, a dedicated helium removal unit can be installed upstream of it. Such a unit operates by the principle of semi-permeable membranes. Small molecules like helium preferentially pass through the membranes. The so created permeate stream contains most of the helium, but still a high amount of methane and light hydrocarbons. The permeate stream can be used as boiler or reformer fuel gas in the plant without problems.

Such a membrane unit has been installed for example by thyssenkrupp Industrial Solutions in the Sorfert ammonia/urea complex in Algeria.

Impurities entering an ammonia plant

Impurities can enter the system via any of the raw material inputs. The hydrocarbon feedstock can contain any combination of sulphur, arsenic, mercury and chlorides (Fig. 2).

Mercury is often only present at ppb levels but is still capable of having a major impact. It's most commonly present in gas in the elemental form and is a highly interactive element, able to form interactions with equipment surfaces and process liquids and sludges. Despite this it has limited reactivity with many catalysts and can move as a vapour through the flowsheet, due to the high temperatures. However it will react with copper containing materials, where sulphur is also present, such as the top of LTS beds, and poison PGM catalysts, such as those used in the downstream process operations on an integrated chemicals site. Mercury can also impact the plant equipment as it is able to form amalgam corrosion, or liquid metal embrittlement. The corrosion mercury causes is a complex area yet through many years of research, our understanding of these risks is now far better understood. This understanding has been highlighted[2] at safety focused events such as the AIChE event in 2008.

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When a failure does occur the magnitude can be large and the impact instant, as seen at the Santos Moomba gas plant in Australia. For this reason there are examples of syngas producers electing to install mercury removal units (MRUs) to protect their flowsheets and to reduce the risk to a level as low as reasonable practicable.

Process air can contain chlorides and sulphur, depending greatly on the location of the plant. Figure 3 shows the levels of sulphur that have been measured in the process air to one ammonia plant. Typically, levels are <50 ppb, however, where adjacent industrial facilities handle high levels of sulphur (e.g.: refineries, steel plants, sulphuric acid plants and some fertilizer blending) sustained peaks of many 100s ppb have been experienced.

The final potential source of impurities is the process water. Although it is supplied via a demineralisation plant, this utility is also susceptible to process upsets where chlorides, sulphides, silicates or phosphides can be carried into the process steam system.

Each catalyst step in an ammonia plant is required to facilitate the conversion of raw materials to products by lowering the activation energy required for the reaction. Moreover the catalysts do this in a way that they remain unchanged and so can repeat the process many millions of times an hour.

The common causes for a catalyst reaching the end of life are well understood, and some of these are shown in Table 2.

The common impacts of poisoning across the flowsheet are as follows:

Purification: The purification section itself can be affected by materials such as dust and debris, excess olefins, heavy metals and other impurities not included in the design.

Pre-reforming: Sulphur in the feedstock causes rapid movement of the reaction profile in the bed. This is due to sulphur chemisorption onto the nickel resulting in catalyst deactivation leading to premature C_2+ slip.

Primary reforming: Sulphur in the feedstock causes catalyst deactivation. This means there is less reforming activity to remove heat from the tube wall. This ultimately causes carbon formation and hot bands that can lead to tube failure if not rectified.

Secondary reforming: Fouling can impact the pressure drop through the secondary reformer. Alumina vaporization can result in mass transfer of support material from the top of the bed that can block material beneath it. This also brings air into the process which can introduce sulphur and chloride that will impact directly on the downstream HTS and LTS.

HTS: This duty can be poisoned but is far less susceptible to deactivation due to the presence of sulphur. Chloride, if present, will deactivate the copper promoter which can lower the activity of the catalyst to the point where over-reduction can occur. Other poisons can have a negative impact on this duty such as silica from refractory linings or the steam system blinding the catalyst surface and making it less active.

LTS: This is a duty for which poisoning is the primary cause of deactivation. Sulphur will readily form a copper sulphide (Cu_2S) which will migrate to form an even more stable zinc sulphide (ZnS) phase. This is illustrated in Equations 1 and 2, and also in Fig. 4.

$$2\ Cu + H_2S \rightarrow Cu_2S + H_2 \qquad (1)$$

$$ZnO + H_2S \rightarrow ZnS + H_2O \qquad (2)$$

Chloride will also readily form copper chloride and zinc chloride phases. The deactivation effect is even more dramatic due to the thermal stability of the chloride phases. These will sublime at typical operating conditions and will rapidly sinter the active copper. An example of this is shown in Fig. 5 which illustrates a large copper crystallite on the surface of a chloride poisoned LTS catalyst.

Methanation: Due to the susceptibility of other catalysts in the process it is unlikely that poisons will migrate this far in the flowsheet. If they do then rapid poisoning of the nickel catalyst would take place. Since this material is well protected by the upstream materials, lives in this duty are often 15 – 25 years. These long lives are possible providing there is no process upsets resulting in carry-over from an upstream liquid CO removal system onto the catalyst surface.

Table 2: **Common causes for catalyst reaching end of life**

Catalyst type	Deactivation mechanism	Primary cause(s)
Pre-reforming	Poisoning	Sulphur & chloride Process upset
Primary reforming	Poisoning Carbon formation	Sulphur & chloride Low S:C ratio Process upset
Secondary reforming	Fouling	Operating conditions process upset
HTS	Sintering Fouling Poisoning	Operating conditions WHB leaks Process upset
LTS	Poisoning Fouling Sintering	Sulphur & chloride operating conditions process upset
Methanation	Poisoning Fouling	Process upset
NH_3 synthesis	Poisoning	COx Other oxygenates

Source: Johnson Matthey

Johnson Matthey purification solutions

Many plants today are operating in areas where gas imports are increasing. Impurity levels in these plants are more likely to fluctuate, especially where there is more than one supplier to ensure energy security. As the gas blend changes operators may see changes in the composition, but they are still seeking to maximise the efficiency and/ or output of their assets.

Mercury removal

Johnson Matthey is the market leader in high-performance, dependable mercury removal systems, with a history of protecting cold boxes in the gas processing and LNG industries stretching back 25 years. Many of the leading oil and gas companies rely on PURASPEC$_{JM}$™, particularly for the most arduous and/or critical applications.

Sulphur removal

For many years a combination of a hydrodesulphurization (HDS) catalyst and sulphur absorbent (ZnO) was considered to

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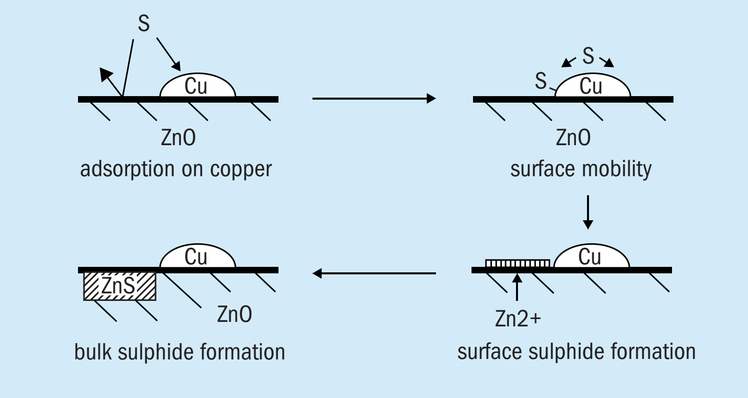
Fig 4: **Absorption mechanism of sulphur into LTS catalyst**



Source: Johnson Matthey

Fig 5: **SEM showing sinter copper crystal on surface of a chloride poisoned LTS catalyst**

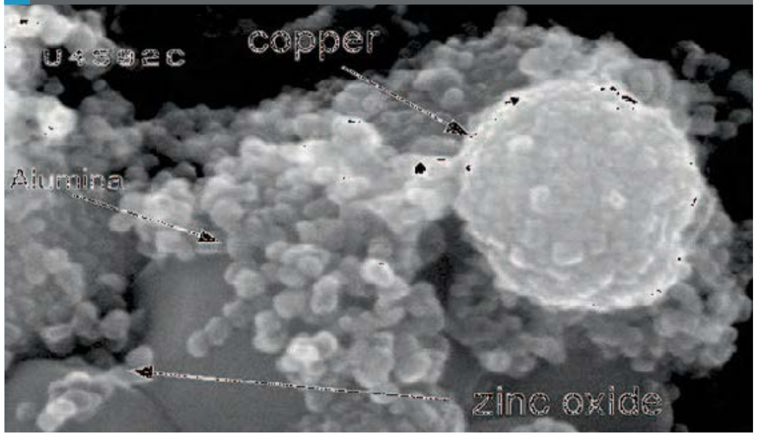
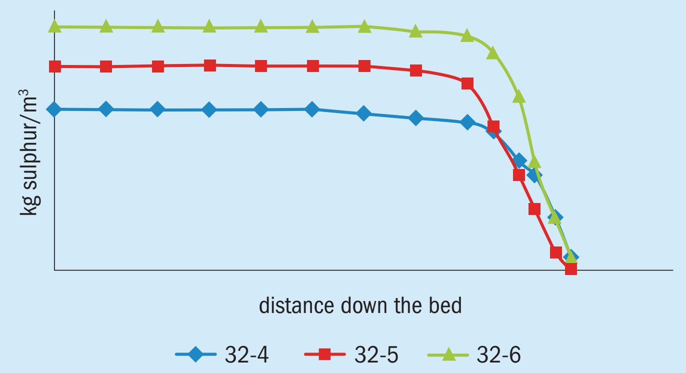


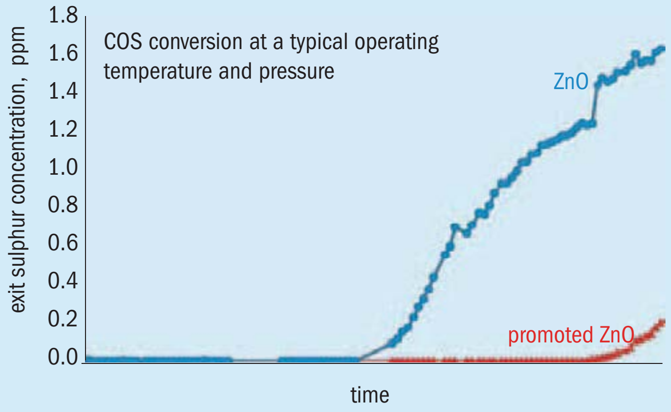
PHOTO: JOHNSON MATTHEY

Fig 6: **Increased sulphur capacity of KATALCO$_{JM}$ 32-6**



Source: Johnson Matthey

Fig 7: **Conversion activity of KATALCO$_{JM}$ 32-6**



Source: Johnson Matthey

be the optimal purification solution. Today this may no longer be the case and the optimum solution should be considered on a case by case basis.

The main considerations in selecting the optimum purification system are the type and level of impurity to be removed.

Unlike other suppliers, Johnson Matthey granulates its absorbent materials so the optimal pore structure is provided to realizs the maximum conversion of ZnO to ZnS. KATALCO$_{JM}^{TM}$ 32-6 is a promoted granulated ZnO material. This latest product builds on the reliability that KATALCO$_{JM}$ 32-4 and KATALCO$_{JM}$ 32-5 have demonstrated since the 1960s and 1990s respectively. As with these well proven products, there is no risk of CO_2 or S release on start-up with KATALCO$_{JM}$ 32-6 due to the high quality of raw material used in the production process. There are also no bespoke reduction requirements making its use a simple 'fit and forget 'solution.

KATALCO$_{JM}$ 32-6 provides two major advantages:

Elevated sulphur capacity: The sulphur capacity is increased by up to 30% as the mass of reactive zinc oxide per m³ is increased. The formulation increases the intrinsic rate of reaction and overcomes the impact of the diffusion limitations that may be caused by an increased mass of zinc oxide per m³ in the product. As a result of this there is still a very sharp profile and short mass transfer zone (Fig. 6).

