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Number 357

March | April 2015

SULPHUR

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Sour gas projects

Sulphur markets

ASRL review

Smelter gas to sulphur



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ISSUE 357
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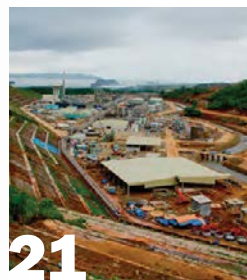
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A momentous year?



“Shah is on course to become one of the most important developments in the sulphur industry this decade.”

Tectonic plates are shifting in the global sulphur industry, as the industry's centre of gravity moves from Canada to the Middle East on the supply side, and two of the most notable manifestations of this are due to come into service this year. In the deserts of the Emirate of Abu Dhabi, the Shah sour gas project is due for start-up imminently (although owner and developer Al Hosn gas recently moved the start date back from Q1 to Q2 2015), while in Florida, Mosaic's new 1.0 million t/a sulphur melter is under construction and also due for a 2015 start-up.

In spite of the project delays (these are actually fairly modest, certainly compared to, say, Kashagan...), Shah is on course to become one of the most important developments in the sulphur industry this decade, adding as it will an extra 3 million t/a of sulphur to the Arabian Gulf's output. The technical challenges that have been overcome are considerable, and the logistical ones have been almost as great. Initial plans for a heated sulphur pipeline have been replaced by building the granulation capacity at the site deep in the desert instead, and then constructing a railway 264km to connect it to the export port at Ruwais. The railway also passes through Habshan, where even more sulphur capacity is being built as other sour gas and condensate fields are plumbed into the Integrated Gas Development there. At a stroke, Abu Dhabi will become the most important sulphur exporter in the world, its output rising from 1.7 million t/a to 7 million t/a, almost 20% of the international market for traded sulphur.

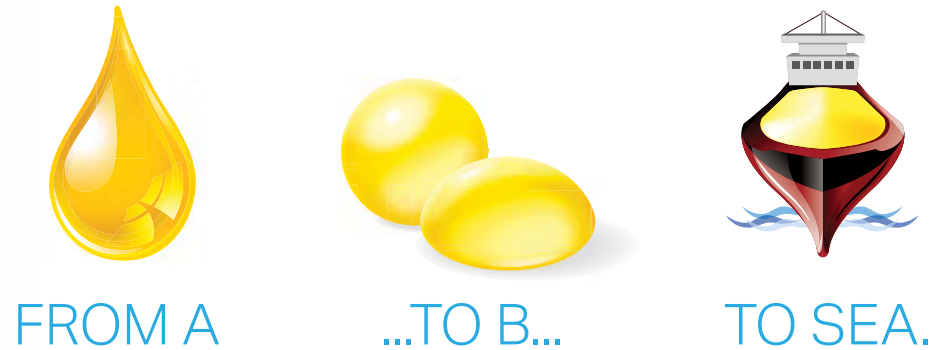
Mosaic's sulphur melter at New Wales, Florida is in some ways equally momentous. Mosaic consumes around 4.5 million t/a of sulphur – almost half of US demand, which totals about 11.5 million t/a. US sulphur production is around 8.6 million t/a, and most of the imports that fill that gap come from Canada, as molten sulphur in rail cars. New Wales in Florida is a long way from Alberta – almost 3,000km from the border, nearly 4,500km from the oil sands patch. This imposes freight costs of up to \$130/t. While US sulphur production is rising incrementally as refineries on the Gulf of Mexico gear up to

handle sourer crude supplies, including Canadian syncrude, there is a great deal of sulphur forming and export capacity on the US Gulf Coast, and the risk for Mosaic is that some of this goes overseas. The sulphur melter allows them to diversify their supply base, taking in cargoes of formed sulphur – perhaps even from Abu Dhabi – when pricing is in favour, and in general this is likely to be cheaper than buying sulphur from Canada.

Equally worrying for the Canadians is that US sulphur consumption is falling at a time when US production is rising, and so is US capacity for cheaper imports. While Canadian sulphur output has fallen from sour gas production, the prospect of losing another 500,000 t/a or more of US demand must be quite a worry, and this is driving a lot of liquid sulphur producers to look at investing in or otherwise gaining access to sulphur forming capacity. But where will Canada export to? China is a long way, and they will have to compete with the cheap sulphur flooding out of the Arabian Gulf. South America is a possibility, and some of the leaching projects in the Pacific and Australasia likewise.

Shah is unlikely to have much of an impact on total sulphur volumes this year, as the gas wells start up and sulphur production ramps up, but according to the best estimates of industry consultants 2015 is nevertheless the year that the sulphur market moves back into surplus. In between Mosaic, Abu Dhabi, and the long-awaited tilting of the market back to surplus, we may look back and see 2015 as a year in which sulphur markets changed for good. ■

Richard Hands, Editor



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Price trends

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

Market reaches ceiling

Improved sulphur demand and price escalation at the start of 2015 was followed by a relatively stable period through February. Chinese buying was notably diminished on the approach to the Lunar New Year holiday as end users had already covered positions. The slowdown in Chinese demand was reflected in the wider market, as buyers started to hesitate before accepting high spot prices. Some interest emerged in India for spot volumes, while interest eased in Latin America following a spike in imports to Brazil in January.

Limited sulphur availability from the Middle East has been a key factor in the run up in spot sulphur prices, heightened by the delay announced for the Al Hosn Shah gas project to Q3 2015. The postponement of the project may keep regional supply stretched in the short to medium term. High refinery run rates in the US meanwhile led to year end sulphur production of 9 million t/a in 2014, but there have also been supply and logistics issues in North America through February as labour disputes led to strikes at both ports and refineries on the US West Coast. Contingency plans at refineries meant that little or no impact on the market was expected, although port closures led to a stack up of California sulphur shipments at export ports.

Middle East producers increased monthly prices for February 2015 for the third consecutive month, reaching \$180+/tonne f.o.b, highs not seen since March 2014. In contrast, North African contract

prices for Q1 2015 were a rollover from Q4 at \$160/tonne c.f.r, despite international price increases in December and January. Buyers' reluctance to accept a price increase may have prompted Aramco's \$5/t drop for its March price, posted at \$175/t f.o.b. Tasweeq decreased its Qatar Sulphur Price (QSP) for March by \$18/t to \$164/t f.o.b. Ras Laffan, a marginal slowdown for the producer. ADNOC meanwhile set its official selling price (OSP) for March shipments to the Indian market at \$175/t f.o.b. Ruwais – a \$5/t decrease.

Prices in China began to weaken in February for the first time since November 2014, dropping to the \$170s/t c.f.r. Spot sulphur prices are expected to continue to weaken going into Q2 2015, although the main counter argument to weaker prices is the low level of China sulphur inventories at major ports – down to around 1 million tonnes at the start of March. Some expect this will spark a spate of buying, which could lead to a short term price spike, or continue to support price highs up to \$190/t c.f.r.

Major oil sands producer Syncrude came back online following maintenance at the end of January due to hydrogen sulphide issues, keeping supply out of Canada healthy in the short term. Prices for sulphur cargoes out of Vancouver are now thought to be fixed on a monthly basis, a change from the previous quarterly contract structure. In South Africa, Foskor is likely to recommence a contract supply relationship for Canadian sulphur in Q2 2015, as it is fully covered until March.

