Number 407

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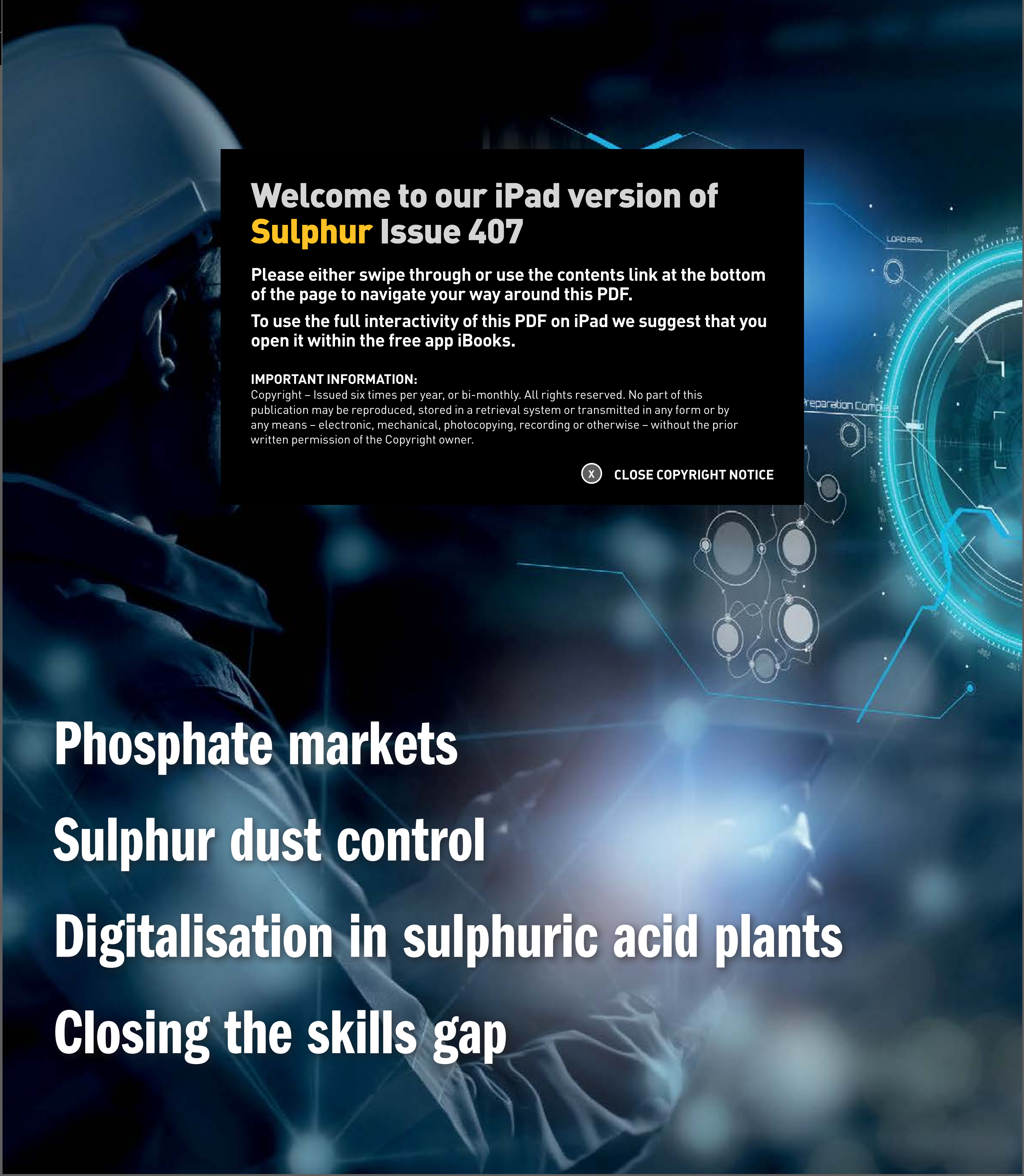

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Phosphate markets

Sulphur dust control

Digitalisation in sulphuric acid plants

Closing the skills gap

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56

1 47 2 48 3 49 4 50 5 51 6 52 7 53 8 54 9 55 10 56 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56

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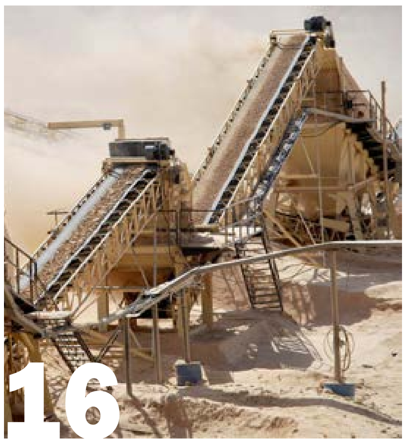
NUMBER 407 JULY | AUGUST 2023

Cover: *A smart industry futuristic technology concept. MangKangMangMee/ Shutterstock.com*

CONTENTS

REGULARS



Phosphate demand

India and Brazil lead new demand for phosphates

Sulphur friability

Producing better quality sulphur

Read this issue online at: www.sulphurmagazine.com

Published by: BCInsight

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56

Unintended consequences?

> "There is a trade-off between sulphur recovery levels and climate action."

The modern sulphur industry is in effect a response to the environmental problems created by the presence of sulphur compounds in oil and gas, and the consequent release of sulphur dioxide when they are burned. The tens of millions of tonnes extracted, formed, traded and used for sulphuric acid production every year would otherwise be entering the atmosphere and causing health issues, especially in major cities, or returning as acid rain. One of the most recent step changes in sulphur recovery has come from the extension of rules on sulphur content of fuels that have been commonplace for road vehicles for many years into the maritime transport sphere. The International Maritime Organisation has mandated a reduction in sulphur content of bunker fuels to 0.5% worldwide, and 0.1% in busy shipping regions that have become designated emissions control areas (ECAs). Because bunker fuels were made from refinery residues, they often had high concentrations of sulphur in them; the limit before 2020 was 3.5%. As a result, a recent paper by two climate scientists calculates that global SO_2 emissions have dropped by as much as 10% since 2020 because of the IMO limits. Given that atmospheric sulphur dioxide is responsible for an estimated 20-90,000 preventable deaths per year, this is surely a good thing.

Now, however, three years into the new regulations, there are signs that this reduction may perhaps be a double-edged sword. As we have discussed before, sulphur dioxide not only reflects incoming sunlight but can also act as a nucleus around which water droplets can form, in effect 'seeding' cloud formation, especially at stratospheric levels. This effectively lowers global temperatures by reflecting sunlight back into space, an effect known as radiative forcing. Indeed, only in January this year we reported on a plan to deliberately release SO_2 at high levels to try and reduce global temperatures, as can happen in the wake of large-scale releases of SO_2 from volcanic eruptions. The well intentioned reduction in SO_2 emissions from shipping has therefore potentially reduced this effect, particularly across the North Atlantic and North Pacific oceans, where the busiest shipping lanes are. At the same time, there has been a spike in global sea surface temperatures to the highest levels recorded, and which is around 0.6°C above the mean for 1982-2011.

In a paper written this month for Carbon Brief, Dr Zeke Haushofer and Prof Piers Forster, director of the Priestly International Centre for Climate, discuss whether the two are linked. There is a major complicating factor in that the oceans are also a source of large-scale natural dimethyl sulphide emissions produced by marine bacteria and plankton, and in fact this is roughly three times the size of SO_2 emissions from shipping. Using a range of widely accepted values for radiative forcing, they calculate that the impact of the IMO ban is probably only an additional 0.05°C of warming, and that the current ocean temperature spike is more likely a result of the move from a La Nina to an El Niño year. Even so, that's 3% of the 1.5°C increase in global temperatures compared to pre-industrial levels that the Intergovernmental Panel on Climate Change is recommending that we try to limit ourselves to, and a reminder that environmental legislation can have unintended consequences. Industry figures such as Angie Slavens have warned before that, beyond a certain level of sulphur recovery in a Claus plant, moving to the most stringent levels of reduction (such as the 99.99% that some bodies have recommended) can lead to a major increase in carbon dioxide emissions. In effect, there is a trade-off between sulphur recovery levels and climate action, and one that should be borne in mind when future restrictions on sulphur content of fuels are contemplated. ■

Richard Hands, Editor

Price Trends

MARKET INSIGHT

Meena Chauhan, Head of Sulphur and Sulphuric Acid Research, Argus Media, assesses price trends and the market outlook for sulphur.

SULPHUR

The global sulphur market has continued to soften through the second quarter as expected, with demand remaining relatively steady, at a time when there is ample availability from key suppliers, with the exception of Russia. Third quarter contract negotiations were ongoing in some regions at the start of July but some settlements had been concluded at decreases. Global spot prices have continued on a downtrend in recent months, but this is expected to turn to stabilisation and a possible uptick during July. The Argus global sulphur trade balance reflects a surplus for the next 12 months, a factor expected to put a ceiling on the uplift we expect at present. The rate and pace of new capacity from new projects will impact how quickly export availability grows, particularly from the Middle East, with both Kuwait and Saudi Arabia expected to lead the rise in 2023.

The ADNOC monthly Official Sulphur Price (OSP) for June was down to $86/t f.o.b., its lowest level since last August, when fears of shortages eased in the wake of the conflict in Ukraine. This was a 36% fall from March in just three months and reflecting weakness in markets across the board, and a massive decline from the highs seen last year. In the US, refinery utilisation averaged 97% in May, and sulphur output was up by 100,000 tonnes compared to the first five months of last year.

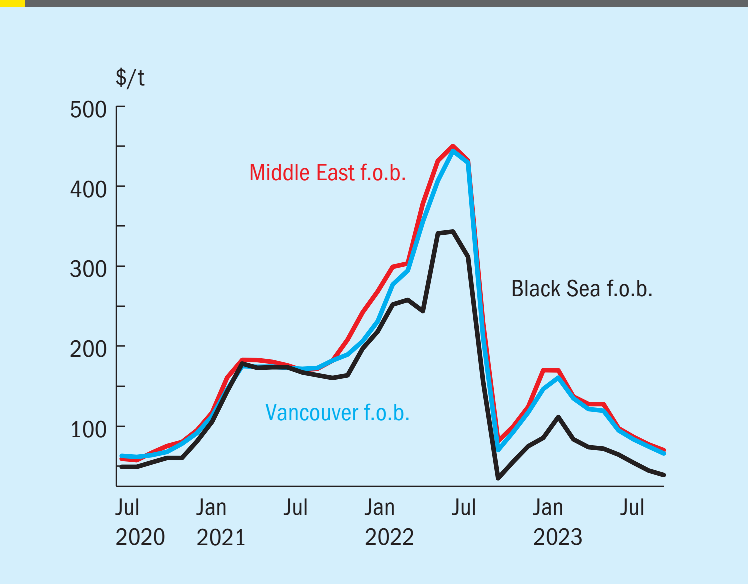
Despite some interest in China briefly lifting and stabilising spot prices, this gave way to further softness in May-June. The high end of the range increased very slightly, up by $2/t in early July to $88/t c.fr. The domestic price increase added pressure to stabilise prices alongside signs of demand emerging from markets outside of China. Port inventory in the country increased in June, with Fangcheng port starting to reach upper limits at warehouses and some cargoes were diverted to Dafeng port instead. A busy arrival schedule in July at Zhenjiang port may also lead to some logistical congestion. Processed phosphate fertilizer fundamentals have remained weak with operating rates at DAP and MAP plants in the 55-60% range on average. The forecast for sulphur demand growth is to see a slight uptick for the production of phosphoric acid, with the first half of 2024 likely to see only a stable to slight uplift in the sector. The main supportive factor for sulphur consumption is the industrial sector.

Battery materials are a sector for potential sulphur demand growth, although historically this has impacted the merchant sulphuric acid sector. New sulphur burning capacity is being added however, and this will lead to an uptick in consumption. Chinese new energy firm Lubei Wanrun has launched a lithium iron phosphate (LFP) material and iron phosphate plant in Wudi county, in north China's Shandong province. The plant has a nameplate capacity of 240,000 t/a for LFP and 240,000 t/a for iron phosphate, with an investment of 6.5 billion yuan ($913 million). Details on including construction schedules and launch dates have not been disclosed. LFP batteries have taken over the majority share of the lithium-ion power battery market because of their lower manufacturing costs. On the supply-side in China, sulphur production continues to be robust, another bearish factor for import demand in the second half of this year. Our current estimate is that sulphur production will rise to more than 10 million t/a this year, up from more than 9 million t/a last year.

Indian demand is expected to see a boost in the short term with new sulphur-burning capacity in 2023-24. Domestic fertilizer producer CIL is still expected to bring on line its burner in the second half of 2023. Iffco Paradeep's burner is scheduled to ramp up in the first quarter of 2024. Combined, the two burners would add over 400,000 t/a sulphur demand at capacity.

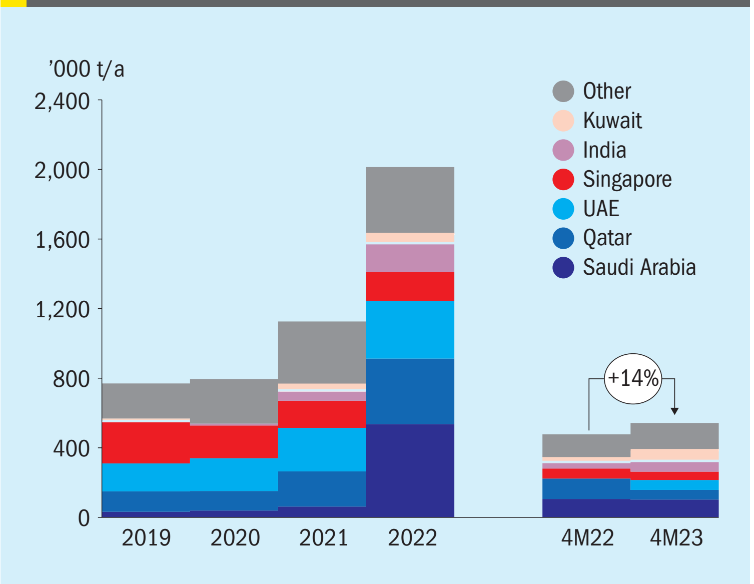
In Europe, molten sulphur contracts settled at a reduction of $40/t on the second quarter, down to a range of $87-103/t c.fr Benelux. The contracts reference molten product moving from the region's refineries to regular local customers. Demand

Fig. 1: **Average sulphur prices, key export regions**



Source: Argus Sulphur

Fig. 2: **Indonesia sulphur imports 2019-23**



Source: Argus Sulphur

in the region has been subdued, amid the economic downturn and downstream product margins being squeezed. On the supply side, the end of refinery and gas field maintenance in Germany may ease supply tightness amid turnarounds, logistics delays and strikes.

Brazilian spot prices have dropped to below $100/t c.fr in June. Logistics-related maintenance is expected to affect short-term sulphur import flows to the country, but we still expect a robust year of demand. Trade data reflect significant year-on-year shipments so far this year. Santos' Tiplam bulk terminal is scheduled to undergo maintenance from 10 July-15 August, and Termag is expected to be down from 15 July-15 October, restricting the discharge of bulk vessels at the port. This has affected sulphur availability from the US Gulf, with the usual spot demand in Brazil not emerging for this period.

Pressure remains from the processed phosphates sector, and we expect to see Brazilian spot prices bottom out in the $70s/t c.fr in July before a slight rebound through the second half of this year as import demand returns.

Meanwhile Chilean sulphur demand has been increasing and is forecast to rise throughout this year and remain healthy in the first half of 2024. Demand is for Noracid's sulphur burner, with the acid produced sold to the local copper industry. Imports increased by 72% in January-April 2023, compared with the same period in 2022, with Canada remaining the leading source of supply. There has been no disruption to trade to the country in the aftermath of ongoing wildfires in Alberta.

SULPHURIC ACID

Global sulphuric acid prices have been under pressure with the downturn in processed phosphates sector and sulphur markets adding even more bearishness. Average acid prices have continued to soften through the first half of the year with a floor yet to be reached at the start of July. Prices in Asia are expected to see further pressure as suppliers look to place cargoes against a backdrop of weak sentiment. Some demand has emerged in Western markets but OCP in Morocco remains out of the market and there is limited spot market interest in Chile.

Average Chilean spot prices were $90/t c.fr at the midpoint in end June – early July, down by $20/t on the previous quarter and the first time the price has dropped below $100/t in this year. A lack of appetite for demand has been cited for the quarter ahead because of ample inventory levels. Chile's state-owned Codelco announced the closure of its Ventanas smelter on 31 May. The company decided to shut the smelter last year in response to environmental concerns. It received permission to close the plant from the geology and mining authority in mid-May. The smelter was in operation for 58 years.

Recent heavy rains in the Chile has led Codelco to estimate losses of around 7,000 tonnes of copper following disruptions at its Andina and El Teniente operations. The state-owned copper company is now expecting 2023 copper production to come in at the lower end of the previously estimated range of 1.35–1.42 million t/a. Chile imported 1.6 million tonnes of sulphuric acid in the first five months of the year, slightly up on the figure of 1.5 million tonnes a year earlier

Base metal prices rose in early July following recent softening, even amid signs the US Federal Reserve will raise its benchmark interest further over the rest of this year. Reports reflecting a tight labour market in July reinforced signals from the minutes of the June Federal Reserve policy meeting that more rate increases are likely this year as the Fed battles to quell inflation, even after hiking 10 times in the last 15 months.

Swedish metal producer Boliden's Ronnskar smelter and refinery has partially resumed production after it was damaged in a fire on 13 June, the company said on 30 June. But the facility will produce only copper anodes until further notice because the electrolytic refinery, which processes the metal into cathodes, was completely destroyed in the fire. Many of its production lines will operate in a limited capacity until July. Because of the switch to anodes, even production lines that were undamaged by the fire will run at only a limited capacity. Ronnskar, Boliden's largest smelter, produced 218,000t of copper and 550,000t of sulphuric acid last year.

Price Indications

Table 1: **Recent sulphur prices, major markets**

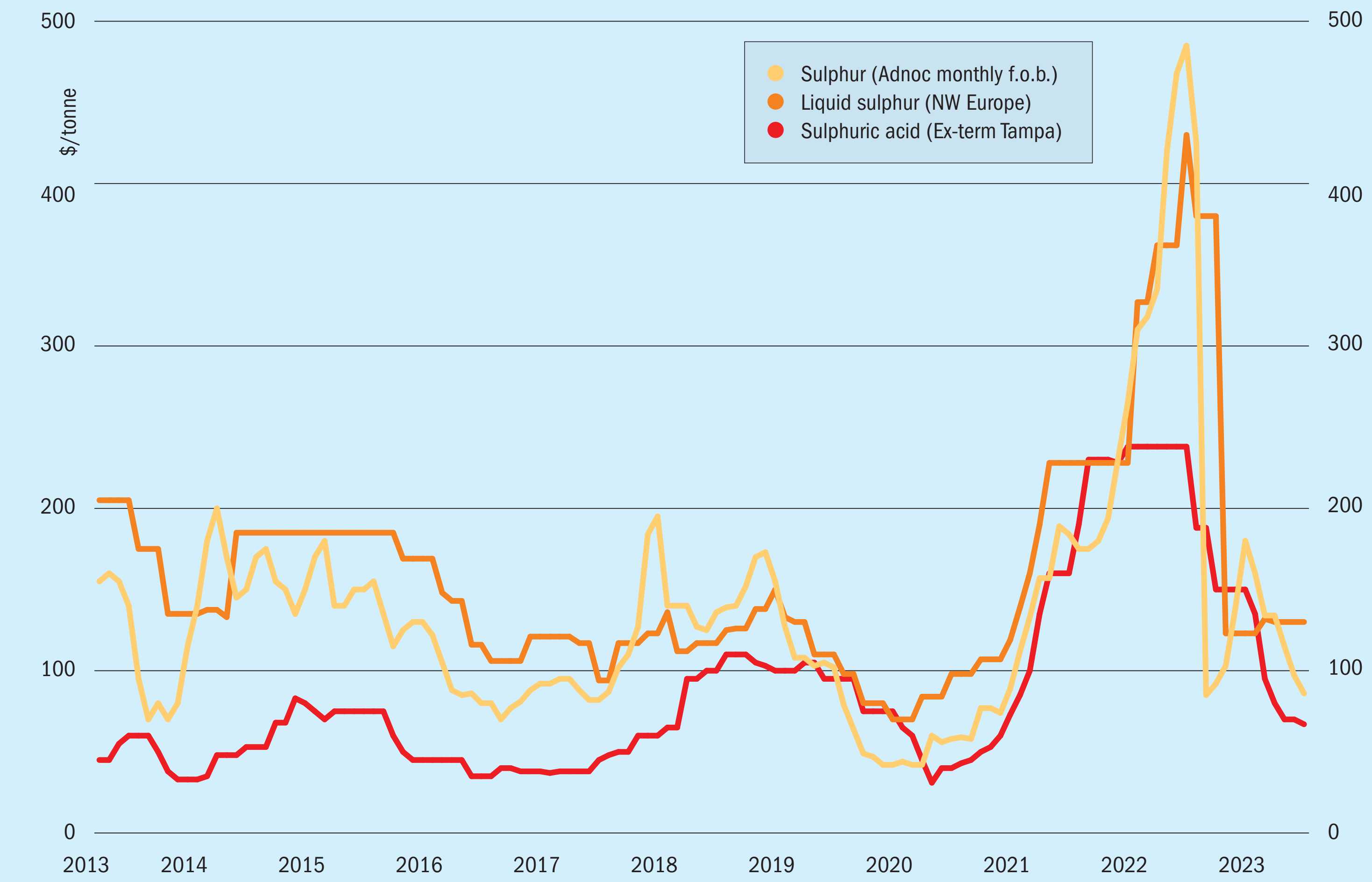
Cash equivalent	February	March	April	May	June
Sulphur, bulk ($/t)					
Adnoc monthly contract	134	134	115	97	86
China c.fr spot	158	150	132	118	112
Liquid sulphur ($/t)					
Tampa f.o.b. contract	125	125	103	103	103
NW Europe c.fr	132	130	130	130	130
Sulphuric acid ($/t)					
US Gulf spot	95	80	70	70	67

Source: various

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56

Market Outlook

Historical price trends $/tonne



Source: BCInsight

SULPHUR

- Indonesian imports have increased in 2023 so far on a year earlier. As new nickel high pressure acid leach projects ramp up, demand for sulphur is expected to increase further. Swing buyers have been importing significant volumes of sulphuric acid, affecting short term sulphur demand in the second quarter. It remains to be seen if this will continue, we expect sulphur demand to ramp up further in the second half of the year, bringing import expectations for the year to around 2 million t.
- China sulphur capacity is forecast to rise even further in 2023, with the first quarter reflecting a 5% year on year rise. After reaching a record 9 million t/a in 2022, we expect production to climb to over 10 million t/a. New gas-based capacity and refining projects will ramp up to support this view.
- In Canada, a strike by members of the International Longshore and Warehouse Union (ILWU) Canada halted commodity loadings across Canadian-Pacific ports, including sulphur. The standstill of sulphur loadings will likely lead to a build up at Alberta prilling sites and inventory builds at production sites.
- Outlook: Global sulphur prices are expected to stabilize and see slight uplift in July following the recent downturn. Consumption is emerging in key markets such as Indonesia, supporting the view for the downturn to turn to stability. Expected softness in DAP prices in the short term remains a bearish factor but with sulphur currently undervalued against DAP, there is room for increases in sulphur pricing.

SULPHURIC ACID

- Morocco remains on the sidelines of the spot market, with no sulphuric acid vessels scheduled in the line up for Jorf Lasfar. OCP is expected to see higher consumption in the second half of the year but attractive sulphur prices are likely to keep interest in acid limited.
- Negative pricing in South Korea/Japan has emerged on the back of cargoes for shipment in the August-September period with lower delivered prices in key markets. Smelter maintenance in Japan during the fourth quarter will limit acid availability, with potential for prices to rise on the back of this.
- India buyers CIL and Iffco are expected to turn increasingly to the sulphur market with the start of new sulphur burning capacity in the 2023-24 period. Acid imports are expected to be impacted as a result.
- Outlook: Further softening is likely before a floor in pricing is reached out of northeast Asia. Market sentiment remains weak from key markets such as Morocco and Chile. There is potential for recovery as demand returns and if sulphur prices start to see uptick, swing buyers may choose to enter the acid market.

Sulphur Industry News

KAZAKHSTAN

Sour gas reinjection resumes at Kashagan

Kazakhstan's oil and condensate output increased by 7% from 1.79 million bbl/d to 1.92 million bbl/d in early June after sour gas reinjection operations resumed at the Kashagan offshore oil and gas development following a recent outage, according to the Kazkah Energy Ministry. Reinjection of sour gas into two wells resumed on 8th June, enabling operator the North Caspian Operating Company (NCOC) to boost oil and condensate production at a large artificial island in Kazakhstan's Caspian Sea waters. Reinjection was paused on May 20th following the detection of sour gas during routine sampling and a subsequent integrity test. Kashagan normally produces about 300,000 barrels of oil per day. Kazakhstan expects Kashsgan to raise oil production this year to 18.2 million t/a from 12.7 million t/a in 2022.

In spite of this, state oil producer KazMunayGaz has reported declining revenues and profits. 1Q 2023 revenues fell by 14% to $4.24 billion, while net profit declined by 13% compared to the same period of 2022 in site of a 9% rise in oil and condensate production to more than 500,000 bbl/d. KazMunayGaz says that it has been able to export crude via the Russian ports of Novorossiysk and Ust-Luga at a significant premium to Russia's Urals export blend, even though Kazakh oil commingles with Russian crude once it enters the country's pipeline network, and has the same density and sour content as Urals, and there are no restrictions on Kazakhstan's oil shipments from Russian ports, with Kazakh crude labelled Kebco to differentiate it from Urals.

Kazakhstan has also said that it plans to change the terms of oil and gas production sharing agreements with foreign majors that operate the three largest developments in the country in a bid to raise more tax.

Kazakh Prime Minister Alikhan Smailov said the government is creating a special joint group to work out proposals to amend existing PSAs to be discussed with international partners on the Tengiz, Kashagan and Karachaganak fields. The group will be led by the Ministry of Energy, and will include representatives of other ministries, the parliament, state investment holding Samruk-Kazyna and KazMunayGaz.

TAIWAN

Quality award for gas scrubbing system

CPC Taoyuan received the 22nd Public Construction Golden Quality Award for the successful startup and continuous operation of its new wet gas scrubbing system for flare gas desulphurisation. CPC partnered with Mascol Engineering and Elessent Clean Technologies for the design and provision of the MECS® DynaWave® system, which features reverse jet scrubbing nozzles designed to remove hydrogen sulphide and waste water chemical oxygen demand using high efficiency soda scrubbing. A typical Claus sulphur recovery unit only recovers 95%-98% of sulphur present. The DynaWave® scrubber is an open duct in which scrubbing liquid is injected through a non-restrictive reverse jet nozzle, counter current to the dirty inlet gas. Liquid collides with down-flowing gas to create the "froth zone," a region of extreme turbulence with a high rate of mass transfer, and this froth zone technology allows for desulphurisation in a wet gas environment, delivering over 99.9%+ sulphur removal.

Eli Ben-Shoshan, Elessent CEO, said of the project, "We are delighted to be a part of this project and to assist CPC Taoyuan with emissions regulations compliance… We are committed to providing clean solutions that are specifically designed to address the demands of the oil and gas industry and reduce the environmental impact of refineries on the environment."

For sulphur recovery unit (SRU) tail gas cleaning, the DynaWave® technology makes it possible to quench incinerated gas, remove potential sulphur particulate entrainment, and absorb SO_2. The technology offers high turndown capabilities and the unique flexibility of handling extremely high SO_2 concentrations when conventional tail gas treatment units (TGTUs) are bypassed during start-up, maintenance, or unexpected outages, ensuring uninterrupted SRU operation with guaranteed low emissions at all times.

CANADA

S&P forecasts increase in oil sands production

Higher crude prices and continued optimisation improvements have driven the first upward revision to the S&P Global Commodity Insights 10-year oil sands production outlook in more than half a decade.

The new forecast, produced by the S&P Global Commodity Insights Oil Sands Dialogue, expects Canadian oil sands production to reach 3.7 million barrels per day (bbl/d) by 2030; 0.5 million bbl/d higher than today. The new projection represents an increase of 140,000 b/d in 2030 from the previous outlook.

"Higher oil prices have driven record returns for the Canadian oil sands," said Celina Hwang, Director, North American Crude Oil Markets, S&P Global Commodity Insights. "Although producers continue to demonstrate capital discipline, stronger balance sheets are now giving oil sands companies renewed confidence in regard to their intentions for capital spending."

The main driver of the upward revision has been the identification of additional opportunities to improve efficiency and/or optimise output, the analysis says. The ongoing ramp-up and operational efficiency gains from learning by doing and step-out optimisation projects are the most significant contributors. Step-out optimisations are a relatively new phenomenon and include, as the name suggests, stepping out from existing operational areas into new high quality adjacent lands.

Capital expenditures for oil sands production in 2022 reached their highest levels since 2015 and could rise further this year. However, most of that increase occurred to offset increased inflation and there has not been a resurgence in large-scale greenfield or even brownfield oil sands projects, the analysis says.

"The Canadian oil sands have entered an 'era of optimisation'," said Kevin Birn, Vice President, Canadian Oil Markets Chief Analyst, S&P Global Commodity Insights. "Learning by doing and step-out optimizations account for nearly 90% of our overall production outlook. The remainder of additions are expected to come from yet another form of optimisation – debottlenecking projects. Optimisations now dominate the oil sands production growth outlook."

A deceleration in growth is expected to begin around the mid to late 2020s, but a very shallow decline only begins to emerge in the early 2030s.

DENMARK

Sustainable aviation fuel plant on track for 2026

Arcadia eFuels, a leading developer of green fuels has signed a license agreement with Sasol and Topsoe for Arcadia's Vordingborg eFuels plant. The agreement represents a major milestone ahead of Arcadia's final investment decision. Once operational, the plant will deliver green fuels for the Danish and European aviation markets to help meet the European Union mandate of 1.2% Renewable Fuels of non-Biological Origin in 2030.