Improved conversion activity: Enabling the conversion of simple organic sulphurs, such as carbonyl sulphide. This functionality is combined with hydrogen sulphide absorption and therefore reduces the level of recombination reactions that are possible. The overall effectiveness is therefore better than having a sequential hydrodesulphurisation (HDS) catalyst and sulphur absorbent (ZnO) system (Fig. 7).

The benefits of installing KATALCO$_{JM}$ 32-6 can deliver value in two ways:

- extend the sulphur capacity and campaign length of the purification system by more than 20% without any equipment modifications;
- reduce the pressure drop across the sulphur absorbent by 20% as a result of installing a reduced volume to achieve the same campaign length.

KATALCO$_{JM}$ 32-6 also provides improved protection compared to an un-promoted sulphur absorbent.

Johnson Matthey also offers a '3-in-1' purification solution, KATALCO$_{JM}$ 33-1, which has additional functionality over KATALCO$_{JM}$ 32-6.

KATALCO$_{JM}$ 33-1 is a dual promoted material that provides an ultra-purification function capability as well as organic sulphur and hydrogen sulphide removal. Its ultra-purification capability reduces sulphur slip to ppb levels but, unlike some ultra-purification products, it does not require a separate reduction step during start-up.

KATALCO$_{JM}$ 33-1 can be used to replace the hydrodesulphurisation material and some or all of the sulphur absorbent. The optimum installation will depend on the level of organic sulphur, the total sulphur in the feedstock and the required campaign length. An alternative option is to remove the hydrodesulphurisation bed completely and take a pressure drop saving.

Using the KATALCO$_{JM}$ purification product range can achieve a system with either a higher sulphur capacity (longer life) or

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Table 3: **Showing the benefits of using the extended KATALCO$_{JM}$ purification range of materials**

Flowsheet and material selections	Relative values S capacity	 Pressure drop
Original flowsheet (HDS over lead lag ZnO)	1.0	1.0
High S capacity flowsheet (33-1 over 32-6)	1.7	1.0
Low pressure drop flowsheet (33-1 lead lag)	1.0	0.7

Source: Johnson Matthey

lower pressure-drop. The relative benefits are illustrated in Table 3.

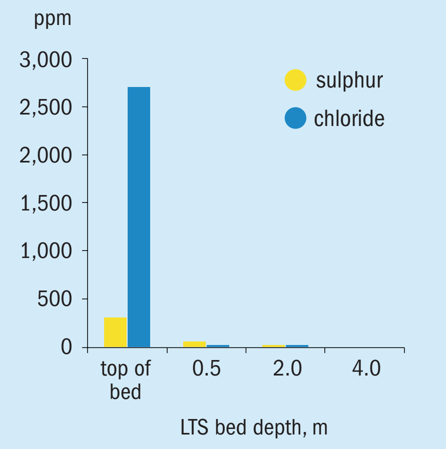
Many plants have a three vessel arrangement with hydrodesulphurisation upstream of a lead-lag arrangement for the sulphur removal absorbent. KATALCO$_{JM}$ materials can still provide the same reliable protection that has been demonstrated over 50 years if no change is required.

Cleaning downstream of the purification system

As mentioned earlier, sulphur and chloride can enter the flowsheet downstream of the feed purification system via the air intake to the secondary. In order to prevent significant poisoning of the LTS catalyst as a result of this, Johnson Matthey has developed a range of self-guarding LTS catalysts.

KATALCO$_{JM}$ 83-series materials employ a guard mechanism that negates the need to install an inactive guard material on top of the catalyst. The catalyst bed itself provides an effective and rapid trapping mechanism. This is a result of the manufacturing process that brings together the copper, zinc, alumina and promoters in an optimized structure, capturing these poisons and preventing the sulphur and chloride from moving into the bulk of the bed.

Fig 8: **S and Cl measured in discharged bed of LTS catalyst after 4 years life**



Source: Johnson Matthey

The sulphur is first trapped by the copper and is then able to migrate to react with the zinc to form a stable material. In cases where chlorides are known to be present a promoted material such as KATALCO$_{JM}$ 83-3X would typically be recommend enabling the formation of a stable chloride.

Where there are known to be elevated levels of poisons, a layer of smaller size KATALCO$_{JM}$ 83-series material can be used. This increases the mass of available material to trap poisons at the top of the bed and increase the surface area available. Figure 8 shows the effectiveness of this solution in a European ammonia plant.

Topsoe feed purification

Topsoe is a leading supplier of both hydrogenation catalyst and desulphurisation absorbent with feed purification products operating in more than 500 units. In addition, Topsoe can provide operating advice and expert technical service, including:

- plant assessment and optimisation;
- troubleshooting;
- sampling and analysis;
- internal visual inspection;
- scheduling of feed purification replacements.

Topsoe TK hydrogenation catalysts

In order to prevent catalyst poisoning with organic sulphur compounds it is important that the hydrogenation (HDS) catalyst has a high activity. The high activity of Topsoe's TK catalysts ensures efficient conversion of all organic sulphur and chlorine compounds, thereby providing maximum protection of downstream catalysts.

Measuring the operational performance of a HDS catalyst directly, e.g. using an on-line sulphur analyser or by adhoc lab analysis for the detection of organic sulphur, can be difficult, as organic sulphur concentrations below detection limits are harmful to some of the catalysts downstream of the HDS.

Examining the deactivation rate of the downstream catalysts, such as the pre-reforming catalyst, can give information about the performance of the hydrogenation catalysts and about the zinc oxide absorbents. Deactivation of prereformer catalysts is generally due to accidental exposure to sulphur. This can occur as a result of an ineffective or exhausted hydrogenation catalyst that does not fully convert organic sulphur to inorganic sulphur for removal by the absorbent, or as a result of an ineffective or exhausted absorbent.

To generate a deactivation plot, periodical Z_{90} values (the height of the prereformer catalyst bed at which 90% of the total temperature difference has been attained) are plotted against prereformer catalyst age. During stable operation, deactivation plots will roughly display a straight line. The slope of the line indicates the deactivation rate. Operating with a highly effective hydrogenation catalyst minimises the deactivation rate and results in significant savings in prereformer catalyst replacement costs.

The deactivation plots illustrated in Fig. 9 have been calculated for three separate plants, all using Topsoe TK hydrogenation catalysts. These examples cover a wide range of sulphur levels in the feed streams, from <1 ppmw to 25 ppmw, as well as different feed types and temperature ranges. The data shown reflects a continuously operating TK charge, i.e., the TK catalyst is not replaced during the time period shown.

The Z_{90} plot for Plant 1 (using TK-261) shows how the prereforming catalyst retains its activity over the first five years of operation.

The Z_{90} plot for Plant 2 (using TK-250) shows how the prereforming catalyst only deactivates very slowly during the first eight years of operation.

The Z_{90} plot for Plant 3 (using TK-250) shows how the prereforming catalyst only deactivates very slowly during the first seven years of operation.

These deactivation plots clearly show a very stable, long-term operation with highly effective hydrogenation catalysts. Adiabatic prereformer (APR) catalyst charges with the optimal protection of TK-250 or

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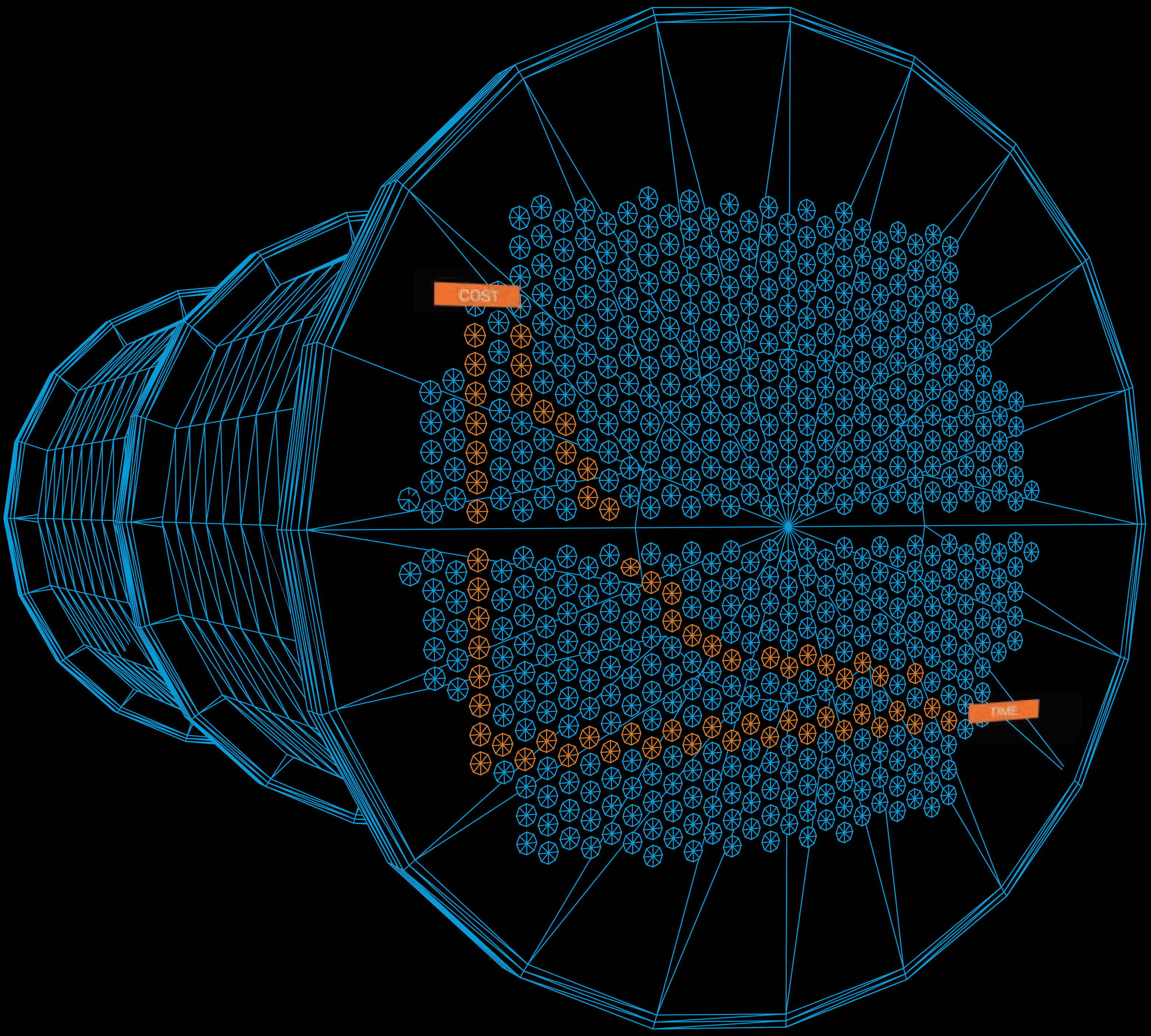