Year-end exports from Vancouver fell by 14% in 2014 compared to 2013 due to logistical issues in western Canada. Australia remained the top export market but there has been a decrease elsewhere, in particular exports to China and Israel. The supply outlook for 2015 Vancouver offshore exports is healthy, with a recovery anticipated. Oil sands supply in Alberta in the medium to long term may be affected by the collapse of oil prices. However, the short term production outlook is unaffected to date. The oil price through the rest of 2015 will be a key indicator impacting the forecast for sulphur production from this sector. Meanwhile, gas based sulphur production in Canada is forecast to continue steadily declining in the year ahead. Increased exports from Vancouver may emerge once the 1 million tonne Mosaic sulphur re-melter project starts up in the latter part of the

year. This may lead to a decline of railed sulphur exports from Canada to the US, thus increasing the likelihood of increased availability for offshore markets from Vancouver.

SULPHURIC ACID

The European acid market has been stable in the year to date. Export prices were unchanged through January and February at \$30-35/t f.o.b. NW Europe, reflecting a flat market and the expected seasonal slowdown following the European holiday period. The 2014 price and supply situation in Europe was positive, and the outlook for the year ahead is also buoyant. Stability is expected to be sustained through 1Q 2015. The recent spike in global sulphur prices has yet to impact sulphuric acid prices in Europe, although some sulphur-based producers have been experiencing increased production costs. For the rest of 2015, the overall outlook remains stable, with forecasts reflecting positive netbacks throughout the year. Prices may firm slightly in coming weeks, should demand in Latin America emerge and supply remain balanced to tight. However, there may be a slight downward correction in Q2 2015, based on any potential downturn in the sulphur market.

European smelter acid producers Aurubis and Boliden released Q4 2014 financial results, both reflecting increased revenues and higher sulphuric acid production rates compared with 2013. Aurubis' outlook included a positive view for demand of copper concentrates. In addition, Boliden reported record production levels at Harjavalta and Odda in 2014.

The market in Chile also started the year on stable footing, with domestic deals in the low-\$80s/t c.f.r during late January. Expectations are for prices to remain in the same range, with some sources describing stock levels as short in mid-February. Interest from end users is expected to emerge during 2Q, with several end users expected to require volumes for shipment in the second half of the year. Chile sulphuric acid imports dropped 21% in 2014 compared with 2013, representing a significant drop in trade to the region. Chile imported 52% of its sulphuric acid from Peru during 2014. Imports from Japan and South Korea fell by 36% and 39%, respectively. This may put pressure on the long term outlook for pricing in Chile, although in 2014 smelter tightness and low inventories led to firm pricing in global markets.



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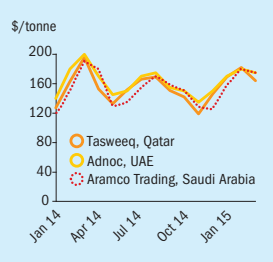
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Fig 1: Middle East FOB monthly prices have started to soften, following the steady upturn since December 2014



Source: ICIS, Integer

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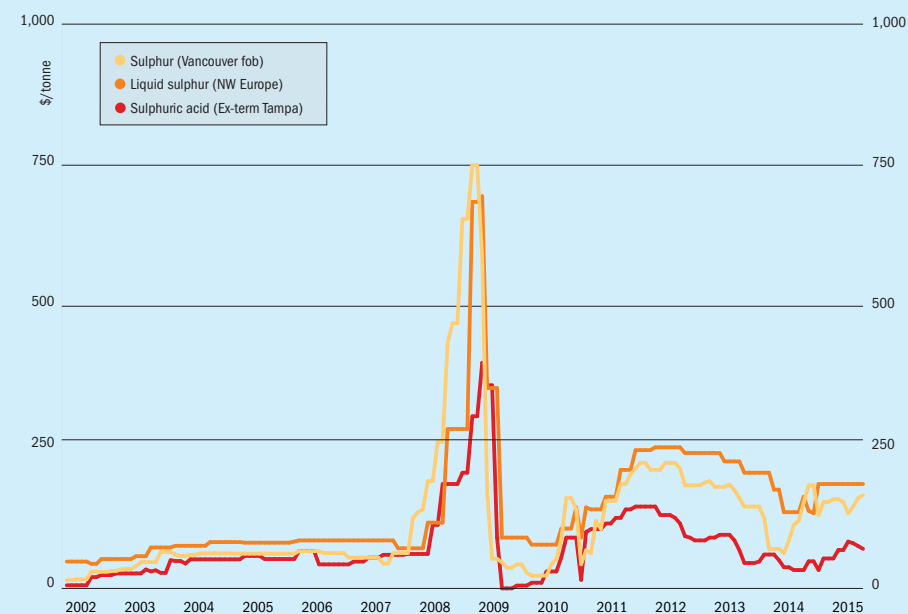
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Market outlook

Historical price trends \$/tonne



SULPHUR

- Spot interest in China may emerge through March due to low stock levels at major ports, providing support to prices. However, high prices may be an obstacle unless the phosphates market also shows support.
- The downward correction in Middle East producer pricing is likely to be short-lived, should support emerge from the phosphates market ahead of the Spring fertilizer season.
- Further delays to the Shah gas project in the UAE pushes the increased export availability forecast later in 2015. However, the rise of sulphur exports from the UAE will likely significantly impact the market from 2016, assuming production rates reach the scheduled levels.
- OCP/Morocco is expected to increase its sulphur imports in 2015, with Middle East producers likely to meet the demands of the buyers processed phosphate fertilizer expansion plans.
- Refinery turnarounds in the Mediterranean region in March are expected to

keep prices firm in the domestic sulphur market, although long term concerns remain for supply security due to the ongoing challenges faced by refiners.

- **Outlook:** Prices are likely to see a downward correction, but the fallout may be limited by the re-entrance of Chinese buyers to the market in March, with a particular view to replenish stocks at major ports, which have dropped down to close to 1 million tonnes. Key markets Brazil and India are also expected to see revitalised import activity as we enter the second quarter, keeping the market in a relatively stable position. The longer term outlook is overshadowed by expectations of increased supply from new sour gas projects.

SULPHURIC ACID

- The PASAR Philippines smelter will undergo maintenance in April, as part of an upgrade which will increase sulphuric acid production. I NW European acid prices are expected to remain stable to firm in the short term outlook. Any downward pressure from the sul-

phur market may not emerge due to the comfortable position smelter producers are in.

- South Korean smelter producers concentrated on shipments to China through Q1 but spot price ideas have firmed to above \$20/t fob for fresh business. LS Nikko had a scheduled turnaround in February, tightening export availability.
- China domestic acid prices may see downward pressure from the re-start of Two Lions at the end of February
- US Gulf acid prices are stable in the short term, but may see firming as demand improves through the spring season.
- Acid consumption in Turkey is set to grow through 2015, with the start up of Meta Nickel's nickel leaching project.
- Dundee Metals' Tsumeb smelter is due to be running by end 2015.
- **Outlook:** The sulphuric acid market outlook for 2015 is positive, based on a stable production outlook, with support from demand in both industrial and fertilizer markets. The market appears increasingly disconnected from sulphur prices.

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BAHRAIN

Bapco awards plant contract



The Bahrain Petroleum Company (Bapco) has selected WorleyParsons to provide process technology for its new sulphur plant, which is being installed as part of Bapco's Sitra refinery modernisation programme. The refinery is being expanded from its current 267,000 bbl/d to 360,000 bbl/d to process additional crude supplied from neighbouring Saudi Arabia, with the project being developed in five packages comprising off-sites and utilities; the crude unit and associated facilities; hydrocracker and associated units; residue conversion unit; and a 70km oil pipeline connecting Bahrain with Ras Tanura in Saudi Arabia.

WorleyParsons will produce engineering design packages (EDPs) for bulk acid gas removal, amine regeneration, sulphur recovery, tail gas treating and the sour water stripper units for the new Sulphur Plant 3. The company is the assigned contractor for the front-end engineering and design (FEED) phase of the modernisation programme.