Amy Hebert, CEO of Arcadia eFuels, said: "We're pleased to conclude this license and engineering agreement with two best in class companies acting as a single point licensor. Their commercial experience in syngas, Fischer Tropsch and refining technology development and licencing combined with Arcadia's novel engineering approach allows us speed to market. Meeting the needs of the aviation and heavy transportation industry's decarbonization efforts is of high priority for Arcadia."

NIGERIA

Dangote Refinery commissioned

A commissioning ceremony was held at the end of my for the 650,000 bbl/d Dangote Petroleum Refinery and petrochemicals plant, presided over by outgoing Nigerian president Muhammadu Buhari. The Dangote Refinery is Africa's largest oil refinery and the world's largest single-train facility. It will also integrate refining with petrochemical production, producing not only various petroleum products but also polypropylene and polyethylene. The refinery will meet 100% of Nigeria's requirements for refined products as well as having a surplus for export, creating a market for $21 billion per annum of Nigerian crude. Nigerian crude oil typically has an API gravity between 28 and 42. Lighter crudes are easier to refine as they contain fewer impurities and require less energy for processing. It is also known for its low sulphur content, which makes it desirable for refining. Low sulphur content reduces the emissions of sulphur dioxide (SO2) during combustion, helping refineries comply with environmental regulations.

Despite having a refinery nameplate capacity that can meet nearly all of its domestic demand, Nigeria, the largest oil producer in Africa, is fully reliant on imported petroleum products to meet domestic demand because its state-owned refineries have been shut in for long-term maintenance or rehabilitation since 2020.

KUWAIT

Al Zour to reach capacity by end of the year

Kuwait's Al-Zour refinery has restored full operation of crude distillation units 1 and 2, with output back up at 345,000 bbl/d, up from 205,000 bbl/d in April. The third CDU is expected to be ready by the end of 2023, bringing production to the refinery's full capacity of 615,000 bbl/d, according to Kuwait Integrated Petroleum Industries Co (KIPIC). KIPIC said in April that its CDUs 1 and 2 were offline temporarily due to a technical problem. The second CDU had only started up operation in early March. However, the CDU 3 planned startup by the year's end is reported to be a "more cautious prediction" than earlier plans for it to be operating by around July.

CHINA

PetroChina producing sour gas from Tieshanpo

PetroChina says that it has begun sour gas production at the Tieshanpo block in the southwestern province of Sichuan. The field was abandoned by Chevron in 2019. PetroChina is aiming to tap the high H_2S content (14.2-15.5%) reserves to boost gas production as part of its energy transition drive. Six production wells have been drilled so far, able to produce 1.71 million cubic metres per day of gas. In the long run, it will add three more production wells at Tieshanpo. The development includes one new dehydration station, two gas collection well stations, one handover metering station and an export pipeline extending 17.3 kilometres, PetroChina said. The two-train gas plant at Wanyuan city is able to treat 4 million m^3/d of gas.

Gas production at Tieshanpo is running six months behind the initial schedule because of safety measures to prevent leaks. PetroChina has put in place a simultaneous leakage monitoring and emergency support system by using fixed gas detectors and a cloud-mounted laser leakage monitoring system in the gas gathering and transportation facilities, as well as infrasonic leak detection, distributed optical fibre acoustic sensing and distributed optical fibre temperature sensing.

Tieshanpo is part of the Chuandongbei sour gas complex, which includes the Dukouhe, Qilibei and Luojiazhai assets. PetroChina brought in Chevron as a partner at the Chuandongbei sour gas project in 2007 after several incidents highlighted its need for expertise in handling the high sulphur content gas. Chevron has a 49% non-operated interest in the Luojiazhai play, which is already in production, but relinquished its interests in the Tienshanpo, Dukouhe and Qilibei natural gas plays four years ago.

UNITED ARAB EMIRATES

Bids in for Hail and Ghasha

Expressions of interest are in for the latest EPC tendering round for the massive Hail and Ghasha sour gas development after ADNOC began a new tender round for the project in April, following the cancellation of the pre-construction services agreements (PCSAs) that had been awarded in January, reportedly due to cost constraints. Firms were initially asked to express interest in the new EPC tendering round by 14 May, later extended until 19 May. ADNOC's execution strategy for the development has now been divided into three packages: subsea pipelines, umbilicals, cables, risers and other offshore structures; offshore drilling centre facilities, the Ghasha offshore processing plant and central living quarters; and the Manayif onshore processing plant, including offsite export pipelines and tie-ins, utilities, the main control building and process buildings. Work on a Ruwais sulphur-handling terminal and other non-process buildings is an optional scope for this package.

ADNOC holds the majority 55% stake in the Ghasha concession, along with Eni (25%), Wintershall (10%), OMV (5%) and Lukoil (5%). Production is targeted at 1.5 bcf/d of sour gas from the Ghasha concession by the middle of this decade. Four artificial islands have already been completed in the Ghasha concession, and development drilling is under way.

Sulphuric Acid News

FINLAND

Metso introduces battery black mass recycling process

Metso is launching an advanced sustainable battery black mass recycling process as part of its battery minerals technology offering, which covers concentration and hydrometallurgical processing as well as related services. Demand for battery minerals is increasing sharply with the ongoing transition to clean energy sources. An electric car battery weighs approximately 200 kg. Recycling of black mass from batteries with Metso's process can reduce up to 60% of embedded carbon compared to use of virgin materials and enables the treatment of mechanically separated and shredded batteries for recovering battery raw materials like nickel, cobalt, and lithium, as well as manganese and copper.

The process is based on Metso's proprietary VSF® X solvent extraction technology and complemented with OKTOP® reactors, Larox® PF filters, dual media and LSF filters, and thickeners and scrubbers. The process flowsheet can be tailored according to feed materials and desired end products with a possible phased approach for adding equipment also for the recovery of less valuable materials.

"With the launch of the battery black mass recycling process, our offering for the battery minerals value chain covers 90% of the end-to-end production process. We can provide sustainable technology and equipment for the entire lithium, nickel, and cobalt production chain from the mine to battery materials and black mass recycling with project scopes ranging from equipment packages to plant deliveries. We can also support our customers in the design of the process with our comprehensive testing and research capabilities," said Mikko Rantaharju, Vice President, Hydrometallurgy at Metso. ■

TAIWAN

SAR awards contract for spent acid regeneration

SAR Technology Inc. has awarded MECS, Inc., a subsidiary of Elessent Clean Technologies a contract to supply of proprietary technology and equipment as part of a new spent acid regeneration plant at Tainan. By incorporating MECS® sulphuric acid recovery technology in its electronic grade sulphuric acid plant, SAR aim to position themselves as a reliable supplier of regenerable high purity sulphuric acid to the semiconductor industry in Taiwan, which is the world's largest consumer e-grade sulphuric acid.

The semiconductor industry is one of the fastest growing markets in the world, and its manufacturing process creates numerous by-product waste streams. Disposal of this waste has become a challenge with the extreme growth experienced by the industry, and one way to combat waste disposal issues is by recycling the by-product spent acid from semiconductor fabrication. MECS designs sulphuric acid regeneration plants to balance operating temperature, acid concentrations and corrosion rates to ensure consistent, productive uptime of the equipment while guaranteeing environmental compliance and offering proven protection of plant equipment from acid condensation.

"Every manufacturer in the industry wishes to embrace a circular economy on waste streams, especially at a time with such high demand. It is exciting to work with SAR Inc. who is shouldering the responsibility to ensure the sustainability of the e-grade acid supply chain." said Eli Ben-Shoshan, CEO, Elessent.

SAR Inc. was established in 2018 to provide a full-cycle electronic-grade sulphuric acid factory to deliver a sustainable solution for by-product waste in the semiconductor industry. Start-up of the plant is slated for June 2023 and is expected to improve utilisation of domestic resources and help address sulphuric acid disposal challenges.

UNITED STATES

Sumitomo buys Saconix

Sumitomo Corporation has announced its acquisition of Saconix LLC, a company engaged in the procurement, sale, storage and distribution of sulphuric acid in the United States. Sumitomo bought Saconix from Dallas-based Copperbeck Energy Partners. Saconix is one of the leading sulphuric acid distributors in North America providing logistics and storage solutions with strategically located assets, adding value through its functions and services that are very similar to Sulphuric Acid Trading Company (SATCO), another Sumitomo Group company. Saconix owns, leases and operates in seven strategic logistics facilities in the US Gulf and West Coast. The acquisition expands Sumitomo's footprint in the US sulphuric acid business, adding operations and access now in both the Gulf region and West Coast region, including California, Nevada and Arizona, and will take the Sumitomo Group's worldwide annual volume of acid handled to 3.5 million t/a via 19 tanks with approximately 330,000 tonnes capacity.

"We identified Saconix as a strategic investment because its business model is extremely compatible with our current investments, and brings a high potential of growth synergy for our sulphuric acid business in the US, increasing our global competitiveness," said Masaya Sato, Basic Chemical Group General Manager of SCOA.

Sumitomo Group's sulphuric acid business began with the export of sulphuric acid produced from the smelting of nonferrous metals from Japan, and since 1994, when it acquired Interacid Trading in Switzerland, the parent company of SATCO. The company handles one of the world's largest sulphuric acid maritime transport transactions and provides a wide range of logistics services in countries such as the US and Chile, including the trading of sulphuric acid by sea, regional distribution, and storage of sulphuric acid. Interacid owns and operates its own storage tanks for sulphuric acid, providing customers with just-in-time delivery of product, and if needed, leasing of storage tank space to customers requiring personalized logistics services.

Approval reversed for phosphate project

On June 2nd, 2023, a federal judge reversed previous US government approval for a phosphate mining project in southeastern Idaho. The judge ruled that the US Bureau of Land Management violated environmental laws when it approved the Caldwell Canyon Mine in 2019. Those include a failure to consider the indirect impact of processing ore at a nearby plant and the impact on sage grouse. The mine has been proposed by P4 Production LLC, a subsidiary of German pharmaceutical giant Bayer AG, and was to include two new open mine pits to extract phosphate ore, over a mine life of 40 years, with ore taken by truck or rail to a nearby processing plant.

ZIMBABWE

Zimplats commissions concentrate plant

Zimbabwe Platinum Mines (Ltd) (Zimplats), part of the mining conglomerate Implats Group, has commissioned its third concentrator plant as part of a $1.8 billion expansion plan. The concentrator, with a capacity of 900,000 t/a, processes platinum ore and separates the most valuable minerals or metals while discarding the rest of the ore as tailings.

Speaking at the official opening of the facility at Mhondoro Ngezi, Implats board chair Thandi Orleyn said: "This is a culmination of a long journey which started over 20 years ago when Implats decided to invest in Zimbabwe and resuscitate the mothballed BHP operations." Zimplats' expansion programme, approved and adopted in 2021, comprises nine projects in Ngezi, including the concentrator, and will be implemented over a 10-year period. The plant will process platinum group metals ore including platinum, palladium, rhodium, ruthenium, iridium, and osmium through crushing, milling, floatation and filtration. Projects include the installation of a 110 megawatt solar power plant with sufficient capacity to satisfy the company's and related mining and mineral beneficiation facilities, as well as a 100,000 t/a sulphuric acid plant which will supply the manufacture of fertilizer in the country, reducing acid imports.

CHINA

Zinc market boosted by Chinese government economic support

Zinc markets have picked up after China's central bank lowered interest rates for the first time since August 2022 to restore market confidence and prop up a stalling post-pandemic recovery. Further interest rate cuts are expected in 2H 2023 to boost the economy. There is also hope that the government will try to support China's depressed property sector, a consumer of a number of commodity metals and raw materials.

Poseidon Nickel's Black Swan project

PHOTO: POSEIDON NICKEL

Chinese refined zinc output for the year stood at 564,500 tonnes in May, an increase of 24,500 tonnes per month and 9.6% up year on year. However, some smelters in Henan have reduced production due to high sulphuric acid inventories. The global zinc market remains in surplus, estimated at 26,700 tonnes in March, according to the International Lead and Zinc Study Group (ILZSG), though this surplus is falling. During the first three months of 2023, ILZSG data showed a surplus of 49,000 tonnes, versus a surplus of 116,000 tonnes in the same period of 2022.

AUSTRALIA

Poseidon continuing with Kalgoorlie restart

Poseidon Nickel Ltd says that it has made significant progress towards the restart of its Black Swan smelter-grade concentrate project, situated 50 km northeast of Kalgoorlie in Western Australia. The restart is based on the November 2022 bankable feasibility study for the 1.1 million t/a mill feed option to produce smelter-grade concentrate. A final investment decision (FID) is due to be made in late June or early July this year. Work continues on the expansion project which is based on treating 2.2 million t/a of mill feed and producing a rougher concentrate. The rougher concentrate product has a lower nickel grade and higher magnesium oxide content than conventional smelter feed and is more attractive as a feed for either pressure oxidation (POX) or high-pressure acid leach (HPAL) plants rather than a conventional nickel smelter.

Construction begins at Arafura

Ground preparation and early works construction has begun at the Nolans rare earths project, being developed by Arafura, 135 km from Alice Springs in Australia's Northern Territory. Arafura says that it has begun equipment procurement including sulphanation bake and cooler units, and has begun constructing roads and earthworks and a construction camp. The company recently signed an offtake agreement with wind turbine manufacturer Siemens Gamesa Renewable Energy for the supply of neodymium and praseodymium for the manufacture of permanent magnets. The company has also signed an offtake agreement with Hyundai. Nolans is considered one of the world's largest rare earth deposits and could supply 10% of the world's demand for the metals used in rare earths magnets. The company plans a hydrometallurgical plant and sulphuric acid plant as part of the project.

TOGO

Phosphate output up

Togo's phosphate industry recorded substantial growth in production and sales in 2022 on the back of high global prices for the commodity. Recent figures from the Central Bank of West African States (BCEAO) show Togolese phosphate production grew by 5.9% in 2022, reaching 1.54 million t/a. Sales rose by 14% from 1.39 million t/a to 1.58 million t/a, its highest value since 1999. Growth was mainly spurred by a 116% increase in global raw phosphate prices over the year, extending an upward trend that took root in late 2020. Togo's phosphate company, SNPT, has also benefited from the rising dollar exchange rate.

SNPT has signed several agreements with OCP Group for the establishment of a fertilizer plant in Togo, and there is also a new NPK fertilizer plant at Adétikopé, being developed by Singapore-based NutriSource Pte Ltd. The factory is expected to produce 200,000 t/a of fertilizer.

INDIA

Vedanta invites bids to restart operations at Sterlite

The Vedanta Group has announced an invitation for Expressions of Interest (EOIs) to restart operations at the Sterlite Copper plant located in Tuticorin. The EOI includes safety assessment/audit of structures and buildings, refurbishment or replacement of plant and machinery, and repair works, aimed at restoring the Sterlite Copper plant to its design capacity (400,000 t/a of copper and 1.2 million t/a of sulphuric acid).

The Vedanta Group says that maintenance and upkeep activities at the plant have recommenced as permitted by a Supreme Court order on May 4th, 2023, in coordination with district authorities. The final hearing on the matter is scheduled for August.

Considering the significant restoration required after the plant's closure for over five years, the procurement and deployment of necessary materials and resources for restoration and restart might be a time-consuming process lasting several months. In this context, the EOI has been issued to ensure the selection of suitable partners as part of the preparatory measures for resource planning. The Sterlite plant has been closed since May 2018 after violent local protests against its operations. Prior to that it had met 40% of India's refined copper demand, and was the largest producer of sulphuric and phosphoric acid in South India.

CHILE

Ceibo attracts $30m of investment

Chilean copper extraction technology company Ceibo says it has raised $30 million in Series B financing. The round was led by Energy Impact Partners, with participation from a syndicate of new mining-focused investors including CoTec Holdings, Audley, Orion Resources, Unearth and Pincus Green along with existing investors Khosla Ventures and Aurus Ventures. The funding round will enable Ceibo to accelerate the scale-up of its proprietary technology, drive several on-site trials and prove the technology's value and versatility at scale.

Declining ore grades, changing mineralogy and constrained permitting processes threaten to limit copper supply growth and challenge the energy transition. Ceibo has developed a novel leaching process and solution that economically increases the production of copper, using existing infrastructure at mines with a lower environmental footprint than current state-of-the-art. Ceibo's technology targets chalcopyrite and other refractory copper minerals which hold 70%+ of known copper reserves. It economically and sustainably unlocks supply of copper from sulphide ores, which traditionally are expensive and environmentally intensive to produce.

"Following positive results from a lengthy validation process using minerals from leading copper companies, we're now poised to rapidly scale our technology," said Cristóbal Undurraga, CEO and Co-founder of Ceibo. "This capital raise, together with the strategic support from investors experienced in scaling clean, industrial, and mining technologies, will help us accelerate plans to deploy our technology globally and meet society's need."

IRAN

Copper leach operations begin in northwestern Iran

Iran has started up a new copper cathode production plant in its northwestern Azerbaijan region where the country's second largest copper reserves are located. According to Islamic Republic of Iran Broadcasting, the new copper cathode plant at Varzaqan will be able to produce 3,000 t/a of copper cathode via heap leaching. Iranian president Ebrahim Raeisi, announcing the start of operations, also said that he would make preparations for the launch of an independent company in the region to control the Sungun copper mine which is the largest open-cast copper mine in Iran. Sungun had been controlled by National Iranian Copper Industries Co. (NICICO) a company that is mostly focused on copper mines and smelters in Iran's southeastern province of Kerman.

INDONESIA

Nickel Industries seeking financing for Excelsior

Australia's Nickel Industries says that it is seeking A$943 million (US$645 million) in funding for its Excelsior nickel project in Indonesia. The company says it has agreed a deal with Chinese-based Shanghai Decent, who will take the right to a 20% equity interest in Stage 2 of the project. Excelsior is aiming to produce 72,000 t/y of nickel equivalent as a mixed hydroxide precipitate, and will be capable of producing both nickel sulphate and cathode, differentiating it from the current generation of high-pressure acid leach (HPAL) plants being built across Indonesia, and providing Nickel Industries with significant operating flexibility through the cycle. Shanghai Decent will provide a capital expenditure (capex) guarantee, whereby total construction and commissioning costs will not exceed $2.3 billion. The capex guarantee includes a tailings solution, which is best of breed for tailings storage and management, and an integrated sulphuric acid plant that will generate significant heat that can be turned into power, significantly lowering the carbon footprint of the operation. Construction is expected to start in either December 2023 or March 2024, or sooner if both parties agree. Commissioning will start no later than 24 months thereafter. ■

People

Metso Outotec's annual general meeting (AGM) in early May approved the board of directors' proposal to change the company name to Metso Corporation. "After the successful integration of Metso and Outotec, we will focus on growing a strong unified Metso company and brand," says President and CEO of Metso, Pekka Vauramo. "We have combined two valuable companies into one strong Metso. Our focus is clear: we continue enabling sustainable modern life and transforming the industry with a clear strategy and strong culture, supported by a name that is short yet established and well recognized among all our stakeholders. Services are an extremely important part of our business, requiring a strong name."

Outotec will continued as a product brand and as the name of some key technologies and products. The change of the parent company name Metso Corporation was effective from May 4th, 2023, but globally the change will be implemented in a phased manner. The visual identity of the company remains unchanged, but the new name is reflected in the company logo.

The company has also announced that **Mikko Vainikka** will take up a post as Group Treasurer of Metso Corporation from August 1st, 2023. The current Group Treasurer **Minna Helppi** has resigned to pursue her career outside of Metso.

"I would like to thank Minna for her strong contribution during the past 11 years leading our treasury operations and wish her all the best for her next career step. At the same time, I am pleased to welcome Mikko as the new Group Treasurer. Mikko has over 10 years of experience in our treasury which enables a smooth transition," said CFO Eeva Sipilä.

First Phosphate Corp. says that **Garry Siskos** has been appointed Chief Operating Officer (COO) and Chief Financial Officer (CFO) of the Company. First Phosphate outgoing CFO, **Bennett Kurtz**, will be moving to the position of Chief Administrative Officer (CAO). **Gilles Laverdière** has been appointed as Chief Geologist for the company.

Garry Siskos has over 25 years of international experience as CFO and COO inside entrepreneurial and multinational public and private business environments. He specialises in strategy, capital raising, structured finance, mergers and acquisitions. He is experienced in the technology, mining, and automotive sectors, and was involved in two of Canada's Top 100 growth organisations.

"I am excited to continue my career journey alongside John and his team in optimizing the value of the Company's unique lithium iron phosphate (LFP) battery related mining assets," said Siskos. "The global transition towards sustainable energy presents a compelling opportunity for First Phosphate. Our LFP related mining assets will play a pivotal role in supporting this global paradigm shift."

The International Fertilizer Association (IFA) has announced that **Tony Will** has been elected as the new Chair of the Association. Will is president and chief executive officer (CEO) of CF Industries, a global manufacturer of hydrogen and nitrogen products for fertilizer, clean energy, emissions abatement and other industrial applications.

"I am honoured to serve as Chair of IFA and help advance this vital organization's mission to promote the efficient and responsible production, distribution and use of plant nutrients," Will said. "Our industry is at the forefront of some of the world's most important challenges, from food security to climate change. I look forward to working with our members and the IFA team to continue our leadership role in addressing these global priorities, collaborating not just within our own industry but with government and other stakeholders as well."

Jeanne Johns, at the time managing director and CEO of Incitec Pivot Ltd., was elected as the new vice chair of the Association.Both Will and Johns serve on the Executive Board of the Association, which also welcomed two further new appointments: **Ahmed El Hoshy**, CEO of OCI Global and Fertiglobe and **Abdulrahman Shamsaddin**, CEO of SABIC Agri-Nutrients. Svein Tore Holsether, president and CEO of Yara, remains on the Executive Board as Immediate Past Chair, along with Raviv Zoller, President and CEO, ICL Group and Alzbeta Klein, Director General, IFA. "Helping to feed the world sustainably is at the core of what we do, and IFA is fortunate to have such seasoned executives on our Board," Klein said.

Calendar 2023

SEPTEMBER

11-15
30th Annual Brimstone Sulphur Symposium, VAIL, Colorado, USA
Contact: Mike Anderson, Brimstone
Tel: +1 909 597 3249
Email: mike.anderson@brimstone-sts.com

11-15
Amine Experts' Amine Treating & Sour Water Stripping Technical Training Course, KANANASKIS, Alberta, Canada
Contact: Daniel Domanko, Senior Manager
Tel: +1 403 215 8400
Email: Daniel.Domanko@SulphurExperts.com
Web: AmineExperts.com

13-14
Oil Sands Conference & Trade Show, CALGARY, Alberta, Canada
Contact: Bruce Carew, EventWorx
Tel: +1 403 971 3227
Email: marketing@eventworx.ca

18-22
Sulphur Experts' Sulphur Recovery Technical Training Course, KANANASKIS, Alberta, Canada
Contact: Daniel Domanko, Senior Manager
Tel: +1 403 215 8400
Email: Daniel.Domanko@SulphurExperts.com
Web: SulphurExperts.com

OCTOBER

9-13
Amine Experts Amine Treating Technical Training Course, NOORDWIJK, Netherlands
Contact: Jan Kiebert, Senior Manager
Tel: +31 71 408 8036
Email: Jan.Kiebert@SulphurExperts.com
Web: AmineExperts.com

16-20
Sulphur Experts Sulphur Recovery Technical Training Course, NOORDWIJK, Netherlands
Contact: Jan Kiebert, Senior Manager
Tel: +31 71 408 8036
Email: Jan.Kiebert@SulphurExperts.com
Web: SulphurExperts.com

NOVEMBER

6-8
CRU Sulphur & Sulphuric Acid Conference 2023, NEW ORLEANS, Louisiana, USA
Contact: CRU Events
Tel: +44 (0)20 7903 2444
Email: conferences@crugroup.com

13-16
European Refining Technology Conference, LAGO MAGGIORE, Italy
Contact: World Refining Association
Tel: +44 (0)20 7384 8056
Web: worldrefiningassociation.com/event-events/ertc

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New phosphate demand – India and Brazil

The centre of gravity of the phosphate industry continues to shift, with Chinese exports less important, and fresh demand coming from India and Brazil.

The phosphate market, which remains the mainstay of industrial sulphuric acid demand, has continued to see-saw over the past few years, hit first by covid and the attendant shutdowns, and then, when the global economy was beginning to recover, by Russia's invasion of Ukraine and the attendant sanctions which restricted availability from key suppliers like Russia and Belarus.

Global phosphate rock production stood at 62.9 million t/a P_2O_5 in 2021, with production split as shown in Figure 1. The world's largest producer remains China, which represents the vast bulk of East Asian production and which produced 21.3 million t/a P_2O_5 of phosphate rock, 34% of the world's supply, mostly for domestic consumption. Next comes Morocco, at 11.6 million t/a P_2O_5 (18%), and the United States, with 6.3 million t/a P_2O_5 (10%) of production. Russia was the fourth largest producer, followed by Jordan and Saudi Arabia.

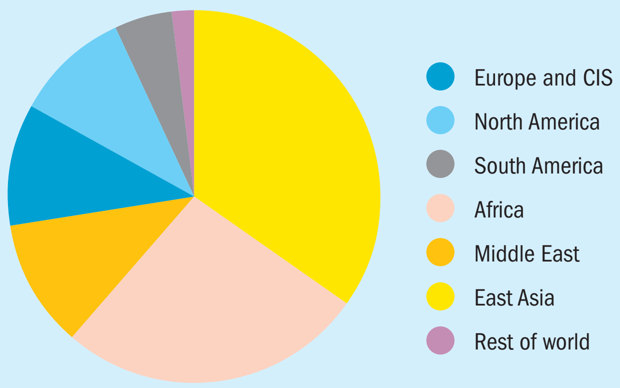
On the processed phosphate side, global phosphoric acid production was 44.9 million t/a P_2O_5, with production split as per Figure 2. On the whole the split is similar to Figure 1, but as Figure 2 shows, Africa produces less finished phosphates a share of world production, and exports phosphate rock to India, China and other places to produce finished phosphates. This is changing quickly however as Morocco expands its downstream phosphate industry.

Overall, the regions that have the greatest influence on phosphate production and trade, and hence consumption of sulphuric acid, are China, North America, India, South America and North Africa. China, India, Brazil and the USA between them account for 60% of all phosphate demand globally.

Global picture

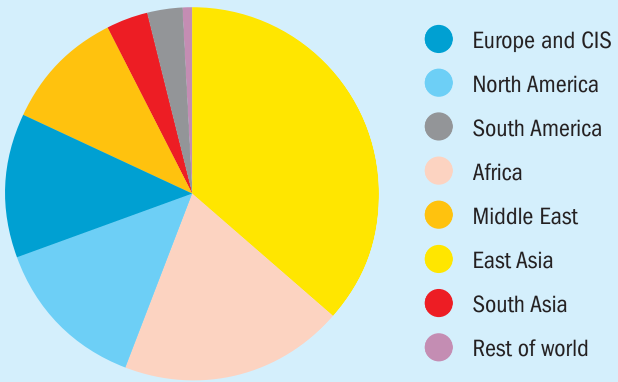
The US Department of Agriculture (USDA) indicates that previously steadily increasing global grain yields per hectare have levelled off in the past few years, and this has contributed to declining global grain stocks since 2018, now down to historic lows as a percentage of demand/use. This has helped support higher crop prices for corn and oilseed, exacerbated by a prolonged drought in North America and Argentina/southern Brazil due to several successive La Nina years. Demand for phosphate tends to be more affected by crop prices than fertilizer cost. Better affordability for phosphate fertilizers bodes well for demand recovery this year. There was a major dip in demand last year due to high prices caused by lack of availability from Russia and Chinese export controls. Combined mono- and di-ammonium phosphate demand (MAP/

Fig 1: **Phosphate rock production 2021**



Source: IFA

Fig 2: **Phosphoric acid production 2021**



Source: IFA

DAP) fell from 64.6 million tonnes product to 58.7 million t/a in 2022. CRU forecasts a bounce back to 63 million t/a this year, though still down on 2021's figure.

China

China remains the world's largest producer and consumer of phosphates. Phosphate fertilizer production capacity stood at 21.4 million t/a P_2O_5 in 2021 according to the China Phosphate and Compound Fertilizer Industry Association (CPFIA), though this was down by 3.3 million t/a from 2016. Production of finished phosphate fertilizers was 16.8 million t/a P_2O_5 in 2021, and consumption 11.3 million t/a, with 5.7 million t/a P_2O_5 of phosphate fertilizers exported. Most of China's phosphate industry produces mono- and di-ammonium phosphate (MAP/DAP); around 86% of all phosphate is produced in these two forms, as compared to 46% of global finished phosphate production. This preponderance of MAP and DAP means that in the past China has dominated the export markets for these products, representing 35-40% of global trade in DAP and 19-24% in MAP in 2021. Production fell sharply in 2022, however, to 15 million t/a P_2O_5, a decline of 12% year on year, as high global phosphate prices due to the Ukraine war and sanctions on Russian phosphate led to consumption cutbacks. Phosphate prices peaked around June-July 2022 and fell back thereafter, with another uptick at the end of the year. High prices for phosphate rock (inland Chinese prices almost doubled) also affected production margins. At the same time, China has maintained export restrictions on phosphates in order to prioritise its domestic market, where demand remained strong in spite of the higher prices. These began in late 2021 and were maintained throughout most of 2022. The amount of phosphate licensed for export was only a fraction of what it had been for the previous year. Chinese MAP/DAP exports were 3.3 million t/a P_2O_5 in 2022, down 43%. The restrictions have continued into 2023, though were eased a little from April, meaning that Chinese exports for this year are likely to be higher than last.

China's phosphate industry is concentrated (85%) in four southern/central provinces. The phosphate industry has been shrinking for most of the past decade, suffering as it does from declining consumption as China tries to rein in overapplication of some fertilizers; over-building of capacity; and environmental pressures on plant operators, especially those in the Yangtse River region. The government continues to strive for greater nutrient use efficiency, targeting an increase from 40% to 43% from 2020-2025, and is promoting new fertilizer technologies such as water-soluble fertigation and new products with greater use of micronutrients, as well as substitution with more organic fertilizer. Domestic demand is thus likely to continue a slow decline. Meanwhile development of new MAP/DAP production plants is very restricted and the structural change in China's phosphate industry continues. China is increasingly viewing phosphate as a strategic resource, and CRU recently reported that there could be cuts to downstream capacity in order to manage China's phosphate rock reserves over the longer term.