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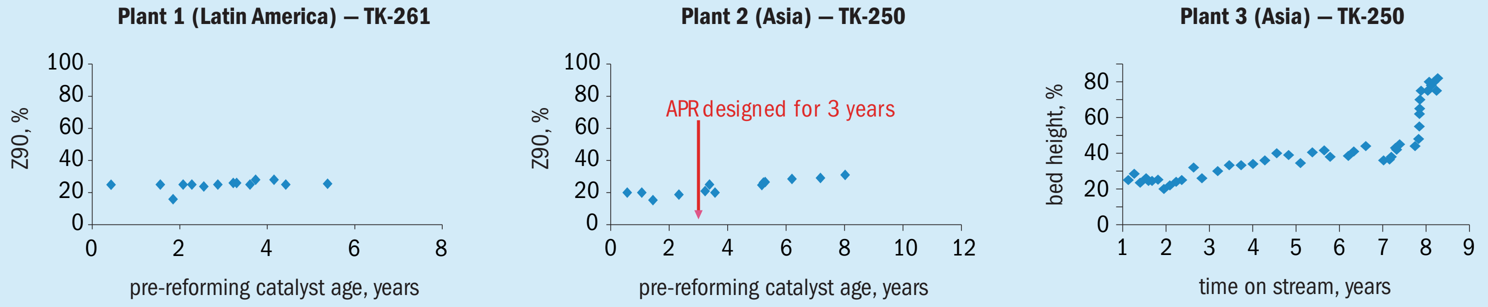
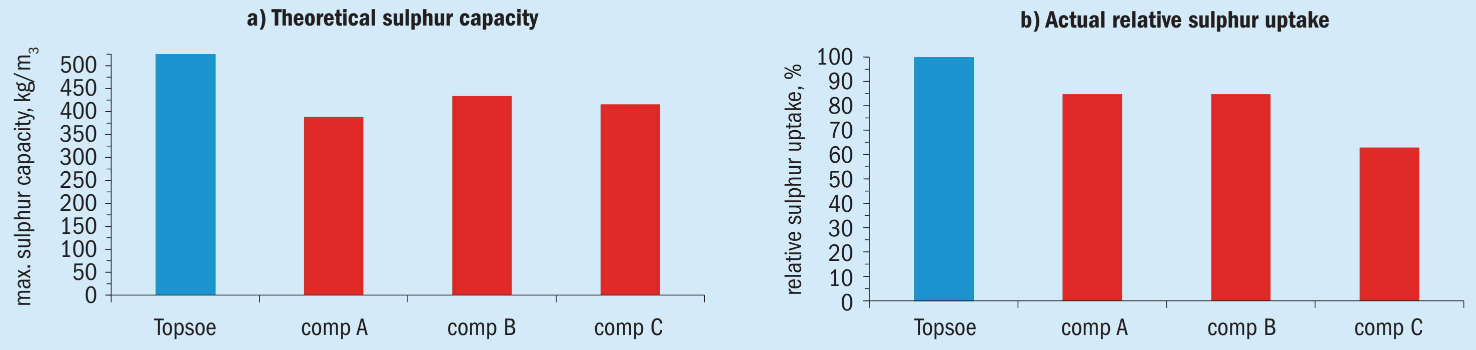
Fig 9: **Long lifetimes and protection of downstream catalysts**



Source: Topsoe

Fig 10: **Performance of Topsoe zinc oxide sulphur absorbents**



Source: Topsoe

TK-261 have a much longer lifetime, resulting in substantial catalyst and operational savings.

The plot from Plant 3 in Fig. 9 also illustrates the potential cost of a poorly functioning HDS catalyst. The 40% progression in APR catalyst deactivation seen around the seven year mark occurred over only 30 days. In reality, an operation error resulted in the complete bypass of the HDS reactor, passing organic sulphur to the APR catalyst. Z_{90} plots are generated manually from APR thermocouple data, and unless Z90 plots are updated regularly on-site, this deactivation could be easily missed.

Optimised shape for low pressure drop

Topsoe's hydrogenation catalyst TK-250 has an optimised ring shape to protect against increasing pressure drop caused by dust or other contaminants present in the feed. Under such conditions, the effective catalyst lifetime can be significantly increased. The large void fraction distributes particulate build-up across a larger volume than cylindrical or lobed extrudates.

TK-261 and HTZ-51

Topsoe's latest generation feed purification products, the hydrogenation catalyst TK-261 and the sulphur absorbent HTZ-51 have unique features that provide for more efficient loading schemes of the feed purification section:

- TK-261 offers a significantly higher activity than its predecessors, which opens up the possibility for reduction of catalyst volume and increase of lifetime.
- HTZ-51 offers not only a higher sulphur pickup relative to the previous generation, but also possesses hydrogenation activity, providing the possibility to reduce catalyst volume, increase lifetime and better protection of downstream catalysts.

Topsoe's ZnO absorbents

The risk of severe financial loss due to sulphur poisoning on downstream catalysts underscores the importance of full and efficient desulphurisation of feedstocks. The theoretical sulphur capacity of zinc oxide absorbents is dependent on the zinc oxide purity and bulk density of the absorbent. Accordingly, high levels of purity and density of Topsoe HTZ absorbents result in a superior sulphur capacity (see Fig. 10a).

The superior sulphur capacity of Topsoe absorbents is supported by their proven performance. Topsoe has optimised the pore system to give HTZ absorbents the high pore volume and surface area needed for maximum removal of hydrogen sulphide. The result is a higher sulphur uptake in practice (see Fig. 10b).

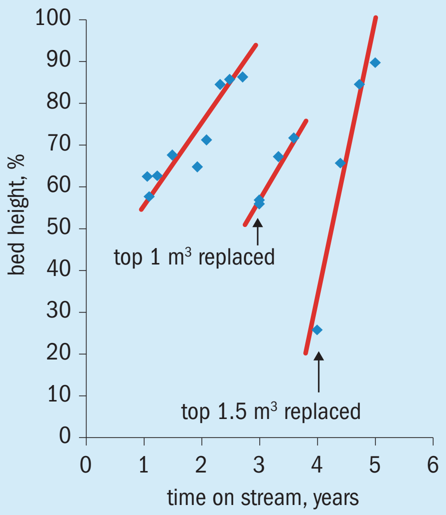
The overall benefits of Topsoe HTZ absorbents include less risk of downstream catalyst sulphur poisoning and significant savings in absorbent replacement costs.

Importance of high quality ZnO raw materials

Impurities in zinc oxide should be minimised in order to prevent poisoning of downstream catalysts and fouling of equipment. Topsoe HTZ absorbents have impurity levels far below those measured in competitor products.

Chlorine impurities in the absorbents can be released during operation and can consequently poison downstream catalysts. Chlorine can also lead to corrosion and possible fouling of downstream equipment.

Fig 11: **Deactivation rates of prereforming catalyst in an ammonia plant**



Source: Topsoe

In addition, chlorine can poison the LTS catalyst in two ways. Firstly, it makes the copper crystal sinter, whereby the sulphur capacity and activity is lowered. Secondly, it blocks the surface so if only chlorine poisoning took place it would also develop a deactivated catalyst zone.

Normally the Cl poisoning will result in the top part of the catalyst having less than expected pickup of sulphur, thereby causing the deactivated catalyst zone to develop somewhat faster.

In really severe Cl poisoning cases, chlorine will cause such severe sintering that the catalyst loses strength and may collapse resulting in increasing pressure drop.

Cadmium and lead impurities in the absorbents can also be released during operation, which can lead to fouling of downstream equipment. Both metals can also poison downstream catalysts and are poisonous.

In some countries mercury impurities are considered hazardous which makes it very difficult and very expensive to dispose of any material with high Hg levels.

Scheduling of feed purification replacements

Getting the timing right of replacing feed purification products is important. Careful sampling and analysis is required in the feed purification section, especially when operating beyond design lifetime. The following examples show some of the consequences of not getting it right.

Figure 11 shows a Z_{90} plot for the deactivation rate of a prereforming catalyst in an ammonia plant. Note that there were also issues with incorrect thermocouple placement in the APR catalyst, making it difficult to observe deactivation while it was occurring.

The three red lines represent the rate of APR catalyst deactivation. The first two have same the same slope, i.e. the same rate of sintering/poisoning but the third line has a much higher deactivation rate. The top layer of the APR catalyst was skimmed and replaced twice. There was a sudden increase in deactivation rate seen after four years on stream.

After laboratory analysis of the feed purification catalysts, it was determined that the ZnO absorbents were fully saturated with sulphur, and the APR catalyst was being poisoned by breakthrough H_2S.

Figure 12 shows the inlet, outlet and accumulated sulphur in a hydrogen plant. Two ZnO beds were installed in lead/lag with Topsoe HTZ catalyst in 2011. Sulphur breakthrough occurred from the lead reactor in 2013, 23 months after SOR. The absorbent in the lead reactor was changed to a competitor product in mid-2014. After only eight months of operation under similar conditions, sulphur breakthrough occurred again, which eventually led to the complete deactivation of the APR catalyst in mid-August 2015.

Further analysis showed that shortly after the complete deactivation of the upstream APR catalyst, the pressure drop across the tubular reformer jumped over two subsequent days to previously unseen levels (3 and 6 kg/cm² from approx. average of 1.5 kg/cm²). When APR catalyst deactivates, heavy hydrocarbons can slip to the tubular reformer, where cracking occurs at high inlet T. The carbon product of this cracking leads to rapid increase in pressure drop. ■

Reference

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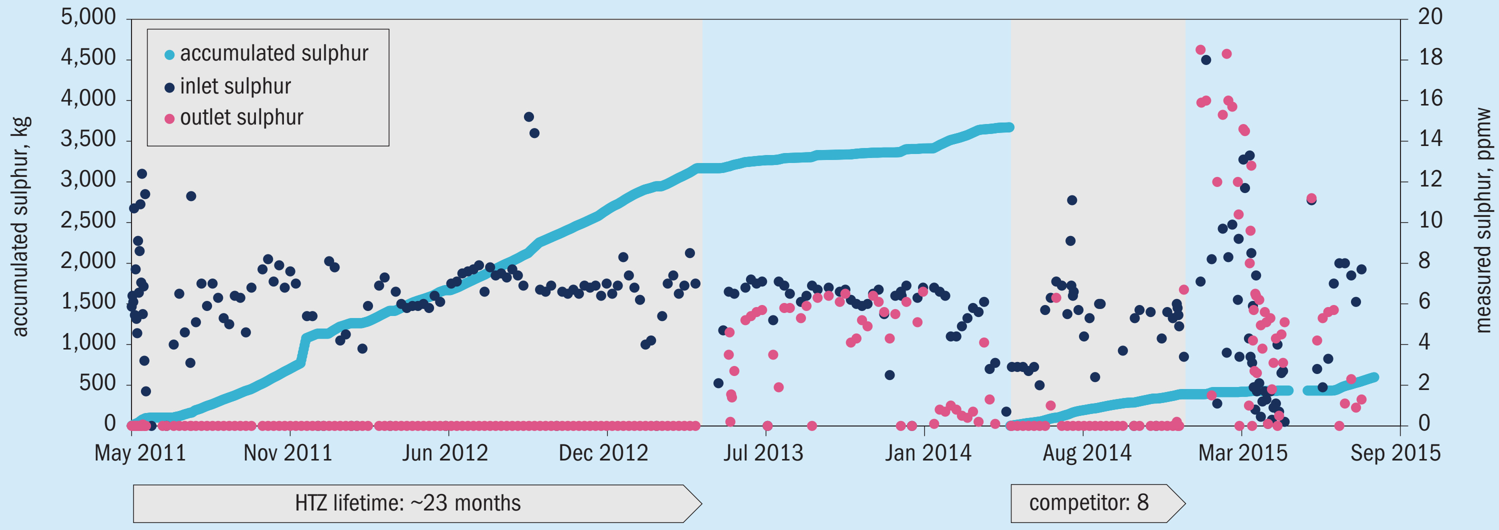
2. Wilhelm S.M.: "Risk analysis for operation of aluminium Heat Exchangers contaminated by mercury"' AICHE, Louisiana (2008).

Fig 12: **Sulphur breakthrough in a hydrogen plant**



Source: Topsoe

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Size matters

Many urea experts and practitioners know that numerous factors affect fertilizer product quality. They include finishing technology, geographical location (climate) as well as how the final product is stored and transported. In this article, **B. George van Bommel** of BioTorTech and UreaKnowHow highlights one of the most easily overlooked but critical aspect determining fertilizer product quality – the end user.

