

SULPHUR

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Refinery sulphur

Sulphur quality specifications

Analysers and SRU control

Sulphuric acid catalysts

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Sulphur quality

The relevance of the SUDIC specification.



Sulphuric acid catalysts

New insight into operating mechanisms.

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FEATURES

20 The return of SSP

For years the most popular phosphorus-based fertilizer, SSP has been eclipsed during the past few decades by the rise of higher analysis fertilizers like mono- and di-ammonium phosphate. However, in the past couple of years the trend of long-term decline seems to have reversed.

24 Mind the gap

For 40 years now the gold standard for formed sulphur has been the Canadian SUDIC Premium Product specification. However, questions have been raised about its very low specification for moisture content, and whether this is necessary, realistic, or even prudent. Friability meanwhile, although vital for sulphur quality and tightly regulated in the SUDIC specification, is conversely often overlooked in sulphur purchase requirements, leading to a gap between buyer and seller in terms of standards and expectations.

30 Sulphur – a refiner’s perspective

The squeeze between regulation, changing product slates and higher sulphur feeds continues to force refiners to invest in new coking and desulphurisation capacity.

32 Analysers and next generation SRU control

Acid gas analysis has arrived and tail gas analysers are now coming into the third generation. S. Simmonds and R. Hauer of AMETEK discuss current trends in SRU process analysers and A. Henning of Jacobs reports on experience to date with Advanced Burner Control+ (ABC+) which now includes sour water acid gas (SWAG) control.

44 A better picture of working catalysts

From nano-scale studies of working sulphuric acid catalysts using new advanced in situ techniques, to improved industrial-scale sulphuric acid production, K. Christensen, F. Cavalca, P. Beato and S. Helveg of Haldor Topsoe present unprecedented new insight into the mechanisms of sulphuric acid catalysts.

50 Selection of sulphuric acid catalysts grows

MECS and Clariant have recently announced new additions to their sulphuric acid catalyst portfolios. Benefits of the new catalyst developments include increased converter performance while limiting SO₂ emissions and the ability to operate at lower bed inlet temperatures.

52 Timely furnace replacement at Burnside

J. Bolebruch of Blasch Precision Ceramics, B. Lamb of MECS and M.D. Harris of J.T. Thorpe & Son, report on the successful recent spent acid furnace upgrade in which HexWall™ ceramic furnace internals enabled DuPont Burnside to conduct a complete furnace replacement in a matter of days.

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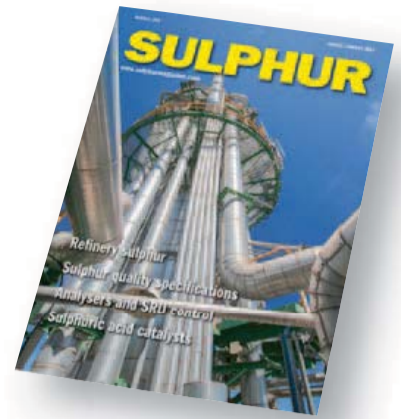
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Egypt – a new phosphate powerhouse?



“Up until now, however, there has been little downstream processing within the country.”

The phosphate industry in the Middle East and North Africa is a major consumer of sulphur, and attempts to monetise the considerable phosphate reserves there in recent years have led to several huge project developments in Jordan, Morocco and Saudi Arabia. But could Egypt soon count itself among that number? Although details are sketchy at this stage, Egyptian president Abdel Fattah Al-Sisi has issued a decree to establish a phosphate industrial zone near Aswan, which is planned to include 12 downstream industrial complexes. Minister of Industry and Foreign Trade Mounir Fakhry Abdel Nour has said the that industrial zone has the potential to be one of the largest phosphate production and exporting complexes in the world, and the General Authority for Industrial and Mining Projects plans to finish establishing seven logistical areas during the first quarter of 2015.

Egypt was once ranked third in the world in terms of phosphate reserves, after work conducted by Soviet geologists in the 1960s along the Red Sea coast and Nile Valley, during president Nasser’s brief flirtation with the USSR. However, although phosphate mines were opened and production rose to 700,000 t/a of rock during the 1970s, mainly for export to eastern Europe, Egypt later became eclipsed as a phosphate producer by developments in other parts of the world. Reserves of high grade phosphate rock are relatively limited, according to the IFDC’s most recent survey, but resources of grades 17-22% P₂O₅ are put at 3.4 billion tonnes, more than double that of nearby Jordan, just across the Red Sea, and almost half that of Saudi Arabia.

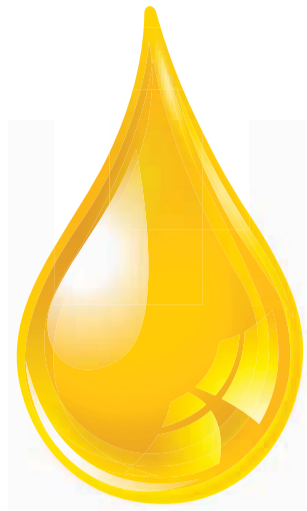
These days are two main complexes involved in Egypt’s rock mining; El Nasr is the largest, based at Aswan in the south of the country, but with some additional mines along the Red Sea coast, and there is a second at Abu Tartour, in the western desert 650km south of Cairo. The latter was plagued by production problems, and while under the control of the Ministry of Industry it produced only 300,000 tonnes of rock phosphate in the course of 30 years. Since 2009, however, it has been under the control of Phosphate Misr Company, and production has been steadily ramping up. Phosphate Misr says that it has plans to reach 5 million t/a of phosphate rock production by the end of the decade, but in the

meantime, about two thirds of Egypt’s phosphate output comes from El Nasr’s mines. Nevertheless, Egypt’s phosphate rock production has more than quadrupled over the period 2002-2012, from 1.5 million t/a in 2002 to 6.2 million t/a in 2012, of which 4.5 million t/a was exported, mainly to India, putting Egypt’s rock output on a par with Jordan, and the fifth largest phosphate rock producer in the world. Up until now, however, there has been little downstream processing within the country. Domestic consumption mostly goes into single superphosphate (SSP) production, but the rest is exported. This makes the new complex an interesting one, as it holds out the prospect of phosphoric and sulphuric acid production, and hence demand for sulphur.

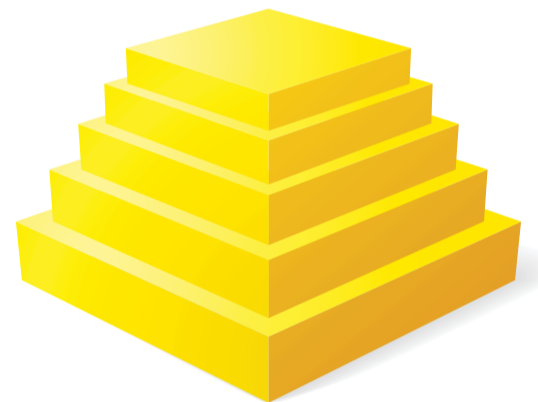
About 10 years ago there were plans for a major collaboration between India’s Iffco and El Nasr Mining – the Indo-Egyptian Fertilizer Company, which would have been a mirror of other Indian collaborations like Indo-Jordan Phosphates. IEFDC planned to develop a major phosphate processing complex at Edfu, near Aswan, where El Nasr’s mines are situated, using 2 million t/a of phosphate rock, and producing 1,500 t/d of phosphoric acid for export to India, and with 4,500 t/d of associated sulphuric acid capacity. Cost then was put at \$325 million. It seems likely that the current announcement by president Sisi is in effect a revival of this project, or something very similar, possibly tying it in with Egyptian ammonia production to manufacture MAP/DAP. Potentially, then an extra 500,000 t/a of sulphur demand; far in excess of Egypt’s domestic production. Egypt’s rapidly growing cities and demand for power and lack of investment in gas extraction has turned the country from a net gas exporter to a net importer, and it seems that, like neighbouring Saudi Arabia, Egypt sees phosphates as a way of tapping into other commodity markets, and adding value to its phosphate rock reserves. ■

Richard Hands, Editor

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Price trends

SULPHUR Meena Chauhan, Integer Research (in partnership with ICIS)

Buoyant market

Renewed demand in the global sulphur market has buoyed an upturn in pricing, led in part by trade activity in China as phosphate fertilizer producers looked to replenish raw material stocks ahead of the New Year holiday. Middle East producer monthly contract prices have seen a significant rise through January, following the market revival in the run up to the end of 2014. Saudi Aramco's January sulphur price of \$158/t f.o.b., an increase of \$33/t from December, boosted price sentiment, followed by increases from Tasweeq and ADNOC of \$23/t to \$169/t f.o.b. Ras Laffan and \$20/t to \$170/t f.o.b. Ruwais respectively. Tight sulphur supply has been a regular feature of the market recently. Producers in the Middle East describe stock positions as very tight with much of supply already committed to be lifted under contracts for North Africa. News that the Shah gas project in the UAE is facing technical problems, delaying its Q1 2015 expected start up date has put a question mark over the expected increase in export availability for 1H 2015 from the UAE.

Confirmation that the Chinese government has set new export taxes for fertilizer products at a flat fee for the duration of 2015 has had a limited effect on the sulphur industry, but some expect the policy to create a more stable outlook for sulphur as phosphate producers could be more likely to engage in regular buying patterns. The phosphate market has reacted positively to the news and demand has been firming through January with trade activity noted picking up in India and in the US. DAP prices have also risen, on the back of tight supply in Russia and North Africa, further

providing support to the sulphur market.

Import prices in China have inflated through December and January. The peak of \$169/tonne CFR in early December which was largely rejected by Chinese buyers was surpassed in January. Sulphur imports in January to November 2014 totalled 9.4 million tonnes (see graph), down on 2013.

Over in Canada, sulphur prices have firmed in line with the global upturn. Spot prices were up to \$165 FOB Vancouver in mid-January. Prices in the short term outlook may continue to rise due to increased activity in key markets, before the market reaches a ceiling or there is a downward correction. Extensive preparations took place to help prevent logistical issues that led to a slowdown of sulphur shipments in 2013.

Sulphur exports from Canada to destinations outside the US are expected to increase from 2015/2016 compared to previous years with the start up of the Mosaic remelter which may displace railed volumes of Canadian sulphur to the US.

During Q4 contract prices in NW Europe for molten sulphur were expected to see a downward correction in Q1 2015, but the global price spike led to widespread rollovers. Future pricing in the region is uncertain, as supply is expected to tighten with the continued challenges faced by the refinery industry while demand may also see a shift as caprolactam producers slow down production due to poor economic conditions.

The Indian spot market has been slow to pick up in the new year, but several end users have been noted enquiring for forward cargoes. FACT and SPIC were both looking for sulphur deliveries in mid-January, and it is understood two cargoes were

purchased of 25,000 and 20,000 tonnes respectively from Swiss Singapore. FACT's purchase was reported at \$175/tonne CFR Cochin and SPIC's at 187/tonne CFR Tuticorin, Fertilizer stocks are understood to be low in India, fuelling expectations that demand will support stable to firm sulphur pricing.

SULPHURIC ACID

The European acid export market remained stable through December and January, with limited enquiries emerging due to the slowdown over the holiday period. At the end of 2014, the supply situation was comfortable at major smelters and the market was not expected to pick up until late January. The price range at \$30-35/t f.o.b. Europe has remained stable in the new year. As sulphur prices in international markets have firmed, this has led to speculation of firmer acid prices, or a sustained level around the \$30s/t f.o.b. for the weeks ahead. There may also be a switch to increased direct acid purchasing in other markets such as India and China due to the sulphur price hike – which may support the European export price, should suppliers in other regions also see supply stretched.

Annual acid contract settlements in Chile have provided end users with coverage for 1H 2015 although some spot purchasing may take place from Northeast Asian suppliers. The Chile spot price ranged \$80-90/t CFR in January and this is expected to remain unchanged in the weeks ahead.

In the absence of fresh spot tenders in Brazil the spot price has been untested in January in Brazil. At the end of 2014, Timac's enquiry for 12,000-15,000 tonnes of acid attracted offers around \$85/t CFR. Mosaic purchased a 12,000-tonne cargo in the low-\$80s/tonne CFR for January/February arrival.

Fig 1: China imports in January-November 2014 totalled 9.4 million tonnes, a decrease on 2013 levels.

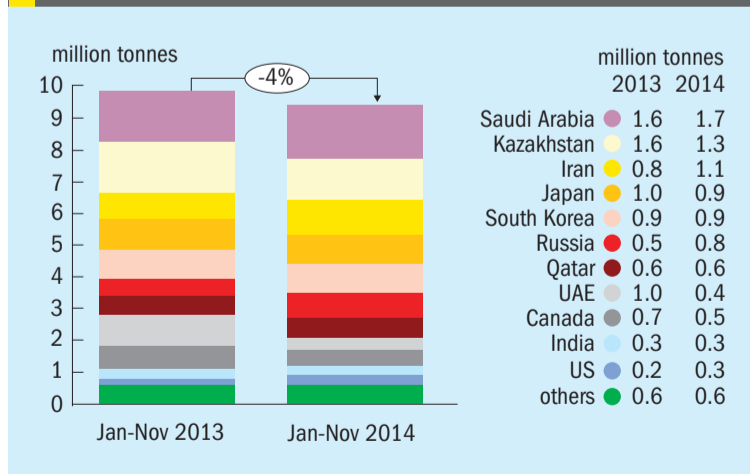
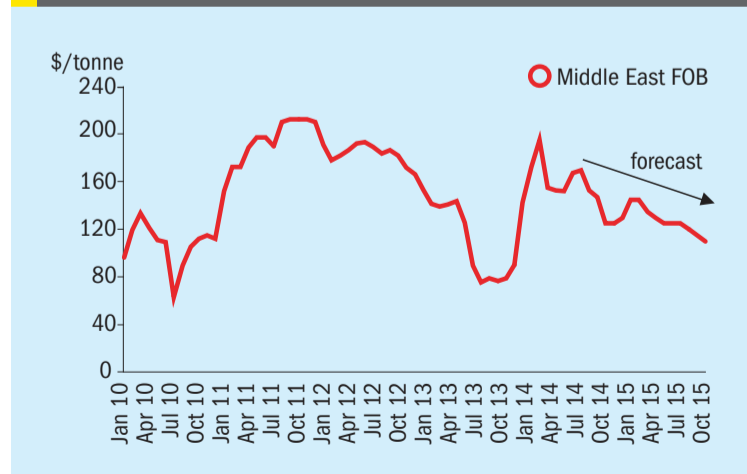


Fig 2: Middle East FOB sulphur spot prices are expected to remain firm in the short term.



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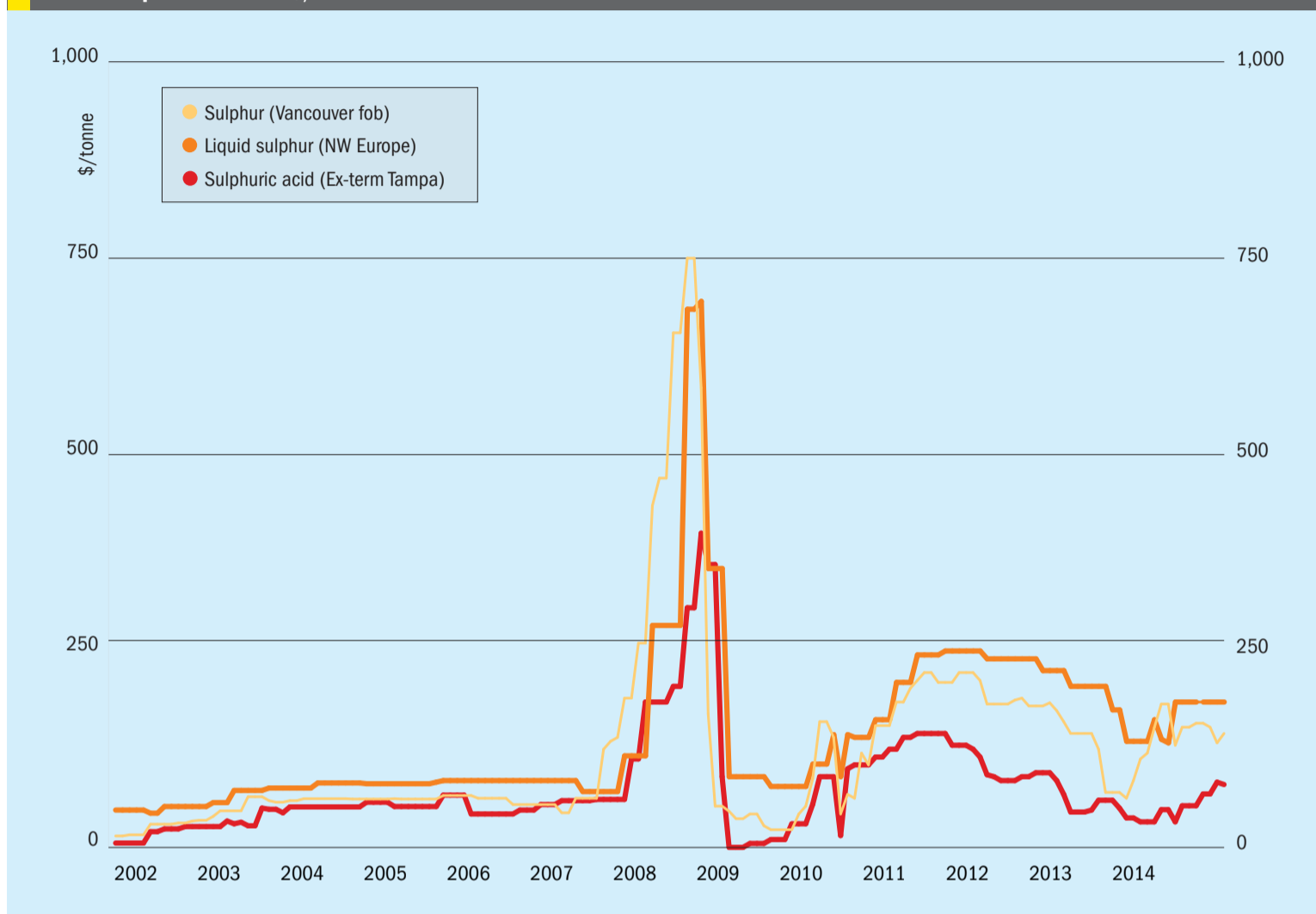
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Market outlook

Historical price trends \$/tonne



SULPHUR

- Activity in the China spot market to sustain sulphur prices in the short term, although a ceiling is likely to be reached once stocks are replenished and port inventories improve.
- Increased Middle East producer prices may lead to breaking point in the market and reluctance from end users unless there is support from the phosphates market, a subsequent downward correction may be a short term occurrence.
- The delay to the Shah gas project in the UAE provides a more bullish outlook for sulphur in 2015, scaling down expected availability of additional export volumes in the short term.
- The impact of the drop in oil prices has yet to be seen in the sulphur market, although recently announced oilsands producer capital budget cuts may impact Canadian production forecasts in the medium to long term outlook.
- OCP/Morocco is heard running its production of phosphates at reduced rates. Any increase in production could

have a bullish impact on spot pricing, while sulphur supply remains tight out of the Middle East.

- The resumption of sulphur exports out of Russia in the spring to improve supply and trade will likely be a bearish factor for the market in Q2.
- **Outlook:** The price upturn may continue if demand remains healthy in key markets such as Brazil, China and India while producers continue to hold comfortable positions. However, producer price increases in the Middle East may lead to a ceiling price and downward correction, as buyers rebuild stocks and resist further price hikes. The change in the Chinese fertilizer export tax to a flat rate is likely to lead to more stable trade through the year, although seasonal demand patterns will remain an influential factor.

SULPHURIC ACID

- Chile contract price for 2015 settled at \$78-85/t cfr, an increase on 2014, but below price expectations for the spot market.

- NW European acid export prices to remain firm in the short to medium term as producers at smelters are comfortable.
- Smelter turnarounds and demand in southeast Asian markets to keep north-east Asian acid suppliers comfortable. One Korean producer will have spot availability due to opting out of contracts for the year.
- China sulphuric acid imports increased in 2014 compared with a year earlier. The spikes in the sulphur price are thought to have encouraged trade activity in the acid market
- Acid demand in the Philippines to remain healthy in 2015 with expected demand from the nickel sector to continue to absorb Japanese acid
- **Outlook:** The acid market outlook for 2015 is stable, supported by demand in major consuming regions and comfortable smelter supply. The upturn in sulphur prices may also provide support to the market. Demand from spot markets Brazil and US Gulf will frame the NW European price outlook in the short term.

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SAUDI ARABIA

Samref completes construction of refinery desulphurisation unit

The Saudi Aramco Mobil Refinery Company (Samref) has completed the construction of major desulphurisation facilities in Saudi Arabia, including a new hydrotreater that cuts sulphur levels in gasoline and diesel. Samref, a joint venture between Saudi Aramco and ExxonMobil, said the project is the largest investment in the company's history, and will help reduce sulphur levels in gasoline and diesel by 98% to 10 ppm, a so-called Euro-V standard.

Khalid Al Falih, the president and CEO of Saudi Aramco, said: "Our long-term partnership benefits from the technology

and innovation from both companies. Our refinery will continue to be an industry leader throughout the Middle East and in the global market place well into the future. It is also testimony of Saudi Arabia's long-standing role as a reliable energy supplier to key geographic areas of the world," he stated.

Darren Woods, the senior vice president of Exxon Mobil Corporation, said the successful, recent start-up of the Clean Fuels Project illustrates the refinery's advancements and preparations to meet global energy demands.

UNITED STATES

Sulphur recovery tail gas analyser

Ametek Process Instruments has introduced its third-generation H₂S/SO₂ tail gas analyser with significant reliability advances for control of the Claus sulphur recovery process. New features of the Model 888 Sulphur Recovery Tail Gas Analyser address the three most common external failure modes encountered by Sulphur Recovery Units (SRU); automatic flow control for proactive response to adverse process conditions, such as liquid entrainment during SRU turndown; flange temperature alarm to give early warning of poor steam quality or defunct steam trap to pre-

vent sample flow failure; extended ambient temperature range to 60°C (140°F) without external cooling, to increase the life of electronics and allow installation in the hottest climates.

The safety advantages of the new Model 888 analyser include double-block process isolation and a Web-enabled interface for remote data acquisition by Ethernet and Modbus. The Model 888 features the AMEvision color graphical user interface and smart diagnostics. The unit's extensive self-diagnostics monitor six temperatures and four separate pressures, with automatic adjustments in process flow. The analyser also is equipped with steam (hot condensate) blowback to removes ammonia salts and an anti-clog-

ging feature that is automatically initiated if plugging is detected.

The 888 uses the proven sample handling technique of an internal demister with two demister pads and face-to-face fittings allowing improved access and maintenance efficiency. All internal analyser connections and internal components are accessible from the front of the unit. The compact, rugged analyser mounts directly on the process pipe eliminating the complexity of fiber-optic coupled photometers or sample lines.

Since the Model 888 occupies the same footprint and uses the same process connection as its predecessor, the Model 880, the new unit can be substituted in less than a day. Its stainless steel housing is rated IP65/NEMA 4X for harsh outdoor operating conditions, a shelter cabinet or house is not required.

Post-conference workshop

Optimized Gas Treating is offering a half day hands-on workshop following the Laurance Reid Gas Conditioning Conference, on the afternoon of Wednesday 25th February 2015. The workshop will cover using process simulation software to dissect and understand some of the nuances of; recognising pinches in deep CO₂ removal applications such as LNG production; distribution of methanol and other contaminants in amine units; the effect of heat stable salts on treating with amines. Ten laptop computers will be provided, which limits the hands-on aspect of the workshop to a maximum of 20 participants. The workshop will take place at the conference centre in Norman, Oklahoma. Attendance is free, but space is limited. To be assured of a place, register at: www.protreat.com

Keystone clears Senate hurdle

Legislation approving construction of the 800,000 bbl/d Keystone XL oil pipeline cleared a Senate hurdle in January, largely thanks to Republican gains in last year's Congressional elections. The 63-32 vote was three more than the 60 required. The Nebraska Supreme Court has also rejected a legal challenge brought in the state by opponents of the 1,900km pipeline, which aims to carry Canadian syncrude across Montana and South Dakota to connect with an existing pipeline in Nebraska for onward transit to refineries on the US Gulf. However, final approval awaits a second vote and the potential threat of a presidential veto by Barack Obama.



The Model 888 sulphur analyser.

BELGIUM**Work begins on Antwerp coker**

Fluor Corporation has begun construction of a new delayed coker unit for ExxonMobil Petroleum & Chemical at its Antwerp, Belgian refinery. Construction on the project is due to be completed in 2016, along with other projects including a 48,000 bbl/d solvent de-asphalting unit, mild hydrocracking unit, and a revamp of residue desulphurisation, all aimed at converting bunker/fuel oil into low sulphur products to comply with new emissions control legislation. Fluor's contract covers design, engineering, procurement, module fabrication, transportation, installation and construction of the new delayed coker unit, which convert heavy, higher sulphur residual oils into transportation fuel products such as marine gasoil and diesel fuel.

"Fluor will provide our full suite of engineering, procurement and construction services on this lump-sum refinery expansion project for the European downstream market", said Taco de Haan, president of Fluor's Energy & Chemicals business for the Europe, Africa and the Middle East region. "We are applying our modular execution and construction innovations in order to deliver schedule and capital efficiencies through an integrated solution to ExxonMobil."

DENMARK**Topsoe wins award for bunker fuel scrubbing system**

Haldor Topsoe A/S has won the Danish Engineering Product Award 2014 for its new ECO-Jet solution. The product is a newly developed catalytic process capable of reducing emission of harmful substances such as soot, hydrocarbons and heavy metals from ships powered by bunker fuel/fuel oil. The award, presented annually by the Danish technical journal Engineering Weekly honours technological products with significant value based on substantial technical expertise.

"When they put out to sea, large ships using bunker fuel emit harmful black smoke, and this smoke represents a major source of air pollution locally and globally. With Topsoe's new process, we have an operational technology in place that is able to reduce emissions of soot and heavy metals. The process has interesting, environmental perspectives and, for Topsoe, promising commercial perspectives as well," Professor Lene Lange, chairperson

of the Engineering Weekly awards committee, said at the award ceremony.