IRAN

Sulphur recovery plant at Pars Phase 12 not yet operational

Phase 12 of the massive South Pars development project – one of the largest – is due to come on-stream by March 21st 2015, according to Petropars. However, the associated 750 t/d of sulphur recovery capacity is not yet completed. Phase 12 includes three main offshore drilling platforms, A, B and C, and a peripheral platform D. Some 41 wells have already been drilled, of which 31 are now productive or ready for gas production at three platforms, and the fourth platform with the remaining wells is due for installation and operations soon. Once full commissioning is completed, the gas output from the phase is due to be 81 million m³/d. Phases 15-18 are now part-completed, according to Petropars, and 1,000 t/d of sulphur production was brought on-stream over the past year.

UNITED ARAB EMIRATES

Gasco awards contracts for Integrated Gas Development Phase 3

Abu Dhabi Gas Industries Company (Gasco) has awarded lump sum turnkey contracts for the third phase of Abu Dhabi's Integrated Gas Development, which will lift associated sour gas output from ADNOC's offshore oil fields by a further 400 million ft³/d by 2017. Gasco is a 68% owned subsidiary of ADNOC. The Integrated Gas Development Expansion Project has three packages, covering expansion of the Das Island facilities (package 1), offshore pipeline (package 2) and onshore

pipeline and modifications to the Habshan gas processing plant (package 3). Package 1, worth \$490 million, was awarded to a consortium consisting of Tecnimont, and Archirodon of Greece, and includes a new fourth gas dehydration train, a new common dry gas compression aftercooler, and additional land reclamation. Package 2, worth \$410 million, was awarded to local firm the National Petroleum Construction Company (NPCC), and consists of the 117 km offshore segment of the new 42" IGD-E pipeline, which will run parallel to the existing pipeline. Spanish contractor Tecnicas Reunidas won the \$700 million contract for the Integrated Gas Development Expansion Project Package 3, which consists of the new 114 km onshore segment of the pipeline, and the associated receiving units at the Habshan 5 gas processing plant, which will also receive additional onshore sour gas from ADCO's North East Bab Development (NEB-III), and will lift the gas processed at Habshan 5 from 1 billion cfd to 1.75 billion cfd. The new sections include a new condensate pipeline, two new boilers for steam generation, and tie-ins with the existing complex.

Wintershall optimistic about Shuwaihat field

Wintershall has said that it is optimistic about prospects for the Shuwaihat sour gas and condensate field in the west of Abu Dhabi, 25 km from Ruwais, and that it hopes to produce a more definitive assessment of resources there in the next few weeks. Gas resources at the field are estimated to be between 28 and 85 bcm of sour gas. Wintershall is developing the

field in cooperation with OMV of Austria and ADNOC – the latter being the majority shareholder. The UAE's demand for gas is expected to rise from the 2013 figure of 79 bcm to as much as 179 bcm in 2020, and gas from every possible source is being tapped, including the major sour gas fields at Shah, under development by Occidental and Al Hosn Gas, and currently in start-up, and Bab, where ADNOC and Shell are in partnership, due for start-up in 2020.

SAUDI ARABIA

Bidding under way for Fadhili gas plant

Saudi Aramco has pre-qualified nine bidders to compete for the engineering, procurement and construction (EPC) contract for the Fadhili gas plant. The bidders include Saipem, Hyundai, GS Engineering & Construction, Tecnicas Reunidas, Tecnimont, JGC, Daelim Industrial and CTCl. The companies have until April 15th 2015 to submit their bids. Foster Wheeler, which undertook the front end engineering and design (FEED) study for the plant, will also provide project management services during the EPC phase.

Fadhili had an initial design capacity of 15.5 bcm/year and will cost around \$3 billion to build. It is due to begin operations in 2018, and Saudi Arabia has indicated that it is pressing ahead with gas developments in spite of the cancellation or postponement of some other major projects, such as the Ras Tanura refinery upgrade, due to falling oil prices. Fadhili will process sour gas from the Khursaniyah and Hasbah fields.

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KAZAKHSTAN

Oil production at Kashagan to resume in 2016

Oil production at the giant Kashagan oil field will be resumed in 2016, according to Kazakh oil minister Uzakbai Karabalin. He said in an interview with local media that 200km of pipe has been constructed to replace the existing lines, damaged by corrosion. Saipem was awarded the \$1.8 billion contract to lay the twin 95km replacement pipelines in early February. Saipem says that this work will be completed "by the end of 2016." Saipem is 43% owned by Italy's Eni, which has been one of the North Caspian Operating Company partners. Saipem also laid the original pipelines which are being replaced. The new pipelines have a corrosion resistant alloy layer to protect against the high hydrogen sulphide content of the gas.

Kashagan has so far cost \$50 billion and run a decade behind schedule. High pressure sour oil and gas from the field, 4.2 km below the sea bed of the Caspian Sea, began pumping in late 2013, but was suspended almost immediately due to leaks of sour gas. Reserves at Kashagan are estimated at 38 billion barrels of oil, with 10 billion barrels being recoverable, as well as over 1 trillion cubic meters of associated sour gas.

Funding arranged for modernisation of Pavlodar refinery

Funding is now in place for the \$5 billion modernisation of the Pavlodar oil refinery in Kazakhstan. Most of this will be provided via a club loan from Japanese banks, and \$1.2 billion will be provided in a 10-year loan from the Development Bank of Kazakhstan. Refinery operator KazMunaiGas says that the project will bring the refinery's output up to Euro-4 or Euro-5 standards, as well as increasing processing capacity from 5 million t/a of oil to 7 million t/a. The refinery, in the northwest of Kazakhstan close to the Russian border, mainly processes western Siberian oil.

The modernisation project is led by Rominserv, a subsidiary of Romania's Rompetrol Group, which is itself owned by KazMunaiGas. On-site works began in 2014 and are scheduled to be completed in 2017. According to Rompetrol, the modernisation project involves upgrading ten

of the plant's existing units – the crude distillation unit, vacuum distillation unit, kero hydrotreater, diesel hydrotreater unit, naphta hydrotreater, vacuum gas oil unit, catalytic cracking unit, catalytic reforming unit, as well as utilities and off-sites – and construction of several new installations, including an isomerisation unit, sulphur recovery unit, sour water stripper, amine regeneration unit, and diesel hydrotreating and dewaxing units. Sulphur output is expected to reach 440 t/d.

Pavlodar is the largest oil refinery in Kazakhstan and the most modern one in Central Asia producing petrol, diesel, jet engine fuel, residual fuel, gases, asphalt, and petroleum coke.

VENEZUELA

Sulphur output at El Palito to increase

Venezuelan state oil firm PDVSA says that processing of Lagoteco crude oil at its El Palito refinery increased by 26% in 2014. El Palito has a capacity of 135,000 bbl/d, but an expansion programme is to double that to 280,000 bbl/d, with completion expected next year. The existing refinery has a maintenance shutdown scheduled for later this year, during which time a sulphur recovery unit expansion is expected to be completed which will increase capacity to 250 t/d of sulphur.

MEXICO

Pemex puts refinery upgrades on hold

Mexico's state oil company Pemex has indicated that it intends to delay several major refinery upgrades to produce low sulphur fuel due to the fall in global oil prices. In a press statement, Pemex said: "the (budget) adjustment is of sufficient size that the execution of the major projects is being deferred". The company's board has approved cuts of \$4.2 billion dollars in expenditure.