Table 1: **Typical specification sheet: urea granules**

Chemical properties	**Typical**	**Range**	**Physical properties**	**Typical**	**Range**
Total nitrogen (N) content, wt-% N	46.2	46.0-46.6	size guide number (SGN)	270	240-320
Biuret content, wt-%	1.0	0.85-1.5	uniformity index (UI), %	52	50-55
Moisture content, wt-% H_2O	0.25	0.1-0.4	bulk density (loose), kg/m^3	720	700-740
Formaldehyde (HCHO), wt-% F	0.4	0.2-0.6	bulk density (tapped), kg/m^3	810	800-860
Free NH_3, ppm	75	50-150	angle of repose, degree	30	38-32
pH in water solution, 10% wt/wt	7.8	7-9	crushing strength, kgf (N)	3.5	3-4

Table 2: **Typical specification sheet: urea prills**

Chemical properties	**Typical**	**Range**	**Physical properties**	**Typical**	**Range**
Total nitrogen (N) content, wt-% N	46.1	46.0-46.6	size guide number (SGN)	190	140-240
Biuret content, wt-%	0.8	0.85-1.5	uniformity index (UI), %	54	50-55
Moisture content, wt-% H_2O	0.35	0.1-0.4	bulk density (loose), kg/m^3	740	730-760
Formaldehyde (HCHO), wt-% F	0.2	0.1-0.3	bulk density (tapped), kg/m^3	780	760-860
Free NH_3, ppm	125	50-150	angle of repose, degree	28	26-30
pH in water solution, 10% wt/wt	7.8	7-9	crushing strength, kgf (N)	1.2	1-3

Many readers will be familiar with the standard specification sheets for urea granules and urea prills shown in Tables 1 and 2 respectively. When comparing the two, a number of physical properties differ due to the different finishing technologies applied.

The granulated product has a much bigger average diameter, 2.7 mm versus 1.9 mm for a prill (d50). The difference in the product is also visible to the naked eye (Fig. 1)[1,2].

Fig. 1: *Typical prill (left) and typical urea granule size (right).*

In the fertilizer industry, product size[2] is commonly expressed in SGN (size guide number), which is the average particle diameter (d50) multiplied by 100.

The thesis of this article is that size does matter in terms of quality and storage as evidenced when exploring the important impact of different particle size at production sites in locations with (semi)-tropical conditions and high relative humidity.

Like most fertilizer products, urea particles can also adsorb moisture from the environmental atmospheric air conditions. Urea particles are susceptible to moisture absorption, decomposition, and caking even at moisture contents as low as 0.25 wt-% during transportation and storage processes.

In fact there is a strong quality relation between the urea product's total surface, number of contact points when exposed to high humidity air and product load during temperature swings. This is not unsurprising and can be demonstrated using easy

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Table 3: **Identification of different fertilizer particles in 1 m^3 volume**

Balls/ spheres	Diameter, mm	Volume, mm^3	Area, mm^2	Perimeter, mm	No. of particles per cu side	No. of particles per m^3	No. of possible bonds per m^3	Total area of all particles, m^2
Ping-pong	40.0	33,510	5.027	126	25	15,625	187,500	8
Prill	1.9	3.59	11.34	5.97	526	145,793,847	1,749,526,170	165
Granule	2.7	10.31	22.90	8.48	370	50,805,263	609,663,161	116
NPK granule	2.2	5.58	15.21	6.91	455	93,914,350	1,126,972,201	143

Fig 2: **Stacking and filling patterns**

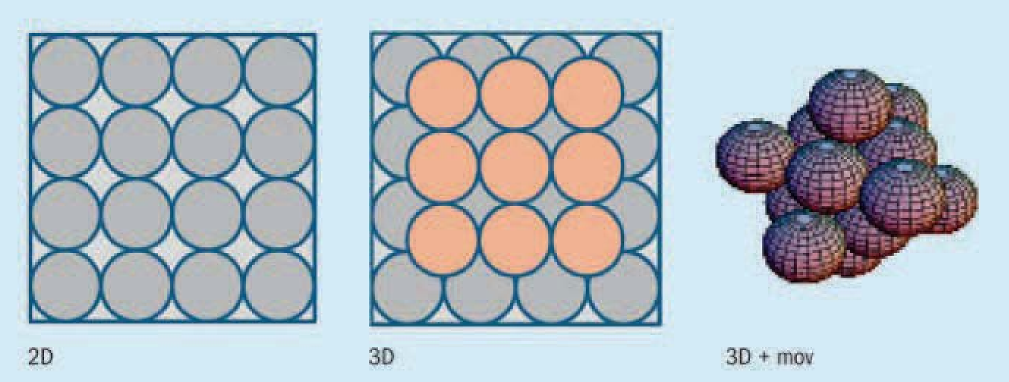


PHOTO: UREAKNOWHOW

Fig. 3: *Urea lumps (left) and severely caked storage pile (right).*

to understand mathematical examples in Table 3.

In the following simplified example, with "one size" 40 mm diameter ping-pong balls, you can stack 25 balls per side in a 1 x 1 x 1 metre cube (length, width, height of 1 metre = 1000 mm/40 = 25), thus you can fit 25 x 25 x 25 = 15,625 balls into the cube with square close packing layers[3].

Each ball touches 12 other balls, as illustrated in Fig. 2, where the grey balls base, are stacked with the yellow balls. If another layer is added, the total amount of contact points is 12 x 15.625 = 187,500 in this 1 m^3 volume and all balls have a total surface area of 8 m^2.

The total surface area on which interaction can take place is an important factor with regard to the exchange of mass force, moisture or absorption. Even in a typical 50 kg bag urea (approx. 70 litres) of prilled product, the total particle surface is 12 m^2, with some 10 million contact points, thus exponentially increasing the possible caking bonds.

Take, for example, 1 m^3 of prills (of 1.9 mm) comprising about 145 million individual prilled particles, with a joint internal surface of some 165 m^2 (Table 3). The amount of inter granular contact points, where the caking bridges start for the 145 million prills is about 1,750 million contact points, versus 610 million for urea granules (50 million individual granules, size 2.7 mm).

It can therefore be concluded that prills have roughly 2.8 times more contact points, and 40% more surface area than granules!

It is very clear that large particles are less sensitive to caking or pressure contact bonding, because they have significantly less contact points. The highest theoretical cubic close packing of equal spheres is 74 % of particles, leaving 26% volume for void (empty) area for air.

This void volume forms an additional problem because this 26% volume can be filled with "moist" air, or in bad cases with ultra-fine, broken and dusty particles. The dusty particles in these void areas can dramatically increase the amount of contact area. This creates a perfect environment for further caking resulting in big lumps and solid piles of product (Fig. 3). It is therefore critical to ensure not only the right size but consistent uniformity of the urea product and to eliminate the risk of dust in the void spaces.

Now that we have covered the impact of size and the risk of dust particles in void spaces let us focus on the additional impact of climatatic conditions.

In tropical climate conditions with temperatures of 30°C and 80% relative humidity (RH), we know that urea can absorb within 72 hours on the exposed pile surface a total of 3,5 kg/m^2 of water, whilst the air only holds some 21 g/m^3. These conditions can therefore create a crust of caked product and lumps.

Bearing in mind that the dew point of the "moist" air is already at 26°C, if the temperature drops from 30°C to 26°C during the night, the "condensing" air will transfer all of its moisture to the warehouse pile. Therefore, reducing fluctuations in the warehouse air temperature and high RH helps to reduce crusting, caking and lumps formation. This is also valid for bulk transshipments when transporting the product to customers.

However, fertilizer products have a certain range of product distribution, depending on accurate sieving, and it is of paramount importance that fines, dust and broken products are removed to prevent void (air) areas from being filled with these off-spec products, thereby increasing the number of contact points and thus cake bridges. These cake bridges need to be eliminated to ensure product consistency and quality.

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PHOTO: CUSTOMER OF ROYAL HASKONING DHV

Fig. 4: *Near miss of a truck coming out of dusty cloud with an approaching pay loader at NPK plant in Brazil, South America.*

Larger granules are also stronger than prills (see crushing strength 3.5 versus 1.5 kgf or kg/particle), and cannot be deformed or plasticised (creating more contact points) so easily by stacking or compacting pressure.

The moisture content in the final product is also a very important quality parameter. A high moisture content is an indication of many capillaries that are still filled with moisture. Consequently, during the drying-evaporation-storage of the prill (or granule) additional needles (fine recrystallised salts) are formed which become a dust nuisance during handling actions such as loading and bagging and bulk transport transfers.

Fig. 4 shows that high dust is also a safety factor; appropriate dust removal systems are necessary to prevent incidents and health inhalation problems which can contribute to respiratory illnesses.

After bulk storage, the static pressure when bagging the product for the customer is important. Static pressure, which can occur in bagged and stacked product, also promotes bridging behaviour resulting in caking. In Fig. 5 the result of static pressure promoting the capillary behaviour can be seen. The porous channels of the granule force out the internal moisture, creating a liquid meniscus of new bridging-caking-crystals on the surface. The static pressure of bagged and stacked fertilizer products therefore plays an important role in caking. The IFDC has developed a method S-106 (small bag method) in which 0.3 kg/cm^2 pressure is applied to the bag equivalent to the pressure applied to a bag at the bottom bag of a 20 bag high stack (Fig. 6). The results of this pressure can then be assessed after one, three and six months by checking the caked lumps and hardness in the bag.

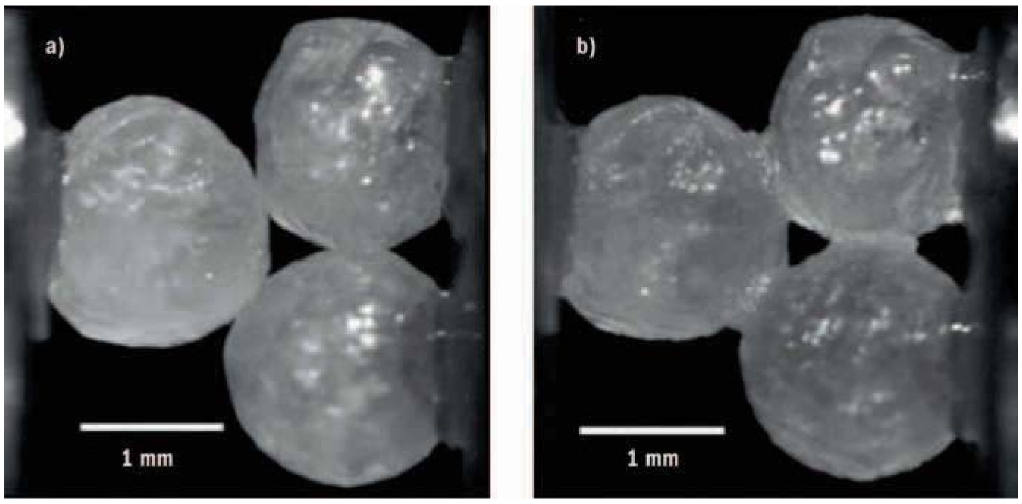

PHOTO: THE FACHSCHULE TRIER, GERMANY

Fig. 5: *Three-particle system of urea prills before (a) and after (b) solid bridge formation*

A promising, faster "company internal" caking tendency method was witnessed at an Asian NPK facility, where 500 ml product sample, was put in a closed bag within a metal cylinder (6 cm diameter and 18 cm length) under a pressure weight of 3 kg/cm^2, for 24 hrs (see Fig. 7).