ECO-Jet was developed over a number of years in cooperation with Italian partner company EcoSpray. The process reduces up to 95% of soot emission from ships using bunker fuel as well as other harmful substances like poisonous hydrocarbons and metals including vanadium, iron, nickel, silicon and sodium. The solution can be combined with a so-called scrubber process to remove sulphur from flue gases; bunker fuel may contain as much as 3.5% sulphur. According to Topsoe scientist Keld Johansen, while ships could sail on environmentally friendly marine fuel with a sulphur content less than 0.1%, such conversion is still much too expensive: "for many years now, the ship industry has discussed using environmentally friendly fuel. The problem is, however, that the global refinery capacity is still too small to replace bunker fuel. That is why we have to treat the exhaust from bunker fuel in another way. And with our catalytic process, this is feasible now," he says.

Topsoe's new emission process is the result of several pilot projects. The largest pilot was the huge cruise ship MS Queen Victoria accommodating more than 2,000 passengers. The process was tested here with promising results under tough, real-life conditions.

INDIA**First sulphur cargo from Mangalore**

Mangalore Refinery and Petrochemicals Ltd (MRPL) says that it has dispatched its very first international cargo of formed sulphur. The sulphur was loaded on board MV Dusita Naree from New Mangalore Port Trust's jetty number 3. Loading of the 16,500 tonnes of the sulphur cargo, which is now bound for Zhenjiang in China, commenced on January 2nd, and was completed by January 6th. Mitsui had bid online for 16,500 t of solid sulphur generated from the phase I, II and III sulphur recovery units at MRPL.

Paradip refinery commissioning 1Q 2015

The Indian Oil Corporation (IOC) says that it will begin the commissioning of its long delayed \$5.5 billion Paradip refinery in Odisha during the first quarter of 2015. "Once fully operational, this 15 million t/a flagship refinery will further strengthen IOC's position as India's premier refiner and also add substantially to our bottom line," direc-

tor (refineries) Sanjiv Singh said in a statement. He said commissioning would begin from the first quarter of 2015, but did not say when it was expected to be completed.

Project costs have escalated due to delays, public order problems, environmental issues, and the Phailin cyclone that struck Odisha in October 2013. The refinery was originally scheduled for completion by April 2012 and cost \$4.7 billion. The Paradip refinery will produce 6.0 million t/a of diesel, 3.4 million t/a of gasoline, 1.45 million t/a of kerosene, 536,000 t/a of LPG, 124,000 t/a of naphtha and 335,000 t/a of sulphur, all of which will be for sale in the domestic market. It is configured to process very heavy and sour crudes such as Mexican Maya.

Gujarat refinery sulphur upgrade

In other news, IOC says that it will also spend \$1.08 billion on revamping and expanding its Gujarat Refinery. The revamp will cover the existing hydrotreating units, but most of the investment will go towards expansion of production at the Koyali-based refinery from its current 13.7 million t/a to 18.0 million t/a. As part of the revamp programme, the refinery will upgrade its production of diesel fuel from the current 350ppm sulphur standard (Euro III) to a 50ppm Euro-IV level, and its gasoline output from 150ppm to 50ppm, as part of Indian environmental improvements which aim to introduce a Euro IV standard nationally from April 1st 2017. This will be achieved by revamps of the diesel hydro-treating unit, diesel hydrodesulphurisation unit, vacuum gas oil hydrotreating unit and hydrogen generation unit. Gujarat refinery is also targeting Euro-V (10ppm sulphur) level compliance by 2019-20, according to IOC.

UNITED ARAB EMIRATES**Shah start-up slips to Q2**

Al Hosn Gas says that start-up at the \$10 billion Shah sour gas project in Abu Dhabi is now likely to begin in Q2 2015 rather than Q1 as had been previously expected. All eyes in the sulphur market are on the impact that this major new project will have on global sulphur prices, as it will be the largest new source of sulphur coming on-stream in 2015. At capacity Shah is expected to produce just under 3 million t/a of sulphur, although this will only occur when all of the targeted sour gas wells are producing, and the figure for 2015 is likely to be significantly lower than this. ■

EGYPT

Egypt to establish \$460 million phosphate industrial zone

Egyptian president Abdel Fattah Al-Sisi has issued a decree to establish a phosphate industrial zone near Aswan with investments totalling 3.3 billion Egyptian pounds (\$460 million), according to the Ministry of Industry and Foreign Trade. The industrial zone cover an area of 2,350 hectares, and will include 12 industrial complexes. Minister of Industry and Foreign Trade Mounir Fakhry Abdel Nour said during the announcement that the

area, like neighbouring counterparts in Saudi Arabia and Jordan, is rich in phosphate ores, which can be used to produce fertilisers and chemical products. The minister said the industrial zone has the potential to be one of the largest zones in the phosphate production and exportation field. The General Authority for Industrial and Mining Projects plans to finish establishing seven logistical areas during the first quarter (Q1) of 2015.

PERU

Outotec to provide gas cleaning technology for a zinc refinery in Peru

Outotec has been awarded a contract worth euro 16 million to supply gas cleaning technology for Votorantim Metais, part of the Votorantim Group, at the Cajamarquilla zinc refinery. Outotec's scope of delivery includes engineering and supply of a *Peracidox*[®] sulphur dioxide gas cleaning system and a scrubber for the production of sulphuric acid. In addition, Outotec will provides installation, commissioning and start-up services, with equipment deliveries scheduled to take place in 2015-2016.

The Cajamarquilla refinery, located east of Lima, produces 320,000 t/a of refined zinc, and the gas emissions from the zinc roasting circuits are processed in sulphuric acid plants. Outotec's solution will further reduce the refinery's SO₂ emissions and improve the quality of acid produced by removing impurities.

"We have cooperated with Votorantim Metais since the late 1970s and this contract once again demonstrates that our customers have a strong confidence in our sustainable technologies. In this case the SO₂ emissions will be significantly reduced, which improves the air quality and allows the Cajamarquilla refinery comply with new environmental requirements with low operating costs", commented Kimmo Kontola, head of Outotec Americas region.

NAMIBIA

Tsumeb acid plant nears completion

The new \$230 million sulphuric acid plant being constructed at Dundee Precious Metals' copper smelter in Tsumeb is progressing well and on target for com-



Tsumeb acid plant.

pletion and commissioning in mid-2015, according to the company. A company spokesman told local press that civil works for the plant were more than 90% complete and mechanical installations were 75% complete. Piping, cables and cable racking were more than 20% complete, and electrical panels have been installed. Preparations for this year's cold commissioning are in progress and the recruitment of maintenance personnel has begun. Civil construction has also begun for the installation of two new Peirce-Smith converters, which upon completion will tie into the acid plant project. The converters produce blister copper from the molten copper output of the smelter. Moving of the converter shells to the converter aisle is scheduled for April 2015 and their installation will commence by June, according to DPM.

The acid plant project is intended to eliminate SO₂ emissions at the smelter, long a bone of contention with local residents. The 300,000 t/a of acid produced will be sold on to Namibian uranium mines for leaching operations.

CHINA

BASF to build electronics-grade sulphuric acid plant in China

BASF has announced plans to build an electronics-grade sulphuric acid facility at Zhapu in Zhejiang Province, China, in partnership with Jiahua Energy Chemical, a wholly owned subsidiary of Huafang Textile. The companies have signed an agreement for the proposed project under which BASF will be responsible for the groundwork to build the plant at the Jiahua production site in Zhapu, while Jiahua will supply the key raw material for plant.

"Establishing our plant within the Jiahua site will allow us to provide electronic-grade sulphuric acid locally," said BASF electronic materials business unit senior vice-president Lothar Laupichler. He added that the new plant "is another step towards our continued growth and expansion in China's electronic materials market. Establishing our plant within the Jiahua site will allow us to provide electronic-grade sulphuric acid locally, in order to better serve the needs of China's fast-growing semiconductor industry."

Work on the project is scheduled to begin this year, with plans to start operations in 2016. The electronics grade acid produced at the plant will be primarily used by the semiconductor industry in China.

Chinese-Israeli phosphate joint venture

Israel Chemicals (ICL) says that it will invest up to \$500 million in a phosphate operation in China as part of a plan to expand abroad in the wake of an Israeli government plan to sharply raise taxes on mining firms domestically. ICL says that it will pay \$269 million in cash for a 15% stake in Chinese government-owned phosphate and

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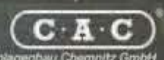


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fertiliser firm Yunnan Yuntianhua. In addition, ICL's Chinese subsidiary and a unit of Yunnan will establish a 50-50 joint venture that will own and operate Yunnan's Haikou Mine Branch Co. ICL said it will invest \$183 million in the venture, in which it will produce 2.5 million tonnes of phosphate rock annually for the next 30 years. ICL said the deal is expected to close in the first half of 2016 and will initially add \$500 million in annual sales, rising to \$700 million a year after four or five years. It will also be accretive to earnings in the first full year of operation, ICL said. The company says that its mining rights within Israel only have another eight years to run unless the government extends them, and developing operations in China is a way to compensate for this potential development.

Yunnan Yuntianhua's phosphate rock mine produces 2.5 million t/a of phosphate, and the joint venture will also reportedly include 1.85 million t/a of sulphuric acid, 700,000 t/a of phosphoric acid, and 1.0 million t/a of downstream fertilizer and speciality phosphates production.

CHILE

Outotec to upgrade Potrerillos copper smelter

Outotec has been awarded a contract to revamp and upgrade the Potrerillos copper smelter and associated sulphuric acid plant for Codelco's Salvador Division in northern Chile. The revamp will allow the smelter to comply with new Chilean environmental regulations that are due to come into force in 2018. The value of the order is approximately euro 64 million. Outotec's scope of delivery includes detailed engineering of the revamp, equipment supply and technical assistance during the construction, commissioning and start up of the smelter and acid plant. Equipment to be supplied includes gas collecting hoods for the existing converters, new dry electrostatic and wet precipitators and gas ducts, a catalytic converter and an effluent treatment plant with additional water management plant equipment.

"This is a good example of how Codelco and Outotec work together, combining their efforts to secure business sustainability and the necessary care of environment in a profitable way", said Kimmo Kontola, head of Outotec's Americas region. "Through advanced technology we can extend the life cycle of our customers' facilities. Specialised technical services are always part

of a long-term business relationship with our customers, providing added value beyond equipment supply", added Robin Lindahl, head of Outotec's Metals, Energy & Water business area.

AUSTRALIA

Ravensthorpe nickel mine closed after acid spill

The Ravensthorpe nickel mine in Western Australia, owned by Canadian-based First Quantum Minerals, has been forced to temporarily shut after a leak led to a spill of acidified leaching slurry on December 15th. FQM says that there was a "structural failure" in an atmospheric leach tank at the Ravensthorpe tank. Acid leaching nickel slurry spilled, but the company says that it was contained within the plant's own protected bunded area, and that there were no reported injuries. In a statement the company said that "The plant is currently shut down and, on the basis of information received to date, no adverse environmental effects are anticipated. Investigations to determine the cause are being undertaken."

The site, 340 km southeast of Perth, has a full production target 38,000 t/a of nickel, which it had been due to reach during 2014, prior to the accident.

Outotec buys Kempe aluminium smelting technology

Outotec has agreed to acquire Australian-based Kempe Engineering's aluminium smelter technologies as well as its service and spare parts businesses in the Middle East and Africa. The acquisition is expected to be closed in February 2015, according to Outotec. Kempe is a privately owned company providing technology for aluminium smelters, operation and maintenance services, modernisation services and spare parts. The parties have agreed not to disclose the acquisition price.

Outotec says that the acquisition of Kempe will strengthen its technology and service business in the Middle East and Africa, doubling the installed base and providing new capabilities to expand the service business in the region. Kempe will also bring additional best cost country sourcing and a manufacturing facility in the United Arab Emirates.

Grant Kempe, founder and Managing Director of Kempe Engineering said: "I believe the acquisition by Outotec will enhance the prospects of the combined

businesses by increasing the service offering to our clients as well as future opportunities for all staff, due to the scale and the magnitude of the Outotec global organisation and growth plans." Outotec CEO Pertti Korhonen added: "the Kempe acquisition supports our technology and service business growth especially in the Middle East and Africa. Through a larger installed base, joint technology offering and new service capabilities we expect to boost new projects, modernisation, operation and maintenance as well as spare parts businesses globally. With our combined resources we will be closer to our customers and a stronger provider of sustainable life cycle solutions in the aluminium industry."

The annual sales of the acquired businesses are approximately euro 25 million, and Outotec plans to double the business volume in the next few years. Nearly 400 of Kempe's employees will join Outotec's team of nearly 5,000 employees worldwide.

UGANDA

Chinese joint venture for phosphate development

The Government of Uganda and a Chinese investor enterprise, the Guangzhou Dong-song Group Co. Ltd, have signed an agreement for the development of the Sukulu Phosphate and Steel project in Uganda's eastern Tororo district. In a ceremony at the State House in Entebbe, Minister of Energy and Minerals Development Hon. Irene Muloni signed the agreement on behalf of the Government of Uganda and Mao Jie, CEO of the enterprise, signed on behalf of Guangzhou Dongsong Energy Group Co. Ltd. The \$620 million Sukulu Phosphate project began site preparation work in August 2014. When completed, it will include a phosphate fertilizer plant with a production capacity of at least 300,000 t/a, as well as a 300,000 t/a steel mill, a 12MW power plant, and a 400,000 t/a sulphuric acid plant.

However, within Uganda, there is a dispute over the ownership of the deposits, with local firm Nilefos Minerals Ltd, which belongs to the powerful Madhvani family, claiming that government denied Nilefos a mining licence over the mineral-rich Sukulu hills even after the company had spent money carrying out studies on the prospects for phosphates in the area, and had showed interest to carry out exploration, and has accused the government of taking bribes to secure the Chinese investment.

UNITED STATES

Florence leaching project clears environmental hurdle

Florence Copper Inc. says that the US Environmental Protection Agency (EPA) has issued a draft underground injection control permit for the four wells that will become the first part of the planned in situ copper leach project. Dan Johnson, general manager of the project, said in a press statement that this represents a "critical milestone" in the company's plans, although Florence still has a way to travel before it will be able to begin injecting sulphuric acid into underground copper deposits. The EPA permit is still subject to public comment, review and possible amendments, and there is still considerable local opposition; a similar permit issued earlier by the state Department of Environmental Quality (DEQ) was appealed, and two years ago the Arizona DEQ rejected the company's request to conduct full-scale commercial operations, allowing only smaller-scale test operations on a temporary basis. Arguments with the state Water Quality Appeals Board are also ongoing.

Mississippi Phosphates idles DAP production

Mississippi Phosphates Corporation (MPC), a wholly owned subsidiary of Phosphate Holdings, Inc., has announced that it has suspended production of diammonium phosphate (DAP) and sulphuric acid at its Pascagoula site in Mississippi. Steve Russo, CEO, said that the company would continue with other aspects of its core operations, including ammonia terminalling operations, maintenance, security and environmental controls, but that "at this time, we do not know when DAP production might resume. The company is actively seeking buyers for its assets while we continue to move forward with the other elements of our bankruptcy case." The Company petitioned for Chapter 11 bankruptcy protection on October 27, 2014. Approximately 50 employees will remain on-site to oversee the remaining core operations.

TOGO

Strike at phosphate mines

Workers at the phosphate mines run by state-owned Societe Nouvelle des Phosphates du Togo (SNPT) have begun indus-

trial action over wages according to local press reports. A member of the local mine-workers union was quoted as saying that SNPT had not respected agreements over wages and premiums, and that while discussions with the authorities were continuing, for the time being the miners "have stopped work until a solution is found."

Togo's phosphate output runs at about 900,000 t/a, although it dropped from almost 1.5 million t/a in 2003 to 670,000 t/a in 2008 due to underinvestment and weak management. New investment to the tune of \$150 million has restored output


to some degree, but it still falls below its target of a return to 1.5 million t/a. Phosphates are projected to earn the country \$160 million in export earnings this year according to the World Bank.

JORDAN

JPMC signs export deals with India and Serbia

Jordan Phosphate Mines Co. (JPMC) has signed new contracts for the supply of 850,000 t/a of phosphate to Indian manufacturers and 250,000 t/a to Serbian






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distributors. The company is reportedly targeting new sales in Australasia, Korea and Eastern Europe following increases to production capacity.

ANGOLA

Licenses granted for phosphate project

Minbos Resources and its joint venture partner Petril Projects have secured new licences for the Cabinda phosphate project in Angola. The two new licences, awarded by the Angolan Ministry of Geology and Mines, are for a term of five years, renewable for a further two. The first licence is for the Cacata deposit, while the second licence covers the remainder of the Cabinda project deposits.

In a press statement, Minbos CEO Lindsay Reed said; "we are extremely pleased that the agreements leading to new licences on the Cabinda project have been executed. The Cabinda project is without peer in the Atlantic basin, and following the issue of the new licences, Minbos and its JV partner Petril will proceed with the bankable feasibility study." He also noted that the company would also continue to pursue capital and operating cost reductions, and to extend the high-grade component of the resource base at Cabinda.

A scoping study over the Cacata project has estimated that a capital spend of some \$157 million would be required to develop the 800 000 t/a phosphate rock concentrate operation, which would have a mine life of around ten years.

RUSSIA

Copper smelter modernisation

Outotec has agreed with OJSC Svyatogor, a subsidiary of Ural Mining and Metallurgical Company, to provide Outotec Ausmelt technology to the Svyatogor smelter modernisation project in Russia's Ural region, in a contract worth euro 10 million. Ural Mining and Metallurgical Company is the second largest copper producer in Russia, and its Svyatogor primary copper smelter currently roasts concentrate in multi-hearth furnaces and smelts in reverberatory furnaces. The switch to Ausmelt technology is intended to improve the efficiency and environmental performance of operations at Svyatogor, as well as reduce production costs.

Outotec says that it will provide detailed engineering, proprietary equipment and

advisory services for a new copper smelting furnace capable of processing more than 500,000 t/a of copper concentrates. In 2013, Outotec provided a technology license and basic engineering for the same project. The smelter is expected to be operational in 2018.

"We are very pleased that OJSC Svyatogor has chosen Outotec's sustainable smelting technology for its modernisation project. It demonstrates their commitment to operating environmentally sustainable metals business", says Robin Lindahl, head of Outotec's Metals, Energy & Water business area.

TUNISIA

Phosphate trains collide in Gafsa

A collision between two phosphate transport trains took place in the area of Charia, between the cities of Metlaoui and Gafsa, according to the Tunisian National Railway Company (SNCF). Three train drivers were injured and taken to hospital and there was "significant" material damage according to SNCF. The accident occurred when a train coming from the railway station of El Aguila, Gafsa to Metlaoui to be loaded with phosphate collided with a train carrying phosphate from Metlaoui while it was waiting at Charia station, 22 km from Gafsa, for another train from El Aguila to clear the line.

BOTSWANA

BCL studies sulphuric acid plant viability

Botswana-based mining and smelting company BCL Mine has initiated studies into the viability of a sulphuric acid plant in order to curb sulphur dioxide emissions from the company's smelter alongside its copper and nickel mine. The Selebi-Phikwe mine reportedly releases approximately 534,000 t/a of sulphur dioxide, most of this emanating from the smelter, the mine's converter and other sources. By the end of this year, the company expects to have completed a feasibility study on the costs involved in setting up such a sulphuric acid plant and metal refinery.

"The studies on sulphuric acid production and our own refinery have been initiated and we are busy evaluating options on the recommendation. These will be completed by the end of the year with the order of magnitude capex indication," said David Keitshokile, Metals Support and Services Manager.

A recent EU-funded study indicated that the cost of developing a 2,400 t/d sulphuric acid plant for BCL at Phikwe would be \$140 million, with an additional infrastructural investment of \$150 million required, including railway tracks, a turn-in and out, ten locomotives and associated works. However, the biggest challenges, as well as the significant cost, will be storage and securing off-take., according to BCL General Manager, Montwedi Mphathi; "a sulphuric acid plant has been the best idea, but the cost of such an operation has been the problem. It would be very helpful for us if we had our market just outside the gates of such a plant because transport of sulphuric acid is another great challenge."

EU officials remain optimistic about a sulphuric plant's project economics, however. Their study indicates that the proposed plant's operating costs would be \$30/t of sulphuric acid, with transport to the envisaged target market, South Africa, a further \$20/t. The EU's researchers also believe markets for sulphuric acid could be found locally in mines such as African Copper, Tati Nickel and abroad in South Africa and Namibia. They also believe a sulphuric acid production plant in Phikwe could spur a fertiliser industry in Botswana, helping economic diversification.

SENEGAL

ZChP to build phosphoric acid plant in Senegal

Poland's Zakłady Chemiczne Police (ZChP) says that it intends to construct a phosphoric acid plant near the phosphate rock mines in Senegal in which it acquired a controlling stake last year. ZChP bought a 55% in the African Investment Group in August 2013 for \$28.9 million, including a concession for reserves of ilmenite sands located in Sud Saint Louis on the northern coast of Senegal, and exploration licenses for calcium phosphate deposits in the Lam Lam, and Kebemer regions.

Establishing the phosphoric acid installation would also help ZChP hedge against the risk of the European Union introducing tough limits on cadmium in fertilizers, the company said. The phosphate rock in Senegal's Lam Lam and Kebemer regions contains relatively low concentrations of cadmium and the costs of cadmium removal at the African subsidiary would be comparatively low, it added. ■

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People

PwC has appointed **Alison Baker** to lead its UK oil and gas sector team. Alison will lead a multi-disciplinary team made up of almost 500 industry specialists operating in areas from strategy, deals and risk assurance through to capital markets, tax, consulting and technology, supporting clients as they navigate fiscal and operational challenges and respond to global growth opportunities across the onshore and offshore cycle of exploration and production through to processing and refining.

Alison Baker commented: "The Oil & Gas industry is going through a period of change; while we've seen record investment of late, it is also operating in a mature oil and gas environment in the UK continental shelf where costs continue to rise, threatening the long term viability of the sector locally. But this doesn't just bring challenges. For those firms who dare to think differently, we believe there are a wealth of opportunities here in the UK and in emerging territories, enabling them to not only survive but thrive. Innovation and collaboration will be key to their success."

An assurance partner for over 20 years, Baker is a specialist in transformational change and capital market transactions, particularly across emerging territories. Over the last two years, she has headed PwC's mid-market energy and mining sector.

Albany, New York-based Blasch Precision Ceramics, Inc., has announced the appointment of **Shailesh Dayal** as Regional Sales Manager, heading up the company's newest office location in Agra, India, focusing on petrochemical and refining facilities in the Middle East and India. Dayal, a ceramic engineer with more than 17 years of experience in the oil and gas and petrochemical industries, will cover the Middle East/Gulf States and the Indian hydrocarbon processing markets. His extensive background includes expertise in business development in those industries. In this role, Mr. Dayal will focus on the continued commercial deployment of the *VectorWall*™ mixing checkerwall system for refinery sulphur recovery systems as well as other product lines in other process areas, and will serve both new and existing customers.

H. J. Baker announced that sales veteran **Shi Dongshen** has joined their Shanghai office in China. This newest hire follows the recent opening of their Lianyungang Sulphur Bentonite Plant. Don Cherry, H.J. Baker's Crop Performance President said; "we are very pleased Mr. Shi is joining our H.J. Baker Trading Shanghai Company. He brings a wealth of knowledge and experience in the China agriculture market that will be extremely helpful as we continue to grow our business in China."

Shi has been involved in the Chinese agriculture market since 2006 when he began working with Mosaic as a sales representative, ultimately supervising all fertilizer sales in the Henan province for the company. Shi also worked with Shandong Denghai Pioneer Seeds Co., Ltd as Regional Sales Manager. At H.J. Baker Trading Shanghai Company, he will be building the brand, developing and expanding regional distributor business as well as working to building strong customer relationships *Tiger-Sul* products. He will be working from his home office in the Henan Province and reporting to David Yang, the China Country Manager.

Mr. Yang said, "Since our grand opening back in August the company has gained much momentum. I am looking forward to working with Mr. Shi to expand sales of our *T9OCR*® and *TZINC*™ sulphur for agricultural use in China. H.J. Baker has a rich history of 164 years selling superior agricultural products. As we enter the China market we will use that history and reputation to build a first class operation selling products that are in high demand."

China has very limited production of sulphur, which has caused massive sulphur deficiencies in soils across the extensive farming regions of the country. The Sulphur Institute (TSI) currently estimates that the annual deficit in China is now more than two million tons, the largest in the world. ■

Diary 2015

JANUARY

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ASRL Chalk Talks and Poster Session, CALGARY, Canada. Contact: ASRL, University of Calgary, Alberta T2L 2K8 Canada. Tel: +1 403 220 5346
Fax: +1 403 284 2054
Email: asrinfo@ucalgary.ca

FEBRUARY

22-25

65th Laurance Reid Annual Gas Conditioning Conference, NORMAN, Oklahoma, USA. Contact: Betty Kettman, University of Oklahoma
Tel: +1 403 325 3136
Email: bettyk@ou.edu

MARCH

22-24

AFPM Annual Meeting, SAN ANTONIO, Texas, USA. Contact: Yvette Brooks
Email: ybrooks@afpm.org
Web: www.afpm.org

23-25

Phosphates 2015, TAMPA, Florida, USA.
Contact: CRU Events
Tel: +44 20 7903 2167
Email: conferences@crugroup.com

23-27

SOGAT 2015, ABU DHABI, UAE.
Contact: Dr Nick Coles, Dome Exhibitions
Tel: +971 2 674 4040
Email: nick@domeexhibitions.com

APRIL

20-22

TSI World Sulphur Symposium, BARCELONA, Spain.
Contact: Marcia Horn (Admin)
Tel: +1 202 331 9660
Email: symposia@sulphurinstitute.org
Web: www.tsi.org

MAY

18-20

SYMPHOS 2015, 3rd International Symposium on Innovation and Technology in the Phosphate Industry, MARRAKESH, Morocco.