The company had previously said it would add deep conversion coking units to three of its six domestic refineries, at Salina Cruz, Tula, and Salamanca as part of a \$20 billion investment package that also included a \$2.8 billion clean fuels initiative. This would upgrade five domestic refineries to be able to produce ultra-low sulphur diesel. Pemex is facing competition from private oil companies following an overhaul of energy legislation last year. It is also renegotiating service contracts in an effort to cut costs.

UNITED KINGDOM

Total to cut output at Lindsey

French oil major Total says it will halve capacity at its 200,000 bbl/d Lindsey refinery on the Humber estuary as part of an overhaul of downstream activities intended to address overcapacity in the European refining sector. Lindsey is one of six refineries still operational in the UK and the one regarded as most likely to face closure. The British refining industry is faced with older, smaller, less complex refineries designed to process sweet North Sea crude, which now commands a substantial premium for its low sulphur content. Furthermore, like many European countries produces too much unwanted gasoline and not enough diesel or jet fuel, while it faces competition from new Middle Eastern and Asian mega-refineries and US refineries which have invested heavily to process higher sulphur feeds. Lindsey has a weighted average Nelson Complexity of just over 6, the lowest in the UK, and Total hopes to use savings made from the output reductions to invest in new equipment at the site.

Other UK refineries have closed in recent years, such as Milford Haven last year, Coryton in 2012 and Teesside in 2009, while Stanlow has cut output by one third, and Grangemouth in Scotland had to be rescued by the government to avoid job losses becoming an issue in the Scottish independence referendum.

BRAZIL

OCP buys 10% stake in Brazilian distributor

OCP, Morocco has completed the purchase of a 10% stake in the Brazilian company Fertilizantes Heringer, providing it with a long-term supply agreement for phosphate products. The deal was concluded at a reported price of approximately \$55 million, after the Moroccan and Brazilian regulatory authorities gave their approval. The agreement follows an earlier deal concluded between supplier Mosaic and the Brazilian distributor ADM and marks further consolidation in the market where OCP, Mosaic and Yara compete for sales. Heringer is one of the largest distributors in Brazil. In 2014, OCP supplied Brazil with an estimated 1.7 million tonnes of DAP, MAP, TSP and NP fertilizers, down from 2.2 million tonnes in 2013. OCP is understood to have lost market share because of an influx of Chinese MAP imports.



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AUSTRALIA

Ravensthorpe to operate at 70% capacity

First Quantum Minerals says that its Ravensthorpe high pressure acid leaching (HPAL) nickel plant in Western Australia will run at 70% of full capacity while the company conducts repairs following an acid spill in December. The company says that it expects output for 2015 will be between 24-30,000 tonnes out of a total production capacity of 38,000 t/a. In December an atmospheric leach tank at the site, 550km southeast of Perth, containing sulphuric acid leach solution ruptured, causing acidic slurry to spill into a bunded containment area in the plant.

First Quantum says that it has

bypassed and demolished the damaged leach tank, enabling full production from one of the two production circuits, but could not give a date on when the site might be restored to full capacity. Investigations into the incident are on-going, according to the company, and reconstruction work will follow.

The shutdown at Ravensthorpe has actually been a relief for nickel markets, which have seen oversupply and near record inventories of 400,000 tonnes on the London Metal Exchange, with prices dropping by 25% over the course of 2014.

Rare earths leaching project passes approvals stage

Alkane Resources says that the New South Wales Planning Assessment Commission (PAC) has completed its review of the Dubbo Zirconia project and has recommended that the project can be approved subject to certain conditions. The key areas considered by PAC related to air quality, water impacts, transport, long term landform and land use. Other issues considered included impacts on Taronga Western Plains Zoo, the local pink-tailed worm lizard and Fossil Hill, hazards and risk (including radiation) and loss of agricultural land for biodiversity offsets. Alkane says that it regards this as a "major milestone in the approvals process" and that it is "optimistic" that consent to the project will be received within the budgeted timeframe.

The A\$1.0 billion Dubbo Zirconia project is based upon large in-ground resources of zirconium, hafnium, niobium, tantalum, yttrium and rare earth elements in demand for batteries, catalysts, alloys and other uses. It is expected to process up to 1.0 million t/a of ore in order to extract the minerals, and will include a 1,180 t/d sulphur burning sulphuric acid plant (100% H₂SO₄ basis). The Front End Engineering and Design contract has been awarded to Hatch Pty Ltd. Following government and finance approvals, early works and long lead time items ordering are scheduled to begin in Q2-Q3 2015, with construction of the project is expected to start in Q3 2015, for completion in early 2017.

NEW ZEALAND

Permit refused for offshore phosphate mining

New Zealand's Environmental Protection Agency (EPA) has refused Chatham Rock Phosphate's application to extract phosphorite from offshore seabed waters up to 450m deep and 450 km east of South Island. In a press statement, the EPA cited the "destructive effects of the extraction process", arguing that they "could not be mitigated by any set of conditions or adaptive management regime that might be reasonably imposed."

The company had been seeking a major extension to its current 820km² permit area, increasing it to 5,200km², and allowing Chatham to produce up to 1.5 million t/a of phosphate rock from the seabed. Chatham Rock Phosphate's managing director Chris Castle said: "To say we are bitterly disappointed is an understatement. We are aghast". The company's shares dropped 92% on the New Zealand stock exchange on the news.

CANADA

Copper North revises leaching plan

Copper North Mining Corp. says that it has revised its processing plan for leaching and recovery of copper, gold and silver at Carmacks in Canada's northern Yukon Territory. A test programme at the end of last year indicated that the oxide mineral resources at the site have very favourable

leach dynamics and that the optimal leach process is an agitated tank leach for both copper and the gold and silver, eliminating the heap leach pads and allowing operation during winter. The agitated tank leach will be a contained system with a smaller footprint and according to the company will provide an opportunity for a significant potential capital cost reduction down to about \$150 million. Processing will move from crushing and grinding to an agitated tank for leaching of copper oxides with weak sulphuric acid, and the leach solution will then be pumped to the solvent extraction electrowinning (SX/EW) plant for recovery of cathode copper.

Start-up for carbon and SO₂ capture project

Operator SaskPower says that its \$1.4 billion carbon capture and storage project is up and running at Boundary Dam, Saskatchewan. The CCS project is based around a retrofitted coal-fired power plant, and is seen as one of the world's first commercial scale operations of its kind, providing power for over 100,000 homes and businesses and capturing 90% of the CO₂ emissions from the plant; about 1.0 million t/a, and pumping it to underground storage. Some of the carbon dioxide is liquefied and will be sold on to oil companies for enhanced oil recovery projects. SaskPower has a 10-year contract with Calgary-based Cenovus Energy Inc. to buy the captured gas. The power plant also captures 100% of the sulphur dioxide generated in burning the coal, around 11,000 t/a, which is converted to 17,000 t/a (100% basis) of sulphuric acid intended for sale to industrial consumers.

Arianne in cost-cutting exercise

Arianne Phosphate Inc has been trying to find ways of reducing the \$1.2 billion cost of its proposed phosphate project at Lac-à-Paul in Quebec Province, ahead of financing talks with its banking syndicate. The company is aiming to produce 3 million t/a of phosphate concentrate from the phosphate deposit, which lies 200 km northeast of Saguenay-Lac-St-Jean, and which has estimated reserves of 500 million tonnes of high grade phosphate rock. Chief Operating Officer Jean-Sébastien David says that the company can save 5% by moving the mining area further west to take advantage of shallower overburden, and by fuelling its fleet of trucks on LNG

rather than diesel. The company is also in negotiations with Hydro Quebec over its electricity costs.

The concentrate will be trucked to a new deep water terminal at Saint-Fulgence, on the Saguenay River, and then loaded into bulk carriers. Arianne will focus on the North America and European markets, competing against Morocco and Russia.