To assess the caking tendency the two metal cylinder shells were removed carefully, the remaining fertilizer particles

Fig 6: **Storage rack to measure caking tendency of fertilizer by small bag method**

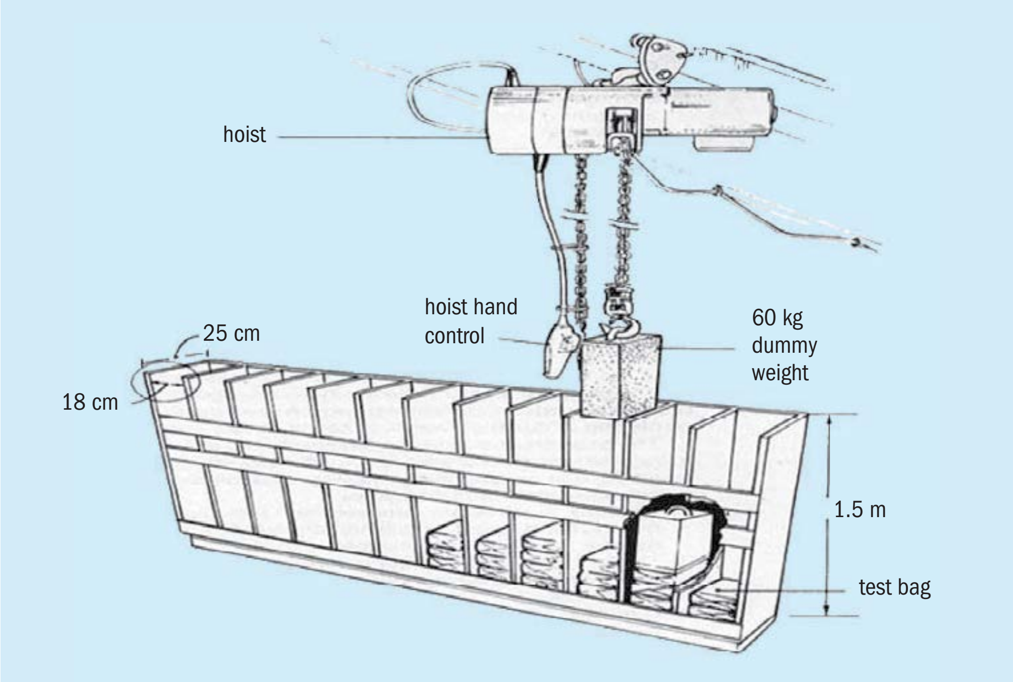

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PHOTO: CUSTOMER OF BIOTORTECH

Fig. 7: *Company internal caking tendency method at an Asian NPK facility.*

cylinder, was put under 1 kg weight. If the tested product has high caking (9-10) the fertilizer cylinder does not collapse, and remains intact. If the product cylinder collapses with the weight medium caking (5-6) was noted. If the cylinder collapses with no weight on it, a very low caking tendency (1-2) is expected.

The size of prills or granules of other common fertilizers (MAP, DAP, MOP, SOP, AN, AS, NPK's etc) are also paramount.

When (steam-physical) drum granulated NPK (17-17-17) is compared to the multi raw material input with all the different chemical properties, the relative end product high moisture of 0.9 wt-% is a very critical factor, as are the size and number of contact points (see Table 4).

Aside from the different chemical composition behaviour (e.g. different salt-out temperatures, moisture absorption sensitivity) the NPK has about 1,121 million contact points per 1 m^3 (see Table 3).

The high number of different NPK chemical formulations, SGNs and UIs make NPKs even more sensitive or susceptible to caking, because they have a lower critical relative humidity (CRH) compared to urea prills-granules. It can therefore be concluded that size does indeed matter in ensuring consistent product quality, as do storage and climate conditions.

Tropical conditions also have a large negative impact on the raw materials applied in the high urea NPK product formulations (Fig. 8). The relative humidity (rainy season) and temperature (day-night) fluctuations of various NPK formulations often surpass the critical relative humidity threshold, initiating water absorption by the product around 50-55% RH.

Bear in mind that if the same 1 m^3 is now filled with this urea based NPK granules, the surface area is capable of absorbing 14.8 % of water in weight after five hours at 25°C and 70% RH as indicated by research figures from Yara (see Fig. 9).

Therefore, compared with urea under the same tropical conditions of 30°C and 80% relative humidity, we know NPK 17-17-17 can absorb some 5,8 kg/m^2 of water (almost twice as a urea granule!) on the exposed surface within 72 hours

Fig. 10 shows a typical pile of high urea NPK after 24 hours in a NPK plant in Asia. A surface crust formation about 5 cm thick and discoloration (darker granules) is clearly visible. This is clear evidence of water absorption evidence (after only 24 hours), even though in this case the NPK (21-7-18) product pile was covered with a PE blanket as a precautionary measure.

Fig. 11 shows the results of spills and high traffic in a fertilizer plant in the tropics with high relative humidity and temperature fluctuations that has created a lot of mush as a result of the water absorbed and the hygroscopic properties of the fertilizers.

Each production location (and client) is unique with regards to the production process and climatic conditions. BioTorTech clients

Table 4: **Typical specification sheet: NPK 17-17-17 granulated**

Chemical properties	Typical	Range	Physical properties	Typical	Range
Total nitrogen (N) content, wt-% N	17.0	16.5-17.5	size guide number (SGN)	220	140-240
Total phosphate (P) content, wt-% P_2O_5	17.0	16.5-17.5	uniformity index (UI), %	52	50-55
Total potash (PK) content, wt-% K_2O	17.0	16.5-17.5	bulk density (loose), kg/m^3	800	760-840
Total sulphur (S) content, wt-% S	4.7	4-5	bulk density (tapped), kg/m^3	833	800-860
Moisture content, wt-% H_2O	0.9	0.8-1.5	angle of repose, deg	29	27-31
pH in water solution, 10% wt/wt	7.8	7-9	crushing strength, kgf (N)	3.1	2-5

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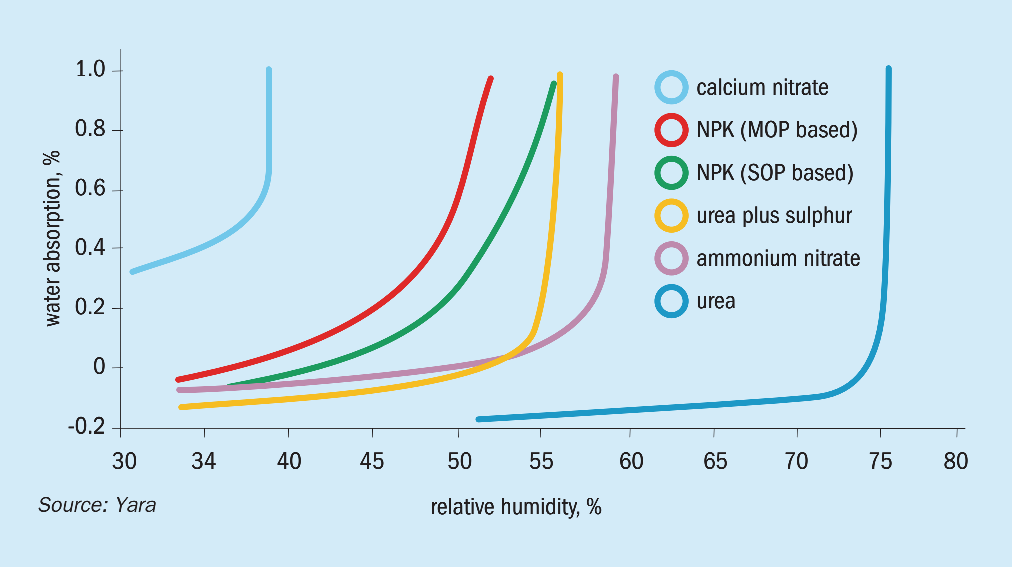
Fig 8: **Critical relative humidity (CRH) of various fertilizers at 25°C**



Source: *Yara*

receive their own detailed report regarding the quick (or hard) wins regarding the production and storage process. Including which ones can have the biggest economic impact for the plant operations and profitability.

Based on the case study, the following non-exhaustive list of general recommendations can be applied to improve product quality:

- large granules, SGN > 250 and 95% between 2-4 mm;
- more uniform size of granules, prills, briquettes, pellets etc, maintain uniformity index > 55;
- low angle of repose <30 degrees, by more and better sieving;
- less Moisture, < 0.8 % moisture content in end product;
- low granule porosity <0.5%;
- hard strong granules, crush test > 3.0 kgf on a 3 mm diameter granule;
- less fines and dust in product, so no < 1mm product;
- allow only limited dT between ambient T and final product to storage pile;
- constant warehouse temperature, prevent moisture migration absorption;
- limit the amount of product formulations, impact and transfers;
- apply first in-first out warehouse principles;
- lower bag stacking of high potential caking products;
- good housekeeping, vacuum clean dusty areas. ■

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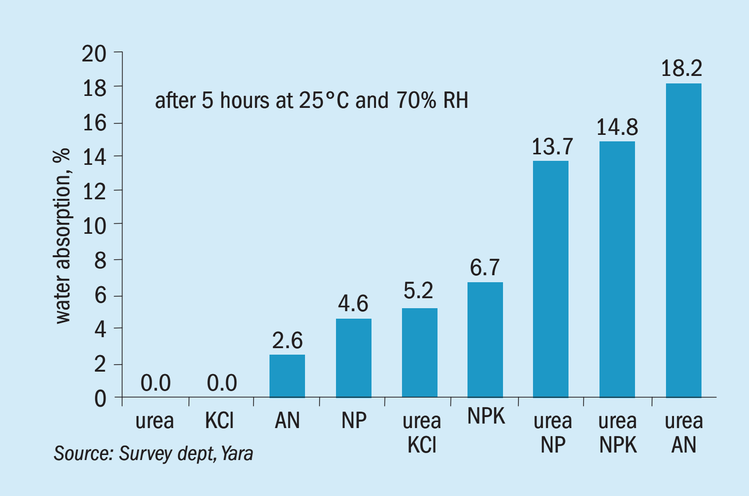
Fig 9: **Water absorption in blends**



Source: *Survey dept, Yara*

PHOTO: CUSTOMER OF BIOTORTECH

Fig. 10: *21-7-18 NPK with surface crust.*

PHOTO: CUSTOMER OF BIOTORTECH

Fig. 11: *Results of spills and high traffic in a fertilizer plant in the tropics.*

Product cooling for optimum quality

Faced with the challenge to increase its production of calcium ammonium nitrate to meet greater market demands and at the same time needing to address problems with product cooling leading to poor product quality, NAK Azot found a solution by replacing its rotary drum cooler with a Solex fertilizer cooler.

Novomoskovskiy Azot (NAK Azot) is one of Russia's largest chemical enterprises. It operates a highly upgraded facility and is the second largest ammonia producer and the largest nitrogen fertilizer producer in Russia as well as a major exporter in the world market.

In 2011, NAK Azot faced a significant market challenge. The international demand for high quality fertilizers was growing and the market was on its doorstep. The opportunity for the company's calcium ammonium nitrate product was significant but internally it needed to find a means to increase its production output at the same time as addressing product cooling practices threatening its quality.

The role of cooling

The requirements for product cooling are not new for the fertilizer industry. Quite simply, product temperature during storage is one of the critical quality parameters. It is generally recommended that fertilizers are cooled to within 10 to 20°C of ambient temperature to prevent moisture migration that can lead to caking and product degradation. Ammonium nitrate and calcium ammonium nitrate, as special cases, are susceptible to phase change; they must be cooled to a stable phase to prevent them from cycling between phases as ambient temperatures vary.

Cooling technologies

For the past 50 or more years, the fertilizer industry has used direct contact air coolers for final product cooling. Rotary drum coolers began as the industry norm, however in the nitrogen fertilizer industry, fluid bed coolers, thanks to their lower install capital cost, smaller footprint and lower maintenance costs, have generally taken their place. The operation of both rotary and fluid bed cooling technologies requires high volumes of air. They depend on large-scale fans for air movement and scrubbers or baghouses to meet ever more stringent emissions requirements. Additionally, they have significant footprints and can be difficult to accommodate and install as part of a plant retrofit.

When it reached the crux of its dilemma in 2011, NAK Azot was cooling its calcium ammonium nitrate after granulation in three parallel fluid bed coolers. The fluid bed coolers were employed to lower the post-granulation temperature of the product to between 65 and 75°C. The calcium ammonium nitrate was then moved into a rotary drum cooler for secondary cooling and then transported to bulk storage. It was held in bulk storage for three to four days until the temperature further decreased to its target range of between 35 and 40°C. It was an expensive and time-consuming process and NAK Azot was finding out the hard way that even with high-cost input it was producing low quality output.