Contact: SYMPHOS Technical Committee
Tel: +212 5 23 34 51 22
Email: symposiumocp@ocpgroup.ma
Web: www.symphos.com

18-22

3rd Annual Vienna Brimstone Sulfur Symposium, VIENNA, Austria.
Contact: Brimstone STS Ltd
Tel: +1 909 597 3249
Web: www.brimstone-sts.com

25-27

83rd IFA Annual Conference, ISTANBUL, Turkey.
Contact: IFA Conference Service
Tel: +33 1 53 93 05 25
Email: conference@fertilizer.org
Web: www.fertilizer.org

JUNE

5-6

Clearwater 2015, 39th Annual International Phosphate Fertilizer and Sulphuric Acid Technical Conference, AIChE Central Florida, CLEARWATER, Florida, USA.
Email: chair@aiiche-cf.org
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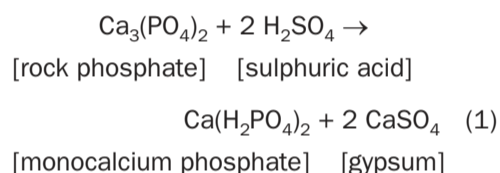
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The return of SSP

For years the most popular phosphorus-based fertilizer, SSP has been eclipsed during the past few decades by the rise of higher analysis fertilizers like mono- and di-ammonium phosphate. However, in the past couple of years the trend of long-term decline seems to have reversed.

Single superphosphate (SSP) is the most simple form of phosphate fertilizer. In essence its production consists only of crushing phosphate rock and treating it with sulphuric acid, as shown in reaction 1.



Production of SSP via this method was first used in 1842, and for many years SSP was in effect the only phosphate fertilizer in existence. In the 1870s it was joined by triple superphosphate (TSP), formed by the action of phosphoric acid on phosphate rock, but the ease of production of sulphuric acid as compared to phosphoric acid meant that SSP continued to predominate. It was not until the 1940s that mono- and di-ammonium phosphates (MAP/DAP) was first produced on a commercial scale, and it did not begin to make serious inroads into the market for SSP until the 1960s.

Since that time, however, the higher analysis of MAP and DAP (52% P₂O₅ and 46% P₂O₅ respectively) as compared to SSP (18-21%) and the fact that MAP/DAP also contain significant amounts of nitrogen fertilizer (12% and 18% respectively) have become far more important. It meant that they were in effect a much more concentrated form of nutrient, and therefore the cost of transporting each tonne of phosphate was much smaller for ammonium phosphates. SSP retained a significant market share in China and India, as its ease of production was still a major factor in its favour, but elsewhere it became eclipsed in favour of MAP/DAP and more sophisticated NPK blends. In 2013, SSP represented about 15% of global

phosphate fertilizer demand, as compared to 7% for TSP and 70% for MAP/DAP. Now, however, there appears to be something of a resurgence in SSP demand, especially in India, but also to an extent in Brazil. This article will look at some of the reasons behind that.

Phosphoric acid production – most of it (93%) for fertilizer or other agricultural (feed phosphate) use – represents about 57% of global sulphuric acid consumption, although SSP production is only a small proportion of this. Table 1 shows current levels of SSP production. Each tonne of SSP requires 0.37 tonnes of sulphuric acid, and thus a global output of 6.1 million tonnes P₂O₅ (i.e. about 33 million tonnes product at a P₂O₅ content of 18%) represents 12 million t/a of sulphuric acid demand. This is only about 4.5% of total sulphuric acid demand, but it still represents a significant slice.

China

China is the largest consumer of phosphate fertilizer in the world, and it also remains the largest producer and consumer of SSP in the world. China has substantial reserves of phosphate rock, and mines about 40% of the world's phosphates; about 80 million tonnes of phosphate rock

in 2012. Chinese phosphate demand has rapidly expanded over the past couple of decades; roughly doubling during that time. SSP was always the mainstay of Chinese phosphate production, due to its ease of production and the relative ease of sourcing of sulphuric acid, but most of the new phosphate production in the past couple of decades has been ammonium phosphates, with production rising to 22 million t/a (tonnes product), and China has turned from an importer to now become a major exporter of MAP/DAP. However, production of SSP continues at relatively high levels. Chinese SSP production peaked in about 1997 at 4.2 million t/a (tonnes P₂O₅), at which time it represented 65% of Chinese phosphate production. Since then there has been a slow decline to the present figure of 2.8 million t/a (P₂O₅). In spite of this, Chinese SSP capacity has run ahead of domestic demand, which tends to prefer MAP/DAP, and China has exported significant tonnages of SSP for some years, peaking at 800,000 t/a in the mid-2000s, and still running at about 700,000 t/a of SSP in 2012 (tonnes product). From the foregoing it can be seen that China is very much in the camp of declining SSP production and use, and does not form part of the recent SSP renaissance that has been seen elsewhere.

Latin America

Latin America is the second largest consumer of SSP, with around 1.2 million t/a (tonnes P₂O₅) consumed in 2013. Of the individual markets within South America, Brazil is by far the largest, consuming 5.7 million tonnes of SSP in 2012 (1.0 million tonnes P₂O₅), or over 80% of the South American SSP market. Moreover, while the actual volumes consumed have fluctuated, the trend has been generally upward, with an extra 1.2 million t/a of SSP demand arising from 2002 to 2012 (210,000 tonnes P₂O₅). This demand has historically been mainly in the southwestern Mato Grosso region, where soybean cultivation is extremely important. SSP demand for Brazil is closely tied in with its sulphur content (11-12% S), as soybeans are major consumers of sulphur as a nutri-

Table 1: SSP production, 2013, million tonnes P₂O₅

China	2.8
Latin America	1.2
India	0.7
Egypt	0.3
Oceania	0.3
Other	0.8
Total	6.1

Source: CRU

ent. Although MAP is about 65% nutrient by weight (12% nitrogen and 52% phosphate), and SSP is only 30% (18% phosphate and 12% sulphur), the fact that MAP is also more than twice the price of SSP in Brazil means that on a nutrient basis SSP is cheaper, and has some of the nutrients most desired by farmers in that region. It also contains calcium, which is an important secondary nutrient. Now that the subtropical savannah of the Cerrado region is also being exploited, stretching from the Mato Grosso further east into Goiás, where soils are generally poor, additional sulphur, phosphate and calcium is also likely to be required there.

SSP capacity in Brazil is about 8.0 million t/a (tonnes product) and clusters in the southern states of Sao Paulo, Minas Gerais and Goiás, and to a lesser extent in the eastern state of Bahia, south of the Amazon river, but several new SSP plants are under development to support the new developments in the Cerrado. One of the largest among these has been MBAC, whose Itafos SSP plant started up in 2014 with a capacity of 500,000 t/a. A second phase, also with a capacity of 500,000 t/a SSP, is under development. A bankable feasibility study was completed in 2013 on MBAC's Santana project further north, which is also slated to produce 500,000 t/a of SSP. Further south at Salitre Vale is developing a 500,000 t/a SSP project which it acquired via its purchase of Fosfertil. Aguiá has another 350,000 t/a plant under development at Tres Estradas

The addition of 2.4 million t/a of SSP production in Brazil may displace some imports in the short term, but it is also sign of the robust health of the SSP market there.

India

India is the world's second largest consumer of phosphates after China, and hence a mainstay of the world's phosphate fertilizer industry. Agriculture continues to be very important to the Indian economy, and as India's population grows, so do its requirements for phosphates. India has a population of 1.2 billion – about 17% of the world's population, and although it has only 2% of the world's land area, a relatively high proportion of this is cultivated, giving India, at 195 million hectares, actually a slightly higher cultivated area than China. About 45% of this area is irrigated, and net cropped area is 141 million hectares. It is also the third largest producer

of phosphate fertilizer in the world, with 19 DAP and other complex fertilizer plants and a total of 82 single superphosphate plants. Phosphorus consumption for the 2010-11 season was 8 million tonnes of P₂O₅, up nearly 10% on the previous year's figure of 7.3 million t/a, but in the past couple of years this has dropped back to 6.4 million t/a in 2012-13 and 5.5 million t/a in 2013-14 because of the effect of the nutrient-based subsidy scheme.

India's single superphosphate (SSP) industry dates back to 1906, and as with elsewhere in the world, until the 1950s SSP was India's sole source of phosphate, but SSP steadily declined as farmers preferred higher analysis fertilizers such as diammonium phosphate and various NP and NPKs. In 1991 SSP represented 19% of India's phosphate consumption, but by 2011 this had fallen to 8%. India still has 8.9 million t/a of SSP capacity (tonnes product), in 80 plants, with both production and consumption concentrated in the west of the country, where about 70% of production and 65% of consumption is based, in particular in the states of Rajasthan, Madhya Pradesh, Maharashtra and to a lesser extent Gujarat.

Subsidy schemes

SSP consumption in India had been relatively stagnant, and this had led to a large overhang of capacity, and utilisation rates below 50%. However, this has been reversed in the past couple of years by changes in subsidy policy. India has tried to encourage use of fertilizer by farmers by a programme of subsidies, but while this has been successful, that success has come at the price of large amounts of government

money; as India must import much of its fertilizer requirements, (especially ammonia and phosphate rock) it must often pay high global prices, while continuing to sell to farmers at low, controlled domestic prices. Furthermore, the relative cheapness of urea compared to other nutrients has led to imbalances in nutrient application, which has been blamed in part for Indian agriculture's plateauing of output. This has been exacerbated by the switch from SSP; some of India's major cash crops such as rice, wheat, oilseed and pulses such as lentils require significant amounts of sulphur, and the shift to DAP has contributed, along with reduction in airborne sulphur dioxide emissions, to lower rates of sulphur in the ground and a major sulphur deficiency in many soils.

The result of this was the Nutrient-Based Subsidy (NBS) scheme, adopted in 2010, which allowed free choice of fertilizer, paying instead a subsidy based on the nutrient content of the fertilizer, at a rate of \$23/kg of nitrogen, \$26/kg of phosphorus, and \$24/kg of potassium but also, for the first time, a subsidy on sulphur content, of \$1.78/kg. At the same time, maximum retail prices (MRP) were allowed to rise. However, as Figure 1 shows, the rise in MRP has been uneven; the government has used the MRP to try and encourage use of SSP, which not only has sulphur content, but may also – it was hoped – would boost the country's balance of payments; India had significant domestic capacity (half of it idle), potentially helping to displace imports of expensive DAP. Thus while the MRP on phosphate fertilizers has gradually been relaxed, it has been relaxed much faster on DAP than on SSP. The falling rupee against the dollar has also



New SSP production at MBAC's Itafos plant.

Fig 1: Maximum retail price for major fertilizers (rupees/tonne)

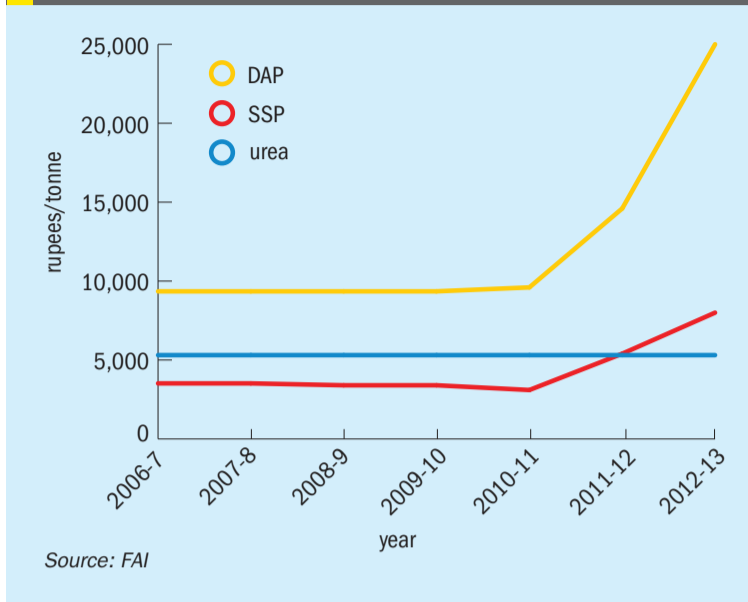
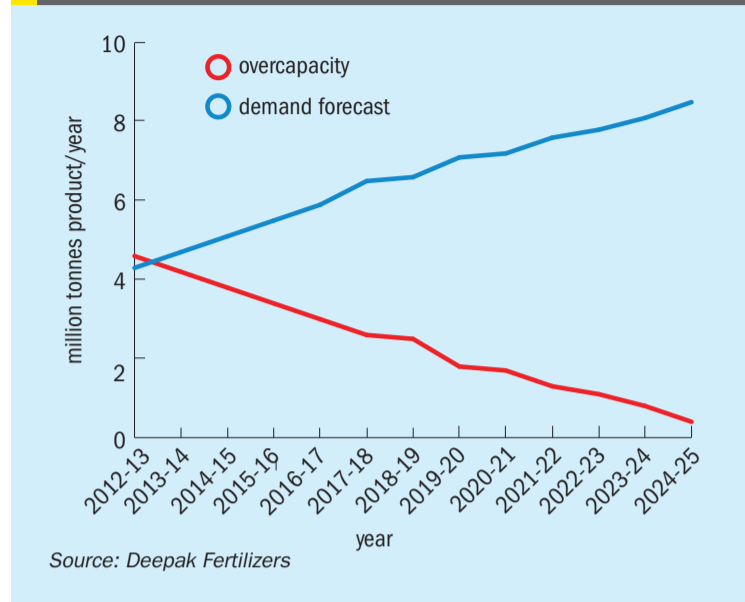


Fig 2: SSP demand forecast and overcapacity million tonnes product/year



played a major factor in this – since 2011 the rupee has fallen from around 45 to the dollar to over 60 at present – a 33% slide and in effect a 33% increase in the price of all imported fertilizer. Since SSP is made domestically (albeit largely with imported phosphate rock), it has not faced the same price pressures as DAP. As a result, SSP has become not only the lowest priced phosphate fertilizer for farmers in terms of kg P per rupee, it has also had additional benefits because of the sulphur content. The impact has been quite a significant a switch away from DAP and towards SSP. DAP use fell from nearly 12 million t/a in 2010 to just over 7 million t/a in 2013, while SSP use rose by 1.6 million t/a over the same period, from 2.7 million t/a (tonnes product) to 4.3 million t/a.

As overcapacity has fallen, so there has been considerable interest in developing new capacity, and the shift in government policy and farmers' preferences has been mirrored in a flurry of new activity in the SSP sector, with some large players in India's fertilizer market now keen to get a foothold in the industry. Coromandel International is setting up a greenfield SSP unit with a capacity of 800 t/d (260,000 t/a) in the Punjab, which is expected to start production soon, and also bought Liberty Phosphate in 2013, who had boosted their own SSP capacity from 600,000 t/a to 960,000 t/a. Chambal Fertilizers has put forward a plan for a 250,000 t/a capacity plant in Rajasthan and is also aiming to set up a 500,000 t/a SSP plant at Dahej in Gujarat by 2015. Rashtriya Chemicals and Fertilizers has also made an ini-

tial foray into SSP manufacturing with a smaller, 60,000 t/a unit, and has plans to eventually increase capacity to 1.0 million t/a. Tata Chemicals has expanded its capacity to 200,000 t/a. According to India's Deepak Fertilizers, there is currently an estimated 3 million t/a of new SSP capacity (tonnes product) under development in the country, in 16 separate plants, taking India's SSP capacity to 11 million t/a by the 2017-18 fertilizer year.

On the other hand, demand is also increasing. Figure 2 shows Deepak Fertilizers' projections for Indian SSP demand across the next decade, and the concomitant effect on the current overhang of capacity. This does not, of course, take into account the extra 3 million t/a of SSP capacity under development, although given that these are mainly larger scale plants to be operated by major, established fertilizer suppliers, there is also no doubt some expectation that it may displace some of the older, smaller, less efficient capacity.

The future for Indian phosphates

However, more worrying for India is the rapid fall in phosphate consumption overall that the NBS has seemingly led to. A drop from 8.0 million t/a to 5.5 million t/a P₂O₅ consumption is a major issue for Indian agriculture, and previous predictions by the Fertilizer Association of India (FAI) that phosphate consumption would rise to 9.52 million t/a in 2016-17 seem very over-optimistic at present. The victory last year of Narendra Modi's government led

to hopes that the subsidy regime might be reformed and indeed there has been some movement in that direction, but so far the government has concentrated on urea; raising gas prices but also vowing to reimburse producers and trying to encourage the development of more domestic urea capacity, while the subsidies for phosphate and potash fertilizers have been reduced still further, making the present nutrient imbalance even worse. With the budget for 2015-16 now under discussion, it remains to be seen how India's phosphate industry will fare. The betting is though that SSP will continue to be favoured, as domestic rather than imported production.

SSP's renaissance?

While China has continued to conform to the familiar pattern over the past few decades of a decline in SSP use as compared to MAP, DAP and NPKs, in Brazil and India it is seeing something of a renaissance, on the one hand for agronomic reasons, and on the other for its relatively cheapness and simplicity of production. As well as India and Brazil, Egypt has added SSP capacity in recent years, and the next few years could see a further 5.5 million t/a of SSP capacity and – if Indian projections are to be believed – possibly an additional 5-6 million t/a of actual production and consumption; an additional 2 million t/a of sulphuric acid demand. With concern about secondary soil nutrients like sulphur and calcium now very much to the fore, it seems that SSP has not yet had its day. ■



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Mind the gap

For 40 years now the gold standard for formed sulphur has been the Canadian SUDIC Premium Product specification. However, questions have been raised about its very low specification for moisture content, and whether this is necessary, realistic, or even prudent. Friability meanwhile, although vital for sulphur quality and tightly regulated in the SUDIC specification, is conversely often overlooked in sulphur purchase requirements, leading to a gap between buyer and seller in terms of standards and expectations.

The Sulphur Development Institute of Canada (SUDIC) was a Canadian non-profit corporation based in Calgary, Alberta from 1973-1994, whose objectives included; “promoting and developing new uses for sulphur; encouraging, coordinating, supervising and assisting with research and development of testing work; furthering use and consumption of sulphur; and furthering ideas, inventions and patents”. SUDIC did not maintain research facilities, but functioned as a project management group via external resources.

In 1977, SUDIC, via the Canadian Industry Sulphur Forming Committee, developed the Standard and Premium Product Specifications (see Table 1)¹. The impetus behind this was a dual concern with safety on the

one hand and marketing on the other. A lot of sulphur was (and is) shipped as a molten liquid, but Canada did not have the facilities for doing this at Vancouver port, nor did many client countries have terminals suitable for handling molten product. Sulphur was therefore exported as a bulk solid, often chipped from sulphur blocks in large lumps. This product was extremely dusty, which produced both a safety and environmental hazard in storage and transport, and eventually in 1972 the shipping of crushed bulk sulphur was banned by the Vancouver port authorities³. The first attempt to ameliorate the problem was via the production of ‘slate’ sulphur on a continuous, water-cooled moving belt, but by the mid-1970s other forming technologies were becoming available, including pel-

letising, wet prilling, and dry or air prilling. The SUDIC standards were designed as a way of encouraging the use of these new forming techniques, in order to produce what could be described and marketed as a ‘premium product’, differentiating it from crushed bulk/lump production elsewhere.

There were numerous ways in which this could have been done, but then, as now, the major issues for transporters and shippers of sulphur were firstly sulphur dust, which is a fire/explosion hazard, and secondly sulphur corrosion/acidity. The standard aimed to correct the former by making the product a uniform granule with good resistance to being chipped and abraded during transport, and the latter by reducing the water content of the sulphur to as low a level as practicable. This relied on the use of one or another

Table 1: SUDIC Product Specifications

Property	Standard Specification	Premium Specification
Shape/size: Generally spherical;	2-5mm diameter	2-4mm diameter 75% between +/- 1mm of avg diam.
Size distribution:	>6.3mm: <5% <1.18mm: <2% <0.3mm: <0.25%	>4.75mm: <5% <1.18mm: <2% <0.3mm: <0.1%
Friability:		
Stress level I (abrasion)	<2% fines (<0.3mm)	<1% fines (<0.3mm)
Stress level II (impact)	<5% fines (<0.3mm)	<0.5% fines (<0.3mm)
Bulk density:		
Loose	>1,040kg/m ³	>1,040kg/m ³
Compacted	>1,200kg/m ³	>1,200kg/m ³
Compaction breakdown: (25psi – equiv to 15m stockpile)	<0.5% fines (<0.3mm)	<0.2% fines (<0.3mm)
Angle of repose:	>25°	>25°
Moisture content:	<1.5%	<0.5%

technology. The Premium specification was based on a Polish technology known as the 'Polish Prill'. This was an air-blown forming technique developed by Siarkopol, the Polish national sulphur company which ran sulphur mining operations in that country. The process sprayed droplets of molten sulphur into a counter-current of cooling air². The resultant spherical prills were, essentially, moisture-free. The theory was that the resulting prills could then be transported, stored and loaded in a virtually dry state, via covered rail cars and covered storage, so that at the time of loading onto a vessel it remained in a 'dry' state, reducing the corrosion problems in shipping (and attendant issues with contamination of the sulphur in transit). However, as Poland was at the time behind the 'Iron Curtain', there was some worry that licensing of the prilling technology might be problematic, and so there was also a 'Standard' sulphur specification developed, which would allow the use of more widely available wet prilling technologies.

In fact there were no issues with licensing of the Polish Prills, and the technology became adopted in Canada and elsewhere. However, it did not come without its own issues, a major one being fugitive sulphur dust from the top of the prill tower during the prilling process. A catastrophic fire destroyed two Polish Prill towers and their associated sulphur storage at Jubail in Saudi Arabia in 1985, and it has been theorised that this was due to ignition of sulphur dust by static electricity. By this time, the adoption of the SUDIC standard had galvanised the development and adoption of other forming technologies, by Enersul, Sandvik, Devco and Brimrock, all of which aimed at meeting the SUDIC Premium specification, and these gradually edged out the Siarkopol process.

Meanwhile, the original theory of keeping sulphur 'dry' all the way from manufacture to customer did not work in practice, because even Premium sulphur product generated quantities of airborne dust during storage and handling operations, and so water was frequently sprayed in order to keep dust levels down. Nevertheless, the specification remained, backed by Canada's power as a sulphur marketing giant; in 1982, Canadian exports represented 42% of world sulphur trade.

The days when Canada was the dominant power in world sulphur are gradually slipping behind us, and the SUDIC organisation itself disappeared 20 years ago. However, one of the lasting legacies of that period has been

the SUDIC Premium Sulphur specification, as there has been no real attempt to develop an alternative since then. Consequently, it has become a standard specification for formed sulphur today. In 2007, Brimrock estimated that just over 60% of all traded sulphur produced worldwide was manufactured to the SUDIC Premium specification⁴, and since then the percentage has increased; perhaps to around 75%.

However, while the standard no doubt leads to a far higher quality product than crushed bulk, lump or slate sulphur, it was developed for a specific time and place, and based around a technology no longer in widespread use, and there have long been niggling questions about exactly how relevant the specification remains to today's market and technologies, especially as regards moisture content. Indeed, since 2007 China – the world's largest importer of sulphur – has had its own sulphur specification which allows up to 2% water content of sulphur, and many buyers are willing to accept higher than the 0.5% specification when purchasing sulphur, as we will see later. It is worth remembering that SUDIC's own, often overlooked Standard specification allows for up to 1.5% moisture content. Finally, there is also the question as to what extent the sulphur which arrives at a customer bears any resemblance to that which leaves the forming plant.

Many a slip

The SUDIC specification for moisture has frequently been taken to mean the moisture content of the sulphur as it leaves the forming machine, although there has in the past been some argument as to whether the SUDIC moisture measurement was intended to be taken 'as formed' or 'as shipped' – i.e. from Vancouver. The fact that some elements of the testing – the friability tests in particular – were to take place "not less than 21 days" following forming, after the full relaxation from the high temperature monoclinic form (stable above 96C) to the orthorhombic form of sulphur had taken place, might indicate that the product could well have already been moved from the forming plant – in Alberta most likely at a sour gas plant in the foothills of the Rocky Mountains – to the wharves at Vancouver port during that time.

Still, it is nevertheless possible that, with careful storage and handling, the moisture content at the point of production may approximate to the moisture content

of sulphur reaching the port of embarkation. Even so, this does not necessarily equate to what will be discovered by the end consumer when the trucks or railcars reach the acid plant at the other end. While the SUDIC working group assumed that there would be covered storage, storage in open silos leaves the sulphur at the mercy of the elements, especially rain. Furthermore, as noted above, water is often sprayed at transfer points to keep the levels of dust down – sulphur dust is otherwise potentially a deadly fire or explosion hazard, with a lower explosion limit of just 35g/m³. Nor, of course, is any ship hold ever likely to be 100% watertight, and during a long voyage temperature differentials are in any event likely to lead to condensation within the hold.

But it is not just moisture content that may change during transit – 'real world' loading and unloading operations may involve hoppers placed too high (so that sulphur granules fall a longer distance and become more broken) and the use of front end loaders which are likely to crush some of the sulphur beneath their wheels. In an attempt to gauge the extent of the difference between sulphur as shipped and as received, consultant Gerry D'Aquin of ConSul reported⁵ on studies he had conducted on the moisture content and particle size distribution of sulphur from various manufacturing techniques, both at the point of forming, and at the point of delivery. The tests included sulphur shipped as slate, *Rotoform* prills, as Devco wet granules and Enersul GX granules, which were taken from Alberta to China, from the US Gulf to Morocco, and from Germany to Morocco respectively. The results are given in Table 2. While all of the premium sulphur products showed a considerable improvement on the slate product, which at loading had 17% of its content below 1.18mm in size (#16 mesh screen), and a moisture content of between 1.6% and 4.1% (large quantities of water were observed being used during unloading because of the dustiness of the sulphur), the transit operation nevertheless took its toll on the formed product, particularly in the generation of fine particles.

Those who argue for the continuing applicability of the SUDIC standard contend that whatever the level of moisture that the sulphur picks up on the way, it is a demonstrable fact that the lower the starting amount of moisture in the sulphur, the lower will be the moisture content at the other end, and

Table 2: Premium formed sulphur, before and after transit

Particle size:			
Sample	Particle range	As produced	On arrival
1	2.36-3.35mm	98%	72%
	<1.18mm	2.0%	25.2%
	<0.3mm	0.3%	4.9%
2	3.35-4.75mm	90%	62%
	<1.18mm	7.2%	22.3%
	<0.3mm	0.4%	3.2%
3	2.36-3.35mm	85%	70%
	<1.18mm	4.9%	21.4%
	<0.3mm	0.4%	0.7%
Moisture content:			
Sample	As produced	As shipped	On arrival
1	0.01%	2.68%	0.8%
2	3.17%	1.6%	2.9%
3	0.04%	1.99%	0.1%

the stronger and more regular the formed sulphur, the greater will be its resistance to breaking. Delivery of the highest quality sulphur is thus a function of forming the best, driest product and then taking as much care as possible during transfer and handling operations. This was certainly the thinking behind the development of the SUDIC specification in the first place.