PHOTO: PHOSPHATE MISR

Phosphate rock mining in Egypt.

North America

The US was the largest producer of phosphate rock in the world throughout the 20th century, but its dominant position has declined over the past two decades as competition has evolved elsewhere. US production of phosphate rock peaked in 1980 at 54.4 million metric tons, and has more than halved since then. As phosphate rock mining and processing has shrunk in North America, the North American industry has consolidated, leaving just four major producers; Mosaic, Nutrien, Simplot and Itafos, at nine sites. Production of phosphoric acid in 2021 was 6.1 million tonnes P_2O_5.

A larger planted area for corn, soybean and wheat is forecast for 2023; roughly a 3.5% increase year on year due to higher crop prices and better spring weather. Itafos says it expects phosphate demand from June 2022-June 2023 to be 3.85 million t/a P_2O_5, a 9% increase on the previous year, though still significantly down from 2019-2021. Imports and exports are expected to be relatively flat year on year.

Looking out to 2026, however, Canada is set to return to large scale phosphate production with the start-up of Arianne Resources' Lac a Paul mine.

India

India has a significant domestic finished phosphate industry but little domestic phosphate rock mining. Consequently the country is one of the most important importers of phosphate rock and phosphoric acid, and the largest importer of processed phosphates like DAP. India's phosphate consumption was 7.83 million t/a P_2O_5 for the fertilizer year 2021-22, according to the Fertilizer Association of India, part of a steady increase over the past few years. This included 9.27 million t/a of DAP (tonnes product), 5.68 million t/a of single superphosphate (SSP), and 11.68 million t/a of nitrophosphates and NPK fertilizer. Domestic phosphate fertilizer production was 4.7 million t/a P_2O_5, including 4.2 million t/a (product) DAP and 5.35 million t/a SSP. Imports of DAP ran at 5.46 million t/a.

India has suffered from systematic overapplication of urea at the expense of other fertilizers. In an effort to improve crop yield and provide more balanced nutrition the government is attempting to push greater application of phosphate and potash fertilizers. As a result, Indian phosphate demand is projected to increase by around 2.2% year on year, including a 3.2% increase in 2023.

South America

Latin America continues to be a hotspot for new phosphate demand is South America, with Brazil far and away the largest consumer. Brazil's fertilizer requirements have more than doubled in the past two decades, and phosphate applications have climbed from just over 3.5 million t/a P_2O_5 in 2010 to 7.5 million t/a in 2021. The rest of Latin America adds another 2.5 million t/a P_2O_5 of consumption, more than half of this represented by Argentina and Mexico. Phosphate consumption has risen equally rapidly in these countries; around a 4% increase year on year in Argentina, 5% year on year in Mexico, and a remarkable 16% in Peru, now the fourth largest consumer in South America. Phosphate demand is continuing to increase, to counter phosphate deficiencies in topsoil in e.g. Argentina's grain-growing Pampas region, as well as to serve new areas being brought into cultivation. Growth is predicted to average 5-6% year on year for the region over the next few years.

As far as domestic fertilizer production goes, phosphate rock in the region is mined mainly in Brazil and Peru. But downstream capacity remains limited. Total phosphoric acid production in 2021 was 2.6 million t/a P_2O_5, 1.5 million t/a of that in Brazil, and most of the rest in Mexico, and the region has to import to cover the gap.

North Africa/Middle East

On the production side, North Africa and the Middle East are hugely important to the global phosphate industry in terms of phosphate rock production, but also increasingly in terms of finished phosphates (and hence sulphuric acid demand). Morocco is the world's largest holder of phosphate reserves and the second largest producer. But with little domestic phosphate demand, it is by far the world's largest exporter of phosphate rock and, increasingly, processed phosphates. State producer OCP is continuing a major expansion programme that it began in 2007 to not only boost phosphate rock production but also to develop more downstream phosphate production and export capacity in an attempt to capture more value from its phosphate resources. As the lowest cost producer of phosphates, OCP's increase has come at the expense of edging out exports from China and the United States. Last year, Morocco was the largest exporter of MAP and DAP with a total of 6.3 million tonnes product, placing it ahead of China, previously the world's largest exporter.

So far, OCP's expansion has seen rock mining capacity increase by 50% to 44 million t/a, the construction of a gravity driven slurry pipeline to take rock from mines to Jorf Lasfar, and a huge expansion of phosphate processing and phosphate fertilizer manufacture at the Jorf Lasfar Hub. Four large integrated facilities have been built at Jorf Lasfar, each with a capacity of just over 1.0 million t/a of MAP and DAP, and each consuming 500,000 t/a of sulphur each to feed sulphuric acid and phosphoric acid capacity. Jorf Lasfar has grown to become the largest fertilizer facility in the world, with a capacity of 6 million t/a of phosphoric acid and 10.5 million t/a of fertilizers and with a production of 5.65 million t/a of phosphoric acid and 10.18 million t/a of fertilizers. This year, the start-up of Jorf Lasfar Line F will add another 450,000 t/a (P_2O_5) of MAP and DAP capacity. OCP has been increasing its sulphur melter capacity at Jorf Lasfar to feed acid production at these plants, with capacity now at 12,000 t/d (4 million t/a).

Other new developments include upgrading of the existing Euro Maroc Phosphore (EMAPHOS) lines 3 and 4 at Jorf Lasfar (originally part of a joint venture with Brazil's Bunge but brought back into full OCP ownership several years ago) by 10% each, as well as the addition of a new 500,000 t/a phosphoric acid plant, and the commissioning of a new 450,000 t/a phosphoric acid line at Laayoune, taking OCP's phosphoric acid capacity to 8.4 million t/a.

Algeria is also a major phosphate rock reserve holder, mainly exporting phosphate rock (ca 1 million t/a). Last year, following a reorganization of state industries to create fertilizer group Asmidal, a $7 billion deal was signed with Chinese firms Wuhuan Engineering and Tian'An Chemical, a nitrogen and phosphate fertilisers production company, to create the Algerian Chinese Fertilizers Company (ACFC). ACFC aims to develop a world-scale integrated phosphates project. Asmidal will own 56% of the new company while the Chinese companies will own the remaining 44% stake. The plan is to develop phosphate deposits at Bled El Hadba, Djebel Onk, and Tebessa, with downstream conversion into MAP and DAP using Algeria's domestic ammonia production. It will also include the construction of port facilities at the port of Annaba. Overall the project is expected to have 5.4 million t/a (product) of phosphate fertiliser production capacity.

Tunisia is attempting to rebuild its phosphate industry after years of stagnation due to industrial and political unrest. Phosphate rock production stood at 8 million t/a prior to the Arab Spring, but declined to less than half that in 2011 and has recovered only patchily since then. More recently, however, efforts at improving production seem to be bearing fruit, with the Gafsa Phosphates Company projecting that output will rise to 5.6 million t/a this year.

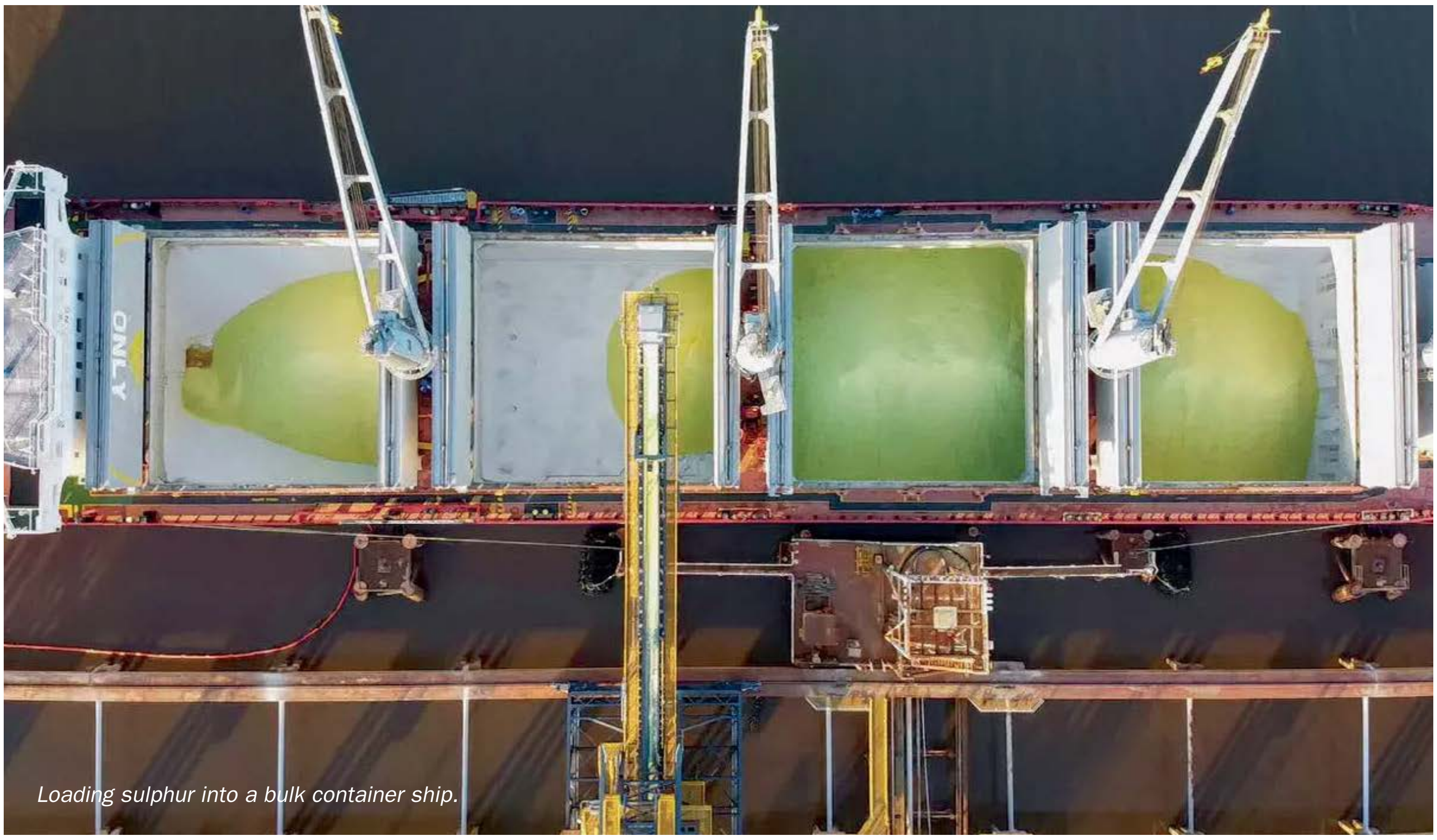
In Egypt, another phosphate rock exporter (ca 3 million t/a), there are also moves to expand mining and greatly expand downstream phosphate production, at Abu Tatour, where 900,000 t/a of phosphoric acid capacity is planned to come onstream by 2024. Jordan is a major phosphate producer, and now Saudi Arabia has also moved into phosphate expansion and processing via state-owned mining company Ma'aden. Two large phosphate complexes are already up and running, the most recent, the Wa'ad Al Shamal joint venture with Mosaic in 2017, and a third mega-complex is planned for 2026.

Sulphuric acid demand

CRU forecasts that total world phosphoric acid capacity will rise from 60.5 million t/a in 2021 to 64.3 million t/a in 2027. This represents annual growth of around 2.0%. Ma'aden III and OCP represent the largest slices of Over the same period, demand is forecast to increase by 3.68 million t/a P_2O_5, only just shy of the new capacity additions (3.8 million t/a). This represents additional demand for sulphuric acid to produce phosphoric acid of around 9 million t/a, with Morocco, Saudi Arabia, India and Russia the major recipients. This will equate to additional sulphur demand for India and Morocco and reduced exports from Saudi and Russia.

Sulphur dust suppression

Sulphur dust is one of the greatest hazards when producing and handling solid sulphur, and methods for its suppression are vitally important to prevent fire and explosion.



Loading sulphur into a bulk container ship.

PHOTO: BRUKS SIWERTELL

While sulphur is a fairly benign substance in terms of toxicity and is a solid at room temperature and pressure, making handling fairly easy, it has various characteristics which can be problematical if not looked after. As is detailed in the article elsewhere in this issue (pages 44-53), it is very brittle and friable, which means that rough handling can cause it to break into progressively smaller pieces.

The condition of the sulphur begins with the sulphur recovery unit (SRU) which is producing it. Running outside of normal temperature limits, the inclusion of contaminants, or even catalysts used in the degassing process can all contribute to the properties of the formed sulphur, and the proportion of polymeric sulphur molecules which can reduce friability.

But undoubtedly the greatest influence comes from the forming process itself. One complication of sulphur is the crystal phase change between the higher temperature monoclinic form to the orthorhombic crystal structure, which occurs at around 95°C. The speed at which the sulphur is cooled during forming and at what temperature it is subsequently stored can affect this phase change. Sulphur has a very high specific heat capacity and acts as an insulator, so the bulk solid can retain heat for a considerable period. The monoclinic form is stronger than the orthorhombic form, and rapid cooling can preserve the monoclinic form in the cooled sulphur, but this will gradually change over time as the sulphur ages. The phase change is also exothermic and contribute to caking.

Forming processes roughly divide into those which produce dry sulphur and those which include entrained water with the sulphur. The main dry forming process is pastillation, whereby water is sprayed under a steel belt on which liquid sulphur drops are deposited to produce pastilles. Wet processes include granulation, where water is sprayed into a rotating drum to cool liquid sulphur that has been sprayed onto a seed curtain to form sulphur particles or 'seeds', and wet prilling, where liquid sulphur is introduced at the top of a forming tank for direct counter-current heat exchange, with water, used to produce prills that are withdrawn from the bottom of the forming tank.

In general wet sulphur generates less dust, as the water allows fine particles to agglomerate, but this is complicated by sulphur's hydrophobic nature, which can prevent water from completely wetting particles. For this reason surfactants are often added to improve the water penetration into the sulphur.

Dust hazards

The main hazard associated with sulphur dust is fire and/or explosion. Sulphur burns if ignited, and when there is a large surface area to air ratio this can be rapid enough to cause an explosion. The lower explosion limit for sulphur is usually quoted as 35g/m³, but finer particles can lower this to around 20g/m³. Finer particles (<75 micrometers) are easier to suspend in the air, and also have a lower ignition temperature, and can be set off by static electricity sparks generated by sulphur particles rubbing against each other. Therefore, care must be taken to prevent static electricity accumulation in areas where solid sulphur is handled.

Smoking and the use of matches must be prohibited in all areas where sulphur dust is likely to be present. Naked flames or lights and the use of gas cutting or welding equipment should also be prohibited during the normal operation of the plant. It is also important to remove potential sources of ignition. To prevent excessive heat build-up, all equipment should be properly and securely installed to ensure the correct alignment of rotating shafts, belt tension etc. The surface temperature of plant or machinery should not be allowed to exceed 2/3 of the ignition temperature of sulphur dust (i.e. 127°C). Once sulphur burns or explodes, it also generates sulphur dioxide, which is toxic.

Sulphur can be abrasive, so care must be taken to use equipment that can withstand prolonged exposure to sulphur. If the sulphur is being pneumatically conveyed into a processing system, the blower used to move this material through the air line must be sized to meet the demands of system. The use of 316 grade stainless steel is usually recommended for any equipment that is exposed to sulphur.

There is also the risk of corrosion, especially where wet sulphur is present. The carbon steel in long term contact with wet sulphur can produce ferrous sulfide on the surface. Water has a catalytic effect on the spontaneous combustion process of ferrous sulfide, and the initial self-heating temperature of fire sulphide can be reduced from 141°C when there is no water to only 40-60°C when water is contained. Humidity in the air can also promote self-heating performance of ferrous sulfide.

Dust minimisation

In order to prevent dust fires and explosions there are two basic strategies; to avoid suspensions of dust in the air, and to exclude sources of ignition. Dust formation can be reduced by reducing the number of transfer points and handling steps to a minimum, and try to minimise the use of front end loaders which can crush sulphur granules beneath their wheels.

Some grades of powdered sulphur can be non-free flowing and the material may tend to cake or pack. This characteristic can affect feeding of the conveyor, as wells as the discharge of the material from the conveyor. Because of this characteristic, it is important to select infeed transitions with the correct geometry as well as the provision of flow-aids, such as vibrators or agitators, to ensure trouble-free feeding of the conveyor.

Fine sulphur particles can adhere to the surface of conveyor belts and fall due to the vibration of the belt and the change in tension, causing the fine sulphur to be scattered along the belt, or be lifted to land on any horizontal surfaces. Storage in buildings can protect dry sulphur from the elements, but – given the explosive nature of sulphur – it also requires greater dust management. To prevent dust accumulation during storage and handling of sulphur, enclosures should be constructed with a minimum number of horizontal surfaces where dust can accumulate, and access to any and all hidden areas. Good housekeeping is a must, with inspection of and cleaning of dust residues at regular intervals.

When reclaiming sulphur from storage, gravity-based systems can produce less product damage, but rely on the flow characteristics of the sulphur, which can be impacted by caking in moist environments. Mechanical systems are more aggressive and impart more stresses onto the product.

Dust suppression

While dust minimisation and good housekeeping is essential for keeping dust levels down, dust formation cannot be prevented completely. The next step then is to suppress dust. Water can be sprayed at transfer points and load-out station; the wetted particles agglomerate to each other and to larger particles, making them more difficult to be picked up by air currents. However, as previously noted, water promotes the formation of acidity and wet sulphur corrosion. For this reason a maximum moisture content of 2% is recommended.

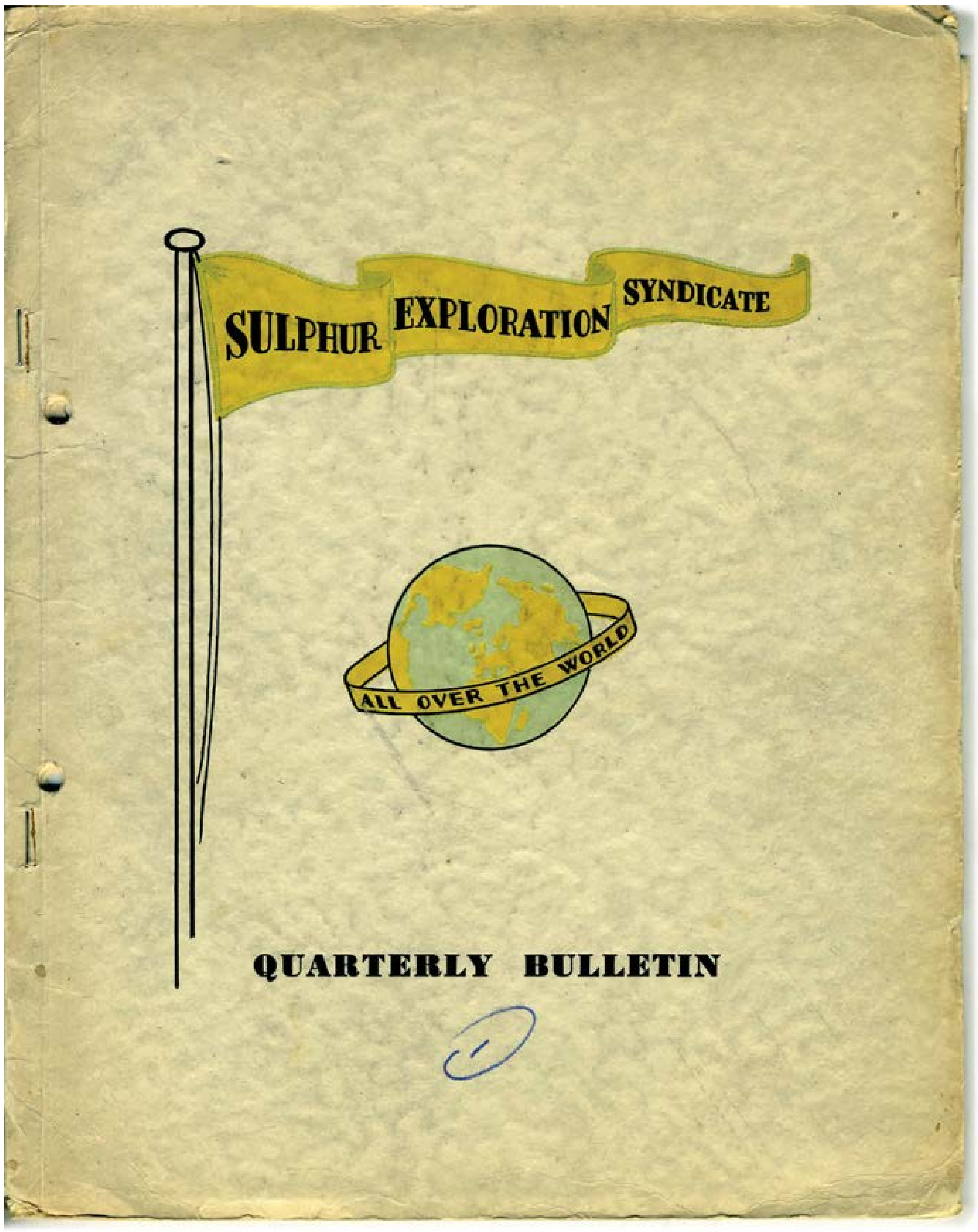
The effectiveness of water for dust control is also limited by the hydrophobic nature of sulphur. This can be overcome by the use of special water-based chemical surfactants. When air is mixed with the surfactant/water mixture, foam is generated. Foam is a very effective means of dust control, made up of small bubbles 100-200 micrometers in size. When it comes into contact with dust particles the bubble breaks, coating the particle. When several come into contact they coalesce into a larger particle and can no longer become airborne as dust. Foam suppression systems create 0.5-1.0% moisture on the specified transfer point according to the transfer rate. Foam is normally applied upstream of transfer points to reduce dust formation during transfer.

Fire

Even with preventive technology in place, there are rare occasions on which fire will nevertheless occur. Fire detection systems should be in place along the conveyor line, perhaps linked to automatic water spraying systems and interrupts to stop the conveyor and prevent fires entering a storage area or building.

When a sulphur pile is on fire, it is necessary to avoid directly impacting the sulphur pile or the ground around the fire point with a direct water jet because the air flow brought by the water column impacting the sulphur pile or the ground will disturb the sulphur dust and suspend the sulphur dust in the air, potentially reaching the explosion limit. The best way to extinguish fire is to use nozzles with good atomisation effect, A water curtain spray system can be used together to form a liquid mist screen of fire extinguishing agent and reduce the possibility of dust and sulphur suspended in the air. ■

Sulphur at 70



The first issue of *Sulphur* magazine – then the *Quarterly Bulletin of the Sulphur Exploration Syndicate* – was printed in June 1953, 70 years ago.

This June marks a milestone for this magazine; a platinum jubilee since the very first issue of the magazine was printed in 1953. It began life as the Quarterly Bulletin of the Sulphur Exploration Syndicate. The Syndicate was created in 1952, and was backed by nine major chemical producers, mainly in Britain and the US, who were concerned about dwindling world supplies of sulphur. Though some of these companies have vanished by the wayside over the years, including F.W. Berk and Co. Ltd, British Titan Products, Brotherton & Co., and Charles Tenant & Sons Ltd, others remain household names to this day, including Monsanto, Courtaulds (now part of Akzo-Nobel), and Dunlop (now owned by Goodyear), while Fisons' fertilizer division was sold to Norsk Hydro in 1982 and today trades as part of Yara.

The syndicate in turn was a response to the publication in 1952 of the Paley Report by the United States Materials Policy Commission. This report estimated that the total available supplies of low-cost Frasch sulphur in the US were around 50-100 million tonnes in the ground, and that a further 250 million tonnes could potentially be sourced worldwide at an additional cost of 25-50% on prevailing market prices. Sulphur (in all forms) consumption in 1950 had been 4.8 million t/a in the United States and 6.7 million t/a elsewhere, though the 'Soviet Bloc', which at the time also included 'Red' China, was excluded from these figures. This figure was anticipated to rise to a global consumption of 25 million t/a by 1975. By that time, over 400 million tonnes of sulphur would have been consumed, and that was more than the 300-350 million tonnes of elemental sulphur reserves that had already been identified. It seemed clear that new sources of sulphur were needed, and the Sulphur Exploration Syndicate aimed to find them.

The Syndicate, and the magazine, was the brainchild of John Lancaster. Czech by birth, Lancaster fled Nazi occupation of his country in 1938 and arrived in England, anglicising his name in the process. Like many exiles from eastern Europe he served with the British Army during the war, in his case specialising in Military Intelligence, with particular responsibility for identifying sources of key raw materials, of which sulphur was of course one. He and colleague Major-General Godfrey Wildman-Lushington CBE were among the founders of the Sulphur Exploration Syndicate, which by 1955 had become the British Sulphur Corporation. At the same time the Quarterly Bulletin had become the *Journal of World Sulphur*, and very shortly thereafter, simply *Sulphur*.

Industry evolution

The sulphur industry was a very different beast in 1953. Production was almost exclusively by Frasch mining,

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with the United States, the place where it was first used by Herman Frasch at what is now Sulphur, Louisiana, still by far the largest producer. In 1952 the US produced 5.5 million t/a of sulphur, or 88% of the sulphur output of what the Quarterly Bulletin somewhat quaintly calls the 'Free World'. Outside of the US, only Italy, Norway and Japan produced more than 100,000 t/a, with Italy, once the world's largest producer in the 19th century, still relying heavily on mined volcanic sulphur deposits from Sicily. The US was of course also the largest exporter, at 1.3 million t/a, with Canada and the UK the main destinations.

> **"The sulphur industry was on the cusp of the seismic change that it would go through during the remainder of the 20th century."**

Of equal importance was 'sulphur in other forms', i.e. sulphur recovered from smelter operations, mainly in Europe, and sulphuric acid production from pyrite roasting. But in its survey of the sulphur industry in mid-1953, the Bulletin does note that the oil industry, a major consumer of sulphuric acid, was actually beginning to generate more sulphur than it consumed, by recovery from cracker off-gases, regeneration of spent sulphuric acid, and, especially in Canada, by treatment of sour natural gas. In the UK, sulphur recovery began at Shell's Stanlow refinery in 1952, to the tune of 12,000 t/a, with a similar amount to come onstream in 1954 from Standard Oil's Fawley refinery, and BP (then the Anglo-Iranian Oil Company) at Grangemouth. Recovered sulphur in the US reached 248,000 t/a in 1952, and the Bulletin comments that "legislation in connection with the sour gas exports to the United States and to eastern Canada is thought to be imminent. It is known that Texas Gulf Sulphur Corporation is actively interested in a large-scale recovery project of sulphur from sour gas." It also comments that; "mention should be made of the vast deposits of bituminous sands in Northern Alberta. The Fuels Division of the Departments of Mines and Technical Surveys at Ottawa and other research stations have developed several different processes for the extraction of cleaned bitumen and for the recovery of sulphur from it."

The sulphur industry was therefore clearly on the cusp of the seismic change that it would go through during the remainder of the 20th century, as sulphur from sour gas and refineries, particularly in Canada and the US, came to dominate world sulphur production. In the US, the Clean Air Act was passed in 1963, and it and its various amendments progressively restricted sulphur content of fuels. Frasch sulphur was still the dominant form until the 1980s, when the rapid expansion of sour gas processing and refinery recovery began to outstrip it. The last US Frasch mine closed in 2000, and today there is only one Frasch mine still operational, in Poland.

Pricing

Then, as now, the magazine covered sulphur markets and pricing, and the figures are instructive. In 1953, US sulphur prices were about $30/t f.o.b., but end users like Italy or Japan could pay $80-90/t c.fr. These prices are, so my inflation calculator tells me, equivalent to $340/t and $910-1,025/t respectively – think on that if you feel your sulphur is expensive! The move from Frasch mining to Claus recovery and the consequent scale up of the industry has brought prices down considerably in real terms, though perhaps not as much as it has for other commodities.

The future

If the 1980s and 90s were the time of the triumph of the recovered sulphur industry in North America, then the period since then has been an equally notable internationalisation of the industry, with sour gas recovery spreading to China, the Middle East and Central Asia, and the Middle East and Asia's rapidly growing oil refining industries taking over from the shrinking output of Europe and North America. Consumption has changed too, with the very rapid growth of China over the past three decades and its need for phosphate fertilizer, and increasing use of sulphuric acid to recover key minerals like copper, nickel, uranium and now even rare earth metals.

During that time, global elemental sulphur output has increased more than tenfold – it turns out that the Sulphur Exploration Syndicate needn't have worried about a looming shortage of sulphur after all. And yet, as the world begins to decarbonise and use of fossil fuels plateaus and, eventually, declines, we are starting to hear similar concerns about how we will source sufficient sulphur in future. *Plus ça change…*

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Middle East Sulphur Conference 2023

The 2023 Middle East Sulphur Conference (MEScon 2023) was the biggest sulphur event in the region to date with over 650 attendees representing 23 countries from across the sour gas and sulphur value chain. More than a conference, this annual event offers conversation, collaboration and community, with leaders and experts from across the industry sharing knowledge, best practice and predictions for the future of sulphur.

PHOTO: CRU/UNIVERSUL

Group photo of many of the women speakers and delegates of MEScon 2023.

The Middle East Sulphur Conference (MEScon) organised by CRU and UniverSUL Consulting, took place 15-18 May at the Conrad Abu Dhabi Etihad Towers in Abu Dhabi. On Day 1, three pre-conference workshops, hosted separately by BASF, BR&E, and Comprimo, took place at the conference hotel as well as a number of technical showcase presentations. A busy exhibition started on the afternoon of Day 1 and ran alongside the conference over four days with vendors highlighting product portfolios, new innovations and technologies to improve performance and/or reduce costs.

In her introductory remarks for the opening ceremony of the main conference on Day 2, Angie Slavens, Managing Director at UniverSUL Consulting, expressed that it was a pivotal moment as the event's lead sponsors, ADNOC and Aramco, the two largest sulphur producing companies in the world (comprising approximately 20% of global production) kicked off the day's activities, delivering inspiring welcome addresses that underlined the vital role the Middle East will continue to play in the future of the industry.