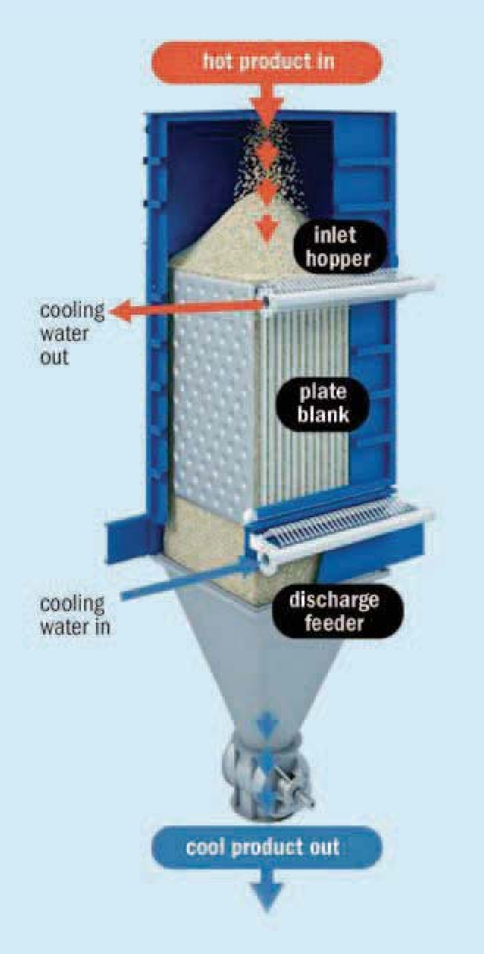
Due to the high temperature of the fertilizer going to storage, the product was highly susceptible to drawing in moisture and caking as it went through its final stage of cooling in the bulk storage. The caked product was then reclaimed using two Kratzer-Crane scrapers, which further degraded the product and led to variable grain-size distribution and a high percentage of fines. Despite its intensive efforts, NAK Azot was getting low-grade product and, consequently unhappy customers.

An alternative to incremental improvements

Unwilling to let its market potential and commitment to quality product pass by, NAK Azot started to explore options to mitigate its caking problem and achieve better quality control. Its first line of defense was to upgrade one of its existing cooling components, the rotary drum. It selected a replacement drum that allowed it to inject anti-caking additives into its product batches before sending them to bulk storage. For this incremental step forward, it was successful in reducing the amount and thickness of the caking. Unfortunately, NAK Azot was still faced with the ultimate barrier to market growth, it needed an effective, efficient and reliable means to produce more fertilizer. Within its current facility, this meant increasing its cooling capacity. NAK Azot realised that incremental investments would only ever yield incremental gains and decided it was time to seek out a game-changing solution with the reputation to change both its position and performance in the world market.

In 2012, NAK Azot engaged Solex Thermal Sciences and chose to implement a Solex fertilizer cooler, a proven product that promised to not only eliminate caking but dramatically enhance its overall product quality, production capacity, facility performance and environmental record. The Solex fertilizer cooler was already at work and demonstrating its value in nearly 150 fertilizer production facilities around the world. It was accepted as the technology of choice for new facilities, including the latest generation of urea facilities being constructed in the United States, and an exceptional option to retrofit mature operations.

Fig 1: **The Solex fertilizer cooler**



The Solex fertilizer cooler

Solex Thermal Science first introduced its revolutionary technology, indirect contact fertilizer cooling, to the market in the early 1990s. In a scenario not unlike NAK Azot's case, it was faced with the high costs and limitations of retrofitting an existing fertilizer facility with direct contact cooling technology. Rather than accept the available options, it developed a simple, elegant heat exchange technology based on indirect heat transfer that delivered and surpassed expectations on multiple industry challenges.

The Solex fertilizer cooler (see Fig. 1) combines the science of mass flow with the thermal efficiency of plate heat exchange design. In the cooler, bulk solids pass in mass flow through vertical banks of stainless steel plates. Cooling water flows through the plates to lower the temperature of the material by conduction. The water is circulated through the plates in counter-current flow for enhanced thermal efficiency. The indirect plate heat exchanger design means air is *not* used in the cooling process, a feature that reduces installation and operating costs as well as guarantees better environmental outcomes. A mass flow discharge device controls the product flow rate through the exchanger. The product moves slowly by gravity through the unit to create sufficient residence time to achieve the required cooling. A level control system ensures the fertilizer cooler operates at its optimum configuration.

Main advantages

Versatility

The Solex fertilizer cooler can be integrated into nearly any facility, new or mature, and used to cool full ranges of fertilizers from urea granules and prills to ammonium nitrate and CAN, NPKs, MAP, DAP, TSP and ammonium sulphate. Certain details are altered to allow for the differing properties of different products but the principles of operation remain the same.

Exceptionally small footprint

With its compact, vertical, modular design, it can be accommodated in even the most compact facility layouts. It is an excellent option for facilities that lack space but still want to increase production capacity either by replacing an existing cooler, as in the case of NAK Azot, or supplementing the existing cooling equipment. It is also ideal for facilities that want to gain all the advantages of modern technology but need to accommodate a retrofit within an existing layout.

Product quality

One of the keys to good product quality is correct temperature to storage. The Solex fertilizer cooler design is based on rigorous thermal modeling to guarantee precise discharge temperature control thus ensuring that the product temperature to storage is correct. Ammonium nitrate and calcium ammonium nitrate create special challenges due to their phase changes and associated heat loads, the Solex modelling accurately allows for these complex phenomena.

Cost savings

When it comes to installation, the cost of integrating a Solex cooling system is typically at least 30% lower than the cost of direct cooling technologies. Its operating costs deliver upwards of 90% annual energy savings – which, in a facility producing 3,600 t of fertilizer daily, can equate to cost savings of over $750,000 each year. Maintenance costs are also virtually eliminated; there are no moving parts and the system operates with a fraction of the components of direct cooling technologies.

Environment

With increasing pressure on fertilizer producers to achieve social license, minimise their reliance on carbon-based energy sources, and curb greenhouse gas emissions, the Solex fertilizer cooler hits multiple targets. It contributes locally by eliminating the risk of dust and odour emissions and ensuring compliance with ambient air quality objectives. It shows industry leadership by reducing power consumption – power that is typically derived from emissions-heavy, coal-fired power plants – by upwards of 90 per cent. It promotes sustainable development, addresses global concern surrounding rising greenhouse gas emissions, and avoids costly carbon levies, all additional benefits of a 90% reduction in energy use.

World-class granulated CAN

NAK Azot recognised the immense potential that could be achieved by replacing its rotary drum with a Solex fertilizer cooler. The equipment took less than five months to deliver and, in this one step, achieved unparalleled gains. NAK Azot was able to eliminate its reliance on bulk storage, thereby recouping the three to four day final cooling time period as well as the high costs and large physical footprint associated with it.

In place of the rotary drum and bulk storage cooling, Solex's technology accepted the granulated calcium ammonium nitrate directly from the fluid bed cooler. The fertilizer product entered the Solex fertilizer cooler at a temperature of between 65 and 70°C and left it at an optimum temperature of between 37 and 41°C. It was discharged from the cooler without breakage or fines and ready to be packed or shipped in bulk to the customer directly from the unit. Its shift from a highly variable three-stage cooling process to a gentle, reliable, non-contact, two-stage process, allowed it to produce calcium ammonium nitrate granules that keep form, resist caking, and hold their value during production, storage and transport.

The solution exceeded its expectations and NAK Azot's calcium ammonium nitrate is now among the best in Europe. With its investment in Solex's indirect cooling technology and other plant process changes, it is also one of the most efficient fertilizer operations in Europe. ■

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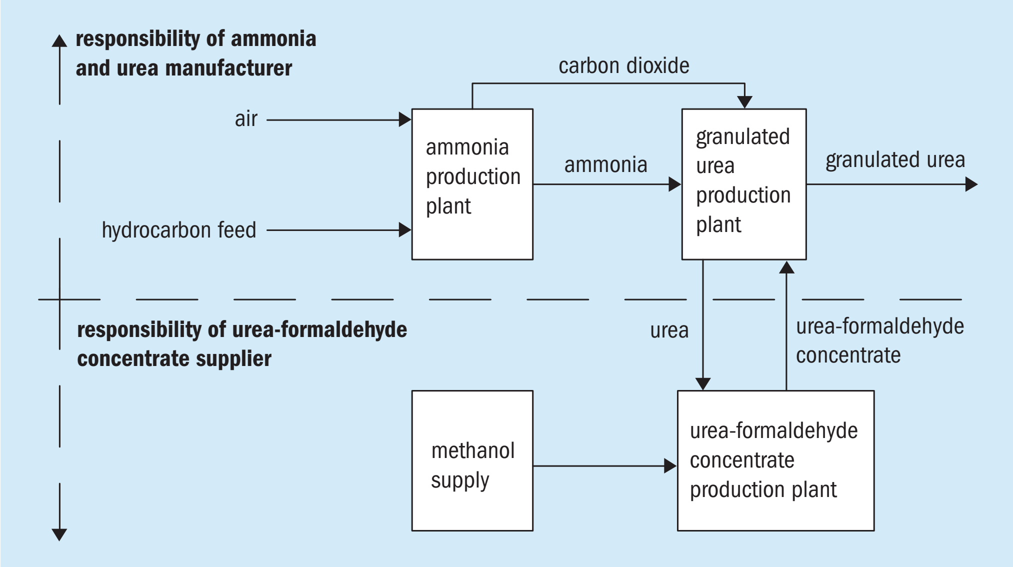
UFC manufacture for urea production

Johnson Matthey has combined its knowhow in ammonia, methanol and formaldehyde production to deliver an innovative new way of producing UFC-85 which will generate significant cost savings when compared to the alternative of purchasing UFC-85 from third parties. The integrated process, which is the subject of patent applications, is now offered commercially.

Fig 1: **Standard arrangement of technologies**



Formaldehyde in the form of UFC-85 is used as an additive in the manufacture of solid urea. Formaldehyde is synthesised from methanol which is normally purchased on the open market and is priced accordingly. In most cases, the quantity of UFC-85 that is required by any individual site is too small to justify investment in small scale methanol production and in a UFC-85 plant.

Johnson Matthey is a leader in catalyst and process technology development for the ammonia, methanol and formaldehyde industries. With a heritage in ammonia dating back to the first decade of the 20th Century, Johnson Matthey (JM) currently offers a range of high performance catalysts, leading edge technologies and diagnostic services to its customers.

Today's methanol industry is based almost entirely around the low-pressure technology and catalysts developed and commercialised by ICI in the late 1960s. Since the acquisition of this business, Johnson Matthey has continued to adapt and improve methanol technology.

Johnson Matthey's range of DAVY technologies offers design, licence and commissioning expertise. The combined skills and experience of catalysts and process design is ideally suited to the development of innovative syngas flowsheets.

Johnson Matthey FORMOX has been developing and selling formaldehyde technology and catalysts since the late 1950s and has supplied more than 20 million t/a (as 37 wt-%) capacity to a wide range of customers. To put this into context, global demand in 2015 was about 45 million t/a. By carrying out both catalyst and technology (flowsheet) development in the same organisation, any catalyst development can easily be implemented in the flowsheet and vice-versa. Johnson Matthey FORMOX typically acts as an engineering and procurement contractor during the project phase and assists during erection and commissioning. After start-up Johnson Matthey FORMOX continue to support plant operation with an extensive technical support program.

Johnson Matthey FORMOX has continuously improved the formaldehyde catalyst and technology and today our customers produce more than four times as much formaldehyde in the same size reactor as in the early 1960s. This increase in production also comes with a considerably improved yield, less than half the power consumption and more than double the steam generation.

The production and use of ammonia, methanol and formaldehyde is linked. Carbon dioxide, a by-product of the synthesis gas generation process for ammonia manufacture, can be usefully combined with product ammonia to synthesise urea, an important fertiliser and chemical product. Solid urea is manufactured as prills or granules and formaldehyde (in the form of urea-formaldehyde concentrate or UFC) is used to condition granulated, and sometimes prilled, urea. One of the most commonly used grades of UFC is UFC-85, a mixture of 60% formaldehyde, 25% urea and the balance water. Formaldehyde is produced from the oxidation of methanol, which in turn is produced from synthesis gas (Fig. 1).

Generally, the formaldehyde additive required for urea conditioning is purchased from a third party as the small quantities required for an individual ammonia/urea plant are below the economic limit that would justify investment in a stand-alone formaldehyde plant along with the associated methanol plant.