Arianne also begins public consultations next week when it files its environmental and benefits statement with the Bureau d'audiences publiques sur l'environnement (BAPE). It hopes to get the key ministerial decree this autumn, start construction in 2016 and hit production in 2017-2018.

RUSSIA

In-situ uranium leach to begin next year

JSC Khiagda has confirmed that it expects to start commercial production of uranium from its Khiagdinskoye deposit from the end of 2016. JSC Khiagda is part of Atomredmetzoloto (ARMZ), the uranium mining subsidiary of Russian state nuclear corporation Rosatom. The company's operations are at Vitimsky in Buryati, north of Mongo-

lia. The pilot plant, producing 440 tU, began operation in 2010 and reached full capacity in 2013, and the new commercial scale in situ leach is targeted to begin operation in 2016, achieving full production of 1,000 tU/a in 2018. The company says that it will be the only in situ leach operation in the world which operates in a permafrost zone. The main uranium mineralisation is a phosphate, requiring oxidant addition to the acid solution. Work has been completed on the construction of the main production facility and the sulphuric acid plant, which has a design capacity of up to 110,000 t/a.

BRAZIL

MBAC considering mothballing Itafós

MBAC Fertilizer is evaluating whether to suspend operations at its Itafós Arraias phosphate complex in Brazil and putting the site into care and maintenance mode following a strategic review to help the company find a "definitive solution" to its current working capital constraints and liquidity requirements. Toronto-based MBAC owns and operates the Itafós Arraias SSP operations, which consist of an integrated fertilizer

production facility comprising a phosphate mine, a mill, a beneficiation plant, a sulphuric acid plant, an SSP plant and a granulation plant and related infrastructure. MBAC says that its options "include, but are not limited to, securing a strategic partner, the sale of the company or its assets as well as other potential value-maximizing transactions." Itafós, in Tocantins state, has been operating at minimum production in an effort to conserve working capital given during the low season for fertilizer sales in Brazil.

TAJIKISTAN

Loan for construction of acid plant

China has made a soft loan of \$88 million to Tajikistan for the construction of several plants as part of the modernisation of the Tajikistan Aluminium Company (Talco), the state aluminium producer. Aluminium is Tajikistan's key export, worth up to 45% of GDP and 90% of foreign currency earnings, and Sharif Rakhimzoda, Tajikistan's Minister of Economic Development and Trade, says that the country plans to increase its output from the 121,000 tonnes produced last year to 190,000 t/a as part of the



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modernisation plan. A deal with Norway's Norsk Hydro will improve offtake. The three new plants that form part of the modernisation programme include a 12,000 t/a crolyite facility, an 18,000 t/a aluminium fluoride plant, and a 100,000 t/a sulphuric acid plant, all to be built at Yavan, 70km from the capital Dushanbe.

VIETNAM

Vietnam approves new SSP plant

According to press reports, the Vietnamese government has approved plans for Borha Industries of India to construct a 400,000 mt/year SSP plant in Nghe An Province, in Vietnam. The plant will import phosphate rock from North Africa, understood to be from Morocco, and supply both the local market as well as nearby SE Asian markets. It will be obliged to supply 30% of the production to the local market but is not able to use indigenous apatite, hence the need for imports.

UNITED STATES

Uranium recovery from phosphoric acid

Australian company PhosEnergy says that it has had positive results from its joint venture with demonstration plant, built in partnership with uranium giant Cameco, designed to extract uranium from the phosphoric acid streams of phosphate fertilizer production plants. The small demonstration plant has been running for several months at a US phosphate fertilizer plant, and has demonstrated uranium recoveries in excess of 90%, suggesting that 400,000 lbs/year (182 metric tonnes/year) of uranium could be produced from that site at a cash cost of approximately \$20 per pound, with potential greater economies of scale obtainable from larger plants, extremely low compared to other uranium production costs.

The PhosEnergy process was originally developed by the Australian Nuclear Science and Technology Organisation, and has moved forward with major funding from Cameco. PhosEnergy estimates that there is the potential for the production of 6 million lbs/year from existing US phosphate plants, and up to 20 million lbs/year worldwide, equivalent to about 15% of global mine production. US and many other sedimentary phosphates contain uranium in concentrations of up to 100ppm.

The joint venture partners will now decide whether to enter a full commercial feasibility process.

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Delays for Paris Hills phosphate project

Stonagate Agricom says that it has temporarily suspended permitting activities at its Paris Hills phosphate project in Idaho, blaming financial constraints. A team of third party consultants has also been retained in order to finalise the groundwater model for permitting applications; there is a wide range of estimates of expected groundwater flow rates into the planned underground mining area. As a result, the company says that it will need to undertake further testing and analysis, which may include additional engineering work, which Stonagate's current financial position prevents it from undertaking at present. The company says that as a result, it is no longer expecting to submit the groundwater model and report in 1Q 2015 as planned. It is reviewing its options with respect to its next steps, and is exploring alternatives to raise additional funds by the beginning of the second quarter of 2015 to cover working capital and fund continuing work on the project.

The Paris Hills deposit is located in Bear Lake County, Idaho and has proven reserves of 7.96 million tonnes and probable reserves of 8.75 million tonnes. Stonagate Agricom plans to produce 904,000 t/a of marketable phosphate concentrate over an estimated mine life of 19 years.

JORDAN

JPMC concludes acid agreement

Jordan Phosphates Mines Company (JPMC) has signed a memorandum of understanding with Gujarat Narmada Valley Fertilisers & Chemicals (GNFC) for the construction of a 300,000 t/a (P_2O_5 basis) phosphoric acid plant in India, for which JPMC will exclusively supply 1.0 million t/a of phosphate rock. This is the first time the company has entered into a partnership venture to build a phosphoric acid plant in India. The new plant will supplement the present JIFCO joint venture with IFFCO, whose 500,000 t/a P_2O_5 phosphoric acid plant at Aqaba is now running at full capacity.

To meet the extra demand, JPMC will ramp up its production of phosphate rock to 15 million t/a by 2020. This compares with a record rock output of around 8 million tonnes in 2014 and planned production of 9 million tonnes this year. JPMC will also supply two JV partners in Indonesia, Kaltim and Pusri, with an additional 1.5 million t/a phosphate rock when their

phosphoric acid plants are complete. JPMC has also indicated its intention to revamp its DAP plant at Aqaba, raising capacity from 800,000 t/a to 1 million t/a.

The Jordan Industrial Ports Company has meanwhile signed an agreement with two Spanish companies to expand the industrial pier at Aqaba. The pier is owned by JPMC and Arab Potash Company. The partners seek to double the pier's handling capacity to enable it to receive vessels of up to 100,000 dwt. The work is expected to take two years to complete.

SAUDI ARABIA

Waad Al Shamal on track for 2016

Ma'aden, the Saudi Arabian mining company, has said that its \$7 billion Waad Al Shamal joint-venture phosphate project is now almost 40% complete. The downstream complex will include an ammonia plant, construction of which was 38% complete as at 31 December, and a sulphuric acid plant, which was 36% complete. Work on the DAP facility was 18% complete, while the phosphoric acid unit was 24% complete. An ore beneficiation plant is also under construction. Ma'aden holds a 60% stake in the project partnership, together with Mosaic (25%) and Sabic (15%). The Waad Al Shamal project is scheduled to be commissioned in 2016, and while some industry analysts questioned whether this target date was an achievable one, the latest progress report seems to indicate that the project is on track.