However, one of the things noted in the study was that, although all three samples generated similar quantities of fines during transit, the actual quantity of dust observed during unloading was effectively zero for the sample with the higher moisture content, but clearly observable for the one with the lowest. Given that there is evidently a trade-off between actual observable dust and moisture content, should we always necessarily be seeking the lowest possible moisture content?

Effect of moisture content

So what is the downside of water content of sulphur? Among the reasons cited for the water content restriction is the additional cost of transporting several tonnes of water with the sulphur cargo, and the heating cost of the latent heat of vaporisation of the water when the formed sulphur is remelted at the end consumer. Water has a very high latent heat of vaporisation – just over 2.2 GJ/tonne, and this has an energy cost for the customer. There are those who argue that this can be overstated – a sulphuric acid producer with a sulphur burning acid plant is generally producing

their own heat and electricity from the sulphur burning process, and the opportunity cost of energy is not the price they would have to pay for gas or electricity from a utility company, but the amount that they would be able to sell excess power at to the local grid, often much lower. Nevertheless, it does represent an additional cost burden. For example, at 1% water content in a tonne of sulphur, this would represent an additional 6kWh of electrical energy in melting it, and at the consumer electrical rates supplied by a utility company here in Europe, that would be an equivalent cost of about \$1.50/tonne, although as noted, the wholesale prices an acid plant might sell electricity at are likely to be lower. Likewise at a bulk freight rate of, say, \$50/tonne, that 1% water is in effect an extra cost of \$0.50/tonne of sulphur shipped. So if there is 2% moisture in your sulphur, the buyer is in effect paying an additional \$4/t over the asking price. Now it could be argued that this may have been more relevant 10 years ago when sulphur was changing hands for \$40/t rather than \$140/t, but it is still a cost nevertheless.

Cost is only one angle, though. Other worries centre around acidity and corrosion – in the presence of thiobacillus and water, sulphur can be broken down into sulphuric acid, which then corrodes ship hulls or railcar interiors. Likewise in the presence of water, direct contact between sulphur and steel can lead to electrochemical corrosion. Electrochemical corrosion is especially problematic, as it can generate hydrogen sulphide, extremely dangerous if

a sealed hold is opened, and it can form pyrophoric iron, which can spontaneously combust on contact with air as the hold is emptied. These days this is often mitigated by treating the interior hold of a vessel with a lime wash prior to loading of sulphur, and to counteract thiobacillus growth, sodium lauryl sulphate (SLS) is generally added to the sulphur at a level below the required organic carbon content of the product. But excess water in the sulphur can wash away the lime wash coating, especially where it collects at the bottom of the hold, and as SLS is water soluble it can also reduce the effectiveness of the anti-bacterial agent. Too much water may lead to other problems like shifting of cargo. In a 2005 paper⁶, Les Lang (then of Brimrock) and Jim Hyne tried to qualify the effect of different moisture levels in sulphur, and the results are presented in Table 3.

One of the teasing questions about solid sulphur is that – given that it is hydrophobic – how does it actually contain water in the first place? Some of it is perhaps surface moisture clinging to individual particles, but one argument is that cracks caused by the phase transition from monoclinic to rhombic sulphur allow water to enter into them by capillary action. The problem of moisture content also thus returns to friability.

Friability

Cooling of sulphur granules happens from the outside inward, meaning that the outside of the droplet solidifies first and forms an outer solid shell before the centre solidi-



Dust generated during unloading operations of dry premium sulphur.

1	47
2	48
3	49
4	50
5	51
6	52
7	53
8	54
9	55
10	56
11	
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Table 3: Effect of moisture content on sulphur handling properties

Moisture content	Handling properties
0 – 0.5%	Too dry to minimise dust generation. Tends to create fines when extensively handled which become airborne as environmentally unfriendly and potentially explosive dust.
0.5 – 1.2%	Suggested ideal moisture content range. Moisture level suppresses fugitive dust, moderates bacterial action, maintains stability in bulk cargo movement. May avoid moisture content penalties if at low end of range.
1.2 – 2.0%	Becoming unnecessarily wet. Encourages bacterial action. Start of slurry flow behaviour in storage and transportation vessels. Vessel corrosion enhanced if steel not protected.
2.0 – 3.0%	Considered very wet product. Will likely wash off lime used to protect ship steel against corrosion. Energy costs during re-melt by user becoming excessive.
3.0 – 6.0%	Excessive moisture content possibly attracting significant price penalties in addition to weight correction. Drainage from stockpile or ship hold may present challenges. Movement in ship hold notable. Almost certain loss of lime wash from steel surface and enhanced corrosion. Check chloride content for possible ingress of sea water.
>6%	Product only acceptable under special circumstances. Transportation may be restricted by local regulations.

Source: Lang and Hyne, 2005

Table 4: Illustrative purchase specifications for formed sulphur

Specification 1:	
Purity (on a dry basis)	99% minimum
Carbon	0.025% maximum
Ash	0.05% maximum
Arsenic	Commercially free
Selenium	Commercially free
Tellurium	Commercially free
Moisture	Moisture >0.5% shall be deducted from Bill of Lading for invoicing purposes.
Specification 2:	
Purity (on a dry basis)	99.8% minimum
Carbon	0.025% maximum
Ash	0.05% maximum
Arsenic	1 ppm maximum (commercially free)
Selenium	1 ppm maximum (commercially free)
Tellurium	1 ppm maximum (commercially free)
Acidity	Typically 0.02% maximum
Moisture	Typically 0% to 3.0% maximum
Specification 3:	
Colour	Bright yellow
Purity	>99.9% minimum
Organic matter	<0.02% maximum
Ash	<0.015% maximum
Arsenic	<1ppm
Selenium	<1ppm
Tellurium	<1ppm
Acidity	<0.007% H ₂ SO ₄
Moisture content	<0.50%
Particle size:	
>6mm	<5%
1-6mm	>90%
<0.5mm	<0.5%

Source: J. Macdonald, private correspondence, Sandvik Process Systems⁹.

fies⁸. When the inner liquid sulphur solidifies it shrinks. This means that a small generally spherical droplet of liquid sulphur solidifies into a generally spherical solid particle with internal voids caused by the shrinkage. These voids can be readily seen in the liquid drop formed sulphur particulate whether it be dry or wet formed and increases the susceptibility of the form to breakage and crushing under pressure. In addition, since the interior cools more slowly (sulphur is a poor conductor of heat) its crystal structure is larger and less well packed, making breakdown to form dust easier if the particle is broken and its interior exposed. Sulphur formers like Enersul have worked hard to overcome this issue via their forming techniques, many of which now rely on building up a sulphur particle in layers to counteract this tendency, and/or paying very careful attention to temperature

control during the process. The result is the excellent formed particles now produced by modern sulphur forming equipment.

However, what many in the industry may not realise is that sulphur producers and sulphur traders/buyers often work with two separate sets of specifications when dealing with the product itself. While producers pay close attention to the SUDIC specification, the purchase specifications may be very different, and tends to focus on purity – especially content of heavy metals like arsenic and selenium, which may render any fertilizer produced unusable. Typical examples are given in Table 4. From this it can be seen that a gap in product quality specifications now appears; there is no mention in the first two sales specifications of particle size distribution, friability, etc. The irony is that the companies that own oil and gas pro-

cessing complexes may spend millions of dollars installing and operating equipment to meet one specification, while the sellers (perhaps often within their own company) and buyers spend millions of dollars on product that must meet a completely different specification criteria. This results in a gap between what a buyer knows about the producer's production operation and what quality of product is ultimately arriving at his company's port of discharge.

There are signs, however, that this may be changing. The third specification, from a major European trader, does quote figures for particle size distribution. In their Sulphur Forming Workshop at the 2014 Sulphur Conference⁹, Sandvik Process Systems argued that there is a "growing awareness" amongst large volume sulphur buyers in addressing these issues with their suppliers, and that as a result, the importance of physical prop-

erties as well as purity are starting to make their way into market specifications.

A happy medium?

So where are we now with sulphur specifications? As we've seen, there is a disconnect between the SUDIC Premium specification which up to 75% of sulphur producers now work to, and the purity-based specifications which buyers tend to ask for. There are signs that this gap may, perhaps, be closing in terms of requirements for friability and particle size distribution, but there is still a discussion to be had over moisture content. Although the SUDIC specifications are – rightly – extremely concerned with fines content (defined as sulphur particles of 0.3mm or less), the fines content of sulphur does not automatically equate to levels of airborne particulates, which are the real danger. While too much water in sulphur is undoubtedly a problem, and an additional cost burden for buyers, too little can be equally problematic during loading and unloading operations. Some of the issues which the water content

specification was designed to counteract, for example the growth of thiobacillus, have essentially solved in the years since the SUDIC specification was written – in the case of thiobacillus by the use of sodium lauryl sulphate. Given that many buyers are willing to accept water content of up to 2%, and given the extent to which moisture content can vary from production to unloading, is there a case for arguing that the minimum water content stipulation of the SUDIC specification could be extended slightly for modern premium products, and perhaps even a minimum moisture level specified? There is no hard and fast answer on this; sulphur formers' opinion of the balance of risks often depends upon whether they are selling a wet or dry forming process. However, it is surely right that the question at least be asked. ■

Acknowledgements

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Sulphur – a refiner's view

The squeeze between regulation, changing product slates and higher sulphur feeds continues to force refiners to invest in new coking and desulphurisation capacity.

The refining industry exists at a unique juncture between the oil industry and both the petrochemical industry and downstream consumers, industrial and individual. It must eke a margin between the cost of oil feedstock and the price of end products, while facing a continual push by governments and regulatory agencies to not only improve product quality, especially in terms of levels of sulphur, aromatics, oxygenates and renewables, but also in terms of refinery emissions of sulphur dioxide, carbon dioxide, etc.

To begin with, refiners must cope with changing demand for products. In the past decade or so, this has come in the form of a move from gasoline towards diesel, a move from OECD to non-OECD countries, and a decline in demand for heavy fuel oil, to name just three of the major trends in the industry. Some of this is down to government intervention in markets, and regulatory issues continue to loom large for refiners. In particular, regulatory limits on sulphur content of fuels are a continually evolving area and represent a major challenge.

A global sulphur standard?

An interesting development is that sulphur in fuel specifications for gasoline and diesel are globally converging on the so-called 'Euro-5' sulphur standard, of 10 parts per million sulphur. The EU has mandated 10ppm sulphur in gasoline since 2009 and diesel since 2011, Japan and South Korea for gasoline since 2007, and California since 2008, but so far only California has tried to move beyond them, and there seems to be a general levelling off of fuel sulphur standards at the 10ppm level.

General US standards are currently at 15ppm for diesel and 30ppm for gasoline, but the forthcoming Tier 3 specifications will require 10ppm from 2017. Other areas are following, and new refineries in Asia and the Middle East which wish to supply into sizeable markets which already have a Euro-5 standard are becoming obliged to match such standards. Worsening air pollution problems have led China to speed up its move to tighter sulphur specifications, and China will move to a Euro-5 fuel standard by 2017. In a way this is good news for refiners, as it means that once they are able to meet the 10ppm standard for diesel and gasoline, they can supply into all markets and do not risk the goalposts changing again, at least, not in the foreseeable future. However, it presents them with considerable challenges in getting to that target.

Marine fuels

For refiners in Europe and North America, the biggest change in sulphur fuels regulations has been the International Maritime Organisation's stipulation of a move to 0.1% sulphur fuels or less in designated Emissions Control Areas – mainly the North and Baltic Seas and coastal regions of North America, although other areas, such as the Mediterranean, may soon follow suit – as compared to the present 1.0% sulphur limit in such areas. Although scrubbing technology exists, there has been some doubt about its cost-effectiveness, and most ships are expected to switch to low sulphur marine gas oil (MGO) in order to meet the limits. The main initial effect is expected to be a boost to demand for distillate fuels and a decline in demand

for residual fuels. The European and North American Emission Control Areas (ECAs) between them are predicted to represent an additional 40-50 million t/a of distillate demand worldwide, and a corresponding decrease in residual fuel demand.

The drop to 0.1%S in ECAs come into force on January 1st this year, but the next major step in marine fuel sulphur restriction will come with the global move to 0.5% sulphur content for all areas. However, the date for this change could be either 2020 or 2025, with the EU looking like it will move to the new lower limit in 2020 even though the IMO regulations allow for an extension to 2025.

Feedstock changes

Crude oil composition is generally fairly uniform, but varies most notably in terms of API gravity and sulphur content. Globally, Middle Eastern crudes tend to be sour, and run from about 1% for Oman to 3% for Saudi Arab Heavy. African crudes, conversely, such as Nigerian Bonny and Libyan and Algerian crudes, tend to be sweeter. Brent crude is about 0.4%, and FSU production from the Urals around 1.3%. In general, supply of oil from sweet sources has tended to plateau, whereas supply of oil from sourer sources continues to increase. The overall effect is that the sulphur content of refinery inputs is steadily increasing, and refiners are having to configure their refineries in order to deal with heavier and sourer feeds, as these feedstocks often come at a discount in cost and hence offer better margins, even with more difficult processing. The downside is that high-sulphur crudes are more corrosive and can cause more rapid deterioration, with associated costs.

In the US, some of this has come from increasing use of Canadian oil sands syncrude. While the Keystone XL pipeline which was supposed to be bringing large volumes of syncrude from northern Alberta to the US Gulf Coast by now remains locked in legal and legislative limbo, deliveries by rail continue, and several hundred thousand tonnes per year are now making their way south. Figure 1 shows the weighted average sulphur content of US crudes over the past 30 years, and the trend has been an increase of about 50% in spite of the production of lower sulphur tight oil and distillates via fracking. Globally, the same trend is observable, with average sulphur content of crude rising from about 1.1% to

about 1.3% sulphur over the course of this decade. The effect is likely to be greatest in regions like the Middle East and Latin America, which already have to deal with very sour crudes. Heavier and sourer crudes tend to produce a preponderance of heavier distillate, exactly the components demand for which is in decline, and again requiring additional investment.

Meeting sulphur regulations

In order to meet these changing feedstock and product requirements, refiners around the world are engaged in a continual programme of investment in new equipment. For the maritime fuel regulations, the conversion of residual fuels to middle distillates is forecast to lead to an increase in coking, residual hydrocracking, and distillate hydrocracking capacity, as well as greater demand for refinery hydrogen. In the EU, CONCAWE says that coking and hydrocracking capacity has increased by about 35% from 2008-2015, with a 10% decline in load on crude and vacuum distillation units, diesel hydrodesulphurisation and FCCs, and a 50% increase in hydrogen demand. However, additional investment will be needed by 2020 in order to permit the production of sufficient 0.5% sulphur marine fuel, with coking and residue desulphurisation capacity most in demand. CONCAWE estimates that around \$30bn of investment has already been earmarked within the European Union (EU) between 2009 and 2015 to meet increased demand for cleaner fuels. But half as

much again – \$15bn – will be needed in the region to meet the post-2020 demand generated by tougher global and EU regulations on shipping.

Meeting the 10ppm limits for gasoline raises the question of whether to treat the feed before or after the fluid catalytic cracker (FCC). New refineries, especially in Asia, have generally used a catalytic feed hydrotreater (CFHT) unit upstream of the FCC. However, the coking units being installed to cope with lower demand for fuel oil and processing of heavier crudes produce significant amounts of vacuum gas oils which need to be hydrotreated prior to conversion in the FCC, and this can make increased demand of catalytic systems and possibly even require post-treatment.

EU refineries have generally met the 10ppm gasoline limit by moderate severity fluid catalytic cracker (FCC) post-treatment, being influenced by the need to produce higher volumes of diesel to take into account local market preference, and the availability of relatively light and low sulphur North Sea crude.

In the US, the move to 10ppm sulphur is complicated by the use of Canadian syn-crude and has led to a mix of feed pre- and post-treatment for the FCC, and in about one third of cases, both. However, in recent years US refiners have been able to take advantage of low shale gas costs to acquire relatively low cost hydrogen, which makes the economics of hydrodesulphurisation technologies more attractive.

Finally, many Middle Eastern refiner-

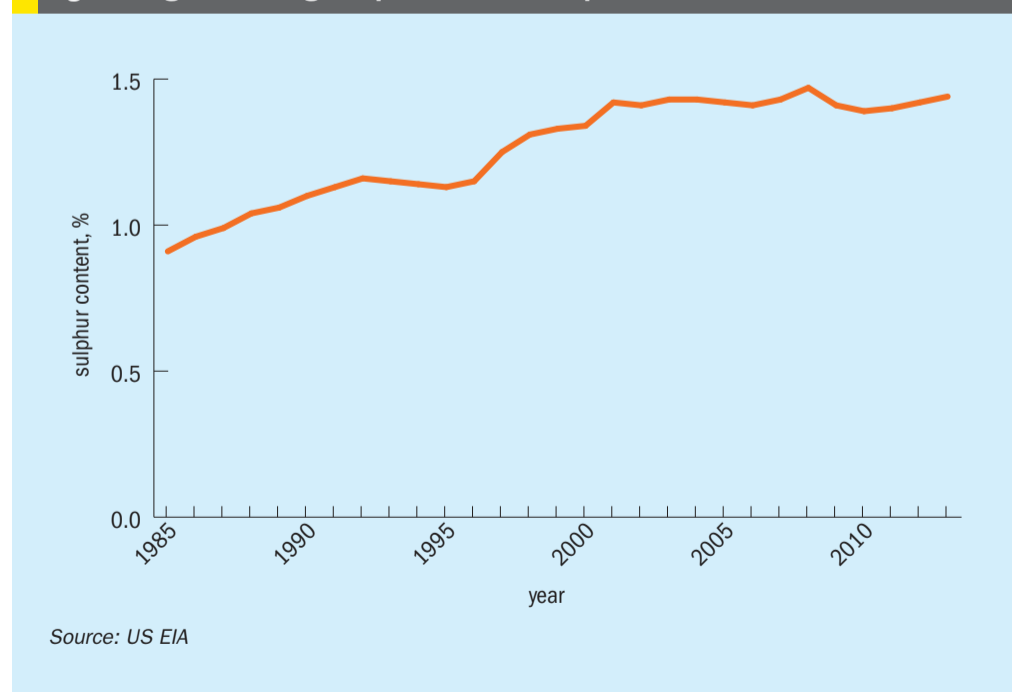
ies are often older and less complex, and have historically had to face generally less stringent standards for sulphur content in fuels, and hence have faced a growing deficit in light and middle distillates, and rising imports of gasoline and diesel. Saudi Arabia had not – until the start-up of the new Aramco refinery at Jubail last year – added any refining capacity since 1990. At the same time, their major markets, such as China and southeast Asia, are moving rapidly towards Euro-4 or 5 fuel standards. This has led to a major new programme of upgrades, revamps and new builds across the region, with 3.3 million bbl/d of capacity being added across Saudi Arabia and the GCC region from 2014-2019.

Overall, the increase in new refining capacity, particularly in China, India and the Middle East, almost all of it geared at meeting Euro-5 fuel sulphur levels, is likely to lead to a surplus in most product areas, and considerable pressure on existing refineries which have not yet made the investments necessary to reach these levels. Older refineries in Europe and the Asia-Pacific region are deemed to be especially vulnerable.

Where will the sulphur go?

Refineries already produce almost half of all elemental sulphur that is extracted worldwide, and as product specifications become more stringent – especially for heavy fuel oils which have traditionally been a ‘sink’ for refinery sulphur – and the average sulphur content of crude oil rises, refiners will inevitably be producing more sulphur. Unlike sour gas producers or processors of oil sands bitumen, who often have the space and volume of recovered sulphur to perform large-scale block pouring operations, refiners must often work in more restricted spaces, close to or within major population centres, with attendant issues relating to emissions, especially of sulphur dioxide or hydrogen sulphide. The old sulphur industry, in North America and Europe, often stored and offloaded the sulphur as a liquid, but new producers and consumers have generally not invested in such capability, and hence formed sulphur is coming to be the default option for refiners, allowing access to a wider international market. This is important since most refiners, as noted, do not have the wherewithal to store large volumes of sulphur for long periods.

Fig 1: Weighted average sulphur content of inputs into US refineries



Analysers and next generation SRU control

Acid gas analysis has arrived and tail gas analysers are now coming into the third generation.

S. Simmonds and **R. Hauer** of AMETEK discuss current trends in SRU process analysers and **A. Henning** of Jacobs reports on experience to date with Advanced Burner Control+ (ABC+) which now includes sour water acid gas (SWAG) control.

The history of on-line process analyt-ics is a relatively short one. Development was driven by the need for process control of high value hydrocarbon-based products. The first on-line analyser applications came during the second war. Rapid development came in the 1970s with the advent of the microprocessor and resultant chemometric techniques. Led by Phillips Petroleum, Union Carbide and Dupont Chemical, amongst others, these initiatives were the antecedents of today's Siemens Applied Automation, ABB Analytics and AMETEK Process Instruments.

Whether or not sulphur can be considered a high value product, in most cases the driving force for process measurement and control of the SRU is largely environmental. The US EPA Clean Air Act of 1970 and the ground-breaking study by Alberta Environment on the capability of the modi-

fied Claus process coincided with the first attempts to control the modified Claus process using an on-line analyser and closed loop control.

The first report of an on-line tail gas analyser was a technical paper based on a gas chromatograph installed at the Dow Chemical Freeport TX (USA) facility in 1970. In 1972 Amoco Oil and Dupont Process Instruments published a paper on an ultraviolet (UV) based tail gas analyser installed at the Amoco Whiting refinery, Indiana, USA. Shortly after this, in 1974 Western Research participated in a pilot study using a prototype UV-based tail gas analyser at the Shell Waterton gas plant in Alberta, Canada.

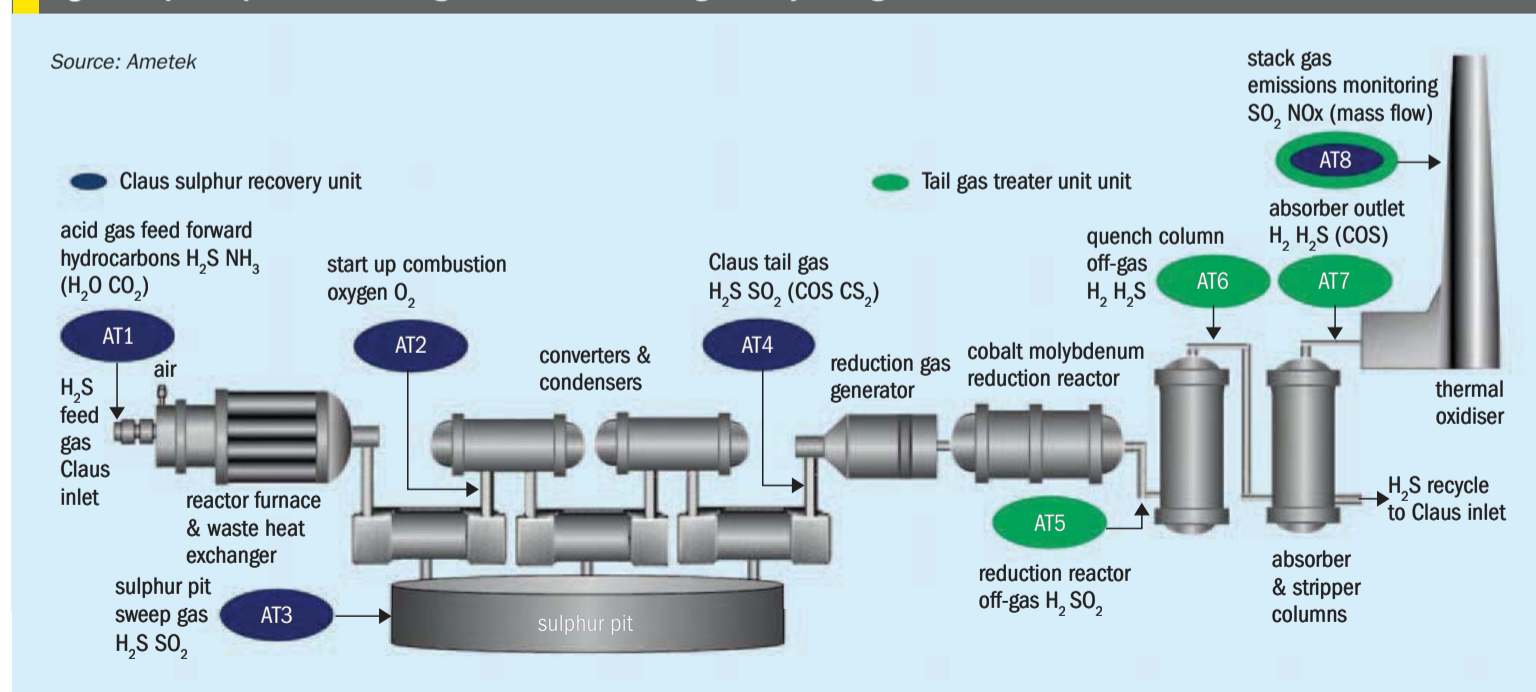
Subsequent developments by various manufacturers were all based on UV spectroscopy and extractive sampling techniques and that remains the case today.

The life cycle of an analyser is expected to be 15-25 years and some analysers can be in the field for 30 years or more if properly protected and maintained. The product life cycle of an analyser is of the same order, 15 years or more and is mostly subject to the obsolescence of the electronic components.