For example, the speakers discussed how growth plans across the Middle East are due to account for more than half of all sulphur production growth by 2030, with global sulphur supply due to grow from 65 million tonnes to 80 million tonnes of sulphur by 2027. Adbulmunim Al Kindy, Executive Director at ADNOC Upstream, proposed a challenge to the delegates to consider ways of extending the dual advantage that ADNOC already has in oil production, lowest possible cost and lowest possible carbon, to sulphur production. Meanwhile, Mohammed Al Abdulqader, Director of Gas Operations, Technical Support Department at Aramco, discussed Aramco's historical and planned future sulphur growth and plans for future investment in SO_2 emissions reduction for existing SRUs.

Later in the morning, CRU's Principal Analyst for Sulphur and Sulphuric Acid, Peter Harrison, delivered a fascinating overview of the sulphur market, noting that sulphur supply in the Middle East saw significant growth from 2014-2016 and this trend is happening once again. Though, the interesting long-term challenge will be managing supply side capabilities as demand growth continues.

This demand for sulphur was a central point of discussion throughout the day, with many speakers highlighting that sulphur is not just a waste by-product of oil and gas, but instead one of the most valuable commodities in today's market. This was evidenced by conversations around its dominance in fertilizers, supporting global food supply; but also its role in emerging markets from EV batteries through to clean energy storage – both of which are only set to expand.

CRU's Principal Consultant, Ionut Lazar, talked about this during his presentation on the evolving role of sulphur in the EV value chain. He predicted that sustained sulphur demand will remain in the phosphates market, but lithium and battery markets will be a growth potential for acid.

The afternoon's technical sessions focused on outside-of-the-box thinking with regards to the value of hydrogen sulphide (H_2S) and elemental sulphur as presented by operating companies, technology

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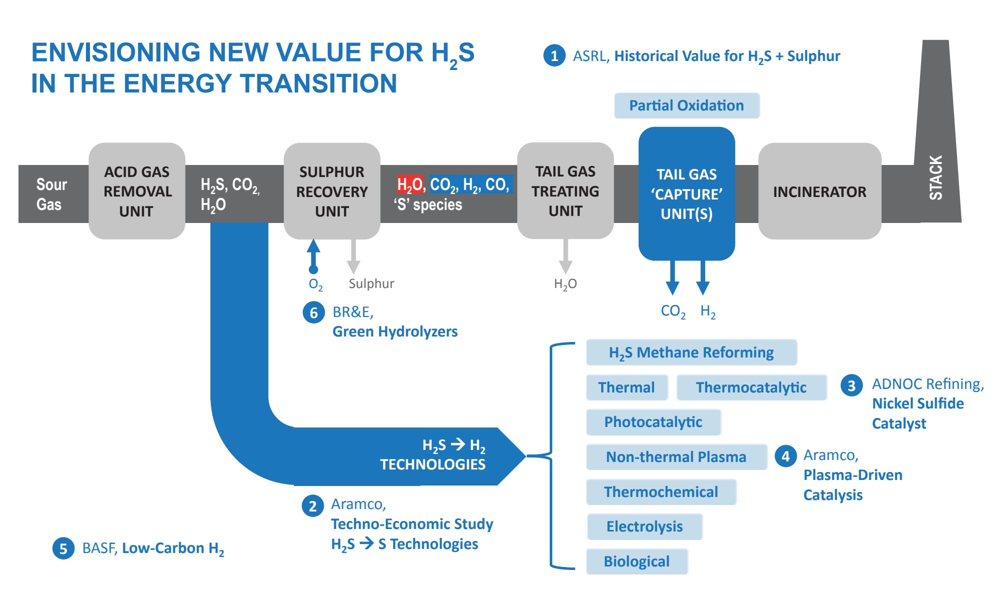
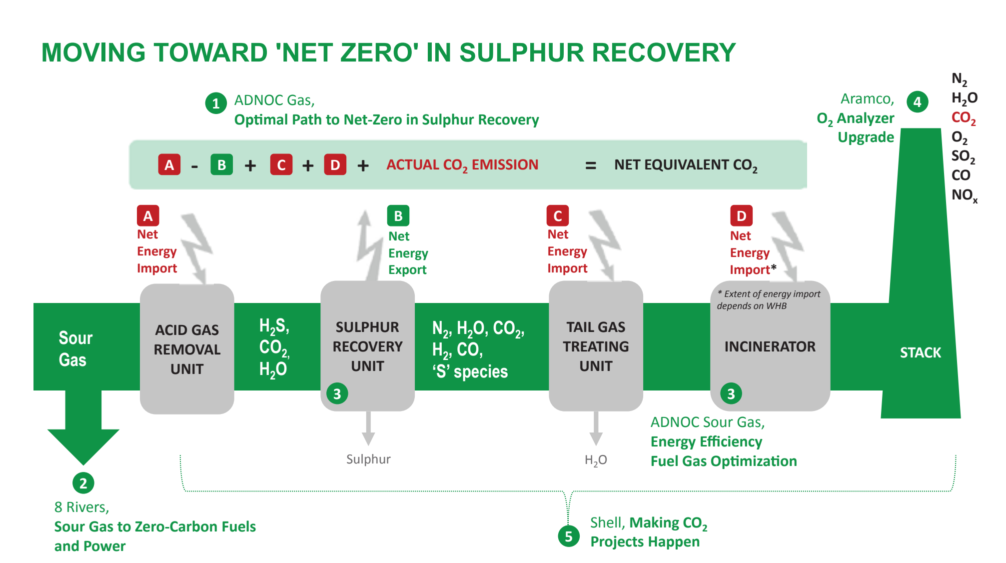


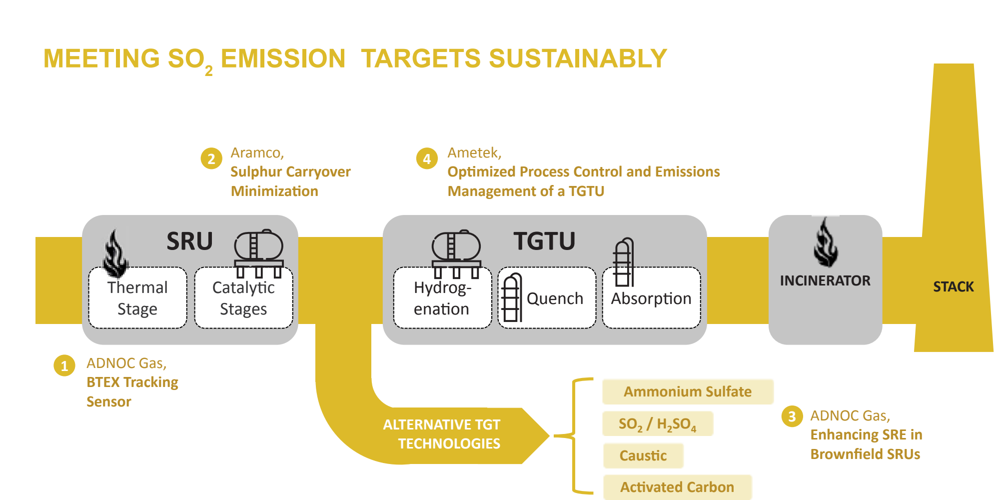
Source: UniverSUL Consulting

Fig. 1: Overview framework for MEScon Day 2, afternoon session.



Source: UniverSUL Consulting

Fig. 2: Overview framework for MEScon Day 3, morning session.



Source: UniverSUL Consulting

Fig. 3: Overview framework for MEScon Day 4, afternoon session.

providers and industry suppliers (Fig. 1). Rob Marriott, Director of Research, at Alberta Sulphur Research kicked off the session with an overview of historical attempts to create new value for H_2S and sulphur, followed by Pramod Patil, Reservoir Engineering Technology Division at Aramco, who presented multiple innovative schemes for H_2S conversion to H_2 and sulphur. These two overarching presentations were followed by specific presentations on research and development in H_2S to H_2 conversion, delivered by ADNOC Refining, Aramco and BR&E, as well as low carbon hydrogen schemes, presented by BASF.

Ali Al Hendi, ADNOC Gas Vice President Technical and Engineering Services, opened Day 3 of the event with a welcome speech describing the importance of MEScon to ADNOC Gas. This ADNOC company operates one of the largest sour gas facilities in the world, equipped with sulphur recovery technologies from many of the major SRU technology licensors, most of whom were attending the conference, and expressed the importance of knowledge exchange between designers and operators.

Day 3 then officially kicked off with a flurry of new participants eager to join the morning deep dive into decarbonisation in the 'Moving toward net zero in sulphur recovery' session (Fig. 2). Saqib Sajjad, Team Leader, Energy Management, ADNOC Gas, started the session with a comprehensive overview of the sulphur plant energy balance and explored interesting options for green energy integration. 8 Rivers' Joe Weiner then presented an innovative sour gas treating technology, with inherent CO_2 capture, utilising cryogenic distillation. Al Sail Al Jaberi, Process Engineer at ADNOC Sour Gas, then described the efforts to reduce fuel gas consumption and corresponding CO_2 footprint in the reaction furnace and incinerator of the world's largest SRU/TGTU trains at the Shah Gas Plant. Bader Alotaibi, Process Engineer at Aramco, then shared Aramco's experience with the implementation of an innovative laser stack analyser to achieve accurate stack O_2 concentration, thereby reducing fuel gas consumption and carbon footprint. Shell's Fahd Fathi then wrapped up the session with a talk on how best to achieve viable economics for CO_2 capture projects in sour gas and sulphur.

A first for the event was an operator poster session, which was briefly summarised by poster presenters during the main conference and conducted on screens

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in the exhibition area during breaks. On Day 3, posters were presented by ADNOC Gas, KNPC, and Aramco, providing delegates with the opportunity to learn and exchange experience directly with operating companies.

The afternoon session was also about sharing operational experiences. Ben Spooner, Principle Engineer at Amine Experts, kicked off the session with a presentation detailing his experience commissioning amine plants in extreme environments – hot, cold and offshore (as featured in *Sulphur* 403, pp48-53). ADNOC Sour Gas process engineers Jawwad Kaleem and Eman Al Ali then presented the many challenges the Shah Gas Plant has overcome in increasing its massive sour gas facility from 1 to 1.45 BSCFD.

More operators took the stage as Muhammad Syu`aib Mohamed Yahaya, Process Engineer at Pengerang Refining Company Sdn. Bhd., shared his learnings from a full sulphur plant complex start-up in 2018, followed by Reliance Syngas Ltd's presentation from Alok Dwivedi and Sarbendu Kar, who explained the optimisations implemented to increase flexibility in the refinery and gasification SRUs in the Jamnagar Refinery – the largest refinery and gasification complex in the world. The session wrapped up with presentations from KNPC's Rizwan Masood, who described the game-changing results achieved during the design and commissioning of AGRP II, followed by ADNOC Gas' Sara Al Katheeri and Pankaj Kumar who described a dual-advantage project which improved the quality of their formed sulphur product while also decreasing capital cost.

The panel session in the afternoon provided a great opportunity for operators to interact with one another and the audience over a wide range of projects; from commissioning to revamp, and types of facilities; from gas plants to refineries to gasification facilities. What was clearly evident was that this type of discussion amongst end-users provides a much-needed feedback loop to front-end engineering designers, which strengthens the entire project execution chain.

Mohamed Bara Adi, ADNOC Sour Gas VP Projects and Technical, opened Day 4 of the event with a welcome speech harkening back to the sentiments expressed by Fahad Al Wahedi in the opening ceremony – that MEScon is a connected community that is extremely important for facilitating knowledge sharing between their operations personnel who are operating the largest sulphur trains in the world, and the global sulphur community. He congratulated the delegation for the lively discussion thus far and encouraged everyone to finish strong on this final day of the conference.

Day 4 then officially kicked off with a session on design innovations to achieve optimal performance and longevity. Gerton Molenaar, Director Business Development Americas at Comprimo, started the discussion with an overview of an innovative development to achieve precise SRU control. Bob Poteet of WIKA and Shaima Almajed of Aramco then took the stage to share their experience developing and implementing a new thermal reactor shell temperature measurement device to detect refractory failures. Johannes Derfler of AGRU then presented an innovative and robust PFA lining that has proven effective in sulphuric acid service, and the audience subsequently proposed and debated potential applications for this material in sulphur plant service in the panel session

Welcome address by Abdulmunim Al Kindy, Executive Director, ADNOC Upstream.

Welcome address by Mohammed Al Abdulqader, Director, Gas Operations Technical Support Department, Aramco.

that followed. Piero Loliva of Axens and Wolfgang Röhrig of OHL then described a subdewpoint process called SmartSulf, and the collaboration between Axens and OHL to incorporate robust, reliable valves for the cyclical process.

The final presentation of the morning was an optimisation of the existing granulation facilities at the Habshan sulphur handling facility to achieve dual objectives of improved process performance and reduced capital cost, presented by Antonia Medina and Nuha Al Hajeri of ADNOC Sour Gas.

Poster sessions (during breaks) and a dedicated poster spotlight (during the main conference) continued from Wednesday with new posters from Fluor, ADNOC Gas and Aramco.

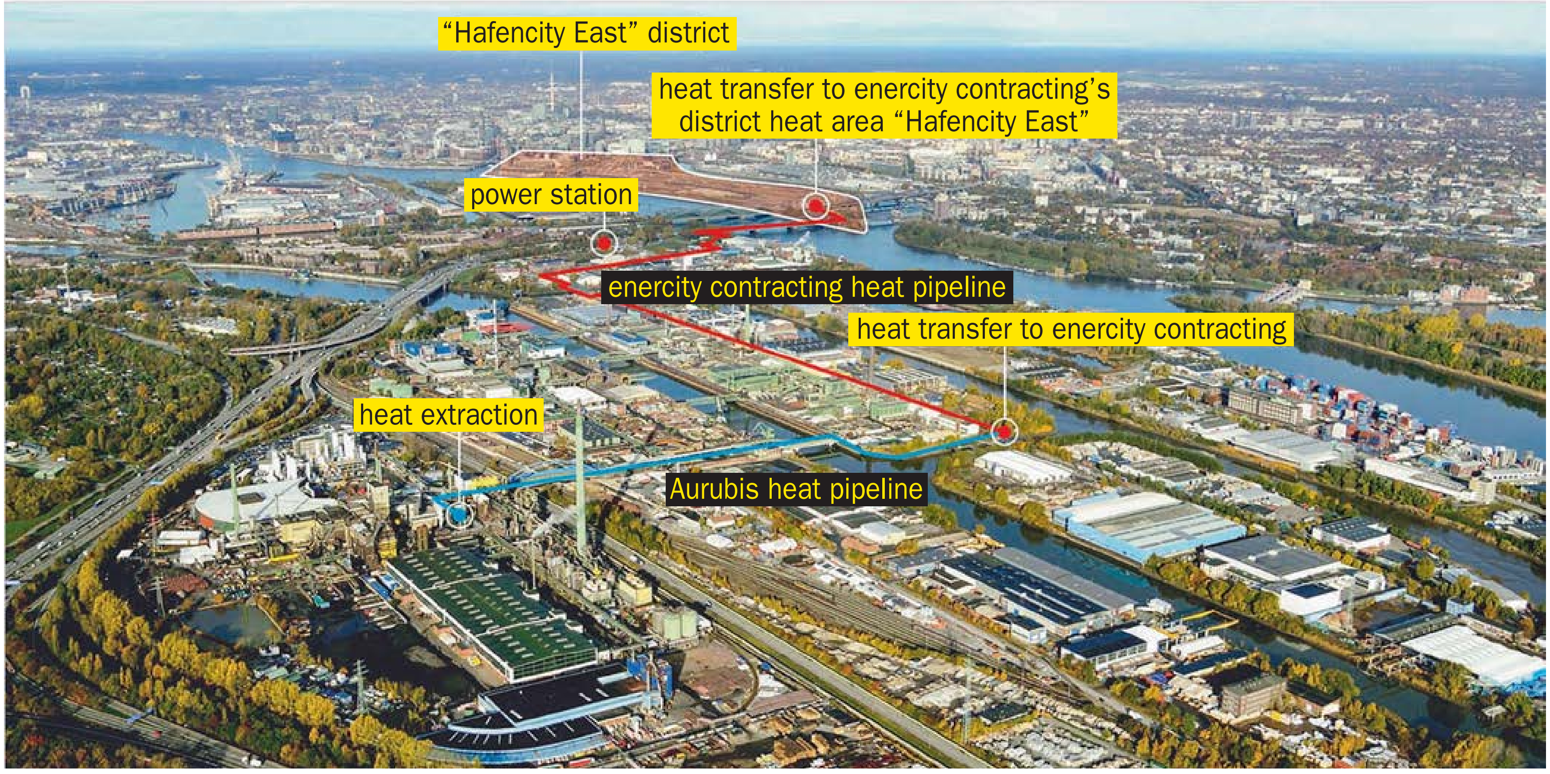
The afternoon session focused on sustainable and efficient SRU operation (Fig. 3). Satyadileep Dara of ADNOC Gas and Rob Marriott of ASRL kicked off the session with a follow-up to Dara's MEScon 2022 presentation about a BTEX soft sensor that was in development at the time and has since been implemented. Operating data from a performance test was collected just days before the conference. The collaboration of industry and academia was noted, as both ASRL and Khalifa University took part in the development. Ibrahim Habib then presented a comprehensive root cause analysis that Aramco carried out to determine the root cause of high SO_2 emissions in one of their SRUs and the steps taken to rectify the matter.

ADNOC Gas' Dinesh Aggarwal then presented a technology evaluation carried out to assess new tail gas treating technologies to improve sulphur recovery efficiency in legacy SRUs at the Habshan complex, noting that both conventional and emerging technologies were considered. He mentioned that the evaluation is still ongoing and alluded to the possibility of presenting the selected technology at MEScon 2024. The session wrapped up with an extremely engaging presentation from Jochen Geiger of Ametek who discussed the historical developments of TGTU process measurement and how to optimise instrumentation for robust and efficient process performance.

In summary, the largest Middle East sulphur event to date was a great success and there was a general feeling from all who participated that the torch is being passed to the region, in terms of future stewardship and advocacy for the industry.

A beacon of energy-efficient waste heat recovery

The recovery of waste heat from Aurubis' copper smelting operation in Hamburg is already helping to reduce global carbon emissions and has the potential to provide heat for up to 20,000 homes through the district heating network in Hamburg's HafenCity. The energy for the network comes from waste heat that Aurubis recovers from their sulphuric acid plant, using unique Alfa Laval plate heat exchanger technology.



Recovering industrial waste heat is one of the most effective ways to improve energy efficiency on a global scale. By recovering energy that would otherwise be lost to the atmosphere or water and reusing it for other purposes, we can greatly reduce fuel consumption compared with today's levels. As a result, we can also drastically reduce global carbon emissions. Aurubis has taken this approach to improve sustainability in one of their smelters. Partnering with the German energy supplier enercity Aktiengesellschaft, the multimetal company has become the driving force behind a district heating network that serves Hamburg's HafenCity neighbourhood. The energy for the network comes from waste heat that Aurubis recovers from their nearby sulphuric acid plant, using unique Alfa Laval plate heat exchanger technology that is designed to resist corrosion and withstand the high pressure and extreme heat associated with the process.

Aurubis, one of the world's largest producers and recyclers of copper, operates a large smelter on the island of Peute, located in the Elbe River, in the heart of Hamburg. Sulphur dioxide gas is a by-product of the pyrometallurgical copper process used here. In the sulphuric acid plant, this gas is converted first to sulphur trioxide and subsequently to liquid sulphuric acid. The sulphuric acid is continuously diluted in the process. Acid dilution is a highly exothermic process, which releases significant amounts of heat. Thanks to Alfa Laval GPHE (gasketed plate heat exchanger), this heat can be recovered by Aurubis for use in district heating.

The sulphuric acid production takes place in the smelter's contact plant. At the heart of the plant lies an intermediate absorption tower. The heat of dilution from this step was originally cooled with river water from the Elbe and thus left unutilised. The aim of Aurubis' collaborative project with enercity was to use heat exchangers to recover energy from this process, and to put that energy back to use sustainably by funnelling it into the HafenCity district heating network.

Equipment design and materials

Aurubis' preliminary challenge was that hot water used in this type of district heating scheme needs to have an initial temperature of at least 90°C. As a result, they had to begin by building a completely redesigned intermediate absorption tower (IAT), increasing the process temperature of the concentrated sulphuric acid to approximately 120°C, a temperature increase of nearly 50°C.

However, this new intermediate absorption tower added further complications to the already demanding project of designing a heat recovery system involving a substance as challenging as sulphuric acid. The higher process temperature substantially increased the corrosiveness of the medium.

This posed a particularly difficult challenge for the heat exchangers in the project, which needed to be constructed of highly corrosion-resistant materials in addition to being able to handle high pressures and temperatures, and to offer extremely high thermal performance to ensure an efficient transfer of heat from the Aurubis process to the district heating network.

Aurubis contacted Alfa Laval, who worked with the copper producer to design customised plate heat exchangers, fully adapted to the demanding process environment. The final system was comprised of eight heat exchangers featuring state-of-the-art channel plate designs. Three of the heat exchangers – the intermediate absorption tower coolers - were semi-welded units, made in Hastelloy D-205™ material, a nickel-based alloy that is particularly resistant to sulphuric acid corrosion. D-205™ has delivered a proven performance at many sulphuric acid plants across the globe since the 1990s. With more than 300 D-205 units installed worldwide, this tried-and-tested material was the obvious choice for ensuring reliable, safe, and efficient operation. The remaining five heat exchangers in the system are fully gasketed plate heat exchangers: three water-water heat exchangers, and two acid-acid interchangers.

Unlike traditional gasketed plate-and-frame heat exchangers, the Alfa Laval semi-welded design is capable of resisting mechanical fatigue in applications with high design pressures and temperatures. The unique plate design also ensures very high thermal efficiency, with a temperature approach as low as 3°C. This means that the water medium used to transfer heat to the district heating network can leave the heat exchanger only a few degrees cooler than the hot acid entering the unit. Additionally, plate heat exchangers are more compact, allowing for easier, more cost-effective installation in a fraction of the space.

Fig. 1 shows the old set-up and the new waste heat recovery set-up.

Improving sustainability

Waste heat that was previously lost to the river Elbe is currently heating up to 8,000 homes through the district heating network in Hamburg's HafenCity, resulting in a reduction in CO_2 emissions of up to 20,000 t/a.

On completion of the project next year when the remaining two lines will be equipped with waste heat recovery equipment the plant will have the potential to heat up to 20,000 homes, with up to 100,000 t/a reduction in CO_2 emissions.

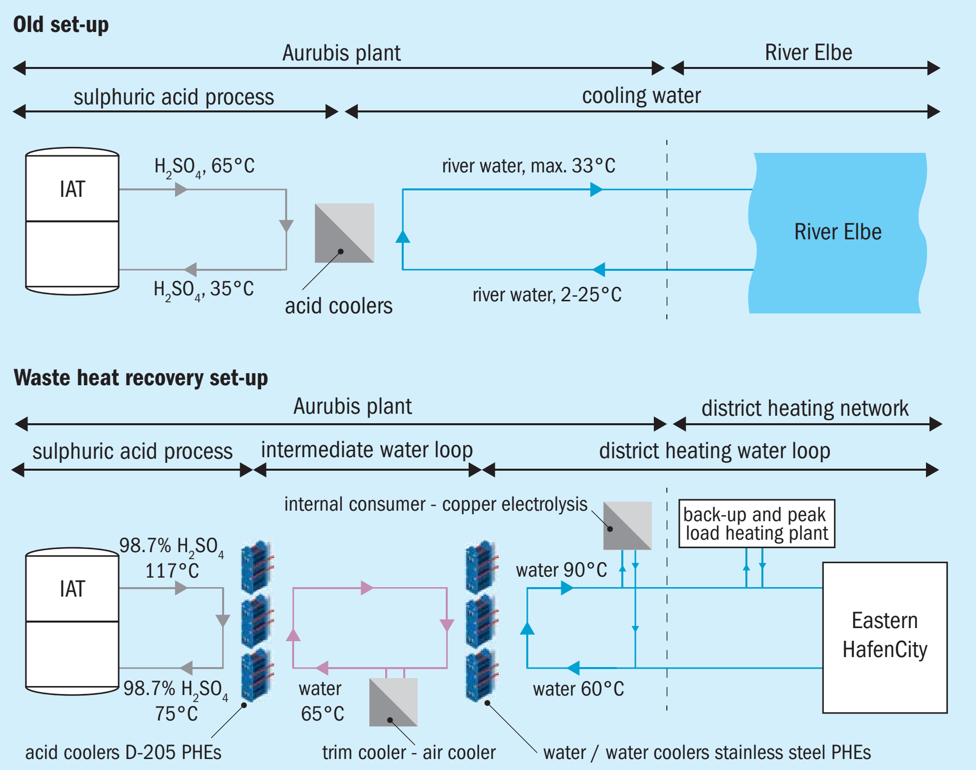
An additional benefit is that Aurubis no longer needs to rely on cooling water taken from the Elbe River, which further benefits the local ecology. The German Energy Agency has recognised the project as a "beacon of energy-efficient waste-heat utilisation."

Almost every city has untapped heat sources like Aurubis and Alfa Laval's heat transfer solutions make it possible to harness that energy and reduce carbon emissions.

Energy efficiency is one of the main contributors to reaching net zero, and increased efficiency could account for 50% of emissions reductions towards 2030.

The challenge though is to figure out what works best for every specific use case. To help with this Alfa Laval has developed a calculator where it's easy to input a system's current configuration and then get suggestions for potential tweaks and upgrades. Case by case the energy saving can be impressive, but the real impact comes from doing this at scale.

Fig 1: **Diagram showing the old set-up and the waste heat recovery set-up**



Source: Alfa Laval

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Closing the skills gaps in sulphur recovery

There is a growing skills gap in the sulphur industry due to the changing nature of the workforce. The traditional ways of doing things are no longer working effectively. Knowledge is lost with retiring subject matter experts (SMEs) and other experienced operations staff leaving a reducing pool of SMEs. In this article a case study reviews how knowledge automation solved the skills gap in a sulphur recovery unit at a major US refinery.

Christian McDermott (Voovio), Angie Slavens (UniverSUL Consulting) and Elmo Nasato (Nasato Consulting).

According to Deloitte's and The Manufacturing Institute's report on the state of the industrial workforce, attracting and retaining a quality workforce is a top focus for 83% of manufacturers surveyed. This perfectly aligns with what is heard across plants in the US and abroad: the loss of experienced people in operating facilities.

Plants are clinging on to baby boomers for as long as they can, fearing the ~30-40 years of equipment specific expertise will be lost forever. As manufacturing facilities expand, thanks to the strong growth of US manufacturing, companies fight to attract an increasingly smaller pool of candidates to run their plants. The workforce is also more transient in nature than it used to be, with less loyalty and shorter tenures. Some people call this a 'skills gap' that is growing and causing severe operational and maintenance challenges in certain areas of manufacturing. The oil and gas industry is further challenged by both political pressures and public opinion with a perception of being an "undesirable" industry; this aggravates the problem of both retaining and also attracting new talent.

The skills gap

Industries must adapt to the changing nature of the workforce. The traditional ways of doing things no longer works effectively. So how is the skills gap affecting the world of sulphur and what can be done to close it?

Voovio has been collecting feedback from its customers, including major refining companies, and is hearing the same concerns everywhere:

- **We are losing subject matter experts (SMEs) and 'tribal knowledge.'**
  - As experts leave, they take their years of valuable and undocumented information and techniques with them decreasing the number of experts that a company can rely on.
- The 'Industrial SME' is becoming a less-desirable career path for younger generations. **Onboarding the traditional way does not work.**
  - Younger generations have grown up with less hands-on mechanical intuition – life is increasingly automated and technology-based.
  - Traditional training cycles are significantly longer due to spending countless hours of classroom training with PowerPoint slides and training manuals, logging time shadowing or under the watchful eye of a mentor or looking up information in a manual.
  - Often, validated training records (particularly refresher training) do not exist and are subject to OSHA violations.
- **We rely on only a few SMEs for hands-on training.**
  - Training is usually people-dependent (highly variable) versus being system based and standardised. As mentioned above, fewer new SMEs are entering the workforce and therefore SME knowledge is not being effectively transferred.
- **Operators have experienced 50% or less of what they need to know on qualification.**
  - In fact, some major petrochemical and refining facilities say it may be as low as 20-30%.
  - Qualification time can be 3-6 months.
- **Practice in the field does not match procedure.**
  - There is variability in how different shifts perform the same procedures or tasks in the field.
  - Procedures are often generic or open-ended in nature, therefore execution and training is not standardised.

This results in:

- workers (SMEs) are overwhelmed;
- unplanned events (equipment reliability);
- near misses and incidents: safety, environmental, quality losses.

The skills gap in sulphur

Let's look at some of the sulphur specific challenges that the sulphur industry is facing, and how we can solve them.

The sulphur recovery unit (SRU) sits at the back end of the refinery or gas plant and is responsible for converting H_2S from upstream units into elemental sulphur. While the SRU produces valuable steam from waste heat recovery, it is typically viewed as a cost centre due to historically low sulphur prices. This often leads to the misperception of the SRU as one of the less critical unit operations in the refinery or gas plant. To the contrary, stable, and reliable operation of the sulphur complex is vital for maximising onstream time of upstream unit operations; thus, although the SRU may not be a revenue generator, it can lead to lost production and revenue reduction for the entire facility if not operated smoothly. Most regions of the world require sulphur recovery efficiency in excess of 99.9%. As environmental regulations become more stringent, optimisation of the sulphur complex continues to be an operating challenge as the industry continues to approach near 100% sulphur recovery levels. In addition, today refiners deal with significant and frequent changes in crude slate which has a direct impact on the SRU operation. The challenge for operating companies is to keep the SRU online for a wide range of sulphur plant loads and to control emissions within regulatory limits, while minimising operating and maintenance costs. Although the process appears to be simple, H_2S, SO_2 and elemental sulphur have unique properties and problems do develop. Unexpected upsets and shutdowns of sulphur recovery operations can result in excess SO_2 emissions and may increase plant maintenance costs due to corrosion and other damage.