INDIA

GSFC begins work on new DAP plant

State-run fertiliser major Gujarat State Fertilizers and Chemicals Ltd (GSFC) has begun work on a new diammonium phosphate plant at its Sikka facility in Jamnagar on Saturday. Chairman and managing director S K Nanda laid the foundation stone for the new plant on January 26th as part of the \$140 million expansion project. GSFC already operates three DAP trains with a total capacity of 722,000 t/a at the site, and the new 544,000 t/a unit will take this to 1.27 million t/a. The company is also building ancillary facilities, including a bagging plant, silos and storage tanks for ammonia & phosphoric acid, and there is also a proposal to undertake dredging operations at the site to allow vessels with a larger capacity to berth at the company's jetty. ■

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People

Sulphur's sister title *Fertilizer International* has a new Editor. **Mark Evans** (left, in picture), the long-standing editor of F.I., is retiring at the age of 65, after a record breaking 27 years as editor of the title, and fully 30 years of journalism within the fertilizer industry, for British Sulphur Publishing, then CRU, who bought British Sulphur in 1994, and of course since January 2007 as a partner in BCInsight Ltd.

Mark's able replacement is **Simon Inglethorpe** (right), who has joined BCInsight Ltd. from Haymarket Media Group, where he was Business Editor at *The ENDS Report*, the UK's leading publication for environmental professionals. A graduate in geology from Kings College London, Simon spent a long career as a mineral resource scientist with the British Geological Survey, a role that involved extensive international project work in Africa, Asia, Central America and Europe.

Simon says that he looks forward to taking the helm of *Fertilizer International*. "It is a great pleasure to be taking over from the estimable Mark Evans," he said. "My background in the earth sciences means I do recognise the underlying importance of mineral resources to the sector, particularly phosphates, potash and mineral processing – as well as the rise of shale gas. But there is much more to the fertilizer industry than resources and my experience in business journalism should serve me well as I immerse myself in technology, products, markets and finance."



"Mark forged strong bonds with the readership of *Fertilizer International* and I intend to do the same. I am particularly looking forward to meeting as many of you as possible over the next few months. Ensuring *Fertilizer International* remains the highly-relevant and essential read that subscribers have come to expect under Mark's editorship is a priority. And meeting many of you in person is one way I can help guarantee that *Fertilizer International* continues to be a well-informed, thoroughly-researched publication full of valuable commercial insights and business intelligence."

Following the completion of its takeover of Foster Wheeler, AMEC plc has announced that the company's name will now be changed to Amec Foster Wheeler plc, with its global headquarters in London, UK. A number of changes to the board of directors become effective as of the

takeover, in mid-January. **Stephanie Newby** has been appointed to the Board of Directors as well as the company's Audit and Ethics Committees. **J. Kent Masters** has also been appointed to the Board of Directors of AMEC following the termination of his employment with Foster Wheeler.

Samir Brikho, Chief Executive of Amec Foster Wheeler, said: "In creating our new company we are building on the proud heritage, skills and customer relationships of two already successful and highly respected businesses. "Amec Foster Wheeler operates in more than 50 countries worldwide, in strong end markets and throughout the value chain. With our strong management team and talented workforce, we will not only serve our customers better, but can also offer our highly skilled employees even better career opportunities. I am proud to be leading the combined business into a new phase of growth."

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23-25

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23-27

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20-22

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JUNE

5-6

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Sour gas update

Where and how much sulphur will be produced from sour gas, and how will it find a market in a time of sulphur surplus?

Right: Al Hosn Gas's new Shah sour gas plant.

Virtually all natural gas contains some hydrogen sulphide, as well as carbon dioxide – the presence of the two gases tend to define what is an 'acid' gas. Sour gas is natural gas containing a 'significant' proportion of hydrogen sulphide, although definitions of what significant actually means can vary considerably. Pipeline sales gas specifications are generally based around customer odour and health and safety requirements, and so any gas with a higher proportion of H₂S than 3-4ppm is regarded as 'sour'. Other specifications are based around corrosion limits, and here US federal specifications define gas >16ppm H₂S as sour, while the state of Texas pushes that definition up to 100ppm (the point at which human olfactory nerves are overwhelmed and are no longer able to detect the presence of H₂S). The 'sour service' equipment definition is based on sulphide stress cracking and is related to the partial pressure of H₂S, but tends to equate to about 50-100ppm. However, natural gas producers have a much higher definition, based on how the gas will be processed to sweeten it. Here sour gas is usually taken to be greater than 1% H₂S (ie 10,000 ppm).

The H₂S can come from various sources, including bacterial reduction of sulphate sources such as dissolution of calcium sulphite, seawater ingress, or oxidation of pyrites. It can also come from thermal decomposition of sulphides in oil and kerogens. But in the sourest gases it tends to come from the reaction of

sulphate minerals and hydrocarbons at temperatures greater than 120-140°C.

Sour gas extraction began in the 1920s in Canada, passing gas through soda ash to remove H₂S via the 'Seebord' process. However, the gas was then released to atmosphere, causing health and other issues, and producers soon switch to solvent-based extraction using monoethylamine (MEA). Shell's Jumping Pound plant in Alberta was regarded as the first large scale sour gas plant which used a Claus plant, with Total at Lacq in France switching from MEA to diethylamine (DEA) in 1957. Since then, production has fallen in North America and Europe, but the expertise gained there has proved invaluable in new projects more recently in Asia and the Middle East.

Imperatives for sour gas production

This tends to vary on a regional basis, according to the availability of sweet alternatives. In North America, which pioneered sour gas extraction, mature and declining fields, especially in western Canada, have faced competition from cheaper US shale gas, which has tended to be much sweeter, avoiding the additional costs of sour gas production associated with higher specification equipment and health and safety concerns, and hence production here is in long term decline.

In the Middle East, conversely, rapidly rising gas demand for power generation amidst rising populations are driving

increased gas consumption, projected to double in countries like Saudi Arabia or the UAE over the next few years. However, much domestic gas production in the region is from associated gas, and hence constrained by OPEC oil quotas, and non-associated gas fields are mainly sour in Saudi Arabia, Abu Dhabi and Oman, and this has driven increased use of sour gas.

In Central Asia, the drivers have so far been mainly oil and condensate production, where sour gas extraction is mainly from associated gas fields, and hence the sour gas processing is in many ways incidental to the oil production that is driving it.

Finally, in China, gas demand far outstrips supply, and so China is turning to unconventional gas of all types to try and meet the gap, including shale gas, tight gas, and coalbed methane, but also sour gas production. Additionally, sulphur extracted from sour gas assists with China's growing domestic sulphur deficit for fertilizer production.

The upshot of this is that the Middle East, China and Central Asia are all seeing major sour gas project developments, which are detailed below.

Middle East

Much of the Arabian Gulf has offshore sour or sour-ish gas reserves, in areas claimed by Saudi Arabia, Iran, Qatar and Abu Dhabi, but the gas fields extend under most of the western UAE and across into Oman in the east. Ample supply of gas from sweeter

sources meant that most of these fields were left untapped for many years but pressure of demand from rapidly expanding economies in Saudi Arabia and the UAE is now starting to lead to widespread exploitation of these sour gas resources. Foremost among the developers of these projects has been the UAE Emirate of Abu Dhabi.

Habshan

Habshan is the home of Abu Dhabi's Integrated Gas Development, which processes offshore gas from Umm Shaif/Das Island, as well as the Bab onshore oil field and non-associated sour gas from the Habshan fields. The Habshan V processing plant started up in 2013, and is currently processing 1 billion cubic feet/day (bcf/d). Gasco has recently awarded contracts for phase three of the Integrated Gas Development, and the aim is for gas processed at Habshan to rise to 1.75 bcf/d by 2017. At capacity, sulphur recovery is expected to reach 5,200 t/d (1.8 million t/a).