Process gas analysers in SRUs

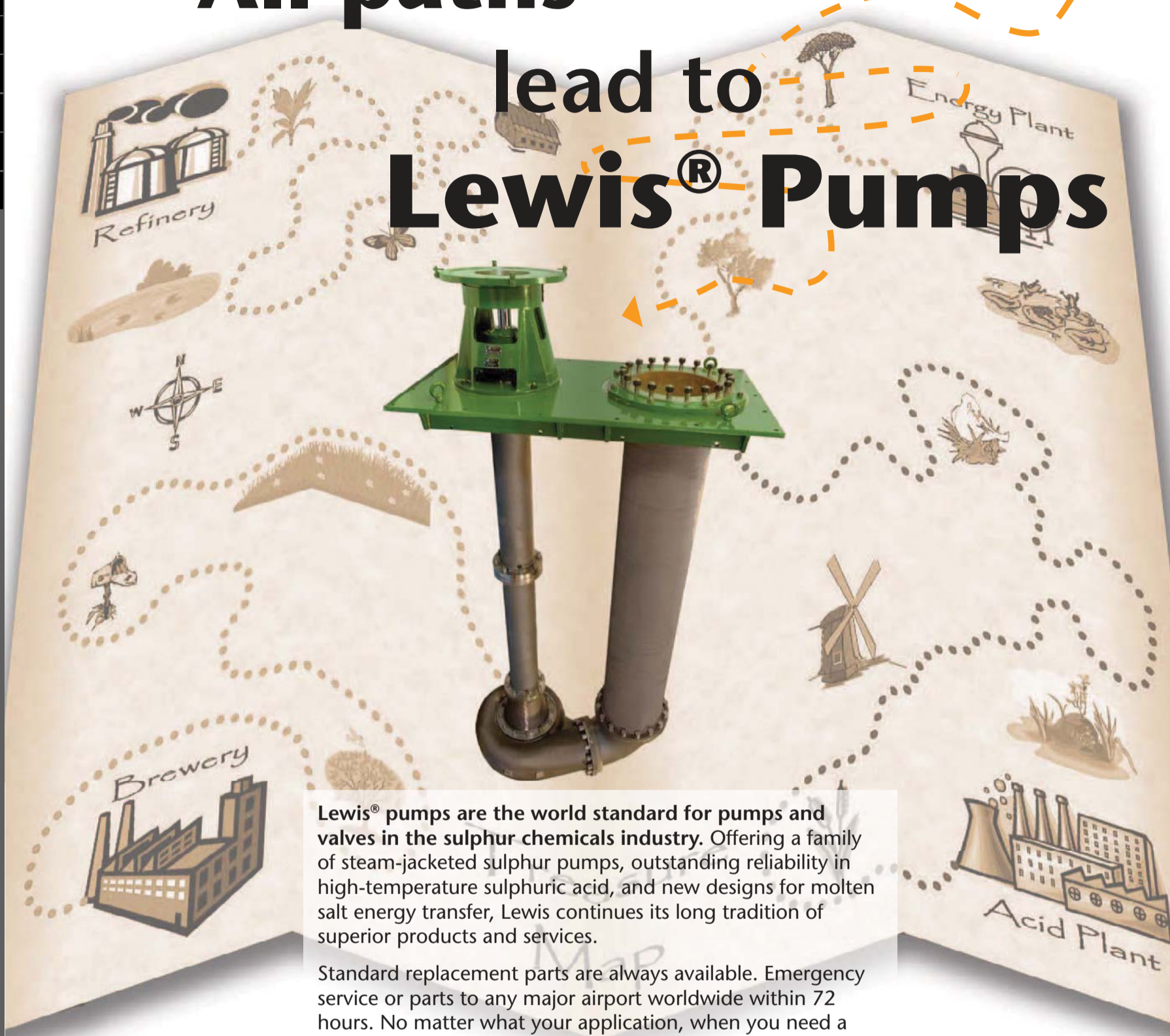
The suite of process gas analysers for a sulphur recovery unit (SRU) can vary from one up to as many as eight analyser tags (see Fig. 1). The (H₂S/SO₂) SRU tail gas analyser is the most common. Every SRU (or enhanced SRU, Superclaus, sub dew-point) will have this critical, primary analytical measurement. If the SRU has an associated amine based TGTU there is a second critical analyser and this analyser can be in one of three locations. The third

Fig 1: Simplified process flow diagram of a SRU showing 8 analyser tags



1	47
2	48
3	49
4	50
5	51
6	52
7	53
8	54
9	55
10	56

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most common analyser tag is the emissions analyser, while not quite yet universal, in some cases (even when an end user is paying for high sulphur efficiency) the governing environmental authorities occasionally do not specifically require an emissions analyser, the emissions analyser is the one measurement that confirms the recovery efficiency on a continuous basis. After these three, the feed gas analyser is becoming more prevalent in front end engineering design.

As the industry drives to tighten control loops and reduce upsets in the process, the analysis of the feed-forward acid gas will become an industry standard. The results are clear that feed-forward control is both practical and worth the (relatively small) capital investment. The industry is still in the education phase and needs to be assured of certain practical implications but the need and benefits of feed-forward control are already well established. If an acid gas analyser has not been included in the FEED, then the design should be revised to allow for simple installation of a feed-forward analyser at a later date by installing the sample tap and associated heated acid gas probe, saving considerable time, effort and cost in the future.

The next most common analyser is the pit gas analyser followed by an analyser to measure the O_2 at the outlet of the waste heat boiler for exact stoichiometric combustion of natural gas during start-up and shutdown.

Tail gas analyser

There is a choice of two tail gas analysers – the “top of the pipe” (close coupled model 888 “no sample line” analyser – see Fig. 2) or the extractive (sample line analyser, model 900 ADA). Over the last five years (~600 tail gas analysers) ~80% of tail analysers for new construction have been top of the pipe (close coupled type). A “sample line” type analyser is selected for three primary reasons; firstly for installation in an analyser house for extreme climates, secondly if the sample point location is compromised due to poor piping design, and finally if the end user wishes to have the additional measurements of COS and CS_2 for process optimisation (only normally required for legacy straight SRUs with no TGTU or for enhanced Claus SRUs).

AMETEK’s tail gas analyser has gone through two full generations and is about to enter a third.

Fig 2: Model 888 tail gas analyser



Source: AMETEK

The first generation analysers proved the measurement to be viable, the second generation analysers improved on sample handling and improved reliability to keep the analyser in cascade control, while the third generation analysers address the common failure modes external to the analyser.

The current design of the close-coupled (“top of the pipe”) tail gas analyser was due for updating which presented the opportunity for some additional features. That said, given the close-coupled analyser has been installed in excess of 1,200 units and the well proven success of the technique, the goal of the update was to be evolutionary not revolutionary. The principal improvements offered in the third generation are;

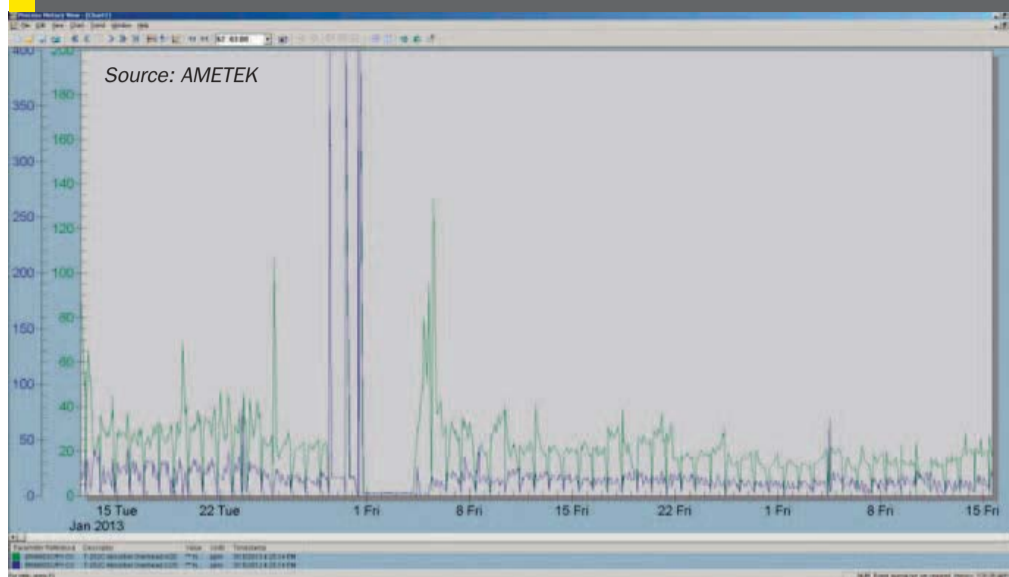
- Flange temperature RTD and alarm: An embedded RTD in the process connection flange provides an alarm for poor quality steam. This advanced warning eliminates unexpected plugging.
- Automatic flow control: This provides a method to automatically adjust the flow rate of the analyser relative to process pressure (especially important if there is entrained sulphur).
- Ambient temperature rating: Improved thermal isolation between the oven and electronics results in an increase in the ambient temperature specification from 50°C to 60°C. This makes installation in regions like the Middle East possible without the need for an air conditioned shelter (with the aforementioned need for protection from solar gain an important detail).
- Double-block isolation from the process: This provides an option for any site specific safety considerations.

TGTU analyser

The sample point location of the TGTU analyser varies depending on who is providing the front end engineering design. In 2002 AMETEK developed a combined ultraviolet (sulphur) and thermal conductivity (H_2) analyser and to date have provided ~180 analysers for various amine based TGTU processes. The measurement of the (excess) hydrogen is the primary measurement in this application. As the H_2 remains essentially unchanged after the CoMo reactor (with a slight increase across the quench tower due to renormalisation after the removal of the water) AMETEK considers any of the three sample points to be non consequential in terms of H_2 measurement based on experience. As to the sulphur gas(es), where and what to measure remains variable according to the designer, the spread of sample point locations are as follows:

- The majority (~150) are at the absorber outlet measuring H_2S and H_2 . Of these 150, ~15 have added the additional analytical capability of COS measurement and it is interesting to note almost all of these have been end users upgrading an old GC and convinced of the utility of the COS measurement. The secondary measurement of the H_2S (after the H_2 measurement) (25-500 ppm) at this location is a measure of the efficiency of the amine absorber and the COS (5-500 ppm) is a measure of the CoMo reactor catalytic activity as illustrated in the process upset in Fig. 3 where the COS momentarily reached 400 ppm during an upset. Both of these sulphur values contribute to SO_2 emissions and can help to isolate the third contribution being sulphur pit sweep gas if vented to the incinerator
- Another ~25 analysers have been installed (further upstream) at the outlet of the quench tower. Here the secondary measurement of (~2.5%) H_2S after the H_2 measurement is a measure of the efficiency of the SRU as it represents all of the sulphur compounds (H_2S , SO_2 , COS, CS_2 , Sv, S liq) leaving the SRU. While the amount of trace COS (5-500 ppm) is the same as the absorber outlet it cannot be effectively measured at this point as it is masked by the %level H_2S
- Approximately five analysers for H_2/SO_2 have been installed further upstream yet, at the inlet to the quench. The util-

Fig 3: COS upset data from TGTU absorber



ity of measuring SO_2 here is to alarm for SO_2 breakthrough from the CoMo reactor before seeing a drop in the pH in the quench water. While this sample point is much less common, one advantage is the analyser can be placed in front of a diverter valve and never be dead legged in the case of TGTU bypass or a sample switching valve for this purpose if the primary sample point is at the absorber (or quench) outlet.

If there is only going to be one analyser (most FEED process packages do this in the interest of cost), AMETEK recommends locating the analyser at the outlet of the absorber. Most importantly add the COS measurement (in addition to the H_2 and H_2S) at this point as the cost is very minimal and of great value when trying to source the cause of a process upset and emission increase. Additionally, if the FEED provides for measurement at two points in the TGTU then add the H_2 measurement to the second point as the cost is again minimal and provides a back up for the primary H_2 measurement which is by far the more important variable. AMETEK has ~20 installations where there are two TGTU analysers as part of the TGTU design most of these being H_2S measurement at both the quench and absorber. Approximately half of these take advantage of adding a second H_2 measurement.

Emissions monitoring analyser

An emissions analyser is normally required as part of the operating permit. Beyond the compliance aspect, mass emission (kg/h SO_2) is a sensible and useful way to evaluate a straight Claus

or enhanced Claus SRU as it can be directly related to sulphur recovery efficiency and used as an optimisation tool. Mass emission requires the SO_2 analytical measurement be made on a "hot-wet" basis because the SO_2 and corresponding velocity measurement must be on the same basis. Mass emission measurement loses some of its utility when there is a TGTU and typically the emissions in these instances are measured on a dry basis and corrected to a zero oxygen base to arrive at standardised measurement. While CO and NO_x are occasionally included as a permit measurement they are not all that common. What is becoming more common is the inclusion of un-oxidised sulphurs (primarily H_2S with some COS and CS_2 , 5-15 ppm total) to be included in the overall SO_2 emissions

Feed (acid) gas analyser

When considering the 'air demand' of the SRU process, primary air control using feed-forward flow ratio of air to acid gas accounts for ~90% of the combustion air. Secondary air control using tail gas analysis (2:1 $\text{H}_2\text{S}/\text{SO}_2$ ratio) controlling a trim air valve by feedback control (or cascading to the feed-forward controller) accounts for the ~10% balance of the required combustion air. Feedback control based on process analysis provides the most precision, however, it is impaired by the ~30 second process lag time, especially if the composition of the acid gas changes rapidly.

Under steady state conditions air control is stable, but in the case of sudden changes in acid gas composition it is not uncommon for air control requirements to exceed the ability of the feedback control loop. In these cases the excursion from the set point of a 2:1 $\text{H}_2\text{S}/\text{SO}_2$ ratio can be extreme, and the cause of a serious loss of recovery efficiency and increased emissions.

With advances in certain technology and process control, the implementation of a feed-forward analyser for real time acid gas analysis has been seen with increasing regularity, particularly on new process projects. The advanced burner control system ABC+ for feed-forward control developed by Jacobs Comprimo® Sulfur Solutions has seen a big increase in uptake in 2014. ABC+ uses real time process analysis of the acid gas to provide information to the closed-loop control.

AMETEK have supplied nine analysers for Superclaus ABC+ projects and an additional 40 analysers for straight SRUs and

Fig 4: IPS-4 acid gas analyser (open)

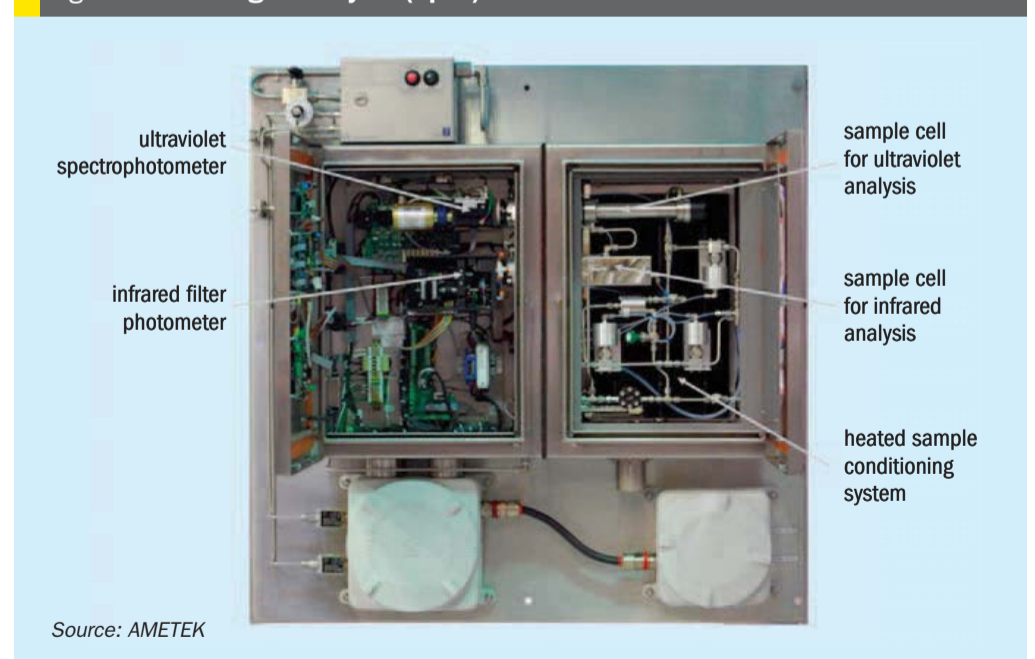
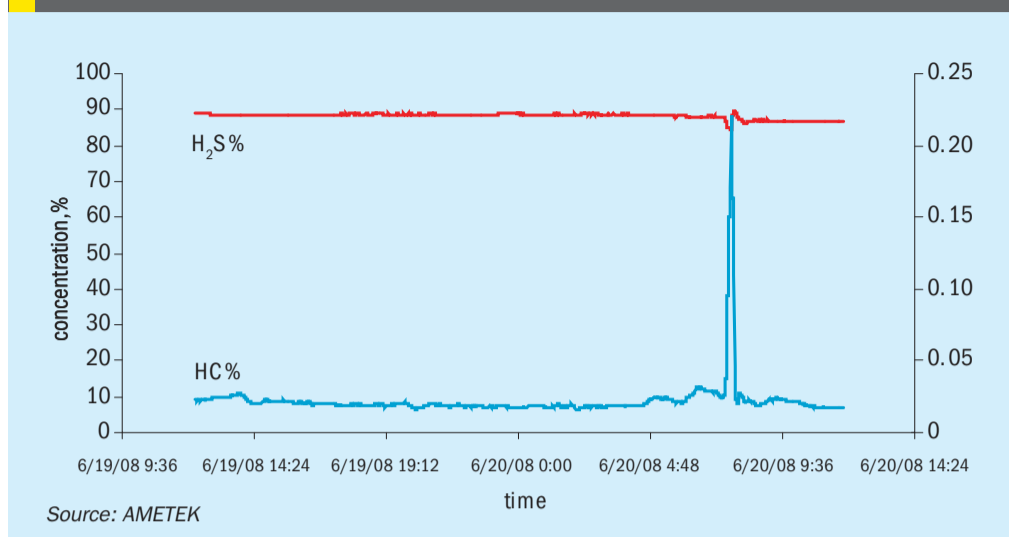


Fig 5: Capture of transient HC variation



SRU-TGTU applications. Figure 4 shows AMETEK's model IPS-4 acid gas analyser and Fig. 5 shows the analyser results for a one time, sudden increase in total hydrocarbons (HC) from 0.02 to 0.25%.

While an easy case can be made to have an acid gas analyser for a gas processing SRU where variable composition in the H₂S content in the acid gas requires feed forward control and the addition of total HC can also be utilised, there is also compelling reason to add a feed gas analyser for an SRU followed by a TGTU. The problem for a TGTU is not when there is a sudden increase in HC (resulting in a dramatic rise in H₂S going to the TGTU) but the resultant dramatic rise in SO₂ going into the TGTU when the HC event is over. Additional work is being conducted to quantify BTEX in amine acid gas for SRUs related to gas processing where these components require co-firing of natural gas.

Pit gas analyser

Hydrogen sulphide exists in sulphur as dissolved H₂S and chemically bound hydrogen polysulphides. The liquid sulphur produced from Claus SRU typically contains a total of 200-350 ppm of dissolved H₂S. Spontaneous or active degassing of the sulphur results in H₂S accumulates in the gas space above the liquid sulphur. H₂S becomes progressively more dangerous as the levels incurred in handling and moving of the sulphur increases above toxic limits (70 ppm), becoming lethal at 600 ppm and reaching the lower explosive limit at ~3.25%. A measurement tag for H₂S and SO₂ in pit gas is sometimes required for certain degassing licences or at locations where there has been an explosion incident. Where there has been an incident there

are records of an explosion occurring at H₂S values well below the 3.25% LEL. AMETEK has been in discussion with Black and Veatch on this subject. Based on CFD modelling Black and Veatch arrive at the conclusion H₂S can exist in pockets and stagnant areas at 16 times the mean value as measured in the sweep gas header meaning 2,000 ppm could constitute a LEL condition somewhere in the pit. There is continuous analyser data to corroborate that where an explosion occurred when there was a measured level of 4,000 ppm H₂S in the sweep gas header. Black and Veatch have asked if the sulphur pit (tank) can be measured at several points and while this is not practical for several reasons it does raise the question as to what level of H₂S constitute an alarm condition, to evaluate the gas flow in the head space and for operations to fully understand what constitutes a hazardous condition. Similarly, events of 3% SO₂ in the pit sweep header have been recorded which clearly indicates a sulphur fire where the condition prevailed for weeks without intervention, again measured values outside of the norm must be heeded.

Process O₂ for start up / shut down

This measurement is made during start-up and shut-down, any time a sulphur recovery unit (SRU) transitions from ambient temperature through natural gas warm up to the introduction of acid gas. The measurement of O₂ stoichiometry is critical in order to maintain a slight excess of oxygen. Historically operators have manually taken samples using a portable electro-chemical type O₂ analyser or even mounting a "sacrificial" analyser that is discarded after start up. While giving more or less satisfactory

results, the requirement for more stringent operating limits, hazard exposure and the non-continuous nature of portable grab sampling are reasons to consider a permanent solution. A fixed system that draws a continuous sample during the operational transition period, without intervention from operations or analyser maintenance can be easily implemented. The O₂ measurement is based on "tunable diode laser (TDL) which is non contact type of analyser. The sample system is the advanced sulphur removal (ASR) probe used on the 900 ADA tail gas analyser, the analyser and sample system are maintained at 150°C so the analyser can be left on-line as the analyser transitions from warm up to acid gas mode.

Industry survey

The analytical instrumentation on a SRU plant is a highly specialised field of such critical operational performance that knowledge and experience are essential to success. In a recent wide-ranging analyser survey, with questions relating to reliability, on-line control and best practices, professionals from within the sulphur recovery industry were surveyed in order to gauge the level of understanding of the needs of the analyser application and to obtain valuable user related experiences. Three distinct stakeholder groups were surveyed who are directly involved in the purchase and maintenance of SRU process gas analysers:

- front end engineering design and start up engineers;
- operations;
- end user analyser engineers and technicians.

Reliability

The survey confirmed that the industry considers tail gas analysers to be reliable.

Trouble-shooting an analyser for sudden failure of an electronic problem is not difficult and a large refinery or gas plant complex has access to adequate skill levels for this. Specialist help is required where the problem is intermittent or electronic component failure is occurring on a frequent basis.

The primary factor affecting reliability of the tail gas analyser is heat integrity at the process connection and the second leading cause is inadequate design of the analyser shelter.

Best practices for heat integrity include:

- The process nozzle should be 150mm

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(6") or less, terminating in a 2" 150 lb raised face flange (maximum).

- For top of the pipe type analysers the vendor must supply the steam jacketed ball valve. The steam needs to be of medium pressure (nominal 7 to 10 barg / 100 - 150 psig) as low pressure steam is, in practice, often wet and unsuitable.
- Use Contra-Trace™ or Contra-Heat™ to overcome any shortcomings. Do not allow the practice of wrapping steam trace tubing and insulation around a problem and thereby masking that problem.
- For sample line analysers, if possible install the analyser above the sample point and in all cases request the analyser vendor confirm the length from on-site measurements and/or isometric drawings. When sample lines are estimated and then the numbers flow through more than one set of hands, the probability the lines will be too long, is almost a certainty.

Best practices for analyser shelters include:

- Provide an adequate shelter that shades the analyser from direct sun at all times. The shelter is no different than what one would see over a local panel, quite inexpensive and almost always neglected at the EPC detailed design stage.
- Involve the analyser vendor at an early stage preferably at frontend engineering design. As end-user insist upon it and continue the dialogue into the detailed design stage. Be very wary of surrendering all analyser design to a systems integrator.

Sample point selection for both tail gas and feed gas analysers is more often than not compromised at the piping engineering stage. It is the lack of analyser specialists at the detailed engineering stage that is the root cause. A constricted and compromised sample point location can affect analyser operability which in itself is bad enough. The problem can extend to safety concerns at HazOp review, requiring an expensive piping change to make it right.

Start-up and turndown

Slow response is sometimes attributed to the analyser at turndown of the SRU when in fact it is process lag time. Although asked frequently to help configure control loops this is typically not a core competency of analyser companies and more the

domain of the FEED and EPC control engineering team.

Reported analyser problems after start-up can often be related to the phenomenon of the Claus catalyst preferentially absorbing H₂S over SO₂ with the result producing values that do not represent the true air control situation. The AMETEK guideline (provided by Sulphur Experts test crews) is that the analyser should only be used for control some eight hours after start-up, perhaps more depending on individual plant conditions.

Acid gas analysis feed-forward control

From the survey it was clear that all respondents saw the benefits of feed-forward control but there were also many misconceptions that should be addressed.

Firstly, respondents were unclear whether feed-forward control can be used in the cascade control loop but from field experience this is exactly the intent. Rapid compositional analysis of the hydrogen sulphide, and a total hydrocarbon measurement provide the air demand components with the addition of a carbon dioxide and water measurement (and in the case of sour water acid gas, ammonia) provide full acid gas composition details.

Secondly, there was clear concern as to whether the information is fast enough for feed forward control, but again this is exactly the intent of these control designs. With the use of real time spectroscopic technology (a combination of both ultraviolet and infrared) the system is able to provide useful data rapidly.

Survey conclusions

The key conclusions of the survey were:

- For large scale project where a systems integrator is involved, ensure close management of the project from field proven end-user analyser technicians, in combination with close communication to the key SRU analyser vendor.
- Ensure that the SRU analyser vendor is involved in the entire project process from the early stages of the front end engineering design (FEED) right through the detailed design phase.
- A successful SRU analyser start-up requires careful thought and planning. The end-user technicians (or alternatively the contractor analyser technicians) need to be factory-trained and authorised to work on the respective analysers, alternatively the analyser vendor must be at site for start-up.

Improved Advanced Burner Control⁺ (ABC⁺)

Since Jacobs Comprimo® Sulfur Solutions (JCSS) first introduced ABC⁺ in 2008, the developments of this SRU burner control concept and the increase in the number of implemented systems have been significant with nine ABC⁺ references running and several more in different design phases.

Following the success of ABC⁺ for feed gas analysis of amine acid gas, Jacobs took the next step to include both amine acid gas (AAG) and sour water acid gas (SWAG) control.