As a result, urea producers tend to purchase UFC-85 from third parties whose cost structure is based on the purchase

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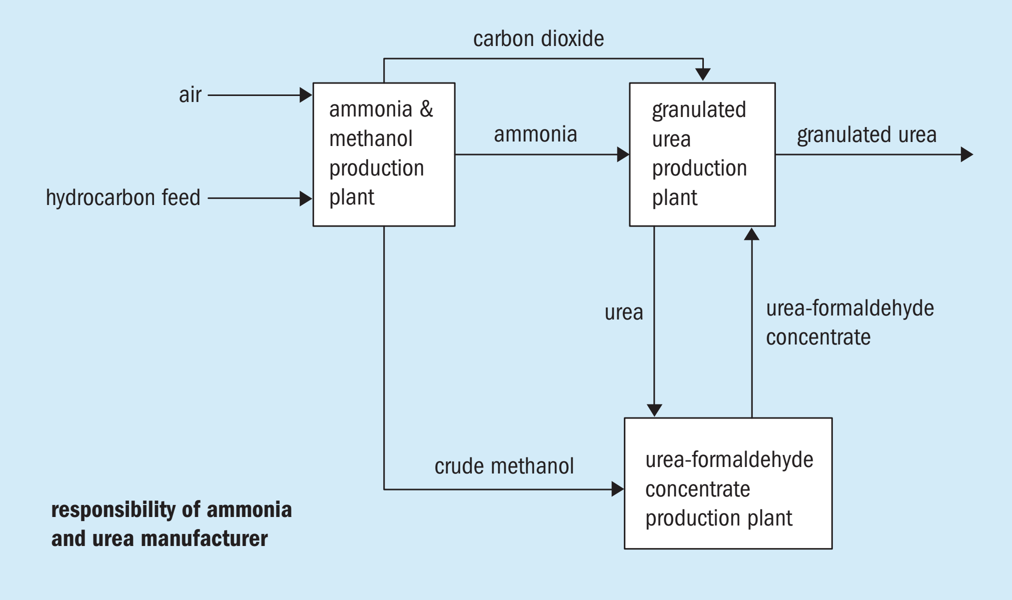
Fig 2: **Arrangement of technologies with Johnson Matthey integrated flowsheet**



(or opportunity cost) of methanol at market price which is normally at a considerable premium to the cost of the raw materials used to synthesise the methanol. The cost of the UFC-85 is then further increased due to transportation costs, the need to cover supplier overheads and return on investment criteria.

If an ammonia-urea producer could manufacture sufficient UFC-85 to meet their own needs in a plant that required only modest capital cost, using feedstock which otherwise only had fuel value and that could be operated with existing staffing levels, significant savings could be realised compared to the alternative of purchasing the UFC-85.

Flowsheet development

Using the knowledge and experience available across Johnson Matthey, a novel scheme, which is the subject of patent applications, has been developed whereby the ammonia, methanol and UFC-85 production plants have been combined to provide reliability of supply as well as capital and operating cost savings (Fig. 2).

The integrated FORMOX™ process uses the residual carbon oxides in the synthesis gas generated on the existing ammonia plant to produce methanol. This methanol can then be used to produce UFC for the urea plant. The bulk of the syngas remains and is then passed to a standard ammonia synthesis loop to produce the ammonia for urea production.

The scheme described here is proposed as a retrofit to existing ammonia-urea plants. However, it could also be conveniently applied to a new build project.

Co-production of ammonia and methanol

The first step in the integration of the three technologies is to produce methanol and ammonia on the same plant.

There are a multitude of flowsheet concepts for integrating ammonia and methanol production. Whilst some have been commercialised, others remain conceptual. This integration is possible as the syngas generation sections of ammonia and methanol flowsheets share many of the same unit operations and features, such as natural gas purification and steam reforming.

However, the purpose of the majority of the previously proposed co-production schemes was the bulk production of both ammonia and methanol. For example, ICI's METHAMM flowsheet would have been capable of producing 1,100 t/d of ammonia and 2,200 t/d of methanol[1].

In the flowsheet considered in this paper for the provision of methanol for UFC-85 production the methanol requirement is much lower than the ammonia production rate.

For example, a typical 2,200 t/d ammonia plant produces enough carbon dioxide to allow around 3,500 t/d of urea to be manufactured.

Depending on the specific design of urea plant the UFC-85 requirement for this amount of granulated urea is 20-30 t/d. To produce this much UFC-85 requires 14-21 t/d of methanol. Therefore, existing co-production schemes for ammonia and methanol are not best suited to this application.

Methanol synthesis

The reactions involved in methanol synthesis are as follows:

$$CO + 2H_2 \rightleftharpoons CH_3OH \quad \Delta H = -90.64 \text{ kJ/mol}$$

$$CO_2 + 3H_2 \rightleftharpoons CH_3OH + H_2O \quad \Delta H = -49.67 \text{ kJ/mol}$$

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \quad \Delta H = -41.47 \text{ kJ/mol}$$

In commercial methanol production plants, the preferred conditions for methanol synthesis are:

- high pressure;
- moderate temperature;
- sufficient CO_2 to synthesise the desired quantity of methanol.

Based on this, there are three options to make methanol with syngas from the ammonia plant, as shown in Fig. 3. The first (shown blue) uses gas from upstream of the shift section, the second (shown green) uses syngas from upstream of the methanator at the synthesis gas compressor suction pressure and the third (shown red) uses higher pressure syngas at an interstage pressure of the compressor, still upstream of a methanator.

Upstream of high temperature shift (HTS)

In this flow scheme, the secondary reformer effluent is cooled and passed through a desaturator before being heated to methanol synthesis temperatures and fed to the converter. The product gas from the converter is cooled and the crude methanol separated. The unreacted gas from the separator is then re-saturated and heated back to the HTS inlet temperature before re-joining the standard flowsheet.

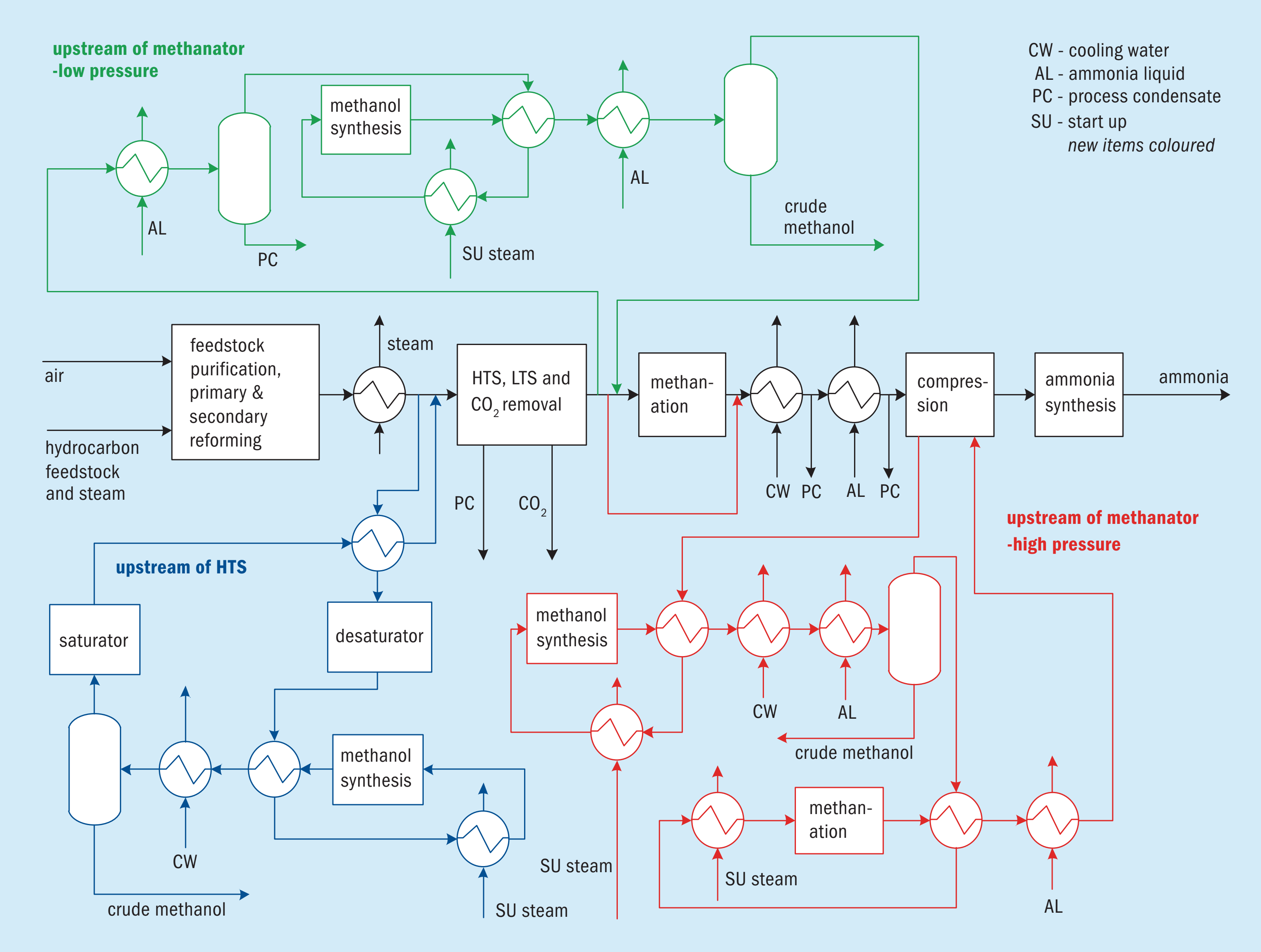
Upstream of methanator – low pressure

In this flow scheme, the syngas that has passed through the shift section and the carbon dioxide removal system is heated before being fed to a methanol converter. The effluent from this converter is cooled and the crude methanol separated from the unreacted synthesis gas. The gas from the separator is then fed to the existing methanator feed/effluent interchanger and methanator.

Upstream of methanator – high pressure

In this flow scheme, the synthesis gas that has passed through the shift section and the carbon dioxide removal system is fed to the

Fig 3: **Ammonia-methanol integration options**



first stage of synthesis gas compression. The relatively high pressure synthesis gas is then heated before being fed to a methanol converter. The effluent from this converter is cooled and the crude methanol separated from the unreacted synthesis gas. The gas from the separator is then fed to a new high pressure methanation section.

Composition of produced methanol

The Johnson Matthey integrated FORMOX process allows formaldehyde to be produced from crude methanol. However, if the water content of the crude is too high, it is not possible to make UFC-85 without a concentration step (typically distillation). Products with a lower concentration of urea and formaldehyde can of course be made. The correct selection of methanol synthesis location within the ammonia plant allows crude methanol to be generated which has a concentration high enough to avoid the need for an energy consuming and capital intensive distillation system.

Option summary

As seen in Table 1, the first option requires a complex saturator and desaturator system and distillation of the crude methanol before it can be used on a UFC plant. Although the carbon oxide content is low in the second option, there is still sufficient residual carbon oxide present to produce the amount of methanol required for UFC production.

Johnson Matthey integrated FORMOX flowsheet

Ammonia plant

The Johnson Matthey integrated FORMOX flowsheet uses the residual carbon oxides downstream of low temperature shift and carbon dioxide removal to produce methanol. The choice between low pressure (compressor suction) and high pressure (compressor interstage) is assessed on a case by case basis.

In both cases, syngas is first chilled to remove moisture and then heated in a feed/effluent interchanger before entering a methanol synthesis reactor. The gas leaving the reactor is cooled in the feed/effluent interchanger before being chilled to remove the crude methanol which is let down in pressure and sent to storage or to the UFC-85 plant. The remaining gas passes through a methanator before being compressed to ammonia synthesis loop pressure.

Bypass option

The production of methanol is limited by the amount of residual carbon oxides present in the gas stream exit the carbon dioxide removal system. In most cases this produces sufficient methanol to meet the UFC demand for an associated urea plant.