Shah

Shah is a joint venture between Adnoc (60%) and Occidental (40%) for an onshore sour gas field 210km southwest of Abu Dhabi city, deep in the desert. The reservoir at Shah is approximately 20-30 tcf, with an H₂S content of 23% (and 10% CO₂). Shah is targeting production of 540 million scf/d (5.2 bcm/year) of sales gas, requiring it to process almost 1 bcf/d of gas. At capacity, sulphur output is estimated at 9,200 t/d (3.1 million t/a). The \$10 billion project is now producing gas, but the indications are that ramp-up may take longer than initially planned.

Bab

Bab has been conceived as a mirror of the Shah project. This time it is a joint venture between Adnoc (60%) and Shell (40%), and the onshore field is 150km southwest of Abu Dhabi city. The CO₂ content of Bab is higher (at 15%), and the H₂S content varies from 15-50%. The gas here is also rich in condensate, and it has been hoped that sales of this will offset the high costs of production, with the carbon dioxide possibly being used for enhanced oil recovery in the nearby oil fields. Target production is again just over 500 million scf/d of sales gas and development costs are around \$10 billion. It is as yet unclear what effect the falling oil price will have on the economics of this project, and whether this

will lead to the projected 2020 on-stream date slipping. Sulphur recovery should be of a similar order to the Shah project, unless some of the H₂S is also used for enhanced oil recovery. Pre-front-end engineering and design (FEED) work was completed at the end of last year, with FEED expected to start next year and contracts expected to be awarded soon.

Hail

Hail is another potential sour gas project in Abu Dhabi, currently at the feasibility study phase. It is offshore of Abu Dhabi city, about 100km to the west. Target production is 400-600 million scf/d of sales gas. The H₂S content of the offshore field is 15%, which is lower than the onshore fields, but the offshore location, albeit in shallow water, will increase production costs.

South Pars

South Pars is the Iranian side of the massive North Field which sits astride the Arabian Gulf; at 1,500 tcf the largest gas field in the world. In spite of delays caused by international sanctions, Iran has been steadily progressing through the 29 project phases, all of which involve gas and condensate recovery and many of which involve sulphur production. Recently Phase 12 was announced as being up and running, with several other phases expected for completion this year. The H₂S content of the South Pars field is only 0.5-1.0%, but the large volumes of gas being processed mean that sulphur production could still be considerable. In 2012, this was estimated at 1.6 million t/a, with the figure for 2017 projected to be 2.0 million t/a.

North Field

On the Qatari side of the field, gas is brought ashore to the massive complex at Ras Laffan, on the northern tip of the Qatar peninsula. Gas is processed for LNG export, export via the Dolphin pipeline to the UAE, and for use in the massive Pearl and Oryx gas to liquids plants at the site. Rasgas and Qatargas between them export 77 million t/a of LNG, making Qatar the largest LNG producer in the world. Sulphur recovered from all of these facilities is sent to the Common Sulphur Facility where it is formed and exported. Sulphur recovery and forming capacity is approximately 3.5 million t/a in total, with actual production in 2012 being 1.8 million t/a, rising to 2.2 million t/a by 2018 as more gas is processed. The new Barzan

gas project, for example, designed to provide gas for domestic use in Qatar, is now in start-up, with Train 1 commissioned in late 2014, and train 2 due to start up later this year. Total sales gas output at capacity will be 1.4 bcf/d, raising sulphur output by a couple of hundred thousand tonnes.

Karan

In neighbouring Saudi Arabia, Karan is the first non-associated sour gas development. The gas is produced offshore and pumped for processing onshore at the existing Kursaniyah gas plant, which has been expanded to handle extra gas from Karan. Karan began production in 2012, and has a design capacity of 1.8 billion scf/d. H₂S content at the field is about 2%, and sulphur production at capacity is 900 t/d (300,000 t/a).

Wasit

The Wasit gas plant was built to process gas from the offshore Arabiyah and Hasbah sour non-associated gas fields. Total gas processing capacity is 2.6 billion scf/d to produce 1.75 billion scf/d of sales gas. H₂S content averages 4-8%, and the sulphur recovery section includes four SRUs with a total capacity of 2,400 t/d (800,000 t/a). Start-up is due to begin this year, with full production by 2016.

Fadhili

Fadhili is another sour gas processing plant, designed to process additional gas from the Kursaniyah and Hasbah sour gas fields. Target production has been increased to 2.5 billion scf/d, with start-up scheduled for late 2018. The plant is currently in its design phase.

Oman

Oman has two major sour gas processing projects. The first, Yibal Khuff Sudair, is operated by Petroleum Development Oman, a company majority owned (60%) by the Government of Oman, with additional participation from Shell (34%), Total (4%) and Partex (2%). Khuff is a deep oil and associated sour gas deposit beneath an existing field, with an H₂S content for the gas of 3%. An 85,000 t/a sulphur recovery plant is due for completion this year, and commissioning of the gas project is expected in 2019.

The other project is the Rabab Harweel Integrated Project, a joint venture between PDO and Petrofac. Again gas is 2-3% H₂S. In the first phase, associated gas will be

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ISSUE 357
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re-injected into the well, but in the second phase the non-associated Amin formation, a 50 tcf deposit, is due to be tapped.

Central Asia

The area of sour gas exploitation in Central Asia is mostly around the Caspian Sea region, in Russia to its west, Kazakhstan to its north and east, and the zone of sour oil and gas reserves extends further south east into Turkmenistan and Uzbekistan. Onshore deposits in Russia and Kazakhstan are the longest standing and most mature, with discoveries going back to the 1960s and exploitation to the 1980s, while new exploration has focused on offshore reserves in the North Caspian and onshore reserves into Turkmenistan.

Asktrkhan and Orenburg

Russia already has two major sour gas processing plants. The first is at Astrakhan on the west side of the Caspian Sea., which processes highly sour (up to 25%) gas from the Krasnoyarsky gas/condensate field, operated by Gazprom. Sulphur output here averages around 4.8 million t/a, and represents most of Gazprom's output, mainly destined for export. The second is at Orenburg, a Soviet era gas processing plant which also processes production from across the Kazakhstan border at Karachaganak, which is run by KPO, a consortium consisting of ChevronTexaco, Agip, BG, Lukoil and KazMunaiGaz. Total sales gas production at Orenburg is 1.5 bcf/d, and H₂S content averages 2-6%. Sulphur production of 1.1 million t/d is mainly for domestic use within Russia. Sour gas is also reinjected, and the Karachaganak Phase 3 project, currently scheduled to be on-stream in 2022, will mostly involve sour gas reinjection.

Tengiz

Tengiz in Kazakhstan, on the northeast side of the Caspian Sea, processes associated gas from oil production at the Tengiz field, both offshore and onshore. The operating company here is the TengizChevroil (TCO) joint venture, in which Chevron has a 50% stake, ExxonMobil 25%, KazMunaiGaz 20%, and Russia's Lukoil 5%. The H₂S content of the gas has varied considerably, and some sour gas is reinjected to boost oil production, but currently sulphur output is running at about 1.8 million t/a. TCO had produced large stockpiles of sulphur but these have been mostly drawn down

now. An expansion project will life oil output from 600,000 bbl/d to 830,000 bbl/d by 2018 and may involve some extra sulphur production.