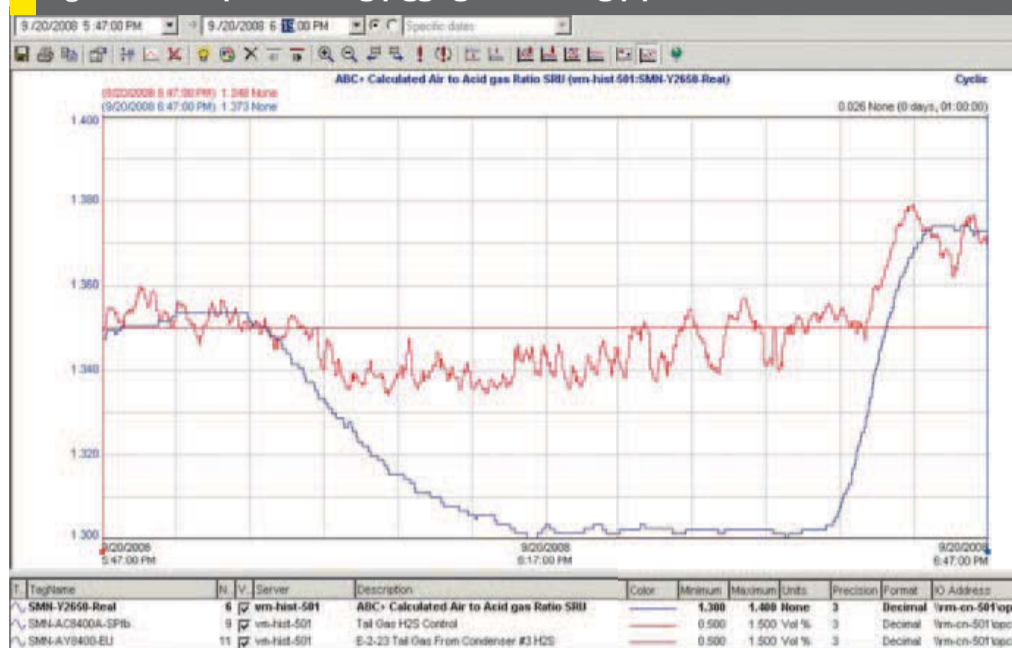
Sour water acid gas differs from other acid gases mainly because it contains ammonia. Further, the hydrocarbon content is often higher and more diverse than in amine acid gas. Luckily, ammonia can be rapidly measured by the combined UV/IR unit that is part of ABC⁺. Together with the molecular weight determination (that comes as part of the ABC⁺ analytics), a robust control is offered.

Conventional SRU control and ABC

Both ABC and conventional SRU control manage feed gas flow rate variations via feed-forward ratio control, but not in exactly the same way. Conventional SRU control uses the main and trim air valves fully independently; the main air valve is controlled in direct ratio to the SRU feed gas flow, while the trim air valve is only controlled by the tail gas quality controller. The downsides of this strategy, such as slow and inaccurate feed gas ratio control via the main air valve and limited tail gas quality control capacity via the trim air valve, are overcome by the different strategy which ABC applies. ABC controls the main and trim air valves simultaneously and utilises each valve's specific strength in the best manner; it provides rapid and accurate total air response to any change in sour gas feed flow rate combined with enough tail gas quality control capacity. While the total air flow controller manipulates the small (thus rapid) and accurate trim air valve in response to any air demand change, the main air valve is continuously adjusted to ensure that the trim air valve stays within its operating capacity. Shortly after that response has been provided, the trim air valve returns to its optimal position to wait for new air demand changes.

If no measures are taken, as in conventional SRU control, the air flow response is

Fig 6: ABC+ response during pigging of incoming pipeline



non-linear in an SRU, especially for main air control. ABC takes this into account using proprietary functions to establish linear responses at any plant load, and repositions the trim air valve to its optimal position. Where conventional SRU control uses separate main and trim air flow transmitters, ABC utilises only a common air flow transmitter, thereby reducing engineering, hardware and maintenance costs, while providing a more accurate combustion air flow. ABC is superior to conventional SRU control in maximising sulphur recovery efficiency (SRE) by optimising tail gas quality control. For this reason all JCSS SUPERCLAUS® processes (currently more than 190 installed worldwide) were equipped with ABC in the past.

ABC+

Although ABC is an important improvement over conventional SRU control it still lacks the ability to react to rapid changes in feed gas composition. ABC+ addresses this problem by adding a feed gas analyser with the related calculating functions to the existing control concept. This revolutionary development determines and uses the dynamic air to feed gas ratio and improves the existing feed gas flow compensation by adding mole weight compensation to precisely provide the required air supply to the SRU main burner. As a result, the combustion air to the SRU main burner is continuously adapted based on all possible changes the feed gas can encounter, such as flow, temperature, pressure and composition changes. This results in a

more accurate air to feed gas ratio control and improved SRU control robustness.

ABC+ at Keyera Simonette gas plant

The first ABC+ unit was installed at Keyera Simonette gas plant in Canada in 2008 (see *Sulphur* no. 326, pp.49-52) and this set-up has been running successfully ever since.

The Keyera Simonette gas plant receives raw natural gas with varying sour gas content due to a network of various wells, pipelines and compressor stations in the wide neighbourhood. Pigging of the incoming pipelines to get rid of liquid and other impurities is required on a regular basis, and is known to cause a considerable swing for the operation of the associated SRU in terms of feed flow as well as composition. In the past, this phenomenon caused frequent selective oxidation reactor bypass trips resulting in SRE losses and other unwanted consequences, such as non-compliance with regulatory SO₂ emission requirements. DCS operators were unable to efficiently determine the precise timing or the amplitude of the upset to be expected, and thus could not take countermeasures. This was due to the fact that tail gas quality control, as part of the implemented ABC, only starts to act slowly at the moment the upset reaches the back of the SRU, which happens several minutes after misoperation started in the SRU main burner. Figure 6 shows ABC+ controlling the tail gas concentration (H₂S to the selective oxidation reactor) which is driven by the rapidly changing air to AAG ratio caused by first stopping

and then starting the natural gas supply from the pipeline being pigged. The pigging process disturbances used to cause frustration and required lots of operator attention to resume normal operation as soon as possible after completion. Implementing ABC+ transformed the pigging process disturbances into a non-event, allowing the operator to divide his attention evenly over the whole plant.

From the trend it can be seen that the moving red line representing the H₂S concentration in the tail gas with a range of 0.5 to 1.5 vol-%, stays very close to the straight red line representing the set-point at 0.75 vol-%, even though the blue air to AAG ratio line is changing significantly and rapidly. It should also be noted that the time involved in the process to shut-off the incoming pipeline is quite different than the time required to open it again (24 minutes versus 6 minutes); during both events ABC+ managed to control the tail gas quality, without initiating a high or low DCS alarm, let alone activating the high high H₂S trip.

During the commissioning period, the AAG ABC+ online % score was found to be just above 85%, mostly due to some small issues and the operators learning curve. After commissioning, a two year period followed with high scores (above 98%). Since that time, there have been a couple of dips which can be explained by some provisional repairs and the associated lead time for analyser replacement parts. After the replacement parts for the analyser had been installed, the high scores experienced during the first two years of operation returned.

Improved ABC+ hardware concept

Shortly after the successful start-up of ABC+ at Keyera Simonette gas plant JCSS decided to add ABC+ to its portfolio, aiming to deliver this concept to owners of SRUs globally. While the idea of how to set-up such a service slowly took shape, the search for an analytical partner with the right qualifications also began. A vendor within JCSS' existing network of known vendors, conveniently located nearby JCSS' main office in Leiden, was approached and preliminary discussions started. Analytical Solutions and Products B.V. (ASaP) in Amsterdam proved to be a company with a flexible, dedicated and experienced team, supplying practical solutions for analytical needs, and locally rep-

Fig 7: ABC+ hardware at ASaP workshop



Fig 8: Sample probe cabinet site installation



representing a broad range of world-renowned vendors of commonly applied brands in the analytical industry. Quickly it became clear that the opportunities and the technological challenges of designing and delivering ABC+ globally created a nice fit between both companies; ASaP providing the ABC+ hardware and JCSS providing the ABC+ engineering services. JCSS and ASaP signed a co-operation agreement at the end of 2010 to collaborate on the technical development of a generic ABC+ hardware concept for SRU feed gas measurement that would meet the requirements of ABC+ on JCSS projects. At the time the agreement was signed, the ABC+ hardware development process had already been in progress for more than a year, and was further accelerated due to the award of one AAG and one SWAG ABC+ system for a client that owns a refinery in Wales as part of the awarded SRU retrofit project.

Having the engineering, start-up and operating experience from a successful AAG composition measurement in the ABC+ installation at Keyera Simonette Gas Plant, JCSS' intention for an improved ABC+ hardware concept was clear. Together with ASaP a clean start was made. All features working well (proven features) based on Keyera's AAG composition measurement were incorporated and the improvement/upgrade process was set in motion (where needed) to design a generic concept for both AAG and SWAG composition measurement. Since SWAG composition measurement requires addressing more stringent design considerations (e.g. contamination prevention, required material quality) than other measurements, the design needed

to be solely based on parts with proven usage track records. The global sulphur community already knows all too well what problems can arise from 'simple' SWAG measurements in an SRU such as flow, temperature and pressure.

In the process that followed, a considerable number of specialists from JCSS, ASaP and external sources were consulted for specific input into the design. The heart of the sample system was extensively tested and optimised for speedy sample transport. Multiple discussions were held with vendors of SRU feed gas analysers, such as AMETEK (IPS4 Dual Bench analyser) and ABB (Multiwave PIR3502 analyser), to ensure proper system integration with the rest of the ABC+ hardware. This resulted in an improved ABC+ hardware design consisting of the following main equipment components shown in Figs 7 and 8:

- sample probe cabinet, to be connected onto the process nozzle;
- sample tube bundle, routing sample gas back and forth from the sample probe cabinet to the sample conditioning system in the analyser walk-in shelter;
- sample conditioning system, directly next to and connected to the sour gas feed analyser;
- utility panel;
- sour gas feed analyser;
- analyser walk-in cabinet, housing the utility panel and the sample conditioning system, as well as the sour gas feed analyser for one or multiple ABC+ system(s);
- PLC cabinet, located in the auxiliary room.

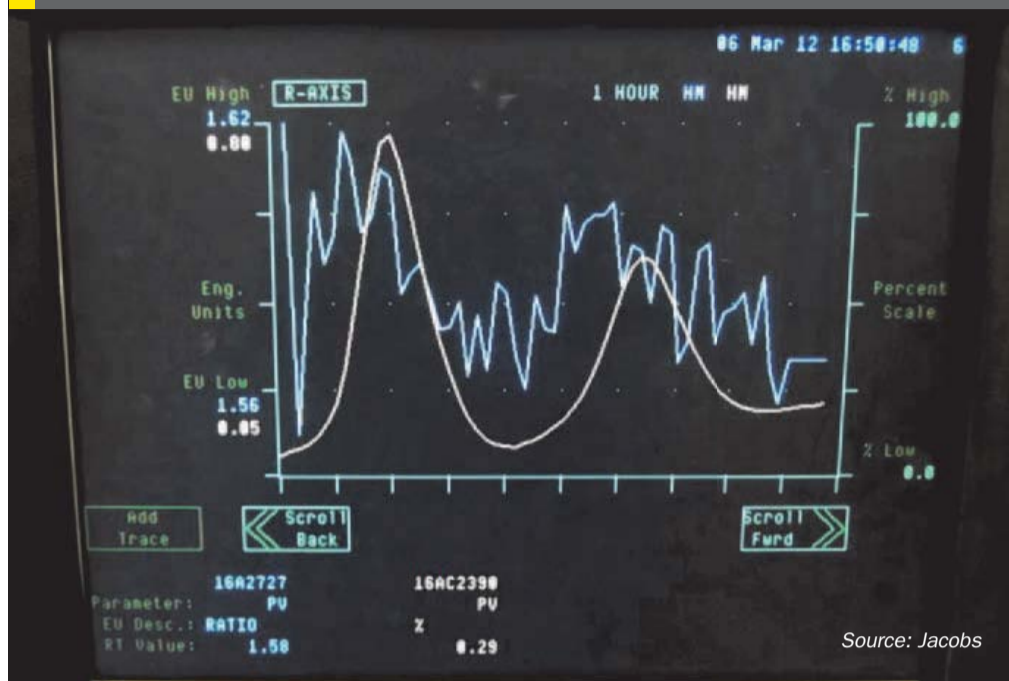
Some highlights of the improved ABC+ hardware design are:

- steam jacketed process nozzle & sample probe;
- all extracted sample gas is returned and processed by the SRU;
- fast sample transport including analytical response (<5 seconds);
- high degree of sample gas monitoring, control and safety by a dedicated PLC. The PLC cabinet includes a local human machine interface (HMI) for easy maintenance access;
- orbital welding, non-traditional fittings of wetted parts;
- steam/condensate cleaning of wetted parts is possible without opening equipment;
- high temperature of complete sample loop to prevent internal contamination;
- special heater and control design to maintain high temperature for T3 (<200°C) classified zones;
- redundant dual sample gas filters (coarse & fine particles);
- manual and scheduled auto-zero calibration of analyser;
- verification of analyser readings using calibrated test gas;
- generic design, which allows focus on 'specials' and potential improvements.

AAG and SWAG ABC+ in a Welsh refinery

The SRU retrofit project in the Welsh refinery included various hardware modifications, such as implementing EURO-CLAUS® technology to improve SRE to at

Fig 9: Calculated air to AAG ratio vs SRU 2 tail gas H₂S concentration



could only run in ABC mode. Restart of SRU 1 followed shortly thereafter, and eventually in March 2012 AAG ABC+ was commissioned. Initially, during commissioning, the calculated air to AAG ratio based on measurements from the AAG feed analyser was not used actively in the control scheme, but the signal was available in the DCS for monitoring purposes.

During those days of stabilisation, monitoring, etc. a huge difference in timing of measurements used within ABC+ was clearly visible, as shown in Fig. 9 below. Figure 9 displays the calculated air to AAG ratio (vivid light blue line, tag 16A2727) in the same trend as the tail gas H₂S of SRU 2 (smooth yellow line, tag 16AC2390). While the amplitude and frequency of both variables match pretty well, there is a timing shift of around 6-7 minutes which is purely due to the residence time of the SRU sections in between the AAG feed measurement and the tail gas analyser downstream the hydrogenation reactor. Switching from AAG ABC mode to AAG ABC+ mode:

- would initiate using information for SRU control that is available 6-7 minutes earlier than what is normally the case;
- would use this early information in feed-forward control, we expect to see a much more stable tail gas H₂S concentration since the amplitude and frequency of the signals match quite well;
- would speed up control response significantly since feed-forward control is much quicker than tail gas quality control; the latter, by definition, is tuned very slowly to avoid unstable control.

The expectations were met in reality when AAG ABC+ was activated for SRUs 1 and

least 99.2% and implementing ABC+ to maintain this SRE during day-to-day operation, despite expected fluctuations in SRU feed gas flow, pressure, temperature and composition. The ABC+ system installed in SRU 1 and 2 controls the H₂S quality of each tail gas and also includes other control features. Both SRUs process around 70 t/d normally and each SRU consists of a thermal reactor followed by two Claus reactors and a hydrogenation reactor. The tail gas from the hydrogenation reactors is routed to a common selective oxidation reactor. The thermal reactors are fed with AAG from two parallel amine regeneration units (ARU) and SWAG from a sour water stripper unit (SWSU) fed by a fluid catalytic cracker unit (FCCU). An additional tie-in is provided for a future connection for SWAG from an SWSU fed by a vacuum distillation

unit (VDU). Fuel gas is used during start-up, stopping and co-firing of the thermal reactors when and if required, and oxygen enrichment up to 28% can be applied to boost the thermal reactor combustion temperatures up to the level required for proper ammonia destruction. Although the oxygen enrichment replaces the existing feature to increase thermal reactor combustion temperatures by AAG split flow control, the ABC+ system still includes this control option as a back-up, in case the completely new oxygen enrichment system is not available.

After the project modifications were partly implemented, SRU 2 was restarted in Claus mode, bypassing the yet unfinished common selective oxidation reactor. Since not all ABC+ hardware had been installed yet, AAG control

Fig 10: AAG ABC+ online % SRU 1 refinery in Wales

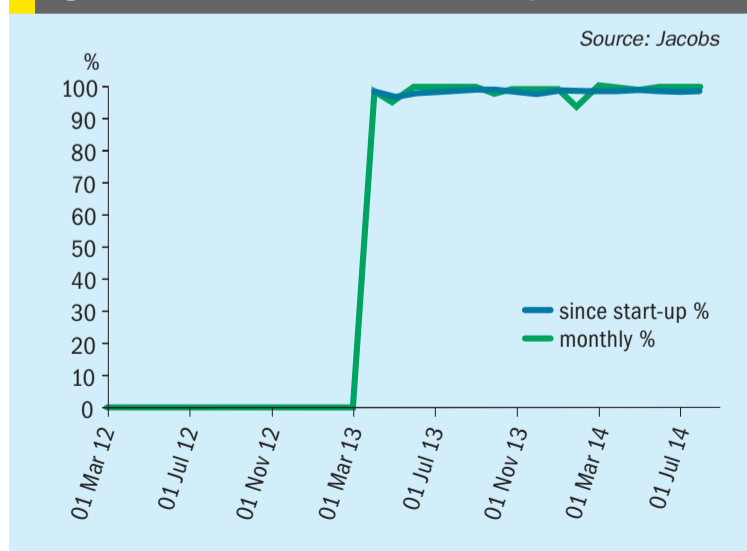


Fig 11: SWAG ABC+ online % SRU 1 refinery in Wales

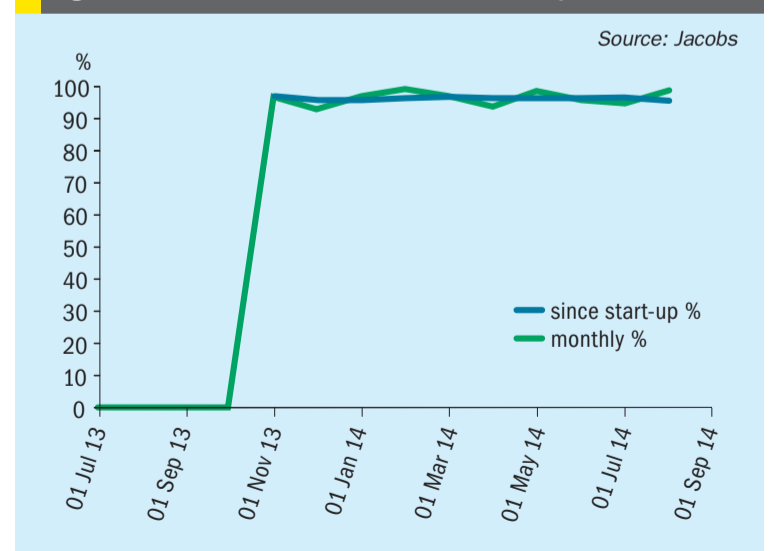


Fig 12: AAG feed concentrations SRU 1 refinery in Wales

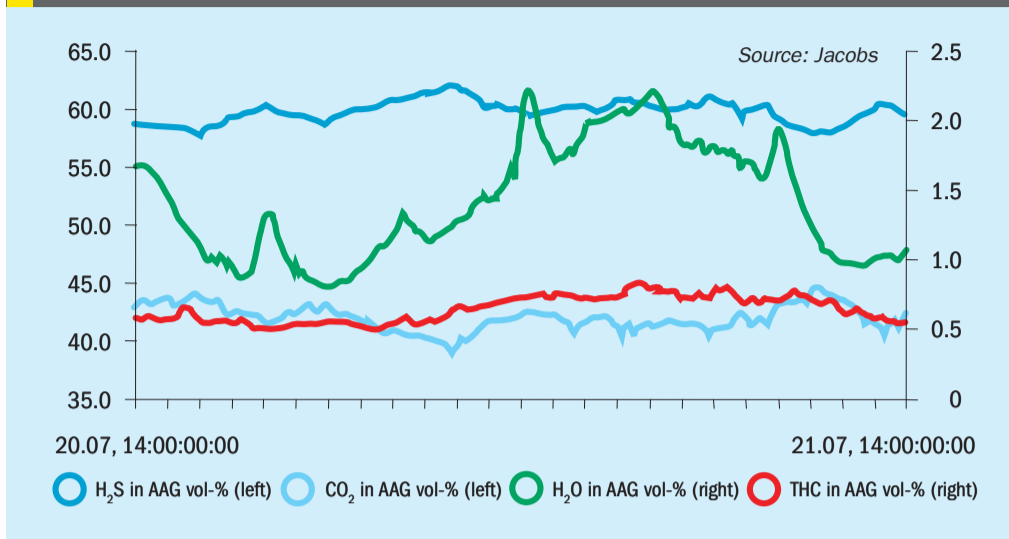


Fig 13: SWAG feed concentrations SRU 1 refinery in Wales

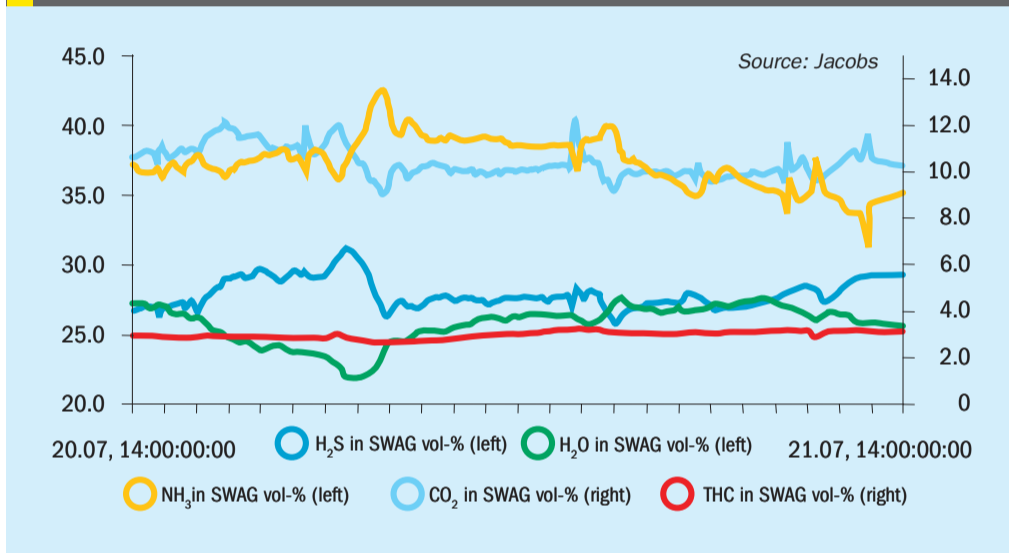
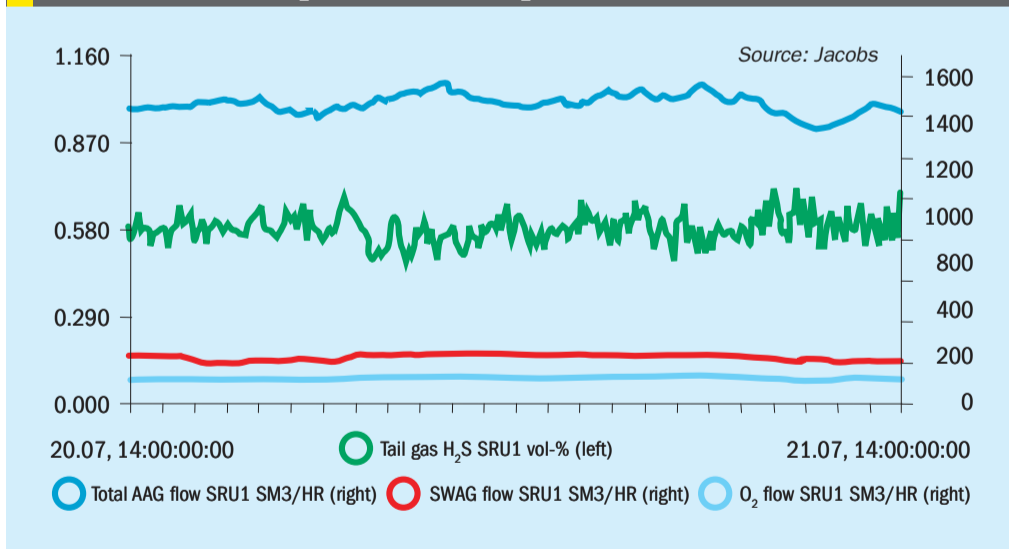


Fig 14: AAG, SWAG, O₂ flows & tail gas H₂S concentration SRU 1 refinery in Wales



2, showing good tail gas H₂S control at set-point +/- 0.05 vol%. In the meantime, AAG ABC⁺ has been running for practically 3 years with excellent online % score, as reflected in Fig. 10, AAG ABC⁺ online % SRU 1. Note that the monthly AAG ABC⁺

online % SRU 1 data prior to April 2013 was unfortunately not stored and therefore not available for publication. However the accumulated AAG ABC⁺ online % SRU 1 up April 2013 ('Since start-up %') score is impressive being 98%.

After some delays, due to a problematic start-up of the oxygen supply unit, the SWAG feed, and therefore SWAG ABC⁺, could eventually be started-up in July 2013 for SRU 1 and 2. The performance results are presented in Fig. 11, SWAG ABC⁺ online % SRU 1.

Note that SWAG ABC⁺ online % SRU 1 data prior to November 2013 was unfortunately not stored and therefore not available for publication. However the accumulated SWAG ABC⁺ online % SRU 1 up November 2013 ('Since start-up %') score is good being 96.2%, considering it includes the period right after commissioning of the first SWAG ABC⁺ system worldwide.

Finally, Figs 12-14 show three trends indicating some important SRU 1 operating variables on a recent day of operation (July 20th 2014), where SRU 1 is processing AAG and SWAG feed while using oxygen enrichment. The variations measured by both AAG and SWAG feed analysers are indicated in Figs 12 and 13, followed by the variations in AAG, SWAG and O₂ flow in SRU 1 and also the tail gas H₂S concentration as main controlled variable. For each trend line reference is made to the associated left or right axis. Despite all sorts of fluctuations in AAG and SWAG feed composition and flow ABC⁺ still manages to maintain the tail gas H₂S concentration in a limited bandwidth around the optimum tail gas operating value (set-point) of 0.58 vol% H₂S.

ABC⁺ looking ahead

In the near future JCSS wants to further improve the concept of executing ABC⁺ projects mainly project organisation-wise and, where possible, also on a technical level. ABC⁺ enables JCSS to offer a fit-for-purpose solution for SRU operators worldwide owning gas plants and refineries that have difficulties to meet stringent regulatory SO₂ emissions on a daily basis. An ABC⁺ implementation is flexible and is suitable for upgrade to an existing SRU or as part of a completely new SRU installation.