Most operational challenges with SRUs occur during abnormal operation, such as feed disturbances from upstream amine and sour water stripping units, start-up, shutdown and/or turndown. The irony is that a well run sulphur recovery complex may have operators who are less prepared to deal with such abnormal conditions since they deal with them less frequently. This makes operator training, practice, and testing/qualification to deal with such conditions crucial for even the best run facilities in the world.

Another critical area that is often overlooked is utility systems. Specifically, the problems associated with the SRU utility systems have always existed, but it appears that in more recent years the frequency and severity of SRU problems associated with the utility systems have increased. It is suggested that the increase and severity of these problems are most likely related, but not limited, to the following items:

- use of utilities may be intermittent;
- utilities are not instrumented to the same degree as the primary process and even when they are instrumented, their maintenance is not prioritised as highly as process systems;
- DCS system has reduced field check-out;
- high turnover/inexperienced operating staff;
- overworked/understaffed operating and technical support staff;
- lack of understanding of the intent and significance of all utility requirements.

By their nature some of the utility streams are used intermittently during start-ups, shutdowns and turndown operation. The industry has moved to lengthening the runtime between scheduled shutdowns. In the past, annual turnarounds were common, today scheduled turnarounds are typically three to four years apart. Several refineries are targeting five years between scheduled turnarounds. This change in turnaround philosophy has resulted in the start-up, shutdown and utility systems being less familiar to operating staff. It is also more likely that the operating staff has changed, and thus operating experience has been lost since the last turnaround. Furthermore, the extended runtime increases the possibility that the utility systems may have been compromised since the last usage.

Sulphur complexes appear to be relatively simple relative to most units in a refinery, however they can be very unforgiving if there is improper attention to detail. Successful operations place emphasis on cross training between process engineers, reliability improvement team members, maintenance personnel, and unit operators. The goal of the training should be to improve operational awareness and the important details in the SRU complex.

Knowledge automation solves a skills gap in sulphur recovery

Knowledge automation has been successfully deployed in the process industry and discrete manufacturing by BASF, SABIC, Halliburton, Tesla, Suncor, Linde, and many more, but what about sulphur? The following case study reviews how knowledge automation solved the skills gap in a sulphur recovery unit at a major US refinery.

The problem

Environmental incidents involving the planned or unplanned releases of substances from plant equipment can be environmentally harmful to air, water, and/or land. Incidents can also involve the breach of plant permits, complaints from local communities and environmental damage. Continued permit violations and regulatory exceedances can compromise a manufacturer's "right to operate" resulting in suspension of operations.

The SRU at a major US refinery experienced several permit violations over a three-year period in fines and expenses of over $500,000. These violations were largely attributed to human error due to the lack of operational knowledge with critical and/or infrequent operating procedures. There were few SMEs who knew how to perform those procedures and the operators lacked the experience and training required (partly due to the high reliability of the unit, which reduced operator familiarity with start-ups and shutdowns).

The site team selected five highly critical and infrequent procedures, where knowledge gaps existed in the workforce and traditional training was not closing them. The procedures were chosen because a) they were not executed properly and/or followed, b) they are not adequate or clear, and/or c) a lack of operator experience exists. The five procedures were:

- tail gas thermal oxidiser start-up;
- Shell Claus Off-gas Treater (SCOT) start-up;
- SRU start-up;
- SCOT unit off-gas warmup bypass;
- sour water system, gas to SRU.

The traditional training process:

- relies heavily on paper manuals;
- contains procedures with open-ended steps (general e.g. one step in the procedure might say 'line up the pump', which in reality may result in ten or more field actions);
- is very "person-dependent" (field training and manual evaluations) which results in 'availability' issues: SMEs need to be on shift and available to train.

An SRU operators' profile was developed (Tables 1 and 2) which illustrates that 2 out of 3 operators who were part of the SRU original start-up team were eligible for retirement very soon, taking critical undocumented knowledge with them and resulting in fewer/no experienced SMEs for training other operators.

The SRU field operator experience indicates that approximately 50% of the operators have more than six years of experience and probably have not experienced certain complex and/or intermittent tasks frequently enough to be fully competent. Three of five console operators have more than two years of experience. Most operators acknowledged that they have never experienced an actual full start-up.

The solution

The refinery chose to implement a knowledge automation platform that captured the critical undocumented SME knowledge (from the original SRU start-up team) within a digital replica (see Fig. 1) and procedure simulators (see Fig. 2). This made this critical knowledge available 24/7 for all

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Table 1: **SRU field operator profiles**

	Years until operator becomes pension eligible?	Is operator qualified on SRU field job?	Is operator qualified on SRU DCS job?	SRU operating experience (years)	Years of service (years)	Is operator considered an SME?
Operator 1	2	Yes	No	20	25	Yes
Operator 2	7	Yes	No	15	15	Yes
Operator 3	12	Yes	No	7	15	No
Operator 4	20	Yes	No	5	13	No
Operator 5	20	Yes	No	10	12	Yes
Operator 6	20	Yes	No	6	6	No
Operator 7	20	Yes	No	2	12	No
Operator 8	25	Yes	No	1	2	No
Operator 9	25	Yes	No	1	2	No

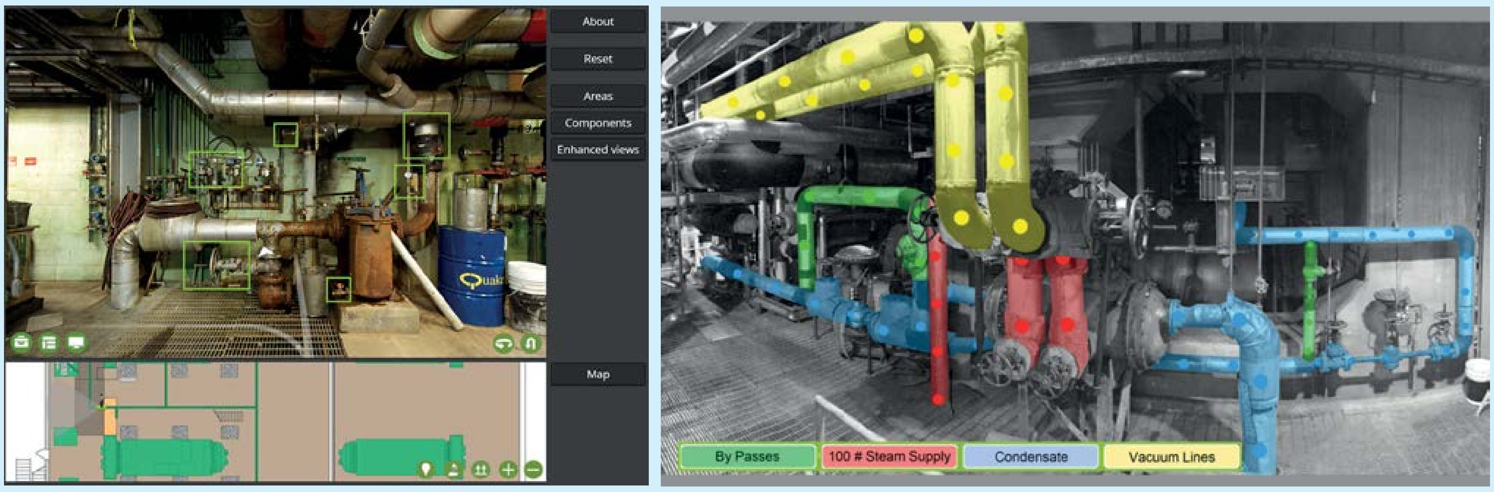
Source: Voovio

Table 2: **DCS (Control Room) SRU operator profiles**

	Years until operator becomes pension eligible?	Is operator qualified on SRU field job?	Is operator qualified on SRU DCS job?	SRU operating experience (years)	Years of service (years)	Is operator considered an SME?
Operator 1	30	Yes	Yes	7	7	No
Operator 2	25	Yes	Yes	7	12	No
Operator 3	10	No	Yes	2	12	No
Operator 4	25	No	Yes	1	2	No
Operator 5	20	No	Yes	1	1	No

Source: Voovio

Fig. 1: **Digital replica**



Source: Image is from a Voovio demo example

operators, allowing them to practice and review the procedures any time, thereby institutionalising and standardising them. There was less reliance on SMEs and the simulation enhanced the scarce field training opportunities.

In Fig. 1 (left image) the digital replica provides a photorealistic immersive replica of the production unit, with all key components (valves, equipment) tagged and accessible in a searchable index. Operators can find things independently of an SME, available 24/7.

In Fig. 1 (right image) the simulation of flows allows operators to learn, review and even practice critical tasks such as valve line-ups in one real image.

In Fig. 2 (procedure simulation) operators can learn, practice and test on procedures within the real digital replica environment of their production unit, accessible 24/7, allowing operators to review critical tasks when an SME or field training is not available.

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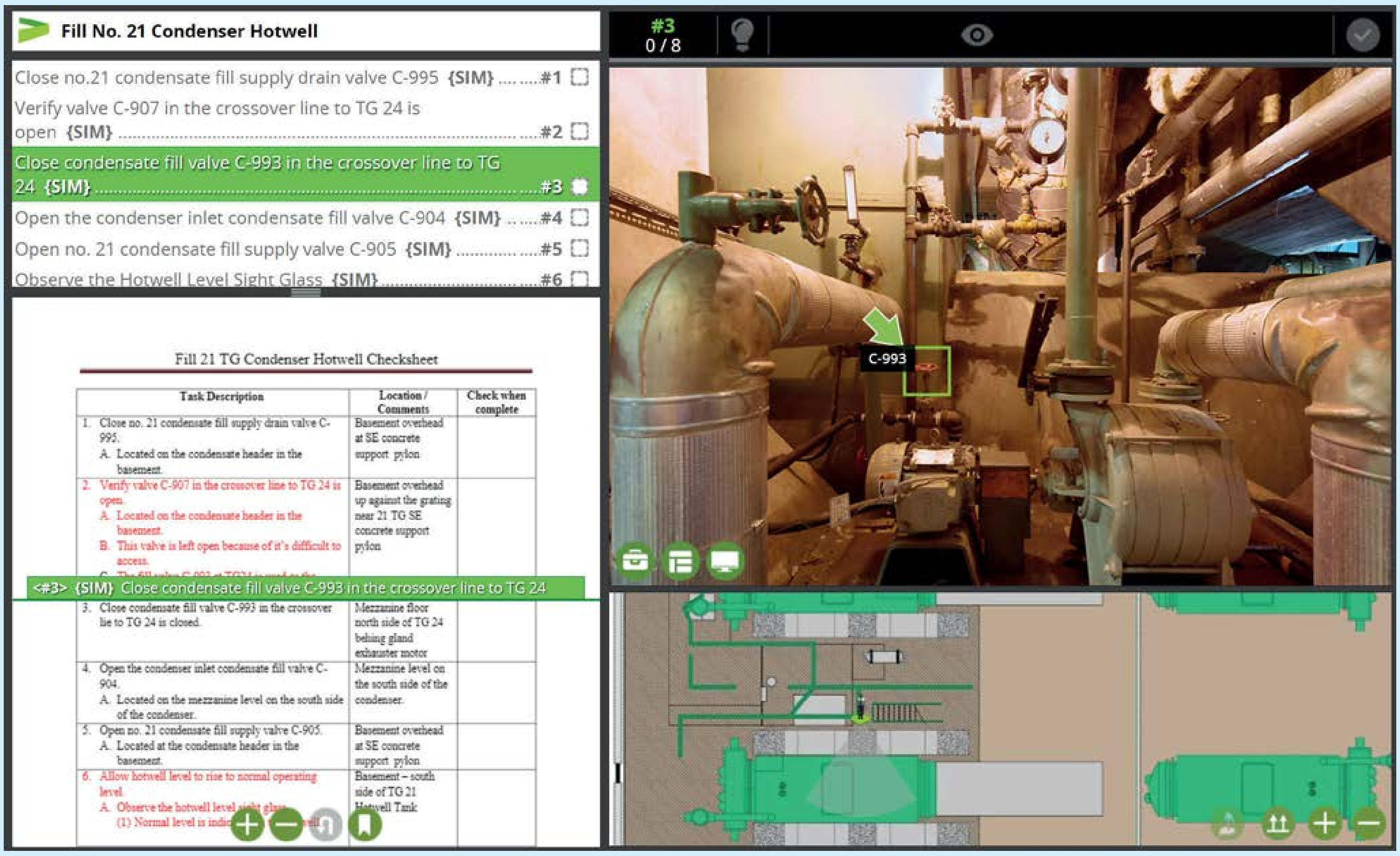
Fig. 2: **Procedure simulator**



Source: Image is from a Voovio demo example

Impact in the SRU

- Reduction in human error related to execution of these five procedures, leading to fewer environmental releases.
- Operators learned and demonstrated adequate knowledge of procedures 60-70% faster than traditional training methods.
- Greater than 70% reduction of SME time dedicated to classroom and other training.
- Operators could access the simulators 24/7 without SME assistance – expertise was now available any time.
- Procedure clarity was achieved in the following ways:
  - Clear, detailed action steps versus general instructions that are open ended (Table 3).
  - Standardisation in the way people learn and review procedures (versus personal preference from one operator to the next).

Table 3: **Action steps in the procedure simulators provide operators with clarity on how they need to perform the task, linked to the actual real equipment in the digital replica**

Procedure	No. of procedure steps in original procedure	No. of action steps in simulator
Tail gas thermal oxidiser start-up	31	98
SCOT start-up	65	190
SRU start-up	65	209
SCOT unit off-gas warm up bypass	5	12
Sour water system, gas to SRU	9	23

Source: Xxxxxx

In summary, the Voovio knowledge automation platform provides tools to standardise and maximise the effectiveness of an operator training and qualification program. This type of tool is essential for any type of manufacturing facility but has proven extremely useful in a sulphur recovery complex where an extensive knowledge gap existed. SRUs are critically important for maintaining overall facility onstream time, yet rarely get the credit they deserve due to their non-revenue-generating status. However, as knowledge is lost with retiring SMEs and other experienced operations staff, the importance of the SRU will be more widely exposed and implementation of knowledge automation tools will become non-negotiable. ■

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4. Data based on Voovo Customer average data as well as exercises performed in the SRU unit. No formal testing was implemented, however, in the SRU.

When digitalisation meets reality

In 2019 Topsoe launched its ClearView™ technology for WSA and SNOx sulphuric acid plants. ClearView™ is a revolutionary process health monitoring software solution to help sulphuric acid plant operators ensure plants run better, more stable and with less downtime. Now, three years later, ClearView™ has been successfully implemented at two WSA plants and several other chemical plants of Topsoe's design, with many others in the pipeline. This article focuses on the results and learnings from the first implementation of ClearView™ at a new WSA plant at Anglo American Platinum's Polokwane smelter in South Africa.

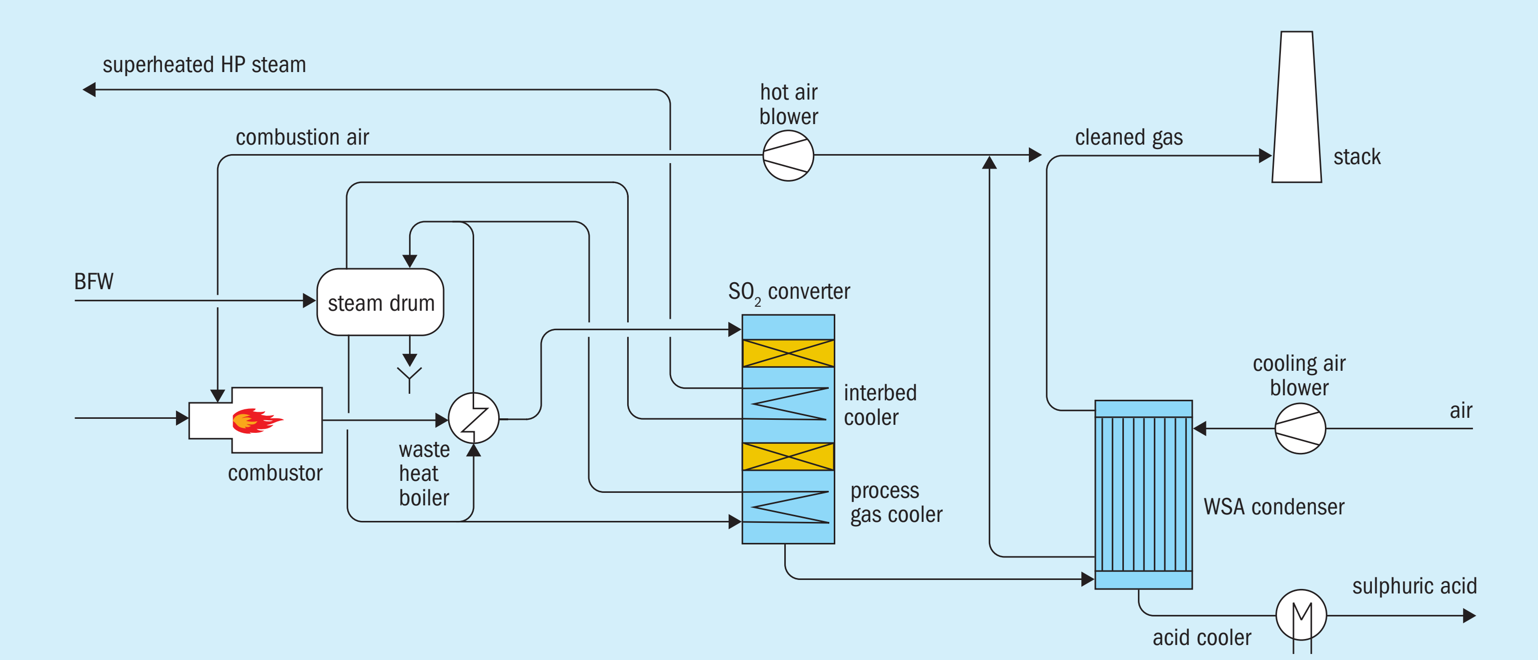
M. Granroth, R. Sverdrup (Topsoe A/S), M. Ndlovu, R. Pinto, (Anglo American), and D. Dippenaar (Anglo American Platinum)

Nowadays, the transformation of all industries through digitalisation, big data, machine learning, and the like has become an almost daily talking point. Topsoe's long history of service to its customers over decades means that it is continuously involved in the real-life challenges faced by plant operators. It is with this in mind that Topsoe undertook to develop the services that will offer the benefits of the so-called fourth industrial revolution to customers in the sulphuric acid sector. Increasing digitalisation of industrial plants results in generation of more and more data; data that on its own can result in increased confusion rather than clarity.

The key to taking advantage of digitalisation is to leverage the torrent of data and new computational capabilities to solve real operational issues in sulphuric acid plants.

As with any industry, there is no lack of challenges that could be addressed. For a bulk chemical market such as sulphuric acid, margins can be small, and at times non-existent as the sulphuric acid plants may also be built for environmental compliance purposes and thus always operated at cost. As such the plant operators are under constant pressure to improve operation to minimise operation cost. Ensuring optimal operation means minimising downtime, maximising throughput, avoiding costly damage to equipment and maintaining emission levels below stipulated limits.

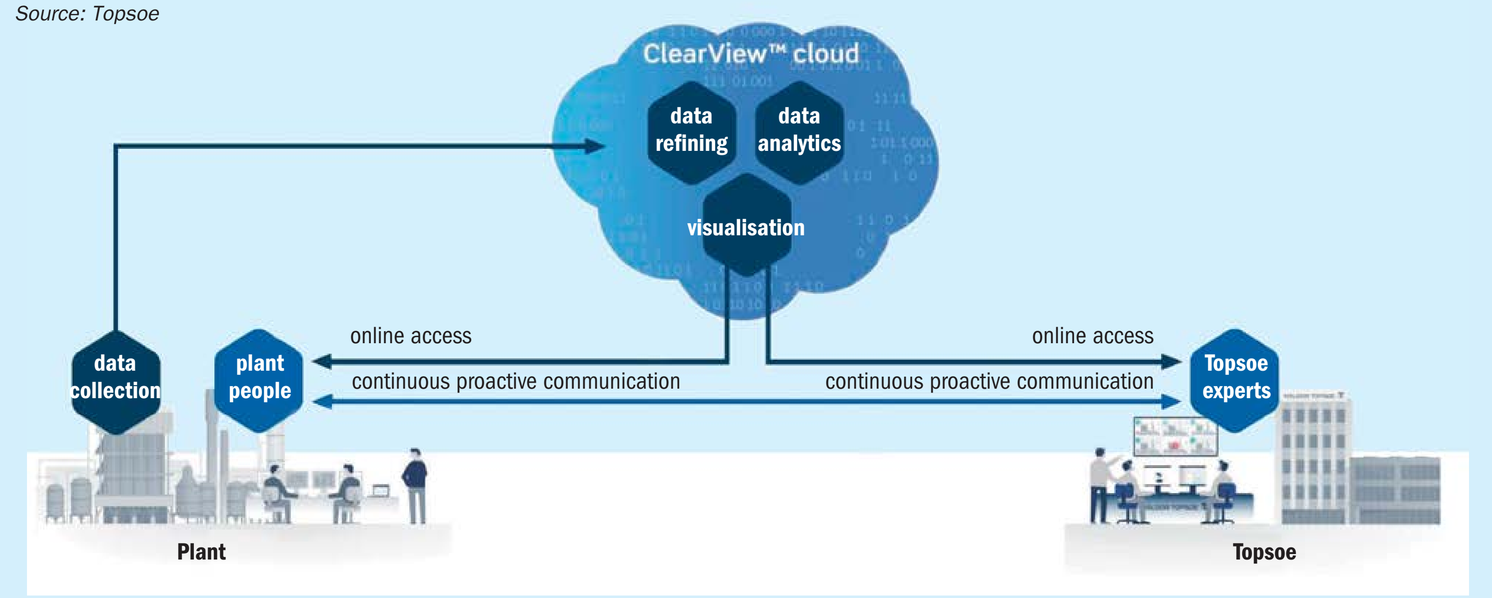
Fig. 1: **General layout of a WSA plant**



Source: Topsoe

Fig. 2: **Illustration of Topsoe connected services solution: ClearView™**

Source: Topsoe



What is WSA?

Topsoe's WSA, or Wet gas Sulphuric Acid, process is a technology which produces commercial grade sulphuric acid from a great variety of sulphur-containing streams. Depending on the stream that is to be treated the layout can vary, but it will always include two key parts: an SO_2 converter where SO_2 is oxidised to SO_3 over Topsoe's proprietary VK sulphuric acid catalyst, and the sulphuric acid condenser, where the product sulphuric acid is condensed in a glass tube condenser.

A simplified flow sheet of the WSA process can be seen in Fig. 1.

The WSA process is highly energy efficient, reducing the facilities carbon footprint and often generates a significant amount of high-pressure steam. A second key benefit of WSA is that it can handle high amounts for water in the gas, allowing it to treat streams that cannot be fed to conventional sulphuric acid plants. Finally, combining the advantages mean the WSA technology can also effectively treat gases which has low to very low SO_2 content, while still producing commercial grade sulphuric acid, and while requiring no or very little combustion of supplementary fuel.

What is ClearView™?

Topsoe launched its connected services, ClearView™ for the Wet gas Sulphuric Acid (WSA) technology in 2019. ClearView™ WSA is a connected solution based on streaming data to an Industrial Internet of Things (IIoT) platform in which rigorous in-house models simulate process data and compare plant parameters to their optimal and healthy values with the aim to provide early warnings of process-related problems. ClearView™ WSA also monitors plant start-up and shutdown and predicts hydraulic and catalytic performance of the unit in order to provide plants with a proactive approach to shutdowns and catalyst screening, before unit capacity is affected.

Online dashboards are designed to deliver up-to-date insights directly to the relevant persons and cater to various roles in the plant, including the overall KPI focus of the senior company executives or directors and plant manager, at a glance. The online dashboards also provide alerts for the daily shift engineer and detailed optimisation screens to be used by the technical department.

The goal of ClearView™ WSA is simple, to detect and mitigate abnormal operation to prevent any surprises in the form of unplanned downtime, and to offer tools and recommendations to mitigate risks, and resolve problems on a planned basis, ensuring world-class availability and reliability.

ClearView™ is a cloud-based system (see Fig. 2), where data is sourced from the DCS historian of the plant. Data refining and analytics are carried out in the cloud, and then visualised live for both the client and Topsoe experts.

Having access to the same results and data facilitates the most powerful part of the ClearView™ service; the direct dialogue and collaboration between the client's operating team and Topsoe technical service.

Data treatment in the ClearView™ cloud

Fluctuations in chemical unit operations are a given in any process industry, but this is especially the case in one dealing with processing sulphur-containing off gases, as is the case with many WSA units.

To make meaningful and actionable conclusions regarding the performance of any piece of equipment, or the process as a whole, requires a consistent mass and energy balance. The starting point is to identify a period of time when the unit is steady enough to perform a mass and energy balance – attempting to do so for too short a period of time will introduce errors due to process fluctuations, while taking a period that is too long risks compressing periods and subjecting them to excessive smoothing, removing necessary details.

Another key parameter to be able to make meaningful evaluation of the condition of the unit is to have consistent data.

It should, however, come as no surprise that mass and energy balances rarely close, with errors arising from a multitude of sources. Instrument measurement errors manifests themselves due to a number of causes, including inherent instrument error, incorrect calibration or compensation, fouling, measurement location, poor process gas mixing (e.g., in the case of temperature measurement), and inaccurate laboratory or analyser measurements.

The solution to this is data reconciliation, or the process of "smoothing out" the error by attributing error to various instruments until the mass and energy balances close.

Data reconciliation requires a model that explains the relationship between process variables. ClearView™ uses the full process model that is used to design WSA units.

Each plant is individually assessed to establish the parameter selection with the strongest links to provide a meaningful result.

Not only does the data reconciliation ensure good data quality for further analysis, by identifying outliers it can also give important clues to troubleshooting or help identify faulty sensors.

Data analytics in the ClearView™ cloud

One of the common questions in operational environments when a piece of equipment doesn't behave as expected and a process value is abnormal is , "What is the value normally?".

If rigorous models to simulate equipment are in place, the expected and measured process parameters can be compared continuously. This is now possible with a reconciled dataset – a single error would have made modelling difficult. Ideally, discrepancies should be detected, before alarms on the DCS system are activated. However, this requires that a rigorous model is developed – this could be a hydraulic model to predict the pressure in a line, or a heat exchanger model to predict the outlet temperature of a heat exchanger.

Rigorous models are, however, not always available. In these instances, a comparison of current and past behaviour for similar process conditions is valid and extremely useful.

Continuous performance assessment through ClearView™ builds up a database of expected values where rigorous models are available, and historical parameters where they are not. Earlier detection means earlier action, and thus lowers risk of unplanned downtime.

Anglo American Platinum, Polokwane smelter

Anglo American Platinum (AAP) operates the world's largest electric furnace for platinum production at its Polokwane smelter in the Limpopo province, South Africa. Implementation of South Africa's new "minimum emission standards", or MES, meant that SO_2 abatement was required for the off gas of the furnace. After having evaluated over 40 different technologies, AAP chose Topsoe's wet gas sulphuric acid (WSA) technology with an associated wet gas cleaning plant from GEA. Fig. 3 shows a photo of the WSA plant.

Fig. 3: The WSA unit at Anglo American's Polokwane smelter.

Commissioning during a pandemic

By the time the new WSA plant was to be commissioned during the second quarter of 2020, the Covid-19 pandemic was in full force. As a result of the pandemic, travel restrictions were in place which prohibited Topsoe experts from visiting the site for normal commissioning activities. Not only were Topsoe experts not able to visit, but other parties and AAP employees' access to site was also severely limited. Had nothing been done, the project would have been delayed, increasing cost and resulting in delayed implementation of SO_2 abatement at the facility.

AAP did not halt the project, however, but implemented a strategy combining digital tools, close contact and careful planning to ensure that the legislative due date could still be met, and commissioning could be carried out safely.

On the technical side, some more advanced tools were applied. One such example is Real-Wear glasses, allowing field work with both hands free, while having live video guidance from Topsoe's experts in Denmark. Most of the work could, however, be done by use of simple, yet effective, technology, such as using video conferencing software in combination with smartphones and tablets.

In preparation for the commissioning, a service level agreement was signed between Topsoe and AAP. As part of the agreement, AAP's commissioning team had unlimited access to Topsoe's experts during business hours. The close contact between AAP and Topsoe allowed the commissioning to proceed without significant delays, despite the limitations brought on by the pandemic.

Even during normal circumstance, the commissioning of any new industrial plant requires detailed planning and careful execution to proceed smoothly. With the limitations introduced by the Covid-19 pandemic this became even more critical. No digitals tools or remote service would have helped if the planning and coordination had been lacking. AAP understood this and ensured that it got the attention it deserved. The result was that the hurdles of the pandemic could be overcome, and the plant could be successfully commissioned with very little on-site support.

Start-up

During start-up much the same strategies were applied as during the commissioning phase, however, with one exception; ClearView™ was also available as an additional tool. While all the parameters had not yet been fully tuned, ClearView™ could still offer a number of important benefits, which made it possible to start up the new plant remotely.