However, if the UFC and therefore the methanol requirement are higher than this limit allows, Johnson Matthey can design the flowsheet to incorporate a bypass around one or more of the HTS,

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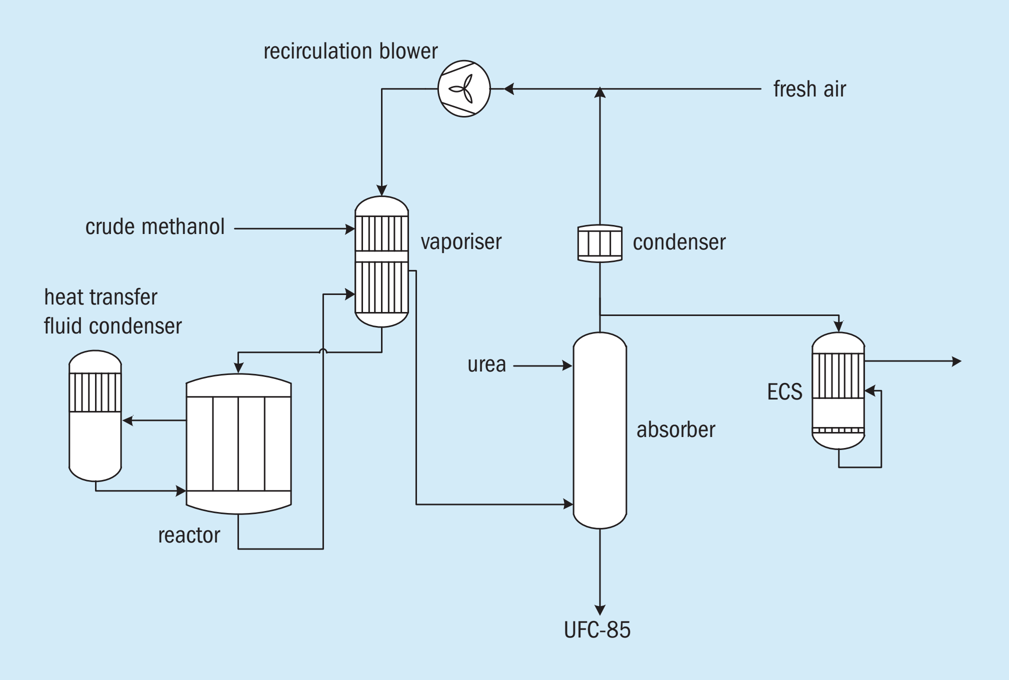
Table 1: **Comparison of options for methanol-ammonia integration flowsheet positions**

	Catalyst volume	Saturator/ desaturator	Distillation	New high pressure methanator
Upstream of high temp shift	high	yes	yes	no
Upstream of methanation at compressor suction	highest	no	no	no
Upstream of methanation at compressor interstage	lowest	no	no	yes

Fig 4: **Integrated UFC flowsheet**



the LTS and/or the CO$_2$ removal system to increase the carbon oxide concentration inlet the methanol reactor. It is important to choose the correct bypass location to minimise water formation in the methanol synthesis section to avoid the need for a crude methanol distillation unit.

If using the bypass to synthesise more methanol, there will either be a reduction in ammonia make, or the feed rate of natural gas will need to be increased accordingly.

Urea formaldehyde concentrate (UFC-85) production

Production of formaldehyde and UFC uses well established technologies. Johnson Matthey FORMOX employ mixed oxide catalyst technology due to its superior yield, high steam production and because it makes it possible to produce UFC-85 directly in the same plant.

The main principle is to partially oxidise methanol in the presence of air to form formaldehyde and water. The gas mixture of formaldehyde, water and air is separated in an absorption column in which water is condensed and the formaldehyde is absorbed into the water. UFC is produced when formaldehyde is absorbed into a urea solution which is fed to the column. The oxidation process consumes oxygen; hence oxygen needs to be provided to the process and oxygen lean gas has to be removed. The oxygen lean gas contains traces of formaldehyde, methanol and carbon monoxide and is passed over a noble metal catalyst incinerator (emission control system or ECS) in which trace impurities are converted to carbon dioxide and water.

Ammonia and UFC plant integration

Standard Johnson Matthey FORMOX plants are designed with flexibility in mind and either UFC-85 or formaldehyde can be produced on the same plant. However, if the plant is only required to produce UFC-85, it is possible to reduce capital cost by replacing the second stage of the absorption tower with a gas cooler/condenser.

The plant can be configured to produce UFC concentrations other than 85%

The UFC plant can be designed to operate with a crude methanol feed. Although operating with crude methanol is uncommon, it is not a new concept. Further integration is also possible and is offered on a case by case basis. An integrated UFC plant flow sheet is shown in Fig. 4.

Operation

Ammonia plant

Catalyst

The methanol synthesis section of the plant uses the KATALCO$_{JM}$™ 51-Series of methanol synthesis catalysts. The high and stable activity of this catalyst allows methanol production to be carried out at low temperatures that minimise the formation of by-products such as high alcohols, hydrocarbons, aldehydes and ketones.

Reduction and start-up

The reduction of methanol synthesis catalyst is similar to the reduction of low temperature shift catalyst. Natural gas or nitrogen can be used as carrier gas and the reductant may be pure hydrogen or synthesis gas. Once the catalyst is reduced, synthesis feed gas can slowly be introduced to the bed and methanol produced.

Operational considerations

The methanol synthesis section can be isolated from the rest of the ammonia plant. This means that the ammonia plant can be started up using existing methods and the syngas which is generated during normal operation can then be used to reduce and start-up the methanol synthesis section.

Despite the inclusion of a chiller to maximise separation of methanol from the gas stream it is inevitable that there will be methanol and other by-products in the feed to the methanator. Johnson Matthey's proven KATALCO$_{JM}$ 11-Series catalysts reduce their concentration to levels suitable for the ammonia synthesis loop.

The Johnson Matthey integrated FORMOX process is designed to avoid issues associated with the reaction of ammonia with carbon dioxide to form ammonium carbamate and the reaction of ammonia with methanol to form trimethylamine.

The final change that may be encountered due to the addition of a methanol synthesis section is that the hydrogen recycle to the front end of the plant could now contain carbon oxides. As a result a nickel-molybdenum hydrodesulphurisation catalyst such as KATALCO$_{JM}$ 61-1T may be recommended.

UFC-85 plant

The UFC plant is of a proven standard design used in plants worldwide with minor adjustments to reduce capital cost by integration with the ammonia complex. However the capacity is lower than that of a normal Johnson Matthey FORMOX plant to match the UFC requirement for an individual complex.

Occasional UFC-85 plant shut-downs are required to replace the catalyst. The catalyst lifetime is dependent on the plant operating rate, but will be a minimum of 8 months. The change-out duration is around 5 days.

The UFC plant can be configured either to consume urea solution generated on the adjacent urea plant or to consume solution generated by dissolving solid urea.

Crude methanol and UFC storage

The crude methanol storage system will follow standard, proven designs. In retrofit cases the urea plant would be likely to have an onsite UFC storage tank and this could be used to store UFC produced in the new UFC plant.

Benefits

Running costs

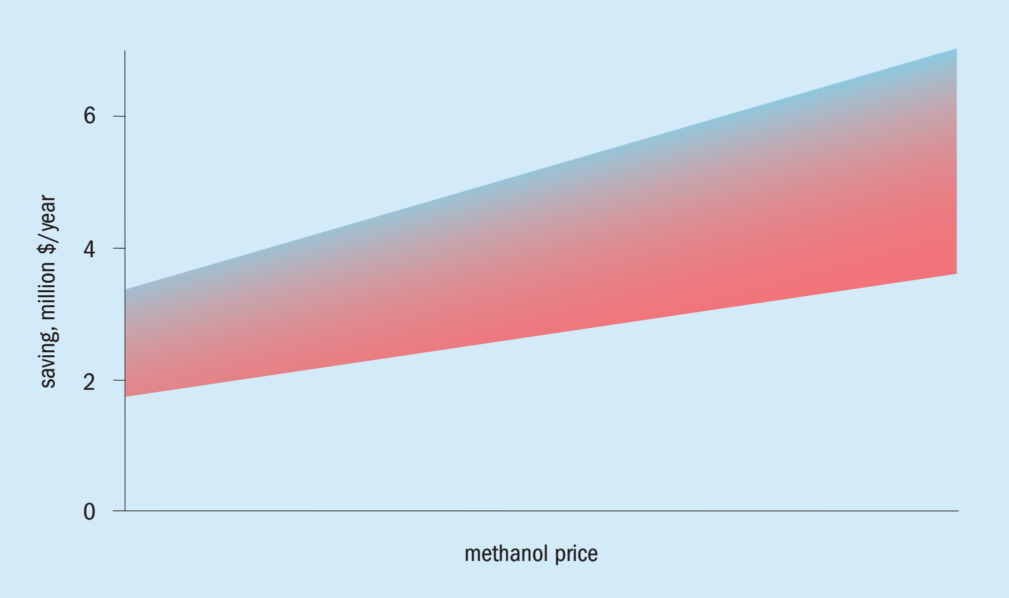
The major benefit of the Johnson Matthey integrated FORMOX flowsheet is the reduction in the cost of purchasing methanol or UFC-85. The operating costs of the UFC-85 plant itself are minimal.

The range of potential savings that can be achieved using the Johnson Matthey scheme on a standard 2,000 t/d ammonia plant making around 3,200 t/d granulated urea are shown in Fig. 5.

The figure uses a range of recent methanol prices for the assessment of savings. The lower range of savings in the figure relate to customers who already have UFC production facilities on site and would save only the cost of importing methanol to their UFC plant.

The upper range relates to customers who import UFC so are paying an additional margin to the UFC producer and additional transportation costs. Further operational savings can be achieved but vary case by case.

Fig 5: **Expected savings with varying methanol price on a 2,000 t/d ammonia plant using JM's integrated flowsheet**



Capital costs

The capital cost of the process is competitive when compared to the alternative of purchasing UFC-85 at prices which reflect those of traded methanol.

Flexibility and reliability

As the methanol synthesis unit can bypassed, there is no impact on ammonia plant reliability. Instantaneous methanol/UFC unit capacity and intermediate storage tank size allows sufficient inventory to be built up to cover a formaldehyde catalyst change without affecting ammonia or urea production.

Synthesis gas and ammonia loop

In the case of a retrofit of an existing plant, the hydrogen content in the synthesis gas to the ammonia loop is increased and the methane content is reduced. This means that the retrofit may allow an increase in ammonia make or an improvement in efficiency.

$$CO + 2H_2 \rightleftharpoons CH_3OH$$

methanol production

$$CO + 3H_2 \rightleftharpoons CH_4 + H_2O$$

methanation

As shown by the reaction equations above, the production of methanol from carbon monoxide uses less hydrogen per mole of carbon oxide removed than the methanation reaction. Therefore, as some of the residual carbon oxides are removed by producing methanol it means that there are less carbon oxides in the methanator feed so less hydrogen is consumed overall than in a standard flowsheet. The second effect is that because the methanator has less carbon oxides to convert there is less methane produced which results in a lower methane content in the loop.

On an ammonia plant using the Johnson Matthey integrated FORMOX flowsheet, the natural gas feed to the primary reformer can be reduced while still achieving the same production rate of syngas and ratio of hydrogen to nitrogen in that syngas. This also reduces the firing required on the primary reformer and gives a total natural gas saving of around 250 Nm^3/h or 0.4% for a typical 2,200 t/d ammonia plant.

Conclusion

With the integrated process, Johnson Matthey's skills in catalyst design, along with ammonia, methanol and formaldehyde process technology development have been used to create a new and cost effective method of synthesising small quantities of UFC-85 which allows a urea producer to make significant savings when compared to the alternative of open market purchases.

Reference

1. "Ammonia or Methanol: The choices", K.J. Elkins, Paper 9, IMTOF 1995, San Francisco

Acknowledgement

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