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A better picture of working catalysts

From nano-scale studies of working sulphuric acid catalysts using new advanced in situ techniques, to improved industrial-scale sulphuric acid production, **K. Christensen, F. Cavalca, P. Beato** and **S. Helveg** of Haldor Topsoe present unprecedented new insight into the mechanisms of sulphuric acid catalysts.

The main purpose of a catalyst is to increase the rate of chemical reactions without being consumed. This is also the main function of the V_2O_5 -based sulphuric acid catalyst, which has been used for more than 50 years for SO_2 oxidation in sulphuric acid production:



In general, however, a catalyst is also a dynamic system for which the detailed chemical composition and nano-scale structure depend on the composition, temperature and pressure of the surrounding gas. As a consequence, catalyst nature and structure are different in the individual beds of a sulphuric acid converter, even if the same catalyst type were loaded in all beds. One implication of this is the different colours of the operating catalyst that may also sometimes be observed after a shutdown depending on the shutdown procedure. Another effect of the dynamic nature of the catalyst is that after a change of feed gas composition, plant load or bed temperatures, the catalyst will adjust to the new local conditions on a time scale from minutes to a couple of days depending primarily on the temperature.

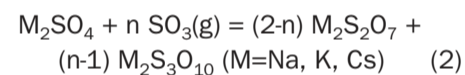
As a catalyst producer, Topsoe continuously aims to improve the fundamental understanding of catalysis in industrial environments and to apply this knowledge to rationally develop better catalysts for improved efficiency of industrial sulphuric acid production. In this article, Topsoe presents results from novel advanced in situ techniques recently introduced in its laboratories and combines the findings with bench-scale investigations and large-scale observations from industrial plants to obtain a more complete picture of the working catalyst.

Background

Commercial sulphuric acid catalysts are based on V_2O_5 dissolved in alkali-metal pyrosulphates on an inactive porous silica support. The carrier is usually made from diatomaceous earth, and the catalyst typically contains 2-5 wt-% vanadium and 2-5 moles of alkali metal promoter per mole of vanadium. The alkali promoters are typically potassium, sodium and cesium, and the catalysts are available as cylindrical pellets, cylindrical rings or finned rings resembling a Daisy as shown in Fig. 1.

Sulphuric acid catalysts are so-called

supported liquid phase (SLP) catalysts, where the oxidation of SO_2 takes place as a homogeneous reaction in a liquid film covering the internal surface of the support material^{1, 2}. The promoting action of the alkali metals is commonly attributed to their ability to form relatively low temperature-melting pyrosulphates, which dissolve the vanadium oxides:



Under operating conditions, the sulphur oxides are bound as alkali metal pyrosulphates, but if the catalyst is blown with hot air for a sufficiently long time, up to 10% of the catalyst weight is desorbed as SO_2/SO_3 leaving the alkali metals in the form of sulphates, M_2SO_4 . The catalytic activity of the melt was demonstrated conclusively in 1948 by Topsoe and Nielsen³, who obtained a high degree of conversion to SO_3 by bubbling SO_2 and oxygen through a column packed with raschig rings and containing molten potassium pyrosulphate in which 14% wt-% V_2O_5 was dissolved. Since then, numerous investigations on the coordination chemistry of the catalytic melt and the reaction mechanism have been published, but in spite of this the reaction mechanism is not understood in detail yet.

For many years, the mechanism was assumed to include reduction of vanadium to V(IV) and re-oxidation to V(V) by oxygen as proposed by among others Mars and Maessen⁴. However, in 1968-1973 Borekov and coworkers^{5,6} found from transient kinetic studies a number of facts contradicting previously suggested redox mechanisms, for example that the rate of SO_2 oxidation is much higher than the rate of V reduction. Numerous subsequent studies have con-

Fig 1: Topsoe Daisy shape sulphuric acid catalyst



Fig 2: The catalytic cycle of a sulphuric acid catalyst

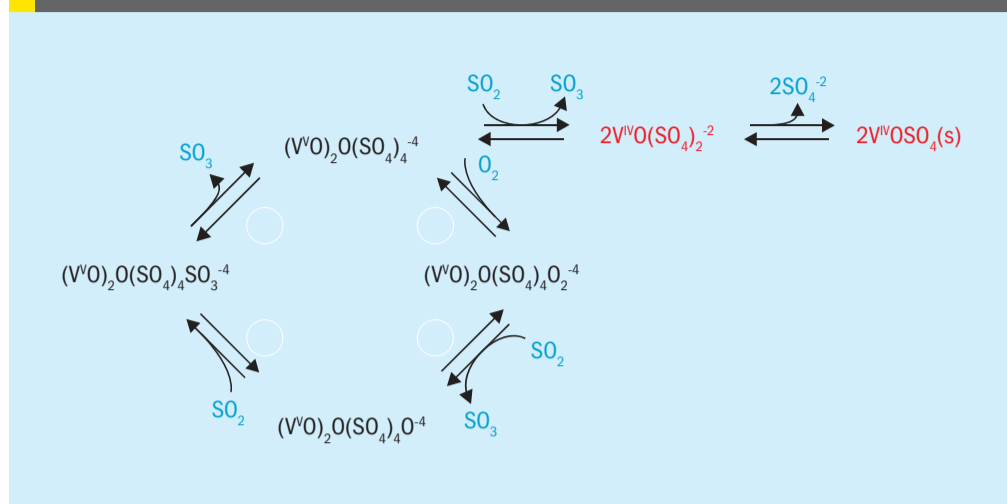
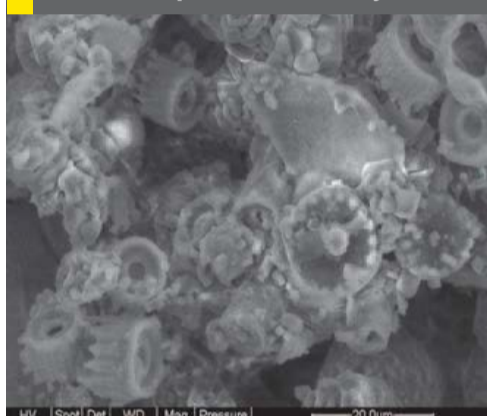


Fig 3: SEM of the inner surface of a cleaved sulphuric acid catalyst



firmed that an associative cycle including only V(V) complexes is the governing mechanism⁷ as illustrated in Fig. 2.

However, V(IV) compounds still play an important role for the activity of the catalyst, because an equilibrium exists between V(V) and V(IV) compounds in the melt. The degree of reduction to inactive V(IV) increases at low temperature and high SO₂ partial pressure. Furthermore, at temperatures below 500°C, some V(IV) compounds precipitate and gradually deplete the melt of V(V), when the temperature is lowered, and this partial solidification eventually causes the activity to drop to practically zero at some minimum operating temperature of 300-350°C. The reduction to V(IV) shown in Fig. 2 should not be regarded as a deactivation but as a dynamic and reversible response of the catalyst to the gas environment.

Understanding the complex composition of the catalytic melt is a scientific challenge that requires advanced characterisation techniques in itself. However, the dynamic response of the catalyst to the gas environment is an additional severe complication, as ex situ studies of

the cold catalyst in ambient air, inert gas or vacuum may lead to erroneous conclusions. Hence, further progress on understanding the dynamics and mechanism of the sulphuric acid catalyst calls for new advanced in situ techniques. We have met this challenge by introducing novel scientific methods, and in the following sections, examples and learnings from current studies at Topsoe are presented.

Dynamics of the active phase

The catalytic melt of a sulphuric acid catalyst typically accounts for 20-40% of the catalyst mass and it is quite mobile under operating conditions. However, this is not revealed at room temperature, where the active phase appears as solid lumps of oxides and sulphates of vanadium and alkali metals on the internal surface of the carrier as shown in Fig. 3. The cylindrical structures seen in the figure are diatom skeletal fractions, which originate from the diatomaceous earth typically used as carrier.

When heated and put on stream in SO₂/O₂/SO₃ gas, the sulphates take up SO₃ and form pyrosulphates according to (2). This transformation causes the active phase to melt and redistribute on the carrier. Due to capillary forces, however, the liquid stays in the pores of the carrier. The redistribution is illustrated in Fig. 4. In this lab experiment, a pelletised porous silica carrier was partially impregnated by submerging one end of the pellets for 30 seconds into an impregnation liquor containing appropriate salts of V, K and Na.

After calcination in air at 400°C for 15 min, visual inspection and a scanning electron microscope (SEM) energy-dispersive X-ray spectrometry (EDX) line scan show how one end is fully impregnated while no V, K and Na are present in the other end,

cf. Fig. 4a. Subsequently, the pellets were put on stream in a glass reactor in a feed gas with 10% SO₂ and 10% O₂ at 500°C for three days. After this treatment, visual inspection and EDX analysis demonstrated migration of the active phase to the unimpregnated end of the pellets as shown in Fig. 4b. After redistribution a 50% increase of catalytic activity was also observed.

In situ TEM

The lab experiment described above clearly shows the mobility of the catalytic melt in the sulphuric acid catalyst. However, in order to look directly at the state of the working catalyst and get direct evidence of the dynamics of melt distribution, we have recently introduced new advanced in situ techniques including high resolution transmission electron microscopy (TEM) to directly observe the catalyst samples interacting with an SO₂/O₂ gas mixture at temperatures up to 600°C and at high spatial resolution. The observations provide unprecedented insight into for example a simulated converter startup.

As the industrial catalyst is quite inhomogeneous on the micro- and nano-scales (cf. Fig. 3), a more well defined model system is used for the first preliminary studies. The model catalyst was prepared by impregnation of 100 nm monodispersed silica spheres with a mixture of vanadium oxide and alkali pyrosulphate to a composition of 4.2 wt-% V content and molar ratios of K/V=3.5 and Cs/V=0.4. The as prepared sample was investigated in a Philips CM300 transmission electron microscope in vacuum (at about 10⁻⁹ bar) using a TIETZ F-114 charged-coupled device camera for imaging and a Gatan Imaging Filter (GIF) for TEM imaging, electron energy loss spectroscopy (EELS) and energy-filtered TEM (EFTEM). The TEM and EFTEM results in Fig. 5 show that the active phase of the fresh catalyst is present in more concentrated lumps of material (marked by red circles). The scanning transmission electron microscope (STEM) image of a cluster of spheres and the EELS data acquired along the line scan in Fig. 6 show the relative vanadium concentration around the silica spheres (peaks at 20 Å and 150 Å). The combined use of these techniques demonstrates that the vanadia phase can be mapped out at nanometer resolution in the as prepared catalyst.

Whereas a TEM normally operates at ambient temperature and very low pressure in the order of 10⁻⁹ bar, the CM300 transmission electron microscope is equipped

with a differentially pumped vacuum system that allows solid samples to be exposed to reactive gas environments up to some mbar in pressure and up to 600°C while still maintaining atomic resolution⁸. Furthermore, as a unique capability, this particular microscope is entirely dedicated to studies involving sulphur-containing gases, in relation to e.g. hydrodesulphurisation and sulphuric acid catalysts, in order to avoid that cross-contamination and sulphur corrosion of the microscope's interior parts interferes with other experiments⁹.

In the present study, few grains of crushed catalyst sample were loaded on a micro-electro-mechanical system (MEMS) heater and placed in the microscope. After an initial inspection in vacuum, the samples were exposed to a gas mixture of 50% SO₂ and 50% O₂ at a total pressure of 10 mbar. Although this total pressure is low compared to that in the industrial reactor operating at 1.1-1.4 bar, the pressure gap has been considerably reduced compared to previous microscope investigations. In fact, a partial pressure of SO₂ of 5 mbar is sufficient to keep the catalyst in its relevant pyrosulphate form. Extensive validation of the method has been carried out to show that e.g. effects of the electron beam itself are negligible on the observations.

A result from the first heating of a catalyst sample is shown in Fig. 7. Up to 350°C, the TEM images show that the nano-scale structure of the catalyst resembles the as-prepared sample as illustrated in Fig. 7a. Upon heating to higher temperature, the images reveal significant dynamic changes in the catalyst structure on a nano-scale. For instance Fig. 7b shows that a redistribution of mass has occurred as the sample is heated to 600°C, corresponding to the high temperature found in the lower part of the first bed of typical sulphuric acid converters. During the course of this experiment, even more pronounced changes are observed after cooling to 350°C and heating to 600°C a second time. Several TEM pictures taken during the experiment are shown consecutively as a movie providing the first-ever vivid impression of liquid phase redistribution in the catalyst. From such dynamic observations, information on wetting properties and dynamics of melt distribution in sulphuric acid catalysts can be derived. Moreover, the observations can also be coupled with chemical composition and catalytic activity from e.g. operando Raman (see next section).

Fig 4: Redistribution of the active phase in a sulphuric acid catalyst

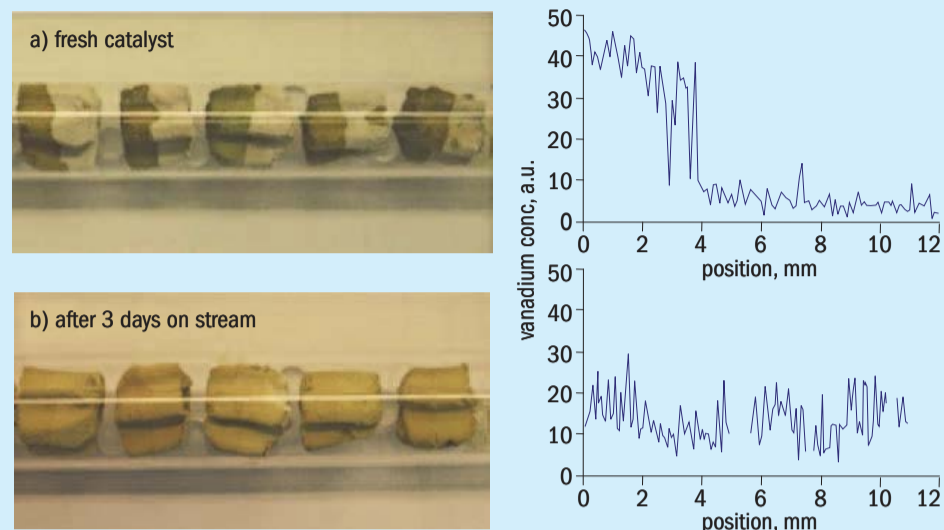


Fig 5: TEM and EFTEM of a cluster of silica spheres with the vanadia phase. The red circles indicate regions of higher vanadium concentration

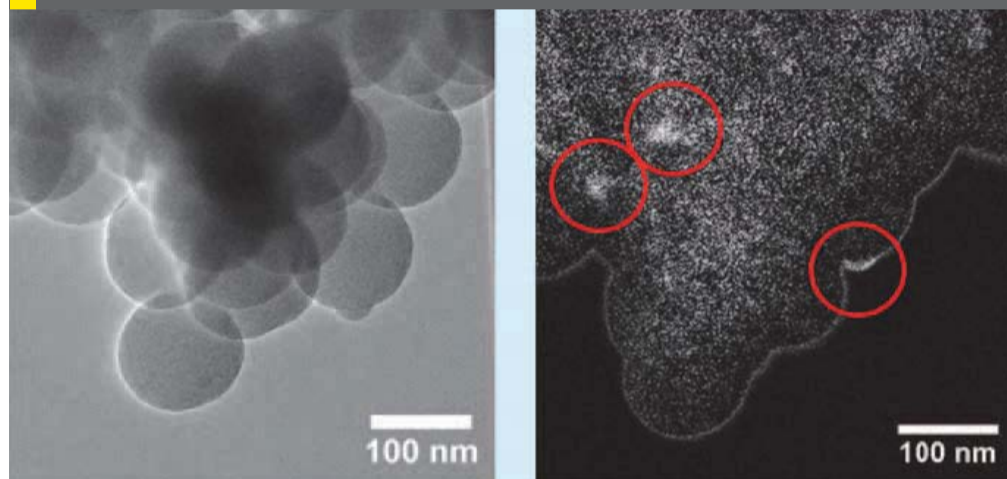
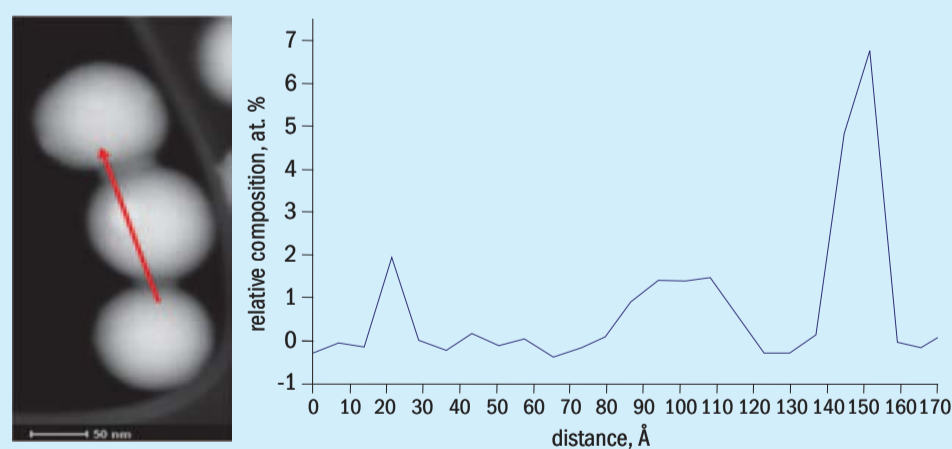


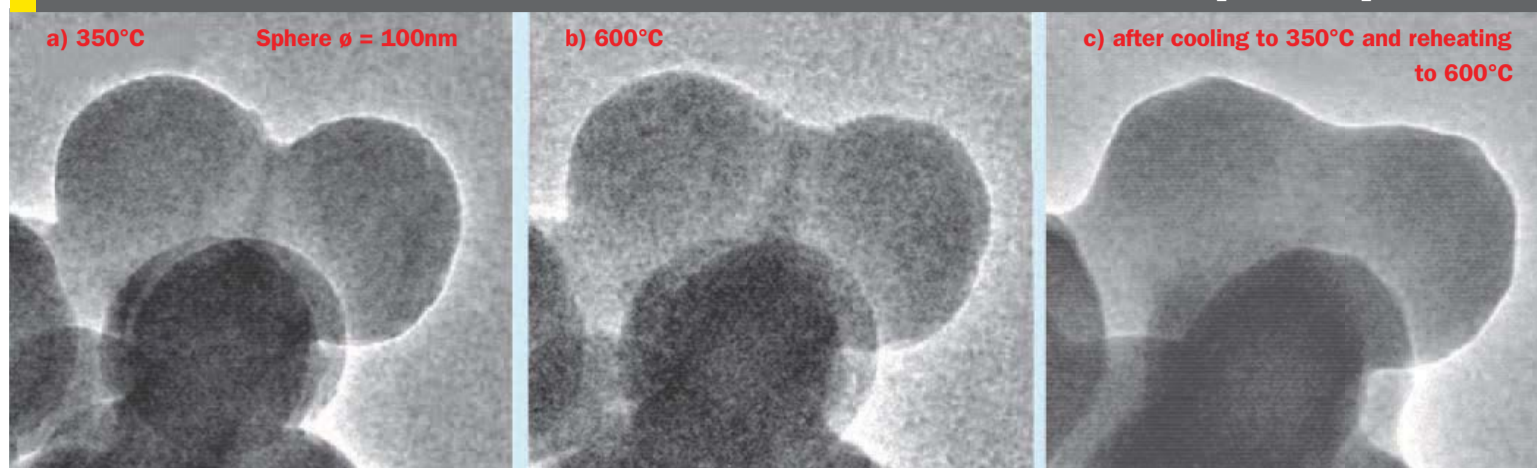
Fig 6: STEM of a cluster of spheres and a STEM EELS line scan showing V content



Liquid loading and dust protection

One learning from the studies of the dynamics of the active phase is that when the catalyst is put in operation, the melt distributes on the carrier after some time. At steady-state, the distribution depends

on the nano-scale surface properties of the silica support, e.g. the wettability, surface tension and carrier morphology. However, in the context of catalyst design another related and very important parameter of an SLP catalyst is the liquid loading defined

Fig 7: In situ TEM of dynamic changes of the model catalyst during exposure to 10 mbar of 50% SO₂ and 50% O₂

as the fraction of the pore volume of the support filled with melt. This is investigated in Fig. 8, where we have varied the melt load on a catalyst in our lab and measured activity in 10% SO₂, 10% O₂ and 500°C. The activity of the catalyst increases proportionally to the liquid loading (which is proportional to the vanadium content) at low loadings but decreases at high loading due to gas phase pore diffusion restriction and liquid diffusion restriction in the deeper melt pools. As a consequence, an optimum melt loading exists for a given melt composition and operating condition. Another implication is that higher vanadium content is not always better for activity.

The melt normally stays in the pores of the porous catalyst due to capillary forces. However, if the catalyst is covered in dust, and particularly fine dust with a high surface area, part of the melt will gradually migrate to the smaller pores of the dust and cause depletion of vanadium and alkali metals. Depending on the initial melt loading, the consequence for activity may be modest in the beginning but at high dust load and repeated screening the conversion will eventually drop and call for catalyst replacement.

Another consequence of the molten active phase is the stickiness of the sulphuric acid catalyst under operating conditions. As a result, dust present in feed gases in sulphuric acid plants typically deposits and accumulates within the top 10-15 cm of catalyst in the first bed of the SO₂ converter. Depending on the dust concentration, particle size, void fraction and catalyst stickiness, this leads to a pressure drop build-up over time which eventually may cause a costly shutdown of the plant necessary for screening of the catalyst. A simple way to prolong the time between screenings is to install a 10-15-

cm top layer of VK38 25-mm Daisy, which distributes the dust further down the bed due to its larger size and high void fraction¹⁰. The top layer should be changed after a few catalyst screenings as it will lose some of its stickiness when the melt is depleted by the fine dust.

Chemistry and colours

Sulphuric acid catalysts are normally characterised by yellow, gold or orange colours indicative of vanadium in +5 oxidation state V(V). If vanadium is in its +4 oxidation state V(IV), the catalyst more typically will have a green, pale green or pale blue colour. However, if the catalyst is exposed to moisture, the appearance and colours also change. All in all, the colour of a catalyst is not a valid method for assessing how it will perform in a converter. When a catalyst is put on stream, the catalyst composition adjusts to the gas composition and temperature, for example with respect to the V(V)/V(IV) equilibrium illustrated in Fig. 2. As a consequence, the catalyst colour also changes although this is not visible in a steel converter.

Operando Raman

In order to study the chemical transformations taking place in the operating sulphuric acid catalyst, we are using an advanced operando Raman setup where both the chemical composition can be studied by Raman spectroscopy and the sample observed visually¹¹. Laser Raman spectroscopy provides a vibrational spectrum, which contains information on the chemical bonding in the sample. Operando Raman spectroscopy is a technique that is able to monitor the structural changes in a sample on a molecular level during the reaction. For operando Raman studies it is particularly important to avoid

laser-induced heating, which, in the worst case, can lead to thermal degradation of the sample. To cope with this problem, we have developed a novel technique, where the sample is studied in a small fluid bed reactor securing uniform and well-defined temperature, cf. Fig. 9.

In order to simulate a converter start-up, the Raman reactor was loaded with a 150-300 μm sieve fraction of crushed VK38, and the fluid bed of catalyst heated first to 380°C in air at 1 atm. Under this condition, the catalyst is yellow/orange, and the Raman spectrum shown in Fig. 10a resembles some characteristic features of the dimeric V(V) complex, which are shown as the active species in Fig. 2.

In particular, the two bands at ~860 and 770 cm⁻¹, which are assigned to V-O-S and V-O-V bridging bonds, have been claimed to be the signature of the active catalyst¹². The lack of V(V)-vanadyl bands (typically at ~1040 cm⁻¹) indicates the extended polymeric nature of the catalyst under this condition. After switching the reactor feed gas to 10% SO₂ and 10% O₂ in nitrogen at 380°C in Fig. 10b, the catalyst colour changes to greenish, and the Raman spectrum reveals structural transformation from a polymeric vanadate to the well-defined molecular structure of the (VO)₃(SO₄)₅⁴⁻ ion implying a reduction of V(V) to V(IV). At this point, the conversion of SO₂ measured by infrared (IR) SO₂ analysis of the effluent is very low. As the temperature is raised to 480°C, a significant increase in conversion is observed, and the colour changes from greenish to ochre, as seen in Fig. 10c. The corresponding Raman spectrum shows a band centred at 1043 cm⁻¹ characteristic for V(V)-vanadyl bonds and a broad and structured band between 940 and 985 cm⁻¹ together with two bands at 665 and 605 cm⁻¹ assigned to the vibra-

tional modes of the SO_4 ligands in different vanadium oxosulphate complexes.

From a detailed analysis of the spectra, it is possible to conclude that the active phase of the industrial VK38 catalyst at 480°C corresponds to a mixture of predominantly monomeric and to a minor extent dimeric vanadium (V) oxosulphate species, which fits well with the proposed structures in Fig. 2.

Colours in industry

The studies presented above show that the composition and colour of the operating catalyst change with the temperature and gas composition. Catalyst containing vanadium primarily in oxidation state +5 is yellow/orange, but if it is loaded in a converter and operating at low temperature or high partial pressure of SO_2 , the colour will change to greenish/bluish because these conditions favour reversible reduction of vanadium to oxidation state +4. Also the catalyst may change visual appearance during a converter shutdown depending on the purging and cooling procedure or during storage. When purged with air while the catalyst is still hot, vanadium will be oxidised to V(V) and attain a yellow colour even if it was green when operating in the converter in e.g. the top of the first bed. As an example, Fig. 11 shows how a greenish catalyst sample from industry can change colour to yellow when heated in an air flow in a glass tube for 24 hours at 500°C.

Catalyst design for optimal performance

The reaction rate for a catalyst may be limited by external mass and heat transfer to the pellet surface, by internal mass transfer in the porous support or by the intrinsic reaction rate itself. For sulphuric acid catalysts operating at a low temperature, the main limitation is the intrinsic reaction rate, which depends on the chemistry in the catalytic melt but, to a great extent, also on the distribution of the catalytic melt on the support and gas solubility and transport through the melt. Advanced in situ techniques such as those mentioned above provide valuable insight into the mechanisms governing the intrinsic catalyst performance, and the learnings are used continuously in Topsoe to develop improved industrial catalysts.