Topsoe engineers could follow the start-up process in real time, and both AAP and Topsoe personnel would be alerted if there

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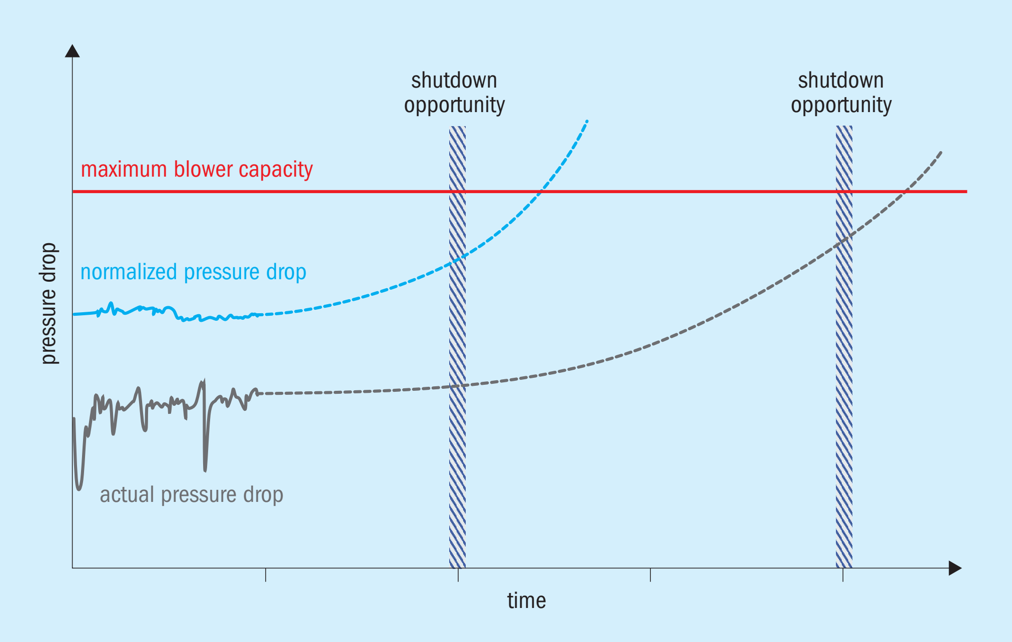
Fig. 4: **Hydraulic and pressure drop projections from ClearView™**



Source: Topsoe

were deviations from the guidelines or if the conditions could affect safety or equipment lifespan.

In the end, ClearView™ was one piece of what made it possible to successfully start up the plant despite the restrictions of the pandemic. The main benefit was how it helped facilitate collaboration between AAP and Topsoe, despite the long distance between the two parties, making the output greater than the sum of the two. This learning shows that rather than being a substitute for good operators and engineers, digitalisation can often be a tool to harness their skills and knowledge better.

Day to day benefits of ClearView™

The practical application of ClearView™ in day-to-day operation of the WSA plant can be divided into two different parts: direct feedback from the system to operators or engineers at the client end, and weekly meetings between the Topsoe technical service engineers and the customers operational team. The experience thus far has been that the largest value is created for the customer during these frequent meetings.

The status meetings focus on supporting the customer with information on their key questions, addressing the most common issues based on Topsoe's vast technical service experience, and finally to identify and troubleshoot actual or potential problems. To summarise, the main points on the typical agenda for the status meetings are:

- hydraulic monitoring and assessment of equipment and catalyst;
- dew point evaluation;
- start-up and shutdown sequences;
- optimisation of operating parameters;
- evaluation of outliers and troubleshooting.

Hydraulic monitoring

The hydraulic monitoring part of ClearView™ seeks to answer three of the most common and important questions operators of sulphuric acid plants have:

- How long can we run before we need to shut down?
- Which part of the plant is having problems?
- Can we do anything to mitigate or counter pressure drop build-up?

The way most sulphuric acid plant operators would try to answer these questions would be to compare actual pressure drop values with design and start-of-run values. For many sulphuric acid plants, and certainly for most WSA plants, the conditions are in a constant state of change. Adding to this, design values often have margins which will mask emerging hydraulic limitations. Together these two facts make such comparisons difficult, and problems may only be discovered once they are severe.

In ClearView™ the issue of comparing to design and start-of-run values is solved by comparing with simulated values for the present conditions. These simulations are done using the full plant model used to design the unit and means that deviations will be noticed as they emerge.

The second part of the ClearView™ service involves the plotting of the deviation between the simulated and actual pressure drops over time for every part of the process. This is illustrated to the customers operational team in a dashboard, giving at a glance information if a problem has arisen in any part of the plant. This answers the second question "Which part of the plant is having problems".

One example of how this approach was applied at AAP was when one of the deviations started to increase at an alarming rate. Both Topsoe and AAP were alerted of the deviation by the Clearview™ system and an action plan could be developed to investigate and potentially rectify possible root causes for the increasing pressure drop. The plan started with simpler explanations, such as instrument failure, and stretched to much more severe, such as critical damage to some of the equipment in the WSA plant. This time around, the deviation turned out to be caused by a common instrument error, but had it turned out that the cause was more severe in nature, the plan would have been ready to address it.

For those parts of the plants where either some deviation has emerged, or based on historical experience needs more scrutiny, ClearView™ allows the pressure drop to be studied in more detail. Other than just looking at the simulated and actual values, the software also predicts when critical pressure drop will be reached, both at design and current gas flows (see Fig. 4). The prediction is an excellent starting point for discussion on when to plan outages, or how to allocate activities such as catalyst screening or equipment cleaning. This answers the first question "How long can we run before we need to shut down", as well as helping the operational team minimise downtime.

In the case of AAP, this is used to plan maintenance activities in already scheduled downtime, ensuring that the time in between outages is spent operating the plant.

This leaves the final question "Can we do anything to mitigate or counter pressure drop build-up?". While ClearView™ has built in tooltips suggesting what can be done to address some of the more common issues, it will be impossible to have solutions built in for all potential issues in the plant. Therefore, discussing action plans to answer the very question of "what can be done" is a key part of the reoccurring meetings. Once again this illustrates how ClearView™ is at its most effective when it is used to augment traditional technical service.

Dew point monitoring

Ensuring that all surfaces within the WSA plant are above the dew point of sulphuric acid is a key point in obtaining optimal plant lifetime and minimising maintenance cost. Disregarding acid dew points will have negative impact on plant lifespan.

Guidelines and procedures have long been available to ensure sufficient margin to the acid dew points are always maintained. To further support clients' ability to maintain safety margins, ClearView™ calculates the margin to the acid dew points continuously for all critical sections of the WSA plant. The margins are visualised on their own dashboard. The calculations are based on Topsoe's proprietary models and provide an accurate estimate of the actual dew point at current conditions, a significant improvement over comparison to single precalculated values at design conditions and a few operating cases.

In addition to simply displaying dew points and margins to dew point throughout the plant, the software will also alert the operations team if any of the safety margins are being violated. With the help of ClearView™, AAP has been able to minimise the number and duration of events where safety margins are being violated. As a result of this work, and Anglo American's diligent maintenance program overall, the first inspections have shown that the plant is in very good condition and is expected to have a very long lifetime.

Start-up and shutdown

Start-up and shutdown are some of the more demanding states of operating a sulphuric acid plant, with a lot of parameters and requirements that need to be managed to ensure this is done safely and effectively. During the initial start-up, licensor supervisors will typically be present (or as in the case for Polokwane project, support remotely) to ensure the plant is started up safely, through adherence to all applicable guidelines. Sulphuric acid plants operating on metallurgical off gases will often experience frequent stops due the upstream operation, and it is not feasible to always have licensor site support for each start-up. With frequent outages, good operating procedures for start-up, hot standby and shutdown scenarios are especially critical for WSA units.

The optimisation of operating procedures improve safety, increase equipment lifespan and reduce time spent on startup and shutdown, and is one thing that sets experienced operational teams apart. This is even more true in the case of the Polokwane smelter WSA plant, where a new operations team had been hired with limited experience in sulphuric acid plants operation in general, or WSA plants in particular.

Topsoe addressed this area of concern through ClearView™ which monitors conditions during a start-up or shutdown and compares them with Topsoe guidelines. As soon as a part of the plant has reached a safe state (right temperature, pressure, flow etc.) it is indicated by colour coding on the start-up/shutdown dashboard, giving go-ahead to continue to the next part in the sequence as part of the plant changes state.

Assisted by the feedback of the start-up and shutdown monitoring, together with the weekly meetings, AAP has already made several revisions of their procedures for start-up and shutdown. Since the latest revision was implemented, every start-up and shutdown has been carried out without violating any guidelines, ensuring that no damage will be sustained to the plant during these critical operations. Not only is AAP now avoiding damage to their plant; they have also been able to reduce start-up time of the plant by a staggering 75%. In short, their own dedicated efforts assisted by the ClearView™ service has allowed AAP to achieve benchmark start-up times in just one year.

Optimising performance

Rarely does any industrial plant ever run completely at design conditions, and there will always be a myriad of parameters that can, or even should, be optimised to achieve good operation. WSA plants are no different, and Topsoe's technical service team has always offered advice on how operation of the WSA plants can be optimised following site visits or when receiving data from customers. Partly due to the time required for the customers to extract data, if there is nothing of particular concern, these manual optimisation with support from Topsoe is generally only done at very low frequency. This all changes with ClearView™, where a reconciled dataset is available to both Topsoe and the customer on an hourly basis. Some optimisation is suggested by ClearView™ itself, and it also forms a natural agenda point for the weekly meeting. Through this continuous improvement and adaptation to new conditions are ensured.

An example of how this was applied at AAP was with regards to inlet temperatures to the catalyst beds. The plant was designed for an SO_2 content of up to 3%, however, so far, the plant has not operated on a gas strenght in excess of 2.5%. As a result of the lower feed SO_2 content, there is excess catalyst available compared to what is needed to achieve the target conversion. This excess catalyst can be utilised to allow operation at lower temperatures without exceeding emission limits. With joint access to cleansed data in ClearView™, the AAP operations team together with Topsoe's technical service engineers could optimise the catalyst bed temperatures, while also making sure that the emission and other key performance parameters were still within acceptable limits.

The output of the optimisation was a new set of operating temperatures, tailored for the actual conditions and current catalyst activity. The new operating point resulted in a saving in fuel and carbon footprint of the unit, while still making sure all targets were met.

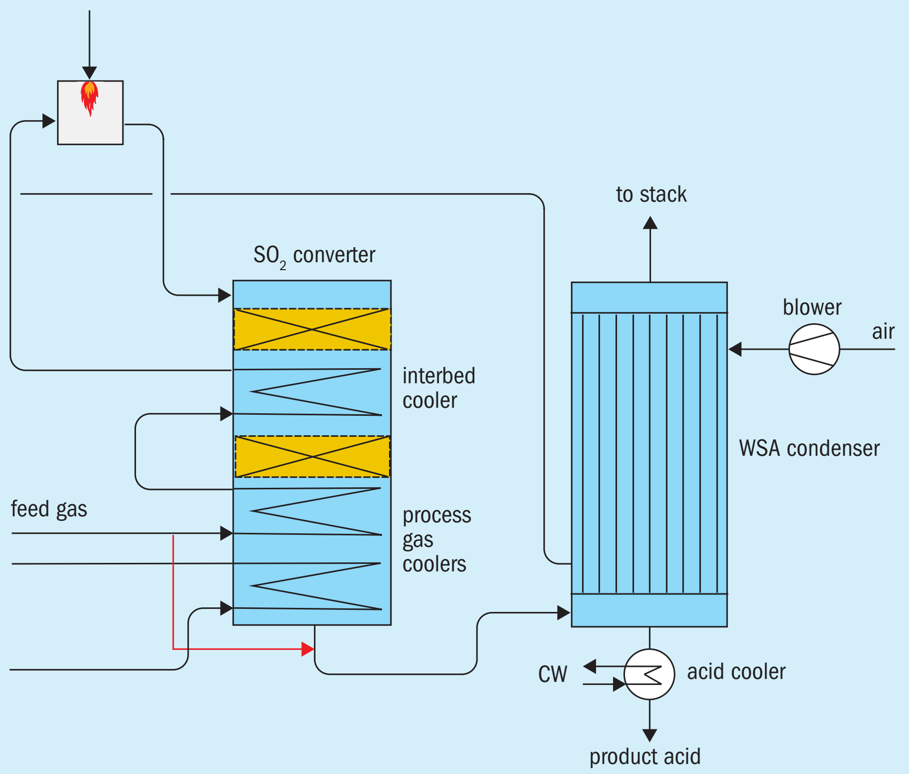
Troubleshooting with digital tools

Not only will ClearView™ help identify looming issues earlier through comparison with built in models and access to reliable up to date data, it also helps the customer and the Topsoe technical service team to troubleshoot those issues.

One example from the Polokwane smelter was tied to an unknown high SO_2 emission following the initial start-up. At first it was thought that the elevated emission would be resolved by finetuning of operating parameters, however, as the plant was operating stable, it became apparent that the emission persisted even with optimised settings. Detailed evaluation by AAP's operational team and Topsoe multidiscipline experts followed.

Fig. 5: Inspection showing the hatch with only 2 out of 16 bolts, and clear pattern of gas having passed through.

Fig. 6: **Effective bypass around the SO_2 converter illustrated by red arrow**



Source: Topsoe

Using the information available through the ClearView™ system, many potential root causes could quickly either be ruled out completely or deemed highly unlikely. Examples of ruled out root causes were low catalyst activity, changes in feed gas composition and faulty readings.

The result of the evaluation was a list of potential root causes, all related to the second feed/effluent heat exchanger:

- second feed/effluent internal hatches;
- second feed/effluent heat exchanger welds to converter shell;
- second feed/effluent heat exchanger tube-tubesheet welds.

Based on the prioritised list, an action plan was developed to prepare for the upcoming shutdown. This meant that the tools and knowhow to efficiently check, and potentially rectify, all of the points on the list were available as the plant was shutting down. When the plant had cooled down sufficiently for safe entry, it was quickly concluded that the source of the high emission was the first point on the list; a leaking access hatch. The hatch was lacking the gasket and was held in place by just 2 out of the 16 specified bolts (see Fig. 5). The result was that some of the feed gas could bypass the entire SO_2 converter, see marking in Fig. 6.

The problem was simple to address, but even if the higher emission would have been caused by damaged equipment, AAP would have been prepared to repair it. Once the leaking hatch had been addressed, the plant could restart with normal SO_2 emission.

Conclusion and future

Experience from the WSA unit at Anglo American Platinum's Polokwane smelter shows how digital tools and strategies can be bring real operational benefits.

During commissioning and start-up, AAP showed how relatively simple and readily available tools with the right strategy and structure could help overcome the obstacles brought on by the pandemic.

From the start-up through to normal operation, ClearView™ has helped answer key operational questions, such as "When do we need to shut down" or "How do we increase equipment lifespan". By supplying the operational team with actionable information, ClearView™ has helped AAP to operate the new plant more efficiently, avoid conditions which might cause long term damage to the plant and facilitate troubleshooting.

Coupled with traditional technical service, ClearView™ can facilitate a positive learning loop between the customers operational team and Topsoe's technical service team. The learning loop and AAP's devotion to excellence has propelled them to a benchmark operator within the first year of operation, while also providing Topsoe with fast and reliable feedback on everything from commissioning and operating guidelines to plant design.

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46
47 48 49 50 51 52 53 54 55 56

Freezing the furnace

Debopam Chaudhuri of Fluor Daniel India Pvt Ltd and **Michiel Baerends** of Fluor B.V. Netherlands investigate how SO_2 impacts the Claus furnace temperature in an SRU and the ways to mitigate it. This article studies the extent of quenching experienced in the Claus furnace with varying amounts of SO_2 in the Claus feed. A case study is presented based on real operating data of a refinery Claus plant with a feed gas cocktail that includes substantial SO_2 recovered from a regenerative flue gas desulphurisation unit.

The classical feed gas streams to a sulphur recovery unit (SRU), originating from the amine regeneration and the sour water stripping units, contain varying amounts of H_2S as the sulphur source. The H_2S in the acid gas streams is captured as elemental sulphur utilising the modified Claus reactions. The process involves burning the acid feed gas with a sub-stoichiometric amount of air – just enough to combust only a third of the H_2S to SO_2. The SO_2 thus formed reacts with the unconverted H_2S to form sulphur.

The main reactions involved are:

$$H_2S + 1^1/_2\, O_2 \rightarrow SO_2 + H_2O \quad (1)$$

$$2H_2S + SO_2 \rightarrow 3S + 2H_2O \quad (2)$$

$$3H_2S + 1^1/_2\, O_2 \rightarrow 3S + 3H_2O \quad (3)$$

Reaction 1 is highly exothermic, while reaction 2 is endothermic, with a net effect of exothermicity for the overall reaction 3.

If SO_2 is also present in the feed stream to the SRU it reduces the air requirement, as well as impacting (lowering) the furnace temperatures.

Another reaction of importance is the destruction of ammonia (NH_3) in the presence of air.

$$2NH_3 + 1^1/_2\, O_2 \rightarrow N_2 + 3H_2O$$

It is generally agreed that the Claus furnace needs to be hot enough to ensure near complete destruction of ammonia to nitrogen, and the target typically followed globally for a standard Claus plant is 1,250°C.

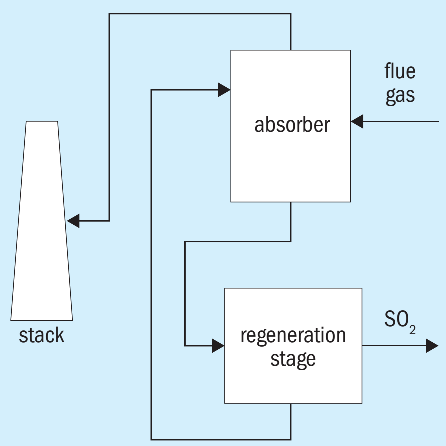
The requirement to achieve at least a certain 'threshold' temperature in the Claus furnace to destroy relevant impurities can clash with the tendency SO_2 has, in terms of reducing the very same temperature. This article explores some ways and means to counteract this tendency; and meet required Claus furnace temperature even in the presence of some SO_2 in the feed gases. First, however, some discussion is warranted on possible sources of SO_2 in Claus feed gases.

Where does the SO_2 come from?

In a refinery setting, FCC regenerator flue gas (if the FCC feedstock is unhydrotreated VGO or a resid feed) can have appreciable SO_2, which in some cases may be removed by a regenerable SO_2 scrubbing system. This implies that the SO_2 removed from the FCC regenerator flue gas is released by the regenerator (stripper) of the regenerative system; and requires processing in the refinery SRU. Similarly, in case the refinery has an oil-fired power plant (power plant steam boilers fired on residue fuel oil having considerable amounts of sulphur), the flue gas would contain high amounts of SO_2, which also might utilise a regenerable SO_2 scrubbing system, resulting in SO_2 ending up in the SRU feed mixture. Other possible SO_2-rich flue gases in refinery settings could be flue gases from various fired heaters using residual fuel oil, flue gas from the fluid coker regenerator or spent acid alkylation flue gas. Even a Claus incinerator flue gas might need to use a regenerable de-SOx process if the sulphur plant is designed without a TGTU, as the incinerator flue gas would then contain appreciable amounts of SO_2.

As indicated above, regenerative SO_2 flue gas removal processes typically remove SO_2 from the flue gas stream using a solvent (for example, an amine-based solvent, such as in the Cansolv process) to absorb the SO_2. The 'rich' solvent is then regenerated to release the SO_2 back. The released SO_2 is then routed to an SRU, along with the normal acid gas streams. Fig. 1 depicts a simplified schematic of a regenerative de-SOx process.

Fig 1: De-SOx with regenerative solvent



Source: Fluor

The impact of SO_2 in feed gas

The presence of SO_2 is both beneficial and potentially harmful for the Claus process.

As each mole of SO_2 replaces 1.5 moles of O_2 required in the furnace, the air flow rate can be reduced (unloading the air blower), which also reduces the 'tail gas' flow through the unit, which, after all, has N_2 from combustion air as the main component. In fact, while increasing elemental sulphur output, additional SO_2 added to the feed cocktail hydraulically unloads the unit. This seems a favourable side effect of having SO_2 in the feed gas.

On the other hand, the SO_2 induces a substantial 'quenching' effect on the Claus furnace temperature, due to the loss of exotherm in the Claus furnace. This in turn presents challenges in meeting the necessary temperature required for destruction of NH_3 and other impurities.

A theoretical assessment of how the SO_2 in the feed stream impacts the overall reaction and capacity of the unit is made here for illustrative purposes. For this it is considered that the Claus furnace receives a constant feed gas flow of 100 kgmol/h, with a varying composition. For the initial case, it is theoretically assumed that the gas fed to the furnace is pure H_2S, and for the subsequent simulation runs, the feed gas composition is changed, increasing the SO_2 content in steps of 5%, but leaving the total feed gas flow the same, and thus leading to the same sulphur product rates, around 75 t/d. The results of the simulation runs are shown in Table 1.

Table 1: **Impact of SO_2 in SRU feedgas**

Feed gas basis, kgmol/h	Claus furnace temperature, °C	Air demand, kgmol/h	Tail gas flow, kgmol/h	Hydraulic load
100 H_2S	1,310	225	280	100% (Base)
95 H_2S, 5 SO_2	1,220	190	250	90%
90 H_2S, 10 SO_2	1,110	156	215	78%

Note: This data is based on simulation results, with a simulation model built in Sulsim software
Source: Fluor

Table 1 illustrates how adding limited amounts of SO_2 (relative to the amount of H_2S), has a significant, favourable impact on the air demand (lower) and tail gas flow / hydraulic load through the unit (lower). At the same time, it can be observed that even small quantities of SO_2 have a very substantial, and unfavourable, impact on furnace temperature (200°C lower when 10% of the H_2S feed is substituted by SO_2), making destruction of impurities significantly harder to achieve.

Managing the freeze

Design modifications are necessary in the SRU to manage the 'cooling' effect of co-processing SO_2. The design options available to maintain proper furnace temperatures in the Claus furnace are well known. They include feed preheat, air preheat, fuel gas cofiring, oxygen enrichment, and furnace design adaptations, or a combination of two or more of these options.

Feed preheat

Feed preheat entails heating up one or more of the feed gas streams, as needed. Similarly, the combustion air supplied to the Claus furnace may also be heated up. Typically, the steam generated in the waste heat boiler of the Claus plant is used as the heating medium, and the amount of preheat available is then limited by the steam pressure available. With steam pressures from Claus waste heat boilers typically in the region of 40 bar (condensing temperature around 250 °C), this leads to practical pre-heat temperatures somewhat below 250°C. Any additional heat input would require the implementation of fired heaters, in-line burners or electric heaters. For the example presented in the previous section, while operating with the 90 kgmol/h H_2S + 10 kgmol/h SO_2 feed gas stream, preheating the amine acid gas to 220°C will ensure a furnace temperature in the range of 1,160°C, while preheating both the air and the feed streams takes the furnace temperature to 1,210°C.

Fuel gas cofiring

Fuel gas cofiring may be applied to increase the furnace temperatures. Typically, natural gas is a preferred for cofiring in the Claus furnace. A continuous application of fuel gas co-firing may not be recommended for various reasons, the major one being the fact that it increases the potential of 'soot' deposition in the downstream catalytic reactors. It should also be noted that cofiring, while helping to increase the furnace temperature, offsets the hydraulic advantages of coprocessing SO_2 as it needs additional air for combustion, adding to the gas flow through the unit. For comparison, adding 5 kgmol/h of natural gas to the 90/10 H_2S/SO_2 feed gas stream increases the furnace temperature to 1,230°C, but also makes the unit run at 93% compared to its base hydraulic load (instead of at 78%, which would have been the case without co-firing). Co-firing also introduces an operating cost for the fuel gas, which may be recovered by the value of the extra steam generated as a result of co-firing.

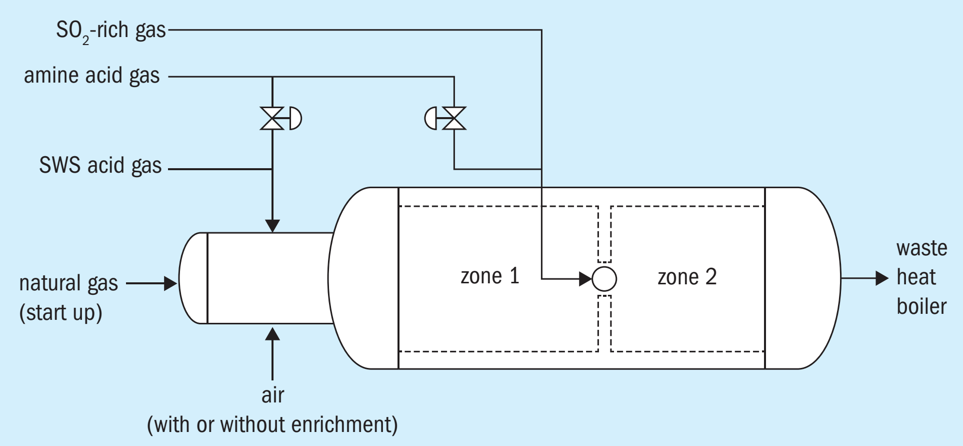
Oxygen enrichment

Oxygen enrichment is a very logical option for enhancing furnace temperatures. Eliminating a portion of the 'inert' nitrogen molecules from the air supplied for the firing in the furnace allows a higher temperature with the same amount and composition of acid gases. Different levels of oxygen enrichment may be implemented – low level (typically up to 28 vol-% oxygen, oxygen added directly to the combustion air line), mid level (typically up to 45 vol-% oxygen) or high level (theoretically up to 100 vol-% oxygen). An important side benefit of implementing oxygen enrichment is that it reduces the hydraulic load on the unit, making it physically smaller. With the example presented in the previous section, while operating with the 90/10 H_2S/SO_2 feed gas stream, in case low level oxygen enrichment level (28 vol%) is implemented, the furnace temperature increases to 1,225°C, while reducing the hydraulic load to 63% compared to the base case. The obvious drawback of this option is the increase in the cost of operating the unit, associated with continuous usage of oxygen. In addition, design complexities of the Claus burner and furnace and even the overall Claus unit are introduced as the enrichment level increases.

Furnace design adaptations

Lastly, there are multiple options of furnace design adaptations available to manage furnace temperatures in the Claus furnace. These are mostly licensor specific, but mostly revolve around regulating gas streams to the furnaces in separate zones. A two-zone furnace in effect allows the combustion to happen in two stages (Fig. 2). The front section, to which all air is directed, operates at higher 'oxidation extent' and therefore at a higher temperature, than would be possible for a single zone furnace. The SWS gas is exclusively combusted here, with a considerable portion of amine acid gas bypassed, allowing the front section to operate with a high enough temperature to destroy the ammonia (typically greater than 1,250°C). For the back end, where there is no NH_3 to destroy (as all NH_3 containing SWS gas was directed to the front end) a temperature above 1,050 or 1,100°C would be acceptable to ensure destruction of hydrocarbons[1,2].

Fig 2: **Two-zone furnace design**



Source: Fluor

Case study

The data presented in this case study is based on the real operating data of a refinery that is undergoing a revamp to replace two small and very old sulphur plants which have been facing age-related issues in operation. They have been heavily dependent on continuous maintenance and have been experiencing emission issues as the sulphur load of the refinery has been gradually increasing.

The refinery processes a moderate to high sour crude. One of the products of the refinery is refinery fuel oil which supplies the power generation plant within the refinery. A major portion of the vacuum residue thus finds its way out as fuel oil for this power plant. A thermal cracking unit 'prepares' the fuel oil, while the co-produced cracked distillates are treated further (desulphurisation) in the hydrotreaters, wherein the cracked distillates are desulphurised along with the straight run light and middle distillates. The balance amount of the vacuum residue is either exported as bitumen or as bunker fuel oil, after adding necessary amounts of cutter stock to it.

The power plant also includes a flue gas desulphurisation unit to capture the SO_2 from the flue gas (originating from the burning of heavy fuel oil, containing significant organic sulphur). The SO_2 removed from the flue gas is subsequently released from the solvent regeneration step; and must be routed to the new sulphur recovery unit for conversion to elemental sulphur.

The refinery is thus opting for a new sulphur plant that is capable of processing the necessary amounts of acid gas streams generated from the upstream amine regeneration and sour water stripping units as well as co-processing a considerable amount of SO_2 as generated from the regenerative flue gas desulphurisation unit of the power generation unit.

The amount of SO_2 that needs to be co-processed in this new unit was proving to be a bottleneck, and hence a detailed assessment was performed on the means and methods via which the plant capacity enhancement could be achieved.

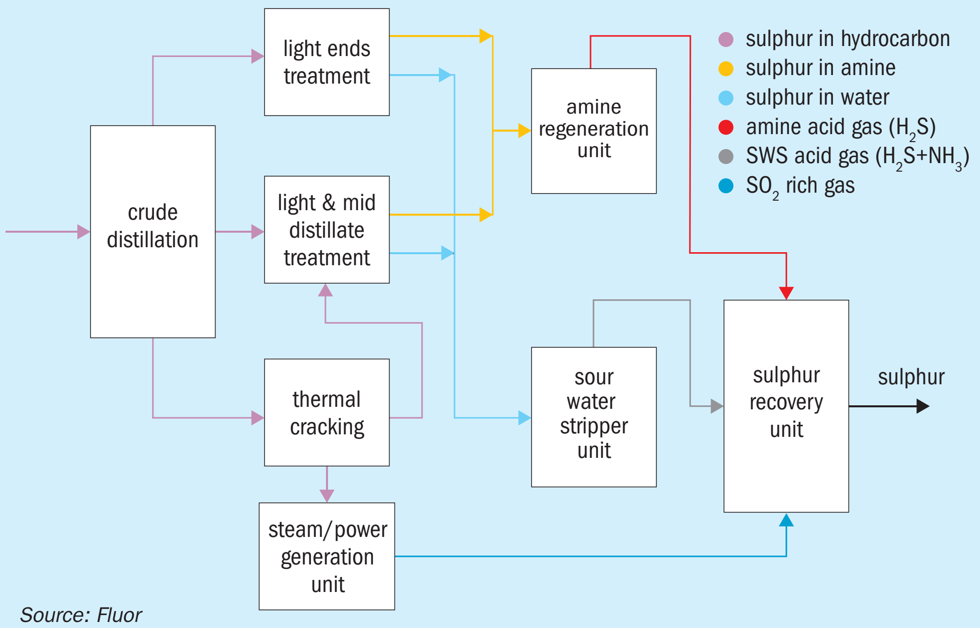
Fig. 3 shows the sulphur map for the refinery.

The expected feed gas compositions and plant capacity are provided in Table 2 corresponding to a sulphur production capacity of 200 t/d.

As can be derived from the Table 2, the feed gas to the new Claus plant contains more than 10 vol-% of SO_2. As already discussed, with such high amounts of SO_2, maintaining proper furnace temperatures becomes challenging. Since the overall gas contains almost 6.5 vol-% of NH_3, the importance of maintaining the right furnace temperature is vital.

The expected Claus furnace temperature when processing this feed gas cocktail

Fig 3: **Overall block flow diagram of refinery sulphur map**



Source: Fluor

Table 2: **Case study feed gas composition**

	Amine acid gas		SWS acid gas		SO_2-rich gas	
Component	**mol-%**	**kg/h**	**mol-%**	**kg/h**	**mol-%**	**kg/h**
H_2S	88.5	6,826	33.0	724	0.0	0
SO_2	0.0	0	0.0	0	90.0	2,598
NH_3	0.0	0	33.0	385	0.0	0
H_2O	4.0	172	33.0	407	7.5	61
Balance	7.5	737	1.0	18	2.5	31
TOTAL	100.0	7735	100.0	1,535	100.0	2,690

Source: Fluor

in a single-stage furnace is a mere 850°C, which is woefully inadequate to ensure impurities destruction. The alternatives available to allow the Claus plant to operate with acceptable furnace temperature, thus allowing to process such a stringent feed gas cocktail, are discussed in the next section.

The alternatives and the path forward

The possible methods for improving the Claus furnace temperature have already been mentioned. In this section each of the methods are explored to improve the furnace temperature for the case study sulphur plant.

Feed preheat

Table 3 defines the impact of preheating on the case study data.

The following options were studied:

- Case 1: no preheating – base conditions;
- Case 2: preheat amine acid gas only (including TGTU recycle) to at least 220°C;
- Case 3: preheat all acid gas streams (amine acid gas, sour water stripper acid gas and SO_2-rich gas) to at least 220°C;
- Case 4: preheat all acid gas and air stream to at least 220°C;
- Case 5: preheat all acid gas stream to at least 220°C and preheat air to at least 350 °C;
- Case 6: preheat all acid gas and air stream to at least 350°C.

Preheating up to 350°C may be achieved by adding an electric heater downstream of the stream air preheater.

From Table 3 it is evident that preheating alone would not be sufficient to achieve the necessary Claus furnace temperatures, and hence alternative methods were investigated.

Table 3: **Claus furnace temperature and extent of feed preheat**

Options	Temperature values in °C				
	Amine acid gas	SWS acid gas	SO_2-rich gas	Air	Claus furnace
Case 1	50	90	50	80	1,025
Case 2	220	90	50	80	1,060
Case 3	220	220	220	80	1,080
Case 4	220	220	220	220	1,125
Case 5	220	220	220	350	1,160
Case 6	350	350	350	350	< 1,200

Source: Fluor

Fuel gas cofiring

Fuel gas cofiring may be applied to increase the furnace temperatures, but due to very stringent feed gas compositions high amounts of natural gas need to be burnt in the Claus furnace. Burning around 175 kg/h of natural gas (containing about 90% methane, the rest heavier hydrocarbons) achieves a furnace temperature of 1,100°C, while to reach a temperature above 1,250°C, 600kg/h of natural gas is envisaged. As already specified, a continuous application of fuel gas firing is typically not recommended for various reasons, and hence was not considered further for any subsequent assessments.

Oxygen enrichment

The furnace temperatures achieved with various levels of oxygen enrichment is shown below:

- Low level (28% O_2): 1,100°C
- Mid level (45% O_2): 1,210°C
- High level (60% O_2): 1,260°C

Thus, when relying on oxygen enrichment alone, the Claus burner/furnace needs to be designed with high level oxygen enrichment to ensure a high enough furnace temperature for ammonia destruction. The amount of oxygen required would add up to around 75 t/d.

Two-zone furnace design

For the case study, a two-zone furnace was conceived where the amine acid gas flow was split between the front and rear end of the furnace.

The front zone furnace temperatures achieved with a two-zone furnace design are represented below:

- 80:20 amine acid gas split: 1,175°C
- 70:30 amine acid gas split: 1,210°C
- 60:40 amine acid gas split: 1,270°C

The above results indicate that a two-zone furnace designed with a 60-40 split just meets the 1,250°C furnace temperature requirement in the front zone.

Using oxygen enrichment, combined with preheat, although a viable option, was ruled out due to the high operating costs to sustain continuous oxygen usage. The case study refinery did not have a ready source of oxygen; and importing liquid oxygen from an external source or investing in a dedicated oxygen generation unit using PSA/VPSA technology was found to be economically unattractive. For refineries that have a ready and cheap source of oxygen this may be the preferred route.

For the case study, the two-zone furnace design was selected as the preferred option, complemented with some feed preheat to allow for operating flexibility, mainly to achieve adequate furnace temperatures at a range of possible operating scenarios especially during partial turn down operation when the amine acid gas flow would reduce more than the SO_2-rich gas flow.

Summary

Small amounts of SO_2 in the Claus feed gas has a favourable impact on the overall hydraulic design of the sulphur recovery unit, as SO_2 replaces air, thus reducing the gas volume leading to smaller sizes for equipment and piping. At higher concentrations of SO_2 the positive impact of reduction of air demand tends to be overshadowed by the quenching effect on the Claus furnace, reducing the furnace temperatures below allowable operating limits for proper NH_3 destruction. Design modifications are required for Claus plants which co-process significant amounts of SO_2 along with the normal feed gas cocktail of H_2S and NH_3.

Relatively small amounts of SO_2 in the feed gas may be easily managed by feed preheat alone. However, with higher amounts of SO_2 in the feed, there is no single, obvious solution available. Instead, application of one or a combination of multiple design modifications will be required. A careful study needs to be performed to select the optimum solution for the specific project application. ■

The age and friability for different forms of elemental sulphur

Aged sulphur products can be friable and fragile, which can lead to sulphur dust during handling. Because sulphur dust can lead to dust explosions and excessive wet sulphur contact corrosion, shipping and handling specifications for the safest products are used by producers, shippers and consumers to limit dangerous incidents. Metastable polymeric sulphur in the solid product limits friability and is rarely cited as a measured quantity within sulphur specifications, but often discussed when explaining best handling and forming practices. In this article, ASRL discusses why sulphur tends to be friable and explores several measurements cited in many specification documents, with the purpose of focusing on several modern solid forms. In addition, the measurement of total and extraneous water is explored.

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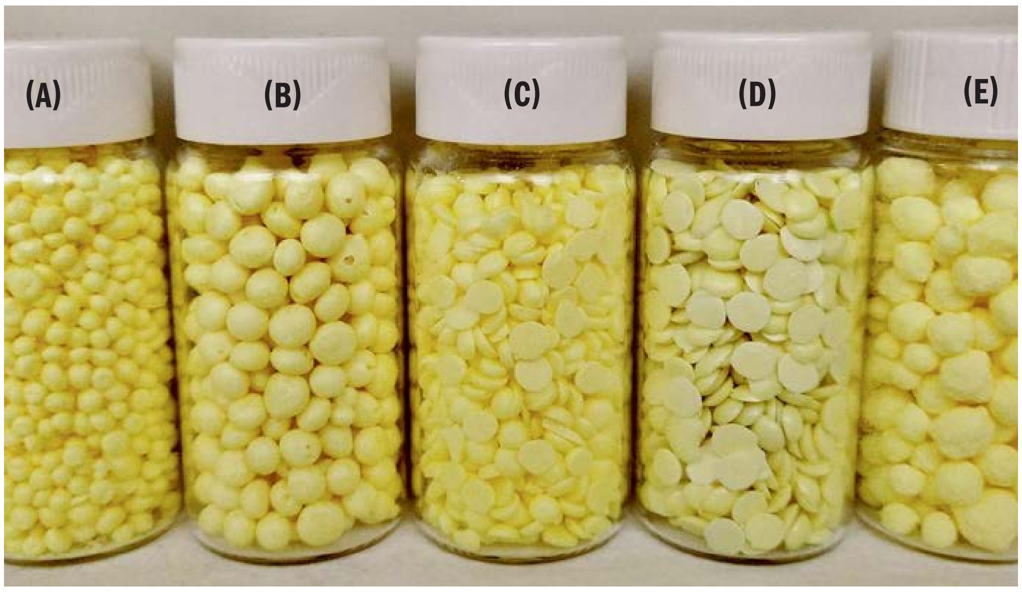


PHOTO: ASRL

Fig. 1: From left to right: (A) commercial air prills, (B) lab formed water prills, (C) commercial pastilles, (D) lab formed pastilles and (E) commercial granules.

Commercially produced sulphur is available in the form of pastilles, granules or prills. To ensure production of a high-quality formed product, several properties are of interest to producers and consumers, such as H_2S content, friability (fragility), water content, porosity, etc. As discussed by many authors, sulphur's properties change during the initial forming of the product, aging of the product and might differ depending on the use of degassed or non-degassed liquid sulphur. With controlled batches of commercial air prills, commercial granules, commercial pastilles, lab formed water prills, and lab formed pastilles (Fig. 1), ASRL has been following several properties during aging to improve the understanding of optimum handling and forming practices.

Upon solidification and further cooling, solid S_8 transforms from monoclinic solid crystal structure (β-solid) to an orthorhombic crystal structure (α-solid) at $T < 95.2°C$ (203°F)[2,3]. Note that the monoclinic form can be kinetically stable below 95.2°C for some time; therefore, it is important to recognise that the solid β-α phase transition may not occur immediately during the formation process and some crystal transition may occur during handling or storage. The transition is exothermic and will exhibit a maximum thermodynamic temperature halt at T = 95.2°C, i.e., if heat is not taken away or if the transition begins at a warm temperature, the internal temperature of a sulphur stockpile could reach 95°C upon transformation. For this and other reasons discussed later, it is important to store solid sulphur at cooler temperatures (T< 50°C).

While the orthorhombic solid is thermodynamically favourable during shipping and handling, this form naturally contains

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56

internal shear stress due to changing the crystal unit cell angles from 96° to 90° during the solid β-α phase transition (see Fig. 2)[3-6]. Note that if the unit cell angles remained the same during the transition, a density change (volume reduction) would not cause shear stress, thus it is not entirely correct to assume that the density change alone induces solid fractures in solid sulphur particles.

The overall consequence of this transition is that pure S_8 (as an α-solid) can be very brittle or friable, which leads to extraneous sulphur dust and fines during product handling. This dust can lead to dangerous and explosive situations. The presence of polymeric sulphur (S_μ) helps to bind and support these stressed shear planes (crystallite disconnects); thus, most forming and handling processes aim to insure some polymeric sulphur content in the product for safety reasons. Note that sulphur specifications rarely include a polymeric sulphur content, which is a critical value.

ASRL and others had previously shown that sulphur with a lower S_μ content results in a higher occurrence of fines when subjected to Stress Level II testing[9,10]. Because S_μ is not thermodynamically stable in the solid sulphur phase at elevated temperature, a warmer solid can lead to conversion to S_8, whereas at colder temperatures the polymeric sulphur is kinetically stable for very long periods of time.

$$S_8 + S_\mu \rightarrow S_8 \qquad (1)$$

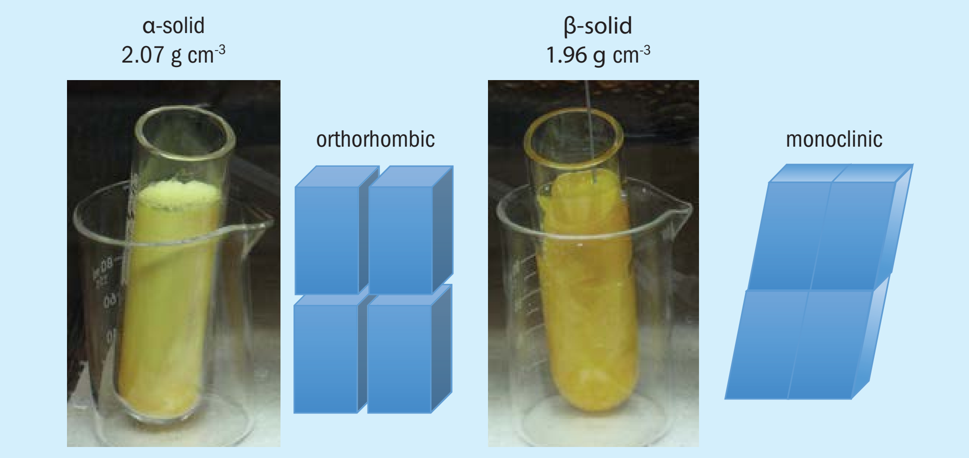
There are multiple questions which can be asked when determining why a solid sulphur product could contain low levels of polymeric sulphur, including:

- How fast was the sulphur quenched from liquid to solid temperatures?
- How cold was the stockpiling temperature of the solid?
- What temperature (and time) was the solid sulphur stored at prior to shipping/analysing?
- Was there any extraneous dissolved chemical left in the sulphur before forming?
- Was the liquid sulphur degassed?
- What temperature was the liquid stored at prior to forming the solid?

In 2020, the first part of this study assessed the last two questions and concluded that fully degassing sulphur (with zero dissolved H_2S; see Fig. 3) did not significantly alter the polymeric content of the liquid sulphur[1], suggesting that over-degassing will not compromise product strength, as long as degassing catalysts do not remain in the product. Here polymeric sulphur is measured as the CS_2 insoluble fraction, S_μ, if carbon content and ash are low[1]. While degassing H_2S is beneficial for safety reasons and over-degassing may lead to excess SO_2 emissions, there is little reason to believe that degassing will improve the quality (strength) of a sulphur product. Thus, H_2S_x in solid sulphur is not necessary. In fact, several of the products investigated in this study were completely free of dissolved H_2S.

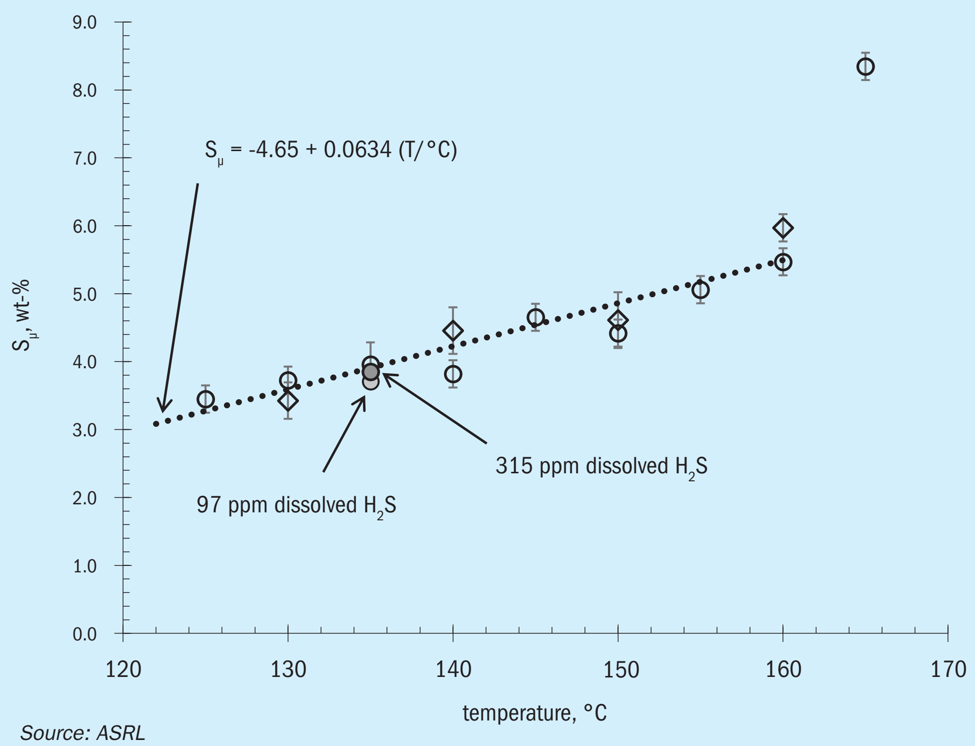
A previous ASRL study also concluded that liquid sulphur storage temperatures between 125 and 160°C do not significantly influence the polymeric sulphur before forming the solid. The current study is an attempt at answering the other questions and aims to quantify the polymeric

Fig. 2: **The solid-solid β-α phase transition for S_8[7]**



Source: Photographs from Dowling and Lau[8]

Fig. 3: **Equilibrium CS_2 insoluble fractions for liquid elemental sulphur from T = 125 to 165°C. Open circles denote measurement Method C and open diamonds denote measurement Method E from Marriott et al[1]**

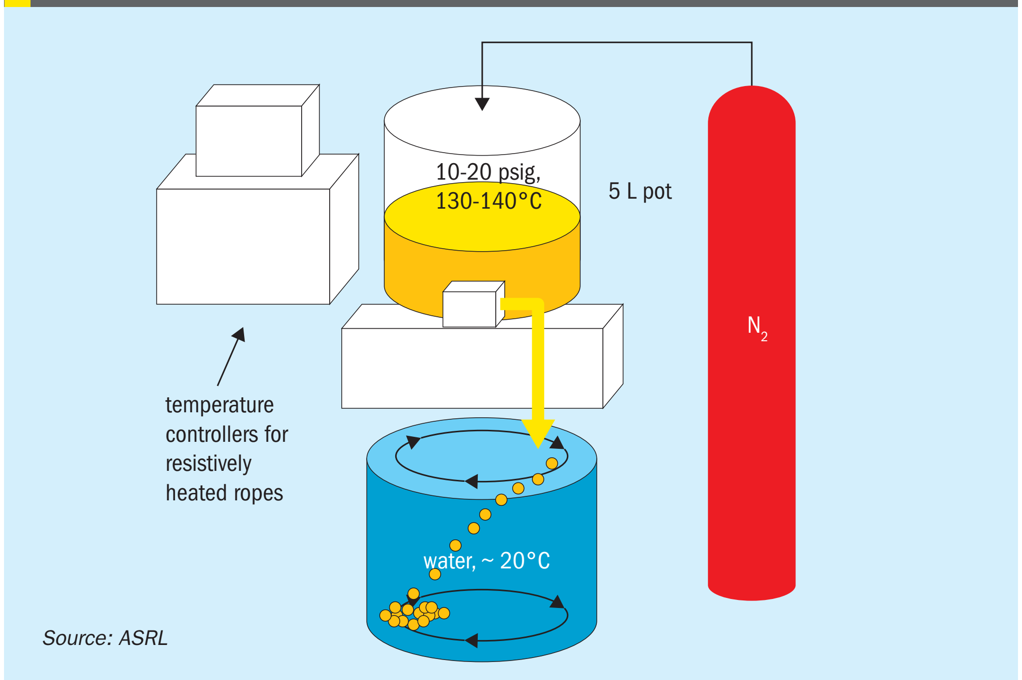


Source: ASRL

sulphur decay after solidification and over time. Finally, ASRL took a brief look at the water content of formed sulphur, where two types of water can be measured.

In order to produce formed prills within the laboratory, ASRL developed its own lab-scale wet forming process. Similarly, ASRL recently commissioned an IPCO Rotoform MI pastillation system to allow for production of small batches of lab-scale pastilles. ASRL also studied field samples of granulated sulphur, pastilles and air-prilled sulphur. While these various forms of sulphur are not directly comparable to each other in terms of morphology, they are all considered premium products and studying their properties allowed ASRL to determine what factors may have an impact on product quality during storage and aging.

Fig. 4: **Lab-scale wet forming process**



Source: ASRL

Sulphur forms

Commercial sulphur forms

The two main commercial sulphur forms used in this study were (C) Rotoform pastilles obtained from the Shell Shantz Gas Plant (> 99.993% purity by combustion) and (A) air-prilled sulphur from the Keyera Strachan Gas Plant (> 99.995% purity by combustion)[1]. Note that these materials were seven and four years old respectively and infra-red (IR) absorbance showed no detectable dissolved H_2S or hydrocarbon in the melts. Additional testing was also performed with (E) commercially produced granules obtained from the Waterton Complex. This product was approximately one month old when used for this project.

(B) Laboratory wet forming

The lab-scale wet forming process consisted of a custom machined sulphur pot (~5 L), sulphur delivery line ($^1/_8$-inch stainless steel) and a circulating water reservoir (~63 L). The sulphur pot, and delivery line were wrapped in resistive heating ropes and insulation. The water in the reservoir was circulated using a Fluval® SEA CP3 Circulation Pump, and ca. 0.5 g of sodium dodecyl sulphate was added to the reservoir prior to forming to prevent surface aggregation. The sulphur pot was pressurised to $p \approx 20$ psig with nitrogen, to facilitate sulphur flow through the $^1/_8$-inch delivery line (see Fig. 4).

Prior to the forming step, a portion of the liquid sulphur was allowed to flow through the delivery line and was directed to a waste collection container to allow for temperature stabilisation of the delivery line. Molten sulphur flow was then stopped briefly, and the outlet of the delivery line was positioned at < 1 cm from the surface of the water. The sulphur pot outlet valve was manually controlled to ensure that the sulphur was emitted as continuous individual droplets (ca. 2 drops s^{-1}). The water circulation allowed the molten droplets enough residence time to completely solidify before hitting the base of the reservoir. At the end of the process, the valve was closed, and the product was collected and allowed to air dry prior to analysis.

(D) Laboratory pastillation

The lab-scale pastillation process setup consisted primarily of a bespoke sulphur pot (~39 L) and a newly commissioned IPCO Rotoform MI lab-scale pastillator (see Fig. 5). The sulphur pot utilised a countercurrent heat exchange system via heated silicone oil, supplied from a LAUDA® Proline P18 heated circulating bath. Once loaded with solid sulphur, the vessel lid was hermetically sealed to prevent the escape of sulphur vapours.

The sulphur vessel was connected to the Rotoform MI system via a $^1/_2$-inch stainless steel braided delivery line. The delivery

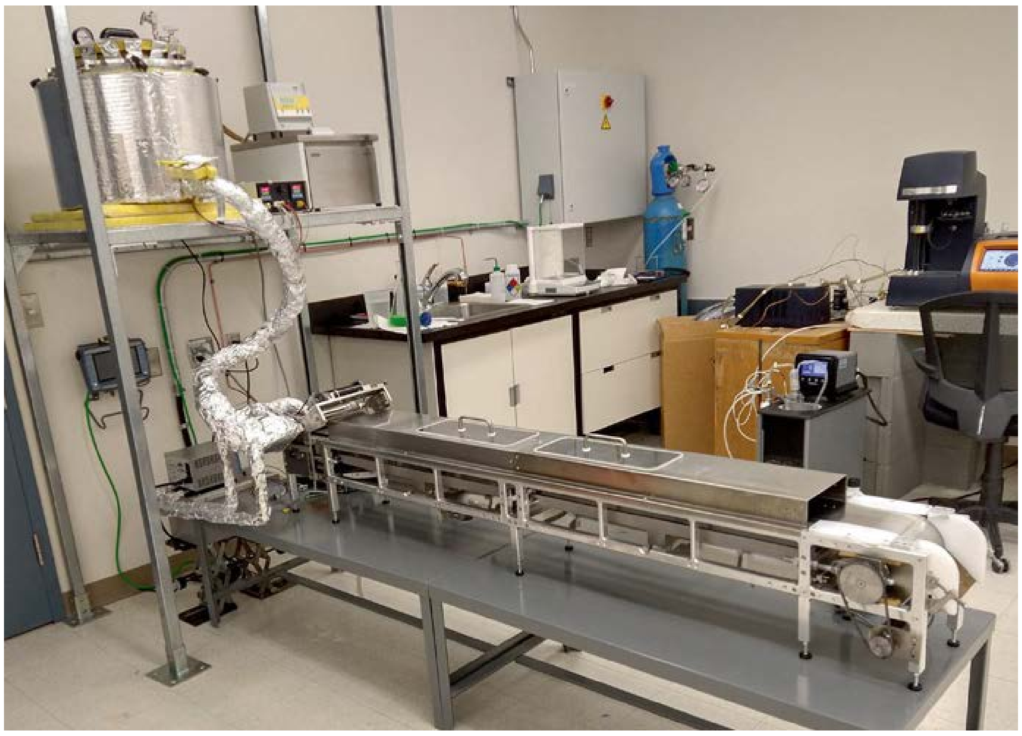


PHOTO: ASRL

Fig. 5: Lab-scale pastillation process.

line and flange connections were resistively heated via heat ropes to prevent sulphur solidification and cold spots. The rotating sulphur outlet drum on the Rotoform MI was heated by a heated silicone oil loop supplied from a VWR® 1136D heated circulating bath. The Rotoform MI system was principally comprised of a steel belt conveyor system and a rotating sulphur outlet drum, set at 10.3 m min^{-1} and 13.6 m min^{-1} speeds, respectively. Cooling water was sprayed underneath the stainless-steel belt to speed up solidification of the molten product as it moved down the conveyor line.

The outlet valve of the sulphur pot was slowly opened while the rotating drum spun, with an operator controlling the valve until the product had a consistent size/shape. The uniform product was then collected for analysis.

Sulphur properties testing methods

S_μ content

Polymeric sulphur content was determined as CS_2 insoluble sulphur by dissolving ca. 10 g of sulphur sample in carbon disulphide, followed by filtering the solution through a pre-weighed medium (10-15 µm) 30 mL sintered stemless Pyrex funnel. The funnel was then dried in a vacuum oven and weighed again to obtain the weight of insoluble S_μ material. Note that the first part of this study confirmed that no detectable amounts of other CS_2 insoluble impurities were present in the stock sulphur[1].

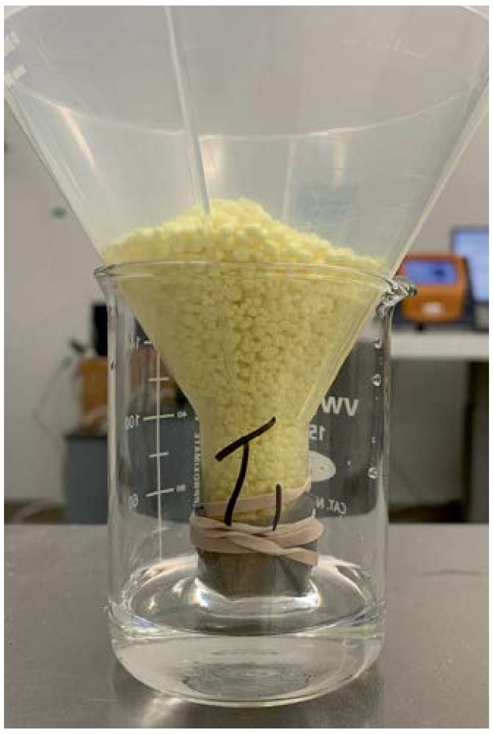
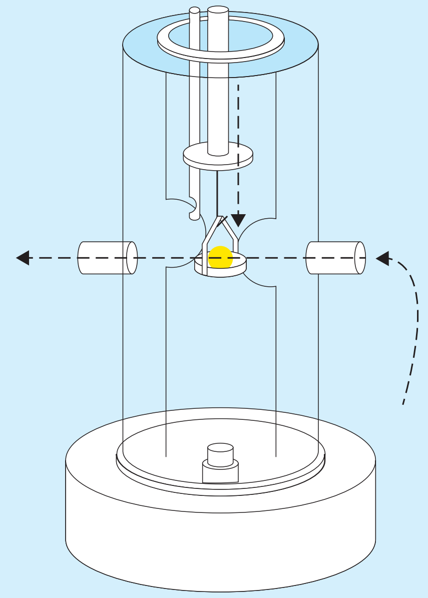


PHOTO: ASRL

Fig. 6: Water retention testing setup.

Fig. 7: **Thermogravimetric analysis setup**



Source: ASRL

S_μ decomposition

For tests requiring holding sulphur at a certain temperature to investigate S_μ decomposition within the formed solid product, ca. 1.3 kg sulphur was loaded into a Bruker 450-GC oven. It was then heated to the target temperature (50-100°C) for the desired time. S_μ content was then determined after quenching to room temperature.

Interstitial water retention (15-minute drainage)

To determine the interstitial water retention of sulphur forms, a mass of ca. 20 g of sulphur form was loaded into a 125 mL beaker and covered with water. The mixture was allowed to sit for 30 min and was then transferred to a pre-weighed beaker-funnel system with a metal mesh (150 µm) occluding the funnel's outlet (Fig. 6). The mixture was allowed to drain for 15 minutes into the beaker. The resulting wet sulphur was then transferred into a pre-weighed crystallising dish and weighed to determine the water retained by the sulphur form.

Thermogravimetric analysis (TGA)

A TA Instruments TGA 55 apparatus was used to determine if internal water was occluded within the sulphur product (Fig. 7). A single sulphur form was placed on a 100 µL platinum pan at 25 to 30°C, followed by a temperature ramp at a specified heating rate (2.5 to 25°C min^{-1}) until it reached a temperature of 145 to 150°C. A dry helium gas purge rate of 30 mL min^{-1} was used.

PHOTO: ASRL

Fig. 8: Stress Level II rotating drum.

Surface area and porosity

The Braunauer, Emmett and Teller (BET)[11] specific surface areas and Barrett, Joyner and Halenda (BJH)[12] pore size distributions for sulphur samples were determined via N_2 physisorption at -197°C on a Micromeritics 3Flex Surface Characterization Analyzer. The samples were degassed ex-situ at room temperature conditions and in-situ (T = 30.0°C) under vacuum to remove volatile species prior to analysis.

Particle size distribution

To determine the particle size distribution for a sulphur sample, ca. 1 kg of formed sulphur (produced at least 20 days prior) was loaded into a pre-weighed sieve stack (>4.75 mm, >2.36 mm, >1.18 mm, >0.600 mm, >0.300 mm, <0.300 mm) on a Retsch AS200 Sieve Shaker apparatus. The apparatus sieved the sample for 20 minutes continuously at a 40% amplitude. The sieves were then reweighed to determine the particle size distribution of the sample.

Stress level II (SLII)

To determine the extent of breakdown of a sulphur sample via a Stress Level II Test, a pre-weighed mass of sulphur (ca. 1 kg) was loaded into a large iron rotating drum (76 cm diameter) equipped with an internal protruding baffle (9 cm) and fully rotated 40 ± 1 times (Fig. 8). The resulting sample was then scrupulously collected from the drum to minimise product loss. The sample

1 47
2 48
3 49
4 50
5 51
6 52
7 53
8 54
9 55
10 56
11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46

Table 1: **Particle size distribution of different sulphur forms**

Product	>4.75 mm	2.36-4.75 mm	1.18-1.70 mm	0.500-1.18 mm	300-500 µm	<300 µm
(A) Commercial air prill	0.04	93	5.8	1.1	0.28	< 0.01
(B) Lab water prill	5.9	94	0.07	0.01	< 0.01	< 0.01
(C) Commercial pastilles	1.1	96	1.2	1.1	0.44	0.38
(D) Lab pastilles	9.6	89	0.95	0.44	0.11	0.03
(E) Commercial granules	50	48	1.1	0.12	0.01	0.04

Source: ASRL

was then loaded into a pre-weighed sieve stack (>4.75 mm, >2.36 mm, >1.18 mm, >0.600 mm, >0.300 mm, <0.300 mm) on a Retsch AS200 Sieve Shaker apparatus, using the same procedure as before.