One example is the significant changes of the melt properties, when some of the potassium and sodium sulphates are replaced by cesium sulphates. The introduction and optimisation of cesium-

Fig 8: Activity vs. liquid loading of a sulphuric acid catalyst prepared in lab

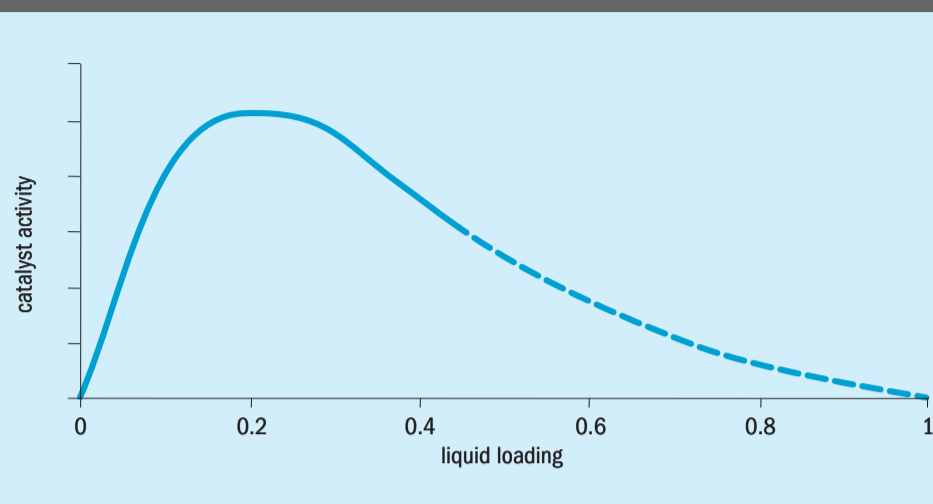
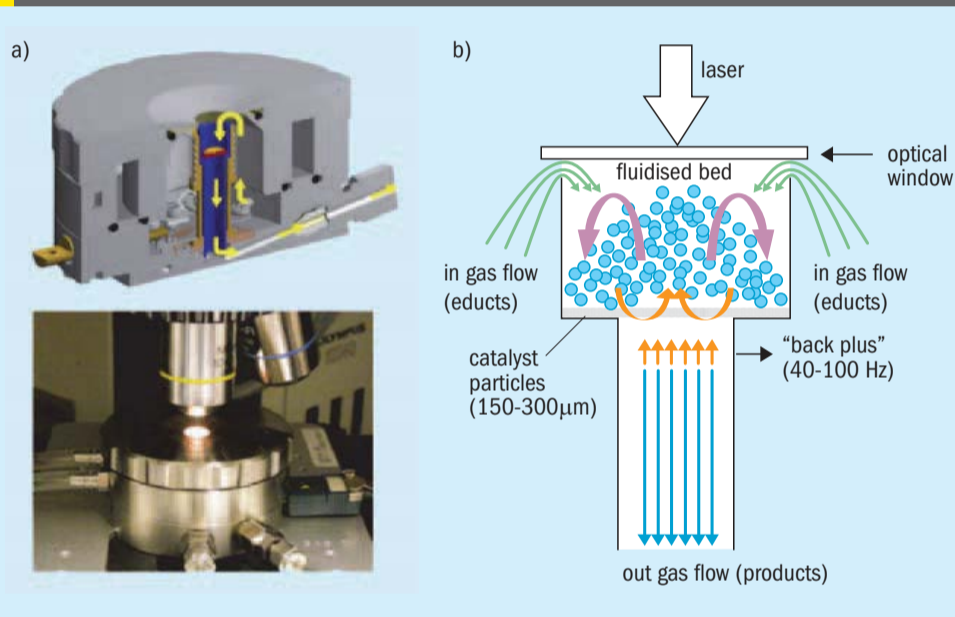


Fig 9: (a) Linkam CCR1000 reactor used for in situ Raman (b) sample holder with flow indications to illustrate the fluidization principle.



promoted catalysts, e.g. VK59 and VK69, for low-temperature operation had a significant impact on possible inlet temperatures and SO_2 emissions in industry¹³. From the fundamental studies, we have also learned how the activity of normal commercial sulphuric acid catalysts is seriously hampered by deep pools of catalytic melt restricting gas transport in the active phase. These limitations have been overcome in the new high-activity VK-701 catalyst based on our novel LEAP5™ technology¹⁴. The improvements, which include a very high V(V) content, were accomplished by changing the intrinsic morphology and surface properties of the carrier and optimising the active phase for high SO_3 concentration.

Conclusions

Catalysts are dynamic systems which interact to a great extent with the local environment in which they operate. As a

consequence, in situ studies at relevant temperatures, pressure and gas composition are necessary to get a true picture of the working catalysts. This is particularly true for sulphuric acid catalysts for which the active phase is a liquid that may account for about one third of the catalyst mass – a supported liquid phase (SLP) catalyst. In order to look directly at the state of the working catalysts, Topsoe has recently introduced new advanced in situ techniques including Raman spectroscopy and high-resolution in situ transmission electron microscopy to directly resolve the dynamic state of catalyst samples interacting with an $\text{SO}_2/\text{O}_2/\text{SO}_3$ gas mixture at temperatures up to 600°C. These techniques provide unprecedented insight into sulphuric acid catalysis.

In situ TEM studies combined with lab-scale results show how the active phase takes up SO_3 and becomes mobile. From

Fig 10: Operando Raman spectra while switching from synthetic air (a) to SO₂/O₂ (b) at 380°C and in SO₂/O₂ at 480°C (c). The photographs to the left of the spectra show the catalyst in the reactor under the respective reaction conditions

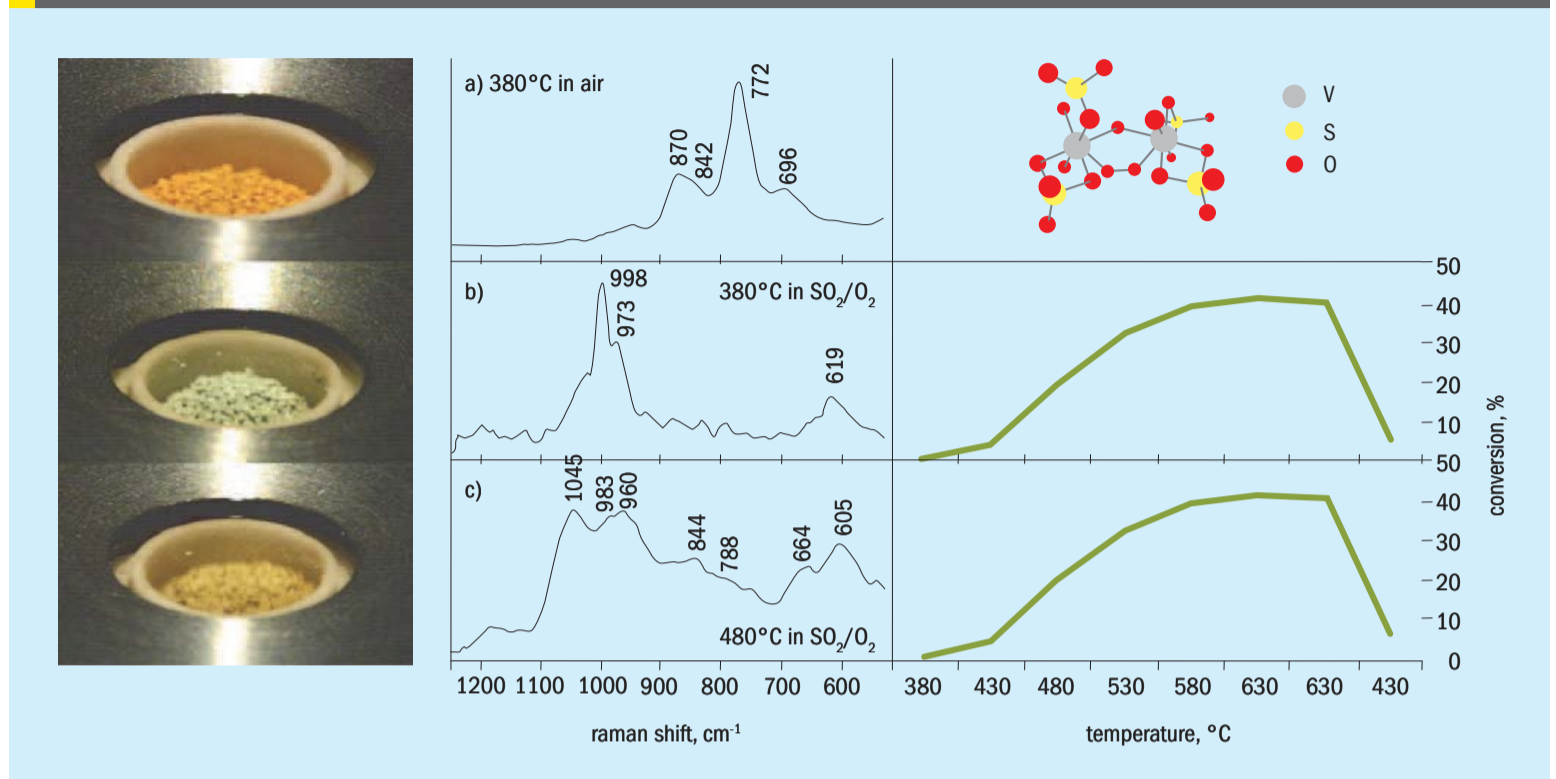


Fig 11: Colours alone cannot be used for assessment of the catalyst performance. Here reduced vanadium is reoxidised by heating of a greenish sample in air



the high resolution TEM images, the rate of the dynamic changes and the final wetting characteristics and melt distribution can be derived. The operando Raman observations demonstrate that colour changes occurring during a simulated converter start-up are directly related to measured catalyst chemistry and observed SO₂ conversion. The results indicate that the vanadium changes from a dimeric V(V) complex in hot air to primarily V(IV) compounds in SO₂/O₂ gas at 380°C. When the temperature is increased to 480°C, vanadium is reoxidised, and the spectral information obtained supports that the active phase of the industrial VK38 catalyst at 480°C corresponds to an equilibrium between predominantly monomeric and to a minor extent dimeric vanadium (V) oxosulphate species.

The studies presented here show that the melt distribution and chemistry interact and that the physical state is just as important as the gross composition of a sulphuric acid catalyst. The detailed in situ observations can also help explain macro-scale phenomena in the industrial converter such as catalyst colours, oxidation states, melt transformation, melt extraction by dust, pressure drop build-up and catalytic activity. ■

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Selection of sulphuric acid catalysts grows

MECS and Clariant have recently announced new additions to their sulphuric acid catalyst portfolios. The latest catalyst enhancements provide an even greater choice of options to tailor catalyst requirements according to sulphuric acid plant requirements. Benefits of the new catalyst developments include increased converter performance while limiting SO₂ emissions and the ability to operate at lower bed inlet temperatures.

New MECS® catalyst developments

Driving innovation in the sulphuric acid market since the 1920s, MECS, Inc. (MECS) remains an industry leader in technology, engineering and equipment dedicated to sulphuric acid producer's needs. As energy savings and environmental concerns create new operational and design challenges for sulphuric acid plants, innovations in catalyst technology provide the solution. Over the past 90 years, the dedicated Research and Development team at MECS has evolved catalyst from pellets to energy-saving rings to low-emission cesium-promoted catalyst. In 2015 MECS commemorates its rich history of catalyst developments with a year-long 90th anniversary celebration.

MECS is the only catalyst manufacturer to offer GEAR® catalyst utilising a unique hexa-lobed ring shape which, combined with an improved catalyst formula, has demonstrated better conversion performance, lower pressure drop and improved dust handling. By geometrically optimising the catalyst shape, GEAR® catalyst offers more surface area for access to active sites than any other catalyst on the market. In addition, when loaded into a catalyst bed, the hexa-lobed ring shape creates a catalyst bed configuration which increases spacing between the catalyst rings, lowering pressure drop significantly compared to standard ribbed ring or daisy-shaped catalyst.

An interesting extension of the unique GEAR® catalyst benefits is now offered for customers who would benefit from combining the low temperature benefits of cesium-promoted catalysts with a GEAR® catalyst shape. An example would be an existing acid plant with the following challenges:

- high dust contamination in the gas stream (such as metallurgical plants or sulphur burning plants with varying quality of sulphur in the feed) or
- desire to increase throughput without increasing pressure drop or SO₂ emissions.

The demonstrated superior dust handling provided by the hexa-lobed ring shape of the GEAR® Catalyst, especially in pass 1, inspired the addition of GEAR® cesium catalyst, GR-Cs, to the MECS® catalyst portfolio. Sulphuric acid plant converters operating with lower bed inlet temperatures have the opportunity to upgrade to GEAR® cesium catalyst for energy savings and excellent dust handling.

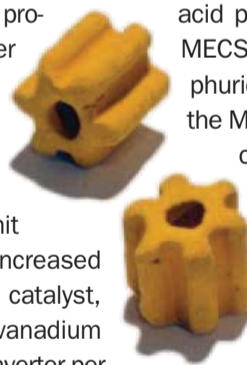
Increasing throughput in an existing plant is often a balance, requiring the process engineer to decide whether additional production is worth the higher energy cost or additional emissions. In many cases, blower capacity or plant emissions permit levels can limit the plant's ability to achieve increased output. With the GEAR® cesium catalyst, GR-Cs, as a replacement for vanadium catalyst, a plant can increase converter performance while limiting additional emissions or avoiding excessive load on the blower.

The most recent product enhancement developed by MECS® Catalyst Research and Development is a minor, but high activity boosting modification to the well-established Super Cesium SCX-2000 cesium catalyst formula. This proprietary formula improvement positively affects the SCX-2000 cesium catalyst activity, offering customers higher performance in the fourth and fifth converter passes. The improved formula

SCX cesium catalyst allows a customer to achieve lower SO₂ emissions through higher catalyst activity and better conversion or the ability to operate at a lower bed inlet temperature of 385°C (725°F).

Extensive lab data on improved formula SCX indicated that it can accomplish the same SO₂ emissions with less catalyst required. In this way, the customer can save on catalyst costs. If lower emissions are preferred, lab data confirmed that improved formula SCX provides lower SO₂ emissions than the former SCX formula at the same catalyst loading. A case study has confirmed lower SO₂ emissions with the improved formula SCX.

The GEAR® and SCX-2000 innovations join the rest of the MECS portfolio offering catalyst solutions to major sulphuric acid producers placing their confidence in MECS technology and know-how. The sulphuric acid industry catalyst workhorse is the MECS® XLP catalyst, a vanadium-based catalyst installed in hundreds of plants worldwide. MECS is unique in proving that XLP and GEAR® are sulphuric acid catalysts that can be applied in all passes, thus simplifying maintenance complexity, reducing inventory costs, and increasing flexibility for unexpected replacement requirements for all standard (XLP) and high performance (GEAR® series) applications. In applications where the use of cesium-promoted catalyst offers the activity and performance needed to achieve more activity with less catalyst, producers rely on MECS® cesium catalysts, especially the improved formula Super Cesium SCX-2000. MECS offers sulphuric acid plant producers a strong portfolio of catalyst products with benefits applicable for a multitude of operating requirements.

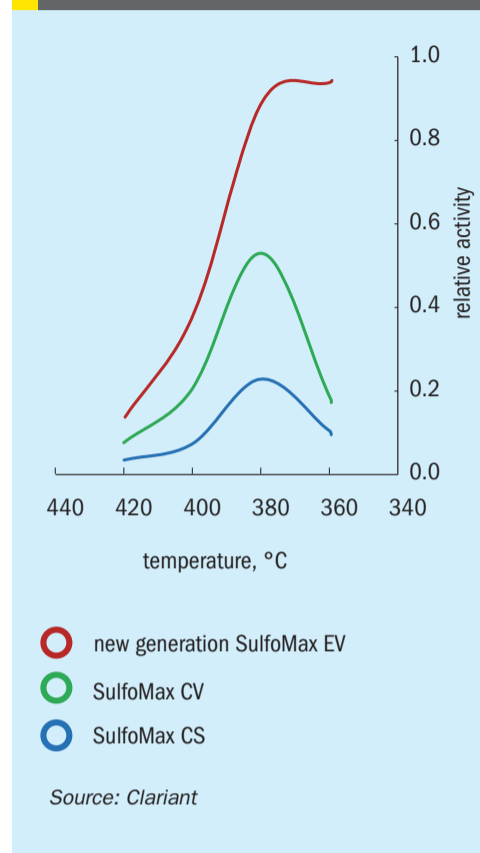


Clariant's new generation catalysts

Taking enough preventive steps to sustain better efficiency is the outlook for the future. As a result of Clariant's continuous research for the improvement of conversion efficiency and reduction of stack emission for a greener world, a new product has been added to the SulfoMax® catalyst portfolio: SulfoMax EV. The interesting fact about the new generation catalyst is that it operates at relatively lower temperature even in comparison with the current cesium promoted catalysts in the market.

Laboratory findings confirm the superior activity of SulfoMax EV, especially at low SO₂ concentrations. The new generation catalysts are tested in industrial plant conditions for performance and lifetime. The test results show that this new formulation has great potential to reduce the emission to greater extent when installed in final passes with much lower inlet temperatures. Figure 1 shows the relative activity of SulfoMax EV in comparison with the conventional SulfoMax catalysts.

Fig 1: Relative activity of new generation SulfoMax in comparison with the existing SulfoMax catalysts



In a sulphur burning plant, the new generation catalyst was installed in a final pass. The conversion improved significantly while reducing the emission at the same time.

The conversion across the final pass before catalyst exchange was 94-95% which increased to 99.5% after catalyst exchange.

By adopting best operational practices, proper diagnosis, troubleshooting and implementing new advancements in the SulfoMax catalyst technology, Clariant's focus on the heart of the sulphuric acid plants allows improving efficiency, reducing emission and creation of sustainable benefits.

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KEEPING YOUR ACID PLANT HEALTHY FOR 90 YEARS

MECS is celebrating 90 years of serving the sulfuric acid industry with breakthrough catalyst products that provide a range of benefits to keep the heart of your plant healthy. If your plant or converter is feeling a little tired, contact MECS for urgent care; you'll soon be pleased with a healthy plant and happy owners. **WE ARE SULFURIC ACID.**

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PHOTO: BLASCH PRECISION CERAMICS

Timely furnace replacement at Burnside

J. Bolebruch of Blasch Precision Ceramics, **B. Lamb** of MECS and **M.D. Harris** of J.T. Thorpe & Son, report on the successful recent spent acid furnace upgrade in which HexWall™ ceramic furnace internals enabled DuPont Burnside to conduct a complete furnace replacement in a matter of days.

The DuPont sulphuric acid plant in Burnside, Louisiana, in the USA is a large and versatile facility with a history of pioneering technology and world-class maintenance and operational practices. It is a 2,300 t/d plant capable of producing everything from standard industrial grades of acid to more specialised products like alkylation grade sulphuric acid, oleum and liquid SO₃. Not only is its size and product slate impressive, but the site also has access to shipment via boat,

rail and truck, making it a site of high strategic importance to DuPont.

Commissioned in 1964, the site has a long history of improvements, capital investments, new technologies and continuous improvement. It was the first DuPont sulphuric acid plant to retrofit from single absorption to double absorption in 2009. It was also the first DuPont sulphuric acid plant to replace older generation burner technology with a modern, fully integrated burner management system. In keeping

Detail of the HexWall™ baffle tied into the hotface brick lining showing the open vs closed blocks in the baffle.

with its tradition and history as an early adopter of modern technologies and an implementer of innovative solutions, in December of 2013, the Burnside plant undertook a major project to replace and upgrade its spent acid furnace using the MECS® HexWall™ ceramic furnace internals.

MECS, Inc. (MECS) entered into an agreement with Blasch in late 2011, appointing MECS as the exclusive distributor of Blasch HexWall™ ceramic furnace internals, and engineered ceramic boiler ferrules for use in sulphuric acid plants and vessels worldwide. MECS had recently become a wholly owned subsidiary of DuPont at that point, and the decision to partner with Blasch was based on a history of successful use of HexWall™ ceramic furnace internals at a pair of DuPont acid plants dating back to 2007, as well as a vision of what could be achieved with the newly fielded VectorWall™ product.

The need for this retrofit at the DuPont Burnside sulphuric acid plant arose out of the world-class preventative maintenance program incorporated at DuPont. Using rigorous inspection protocols, fitness for service tools, and good MIQA practices, DuPont was able to get a 15-year service life out of the furnace, and still retire it before having a catastrophic failure. Upon realising the need for a furnace replacement, DuPont set out to replace the existing furnace efficiently, but also using modern technology to its best advantage.

The first item of interest for DuPont with regard to furnace design was that of pressure drop, which is a significant concern for sulphuric acid plants because a high pressure drop can lead to plant bottlenecking and/or high energy consumption for main blower operation. Since the furnace volume was fixed, the only way for DuPont to reduce the furnace pressure drop was to reduce the total number of baffle walls inside the furnace.

A reduction in the number of baffle walls had positive implications from the standpoint of pressure drop, but there was a legitimate concern about getting the proper level of mixing inside the furnace in the absence of the previous baffle con-

figuration. In order to evaluate the effect, DuPont performed CFD modelling and consulted with a variety of industry leading experts. The result was that by using HexWall™ ceramic furnace internals, DuPont could reduce the total number of baffle walls without having a material impact on the mixing inside the furnace.

The final item to be considered in the new furnace design was that of mechanical stability. In this area, HexWall™ technology provided a clear and proven advantage. With mechanical support around the entire inner circumference of the furnace and an unblemished “no wall fall” record, the MECS® HexWall™ system presented a simple, cost effective and proven means of accomplishing this goal.

HexWall™ system installation

DuPont employed the services of JT Thorpe & Son, North America’s largest industrial refractory contractor, to install the system. JT Thorpe & Son has a long history of providing design and installation services and expertise to DuPont and has itself a long history of being on the cutting edge of new technology in its own industry.

JT Thorpe & Son worked closely with DuPont to optimise the refractory lining at furnace penetrations, end-wall construction and HexWall™ and VectorWall™ integration. This coordination resulted in a refractory lining that took advantage of the latest material and design technologies while also improving constructability, thus providing for a cost-effective installation. JT Thorpe & Son was very familiar with the VectorWall™ system, having installed this design in new installations, as well as upgrading existing furnaces during maintenance outages. The VectorWall™ was installed in approximately one-third the time normally required for a comparable brick baffle/checker wall.

A tight schedule and a tight footprint

This compressed timeline proved to be crucial due to an extremely tight window for replacement of the furnace. The existing furnace was located in an area that had seen considerable encroachment over the years, and consequently there was not enough real estate to place the new furnace in a location that would allow it to be incorporated into the train quickly upon completion of the refractory installation.

It was going to be necessary to cut out the old furnace prior to the siting and lining of its replacement. The problem was that there was not time to accomplish all this work.

Meetings were held, and options weighed, and a bold idea was presented. Why not line the entire furnace and then lift it into place ready to go? This approach was sometimes used with much smaller furnaces, but the new furnace was large by anyone’s standards, at nearly 20 feet in diameter, and greater than 60 feet in length. The weight of the furnace and refractory was estimated to exceed 1.1 million pounds. This made it a heavy lift that could only be accomplished by a very small number of people.

Solidifying the decision was the fact that the HexWall™ ceramic furnace baffles were tied into the lining and were mechanically engaged through a series of tabs and slots on all six sides of each block, while not being mortared together. Thus, the HexWall™ Ceramic Internals could tolerate a certain amount of movement without the threat of damage.

Once the installation was complete, the time required from the point the existing train was brought down until the new furnace was fired up was 11 days.

While this was going on, extensive modelling was underway to optimise the spent acid decomposition furnace design through the use of the VectorWall™ mixing checkerwall.

Furnace design optimisation

The VectorWall™ is a variation of the HexWall™ baffle that utilises a series of mixing inserts in each wall to create very specific flow patterns in the decomposition furnace. The VectorWall™ has been used with great success in the thermal stage of the Claus process for sulphur recovery in refineries and gas plants, and it was felt that many of these same advantages could be realised here.

Part of Blasch’s experience in the Claus process showed that it was possible to configure the VectorWall™ in such a way as to create distinctly different zones within the same furnace, and to include a secondary stream entering the furnace behind the wall. This capability made it possible to greatly increase the volume and efficiency of ammonia destruction accomplished within the furnace. This split flow, or staged combustion approach would prove

to be of great interest to the decomposition furnace community as well.

Prior decomposition furnace designs at MECS incorporated the use of a series of partial brick walls, as many as four, with alternating open areas designed to create a high degree of mixing as spent acid and air passed through the furnace. Combustion of spent acid is a complicated and difficult process and mixing is critical to the efficiency of the process. The downside of this baffle design is that it creates relatively high pressure drop and its broad residence time distribution drives furnace design to longer furnaces with greater cost and real estate requirements.

Modelling results indicate that decomposition furnaces can be designed using a combination of HexWall™ baffles and VectorWall™ systems that ultimately serve to reduce the total number of furnace walls required, while preserving (and often improving) the requisite level of mixing. Further to that, DuPont and MECS have used various combinations of HexWall™ baffles and VectorWall™ systems in the design of decomposition furnaces in conjunction with secondary air addition to create a staged combustion environment. This approach to decomposition furnace design has demonstrated the ability to reduce furnace size as well as have a significant impact on NO_x formation.

The design of the HexWall™ Baffle incorporates the use of a counter bore at the round opening on either side of the block. It is this counter bore on the upstream side that accepts the plugs which form the solid portion of the HexWall™ baffle. These plugs may be removed without dismantling the wall and replaced with a series of vector tiles on the downstream side, or a combination of plugs and vector tiles may be used concurrently, thus forming specific flow configurations in the walls.

The HexWall™ baffle installation at the Burnside plant is just one example of DuPont and MECS implementing cutting-edge technology within an age-old industry to challenge the status quo and to drive continuous improvement. Through MECS’ partnership with Blasch Precision Ceramics, VectorWall™ technology continues to evolve. By leveraging Blasch’s knowledge and experiences in Claus applications, and applying the technology in the sulphuric acid industry, sulphuric acid plant owners and operators can be the beneficiaries of significant operational and maintenance-related improvements. ■

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