Results and discussion

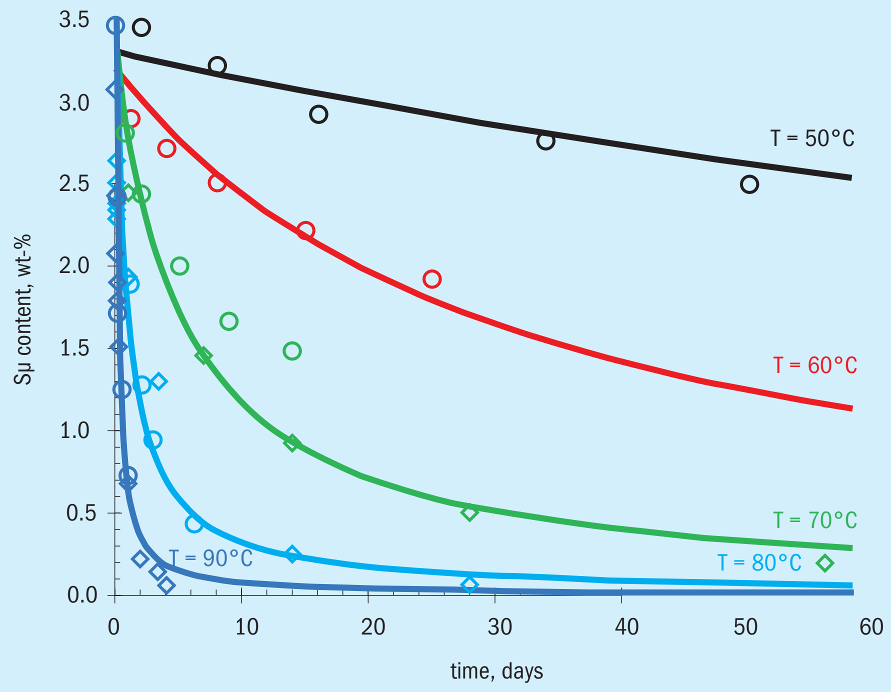
The sulphur industry in general, recognises several tests to determine if a sulphur form is of sufficient quality to be used by customers. The pass/fail criteria are not universal but are in large, inspired by the Sulphur Development Institute of Canada (SUDIC) recommendations from 1978. Typically, criteria include shape, size, size distribution, friability, bulk density, compaction breakdown, angle of repose, moisture content and chemical purity. Additional specifications are often negotiated between different parties when trading sulphur. These typically specify the maximum concentration of different impurities, for example, ash, carbon, hydrogen sulphide, arsenic, selenium, tellurium, water, acidity, chloride, etc.[13]. While only a few of these parameters were studied within the current project, they provided valuable information. Interestingly enough, CS_2 insoluble or polymeric sulphur content is rarely specified or discussed despite our understanding that polymeric sulphur is required for sulphur strength and dust mitigation.

Initial sulphur fines

Initially, the different forms of sulphur used in this study were tested for particle size distribution, as this is one of the tests that is typically quoted to determine if a form is a premium product. This test was performed on each form, prior to any heat treatment. Results are shown in Table 1.

The 1978 SUDIC specifications state that a premium product should contain < 0.1 wt-% of fines (< 300 µm). Most forms tested met this specification regarding fines. Note however, that the commercial pastilles contained marginally more fines, with 0.38 wt-%. 0.38 wt-% is still far below the 5% sited by SUDIC for

Fig. 9: **The decay of polymeric sulphur content at different temperatures for sulphur in the orthorhombic form. ○, this study; ◇, Dowling and Wan[14]**



Source: ASRL

Table 2: **Polymeric content of air-prilled sulphur in the orthorhombic form after aging at 50 to 90°C**

T = 50 °C		T = 60 °C		T = 70 °C		T = 80 °C		T = 90 °C	
t (days)	S_μ (wt-%)	t (days)	S_μ (wt-%)	t (days)	S_μ (wt-%)	t (days)	S_μ (wt-%)	t (days)	S_μ (wt-%)
0	3.47 ± 0.03	0	3.47 ± 0.03	0	3.47 ± 0.03	0	3.47 ± 0.03	0	3.47 ± 0.03
2	3.45 ± 0.06	1.2	2.90 ± 0.04	0.67	2.81 ± 0.03	1	1.90 ± 0.06	0.10	2.42 ± 0.05
8	3.22 ± 0.06	4	2.72 ± 0.01	2	2.44 ± 0.03	2	1.28 ± 0.02	0.27	1.71 ± 0.05
16	2.93 ± 0.06	8	2.51 ± 0.01	5	2.00 ± 0.02	3	0.95 ± 0.01	0.47	1.25 ± 0.02
34	2.77 ± 0.09	15	2.22 ± 0.02	9	1.66 ± 0.01	6.2	0.43 ± 0.02	1	0.73 ± 0.01
50	2.50 ± 0.03	25	1.92 ± 0.01	14	1.48 ± 0.03			4.75	0.08 ± 0.02

Error provided as 1 s.d.

Source: ASRL

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46
47 48 49 50 51 52 53 54 55 56

the minimum specification. The fact that the laboratory version (custom pastilles) contained only 0.03 wt-% fines seems to suggest that this form may also meet the specification when freshly produced but that perhaps, through handling the sample, it had degraded further. Later results will show that these pastilles produced virtually the same quantity of fines, following SLII stress tests.

Polymeric sulphur decay

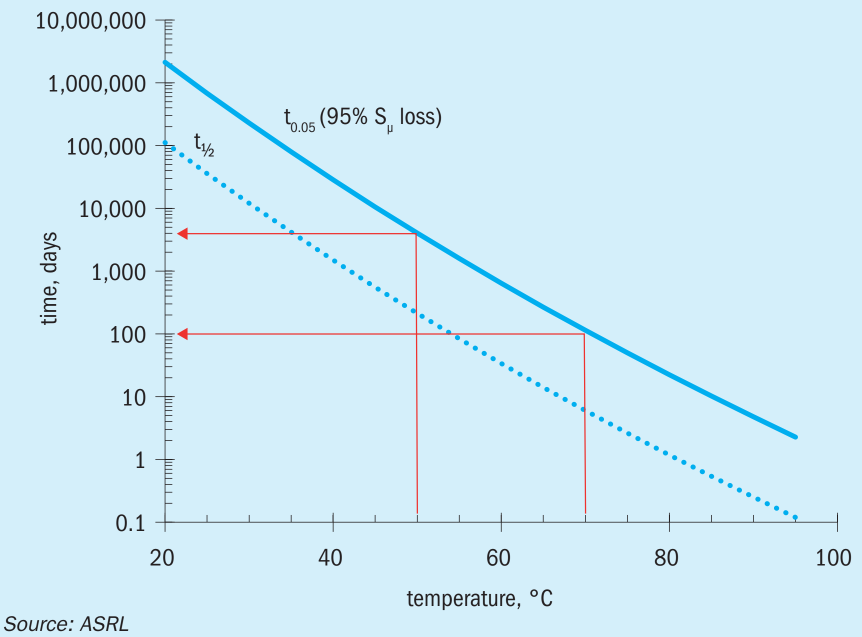
As discussed in the introduction, the friability of elemental sulphur and its propensity to form dust should be significantly affected by the amount of polymeric sulphur retained in the product (S_μ as a CS_2 insoluble wt-%). Polymeric sulphur serves to bind crystallites which undergo stress due to the monoclinic to orthorhombic crystal transition. ASRL showed previously that the liquid storage temperature before forming a product has little effect on the retained polymeric sulphur in the formed solid; however, ASRL also knows that warm solid sulphur can lose its polymeric sulphur over time.

In order to get a clearer understanding of how fast polymeric sulphur would deteriorate when solid sulphur is stored at higher temperatures, ASRL measured the S_μ content for commercial air prills at T = 50 to 90°C (Fig. 9 and Table 2). Fig. 9 also includes data from Dowling and Wan, where their data at 70 and 90°C complement the new data reported in table 2. Dowling and Wan were investigating sulphur block storage, which would suffer the same polymeric decay; however, friability would be more important for block integrity. ASRL found that the formed sulphur still contained S_μ > 3.4 wt-% after more than 4 years of being stored in our laboratories (ca. 20°C), which indicates that sulphur can maintain its important polymeric binder for a very long time, provided that the storage temperature is cool enough.

Fig. 9 shows that orthorhombic sulphur stored above 50°C shows significant loss of polymeric sulphur over time, where warmer storage temperature shows a more significant rate of decay. The decay of polymeric sulphur shown in Fig. 9 has been fit using the global data and an exponential decay (shown in solid lines within Fig. 9):

$$\frac{1}{\text{wt-\%}} = \frac{1}{\text{wt-\%}_{t=0}} + e^{\left(43.37 - {}^{19775}/_{T}\right)} t \qquad (2)$$

Fig. 10: **The time for 50% and 95% polymeric sulphur decay in orthorhombic sulphur at storage temperatures from 20 to 100°C**

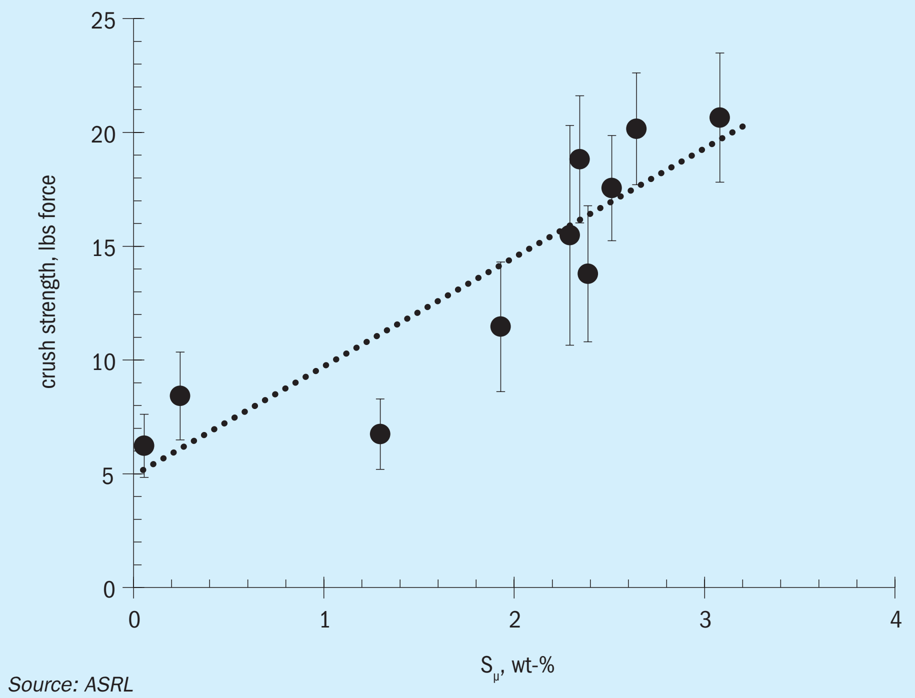


Source: ASRL

In Equation 2, the temperature, T, is in units of Kelvin and time, t, is in seconds. Using this equation, both the half-life, t0.5, or when one expects to lose 95% of the initial sulphur, t0.95 can be calculated. These times versus the temperature are shown in Fig. 10. Using Fig. 10 for a storage temperature of 50°C, one can see that 95% of the polymeric sulphur will be lost after approximately ten years; however, at a nominal storage temperature of 70°C, 95% of the polymeric sulphur will be lost at approximately 100 days. 90°C storage temperatures would result in less than half of the initial polymeric sulphur in less than seven hours and less than 5% of the initial polymeric sulphur in five days.

To make matters a little more complicated, sulphur initially solidifies by crystalising in the monoclinic form. The monoclinic form can persist for some time below its thermodynamic transition point of 95.2°C[2]. If sulphur products were to be piled up before this transition occurred,

Fig. 11: **The relationship between polymeric sulphur, S_μ, and crush strength[14]**



Source: ASRL

Table 3: **Polymeric content and post Stress Level II fines for various sulphur forms**

	Stock		[a]70 and 80°C		[b]90°C		[c]100°C	
	S_μ (wt-%)	<0.3 mm (wt-%)	S_μ (wt-%)	<0.3 mm (wt-%)	S_μ (wt-%)	<0.3 mm (wt-%)	S_μ (wt-%)	<0.3 mm (wt-%)
(A) Commercial air prill	3.5	0.7	0.43	1.5	0.08	2.8	0.04	2.3
(B) Lab water prill	2.8	2.3			0.07	3.4	0.02	3.9
(C) Commercial pastilles	3.4	1.7			0.03	1.7	0.03	1.9
(D) Lab pastilles	3.8	1.8	3.0	2.3	0.44	2.0	0.10	2.4
(E) Commercial granules	1.8	1.4			0.04	1.4	0.04	2.5

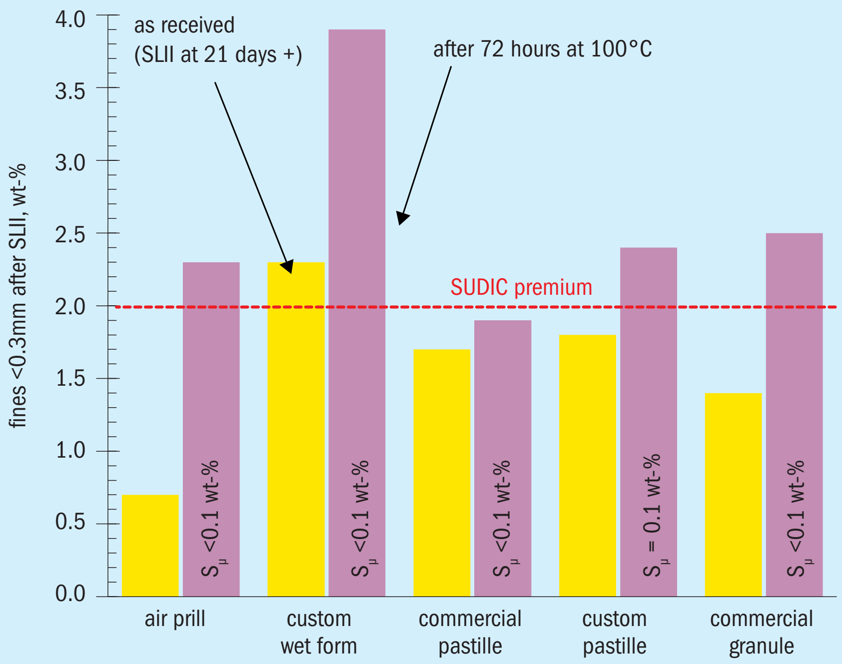
[a]Air prill sulphur was annealed at 80°C for 148 hours and laboratory pastilles were annealed at 70°C for 143 hours; bsulphur products were annealed at 90°C anywhere from 96 to 167 hours; csulphur products were annealed at 100°C anywhere from 24 to 72 hours, after which the sulphur was allowed to convert back into the orthorhombic form at room temperature.

Source: ASRL

the exothermic transition would spontaneously heat the interior of the stockpile (to a maximum temperature of 95°C). This form of sulphur appears translucent and orange, versus the opaque and yellow orthorhombic form. ASRL also measured the decay of polymeric sulphur in the supercooled monoclinic form, by heating to 100°C (converting to monoclinic) and then cooling to 90°C (metastable monoclinic). The rate of decay was ten times faster than in the orthorhombic form. Thus, monoclinic sulphur could lose 95% of its polymeric sulphur in ten days at 70°C or 12 hours at 90°C. Thus, there are two issues with stockpiling sulphur in its monoclinic form: (1) it could self-heat if the heat from the exothermic phase change can't be removed and (2) the polymeric sulphur will decay much faster if the heat is not removed. Another added issue is that monoclinic sulphur products may tend to agglomerate.

Based on the previous calculations, storage temperatures at 50°C (internal stockpile temperature) or less still provides a cold enough environment for polymeric sulphur to persist for years. If sulphur forms are initially handled in the monoclinic form (translucent and slightly orange), then the heat from the solid transition will need to be monitored or removed (through perhaps smaller piles and/or aerial fans).

Fig. 12: **Comparison between SLII produced fines and S_μ content for different forms of sulphur**



Source: ASRL

Friability, strength and dust

Dowling and Wan (2012) previously showed that the crush strength of sulphur was correlated with the polymeric content (Fig. 11)[14]. Their study focused on formed sulphur blocks, where laboratory sulphur slabs were tested for compressive strength using a load cell. ASRL attempted to measure the crush strength of the various sulphur products using the same load cell apparatus; however, the apparatus can only measure one prill or pastille at a time and the results are not reproducible. The variability in morphology of the products requires other methods. ASRL has commissioned a bulk crush strength apparatus to be reported to ASRL member companies in a future report.

Single prill or pastille crush strength is not a specification that is typically reported, likely because the results are inconsistent and not directly comparable between different sulphur forms. In this study ASRL sought to show that S_μ has a significant impact on formed sulphur quality via the Stress Level II test, which is a more traditional test for sulphur product friability. Results are shown in Table 3. To obtain different concentrations of S_μ, the sulphur forms were held at 70, 80, 90 and 100 °C for various lengths of time.

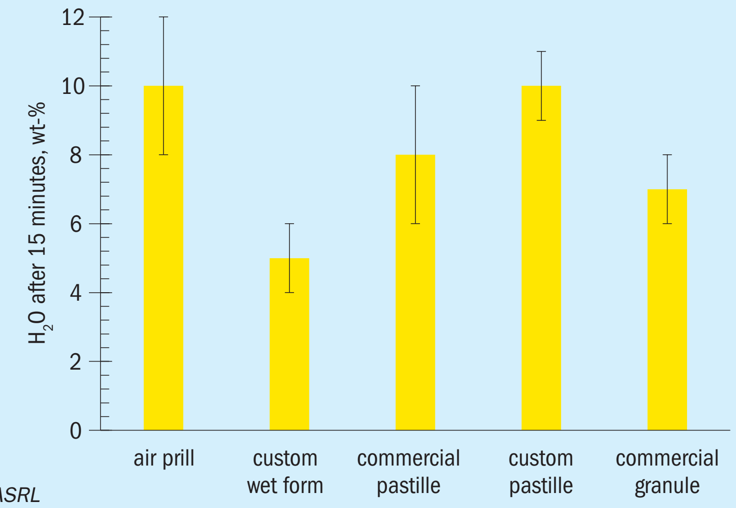
The SUDIC specifications mention that for premium sulphur products, fines following the SLII test should be < 2 wt-%[13]. Based on the results in Table 3, all tested forms would have met this criterion, except for the ASRL custom wet form which could not be considered a premium form as it produced 2.3 wt-% fines. A closer look at the fines results from Table 3 show a soft correlation between polymeric content and produced fines. Fig. 12 shows a comparison between the SLII fines for the non-annealed sulphur products (high polymeric content) and the fines produced from product which was annealed at 100°C (low-polymeric sulphur). The annealing at 100°C and nearly complete removal of polymeric sulphur resulted in a failure for all but one of the sulphur forms.

While it can be seen that the SLII fines increase with reduced polymeric content, it is noted that the SLII test is not as sensitive as one might expect for modern sulphur products. When SUDIC and other groups were looking into SLII test fines to assess sulphur products, slate sulphur was still commercial. It was these forms which were severely failing the SLII test, whereas all the modern forms perform quite well. In terms of laboratory time and reproducibility, it might be easier to measure and report polymeric content in the future, where all products with > 0.4 wt-% polymeric sulphur passed the SLII test. A more precise target for polymeric sulphur may be revealed in future bulk crush tests.

Finally, while sulphur storage temperature, solid phase and polymeric content must be considered to reduce the risk of dust, handling practices are just as important.

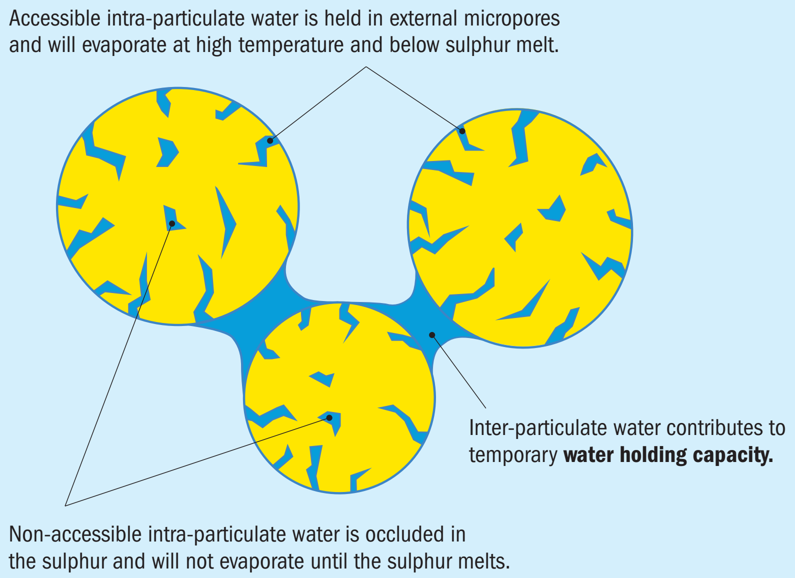
It was also decided to investigate the effect of the presence of different amines on S_μ and SLII test performance. Amines are often used to breakdown polysulfane bound H_2S as degassing catalysts; however, it's well known that too much amine or not allowing amine to evaporate will compromise the product. While ASRL could dose the sulphur feed for the laboratory pastilles with amines, it was difficult to determine the actual amount in the liquid sulphur before forming. ASRL chose to assume that all the amine put into the feed vessel resided in the liquid sulphur. 12-14 ppm of MEA, MDEA and morpholine did not change the polymeric content or the SLII fines. 100 ppm of morpholine in the elemental sulphur resulted in no polymeric sulphur; however, the SLII test passed at 1.4 wt-% fines. Again, this may point to the SLII test as not being as rigorous for modern forms of sulphur. Future investigations might consider looking at how the polymeric content decays in the presence of an amine.

Fig. 13: **Maximum amount of inter-particulate water within a pile of formed sulphur after 15 minutes of water drainage**



Source: ASRL

Fig. 14: **Depiction of the different location of water within a pile of formed sulphur**



Source: ASRL

Water and sulphur products

After looking at polymeric content effects or addition of amine contaminants, the different forms were also tested for how much water remains on the particles after soaking them with water and allowing drainage for 15 minutes. This can approximate to, for example, how the top of a pile of sulphur would behave after a heavy rainfall. Results are shown in Fig. 13 and reported in Table 4. Note that this test assumes that the interaction between the wall and the particles are the same as the interactions between particles and that any wall effects are negligible. The application of water is necessary during handling to reduce dust formation evolution, but this is not typically enough to appreciably increase water content. Due to the extreme nature of our test, the measured values shown in Fig. 13 for all form types are much higher than the recommendations by SUDIC (< 0.5 - 1.5 wt-%). It is important to note that the water retention test results should be viewed as the maximum amount of water that could exist within a pile of formed sulphur 15 minutes after a rainfall (see Fig. 14), whereas the SUDIC values represent an arbitrary target, aimed at lowering transport costs[13]. Note that this 15 minute drainage would eventually evaporate on its own.

Each form also was tested for its internal water content after being wetted and allowed to dry, using thermogravimetric analysis. Prior to testing, each form was allowed to air dry at room temperature for a minimum of 48 hours. This allowed any external water to evaporate. The water remaining within the sulphur form can either be trapped within accessible pores or within inaccessible pores, occluded within each particle. The water within the

Table 4: **Maximum measurable water retention after 15 minutes drainage and natural internal water**

Product	External water 15 minute drainage (wt-% dry basis)	Water from accessible pores (wt-% dry basis)	Water from inaccessible pores (wt-% dry basis)	Total internal water (wt-% dry basis)
(A) Commercial air prill	10 ± 2	< 0.003	< 0.012	< 0.015
(B) Lab water prill	5 ± 1	0.022 ± 0.007	0.059 ± 0.011	0.022 ± 0.013
(C) Commercial pastilles	8 ± 2	< 0.003	< 0.012	< 0.015
(D) Lab pastilles	10 ± 1	< 0.003	< 0.012	< 0.015
(E) Commercial granules	7 ± 1	0.43 ± 0.09	0.33 ± 0.09	0.76 ± 0.12

Error provided as 1 s.d.

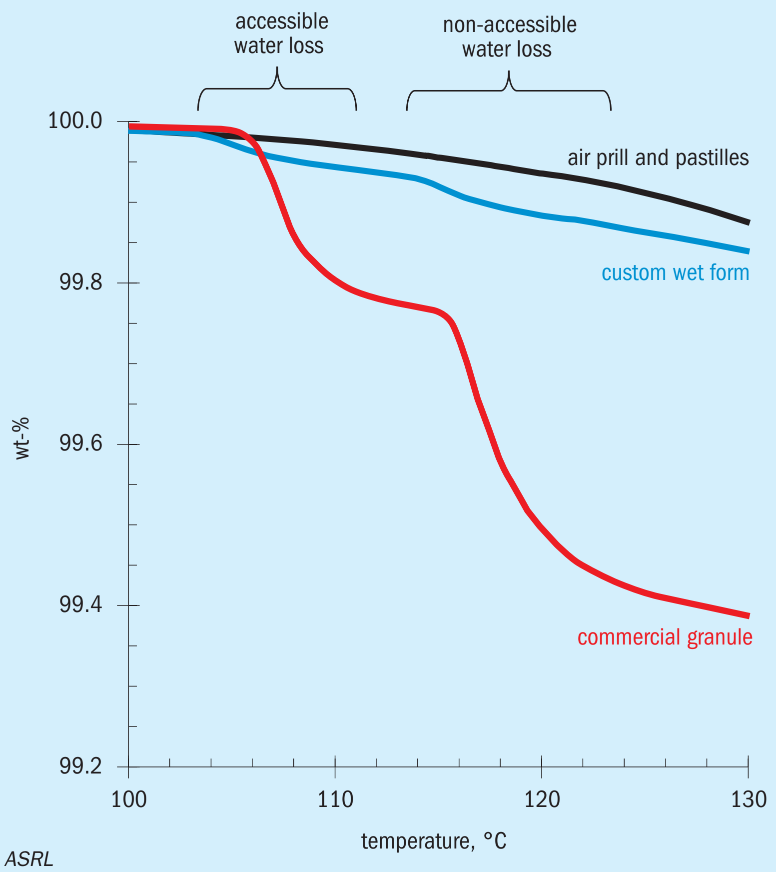
Source: ASRL

inaccessible pores is only accessible once the sulphur particle is melted (Fig. 14). A 5 °C min^{-1} heating rate led to less noisy scans and provided the best sensitivity definition. The overlay of the TGA thermogram of all tested forms is shown in Fig. 15 and the data reported in Table 4.

In Fig. 15 it is apparent that the commercial granule and the custom wet form curves show a different trend than the other three. This is because the water content of the other three forms is below the limit of detection for this analytical technique. The mass change at ~107°C corresponds to water from accessible pores and the peak at ~117°C corresponds to water that sits within inaccessible pores. As the sulphur particle starts to melt, water can escape, and the mass is reduced further. A similar behaviour can be seen for the custom wet form curve, although to a much lesser extent. The occluded (second type) of water is not likely a risk to transportation; however, it might result in frothing during remelt. Note that the increased mass loss near the end of the thermogram is due to the increased sublimation rate of sulphur at higher temperatures.

Finally, both BET surface areas and BJH pore size distributions were measured, as it was originally thought this might give insight into water holding capacity. All sulphur forms were found to have very low surface areas at 0.05 to 0.13 $m^2 g^{-1}$ and only a very small fraction of micropores. Note that after thermal treatment and aging, the sulphur specific surface area dropped to 0.02 to 0.07 $m^2 g^{-1}$. Thus, standard and rapid surface area and pore size distributions were not a reasonable method for looking at sulphur product quality.

Fig. 15: **TGA thermograms of five sulphur forms**



Source: ASRL

Conclusions

In this study, ASRL measured several properties of controlled batches of commercial air prills, commercial granules, commercial pastilles, lab formed water prills, and lab formed pastilles. These results are not meant to compare the different forms but are useful to understand how these properties may be affected by aging. All modern forms of sulphur tested appear to be premium quality with very little propensity to form dust within a SLII test unless the polymeric content of the solid form was allowed to decay below 0.4 w-t%.

Although sulphur polymeric content (CS_2 insoluble) is not mentioned in the original SUDIC specifications, it was found to be a useful parameter when assessing the quality of a sulphur form. In particular, loss of polymeric sulphur can lead to excessively friable sulphur and a moderate failure of the SLII test for premium specification. At 20°C, the concentration of S_μ was determined to remain constant for several years but at 90 °C, freshly formed sulphur can lose over 90% of its S_μ content in less than two hours. It is thus important to cool formed sulphur adequately, to avoid a reduction in crush strength and an increase in fines production. Piling sulphur while hot or before it has transitioned into its orthorhombic form many lead to further polymeric sulphur decay. The delayed transition may cause self-heating within the stockpile (up to 95°C), which will exacerbate the polymeric sulphur decay, in addition to likely leading to product agglomeration. When troubleshooting low quality sulphur product, measuring the S_μ content would likely provide valuable information.

It was previously determined that liquid sulphur storage temperatures do not significantly influence the final product (assuming solid products are rapidly quenched). Degassing also did not alter the polymeric content of the liquid sulphur, suggesting that over-degassing will not compromise product strength, as long as degassing catalysts do not remain in the product. In addition, the latter suggests that, while degassing H_2S is beneficial for safety reasons, there is little reason to believe that degassing will improve the quality (strength) of a sulphur product.

ASRL looked at the maximum water retention after sulphur was saturated with water and allowed to drain for 15 minutes, where no differences were noticeable between forms. In addition, internal water content was found to be below the limit of detection for all tested forms, except for custom wet form and granules. Completely melting the sulphur reveals how much occluded water can be retained, where occluded water may lead to frothing upon remelting the product downstream. ■

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