Kashagan

The greatest variable among the Central Asian sour gas processing plants is Kashagan. Kashagan is a very large offshore oilfield with a very deep (4.2km) high pressure reservoir and very sour (17%) H₂S associated gas. Difficult conditions such as winter ice and the high partial pressure of H₂S have posed problems for the North Caspian Operating Company (NCOC) which is developing the project, a consortium of ExxonMobil, Shell, Total, KazMunaiGaz, Inpex, CNPC (which bought out Chevron's stake), and led by Italy's Eni. The massively expensive (\$46 billion at last estimate) and long-delayed project finally came on-stream in late 2013, but sour gas leaks and pipe corrosion have forced the replacement of the entire double pipe system which brings sour gas from the artificial island where the wells are to the onshore processing plant, and no production is now expected until 2016 at the earliest. Sulphur production was to have been 1.1 million t/a at capacity.

South Yolotan

Meanwhile, in Turkmenistan, there is another large reservoir (700 tcf according to some estimates, making it the world's sec-

ond largest gas field, though not all is recoverable) at South Yolotan, and other, smaller nearby fields like Dauletabad and Shatlyk. The Galkynsh processing plant came on-stream in September 2013 and is expected to be recovering 1.8 million t/a from gas with an H₂S content of 6% once full capacity is achieved some time in 2016. There are also other sour gas fields across the border in Uzbekistan which are as yet untapped, and potential for further expansion.

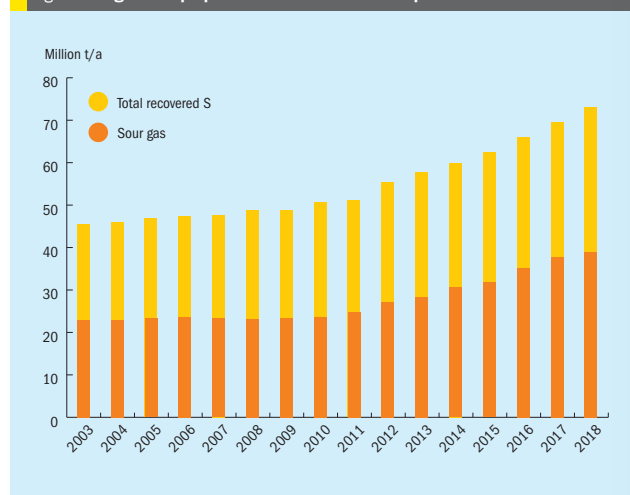
China

China's sour gas fields are mostly in the southern province of Sichuan. Most of the fields were discovered in the late 1990s, and exploration and discovery continued throughout the 2000s, with China's rapidly rising demand for natural gas driving large scale exploitation in the past few years. The move is likely to change China's sulphur picture dramatically.

Puguang

China's first sour gas field to be exploited is Puguang, where there are 410bcm of reserves with an H₂S content of around 15-17%. The Puguang sour gas processing plant, operated solely by Sinopec, became operational in 2012, and has ramped up towards its maximum capacity of 12 bcm/year, which was achieved last year. Sulphur output at Puguang is now over 3 million t/a.

Fig 1: Sour gas as a proportion of total recovered sulphur



Yuanba

Another field operated only by Sinopec, Yuanba is of similar size to Puguang – estimates say 210 bcm – but extremely deep; the field is over 7.5 km down in places, and averages 6.7km deep. The H₂S content at Yuanba is lower than Puguang, at 5%, but the high pressure makes the demands upon the equipment just as severe. There are two phases to the Yuanba development, each of which will process 1.7 bcm/year of gas. The first phase began operations at the very end of 2014, and the second phase is earmarked to begin operations in late 2015 or early 2016. Total sulphur output from both phases is expected to be 300,000 t/a.

Chuangdongbei

Chuangdongbei is one of the few sour gas projects in China with foreign participation, in this case Chevron, who have a 49% stake in the project against China National Petroleum Corp's 51%. Total proved reserves at Chuangdongbei are put at 175bcm, with H₂S content between 7-11%. Target production is 740 million scf/d (7.4 bcm/year) by 2018, with gas from the Luojiazhai, Gunziping, Tienshanpo and Dukhouhe-Quikibe fields all being fed to central processing plants. The first of these, processing 250 million scf/d, has been delayed but is due to come on-stream this year. At capacity, sulphur output from the entire project is expected to be 1.5 million t/a.

Total recovered sulphur

Figure 1 shows total recovered sulphur production, and the proportion derived from sour gas. As can be seen, the total recovered from sour gas remained relatively steady during the 2000s, with falling production in Canada being balanced by increases in the Middle East. However, this decade the production from sour gas can be seen to steadily ramp up, taking the total elemental sulphur production with it. From 2014-2018, an extra 10-12 million t/a of sulphur is likely to come from the sour gas projects that we have described here.

The accompanying article in this issue on global sulphur markets discusses the ramifications of this in more detail, but in short, the projects describe include an extra 3.3 million t/a of recovered sulphur in Central Asia, an extra 4.8 million t/a in China, and 8.0 million t/a in the Middle

East, with perhaps another 500,000 t/a of reductions from processing in Canada over the next five years. This is likely to push sulphur markets overall into surplus. The question is then will all of this sulphur enter the market, or will some be forced to be stored. This depends very much on the different regional conditions.

In China, there is a sulphur deficit forecast for the foreseeable future in spite of new sour gas production, and most sulphur should be capable of being absorbed by local demand. Sulphur production in Central Asia, conversely, is logistically con-

strained as regards export to e.g. China or reaching major ocean-going ports. There is some local demand for uranium leaching and fertilizer production, but some of the extra sulphur may end up having to be stored. In the Middle East, there will be a large sulphur surplus in spite of some major phosphate projects in Saudi Arabia, but the region is better placed for sulphur exports to China, India and North Africa than Central Asia. Here the balance of storage and export will depend on the economics of production and prevailing market prices. ■

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Sulphur World Symposium 2015

The Sulphur Institute (TSI) will hold its 2015 Sulphur World Symposium at the Hotel, Barcelona, Spain, from April 20th – 22nd

Based in Washington, DC, The Sulphur Institute (TSI) is the main industry body for the sulphur industry, representing all stakeholders involved in producing, buying, selling, handling, transporting or adding value to sulphur on a global basis. TSI's annual Sulphur World Symposium will this year be held in Beijing, and as usual will bring together sulphur and sulphuric acid industry specialists from around the globe.

Programme

The following are abstracts of those presentations to be made during the Symposium received at time of publication:

The European refinery industry: at a crossroads

Marina Ivanova, Douglas Westwood

Since 2009, 22 European refineries have closed down or converted to storage depots. It is widely expected that this trend will continue, amid historically low demand and a supply glut that has resulted from cheaper, more energy-efficient imports from the US, Russia and the Middle East. European refineries are also facing pressure from inside the European Union to drastically reduce sulphur content in a bid to reduce greenhouse gas emissions. This has compelled the surviving refiners to embark on a very expensive upgrade programs in order to both meet stringent requirements and compete with technologically advanced refiners outside Europe. The presentation will first investigate the market dynamics that are impacting refineries in Europe, and secondly determine how refineries can remain competitive in these fast-changing times, as well as what the impact will be on the sulphur market.

Dynamics of the sulphur supply chain

Craig Jorgenson, The Sulphur Institute

With growing speculation about increased supply in the sulphur market and evolving changes in regulatory requirements, industry professionals will face many challenges managing the sulphur supply chain in the years to come. To better address these challenges, industry leaders will benefit from a thorough understanding of rules and regulations that influence transportation and safe handling of different forms of sulphur. This presentation will summarize recent and proposed changes in regional and international regulatory requirements that can affect the sulphur industry. Examples of environmental and safety incidents that occurred during loading, unloading, transportation, and handling of sulphur over this past year will be reviewed. This presentation also will include discussion of alternatives for a growing sulphur supply under evaluation by various organisations.

Global energy outlook – the peril of forecasting

Francis Osborne, Argus Media Limited

The recent collapse in oil prices once again demonstrates that forecasting can be a mug's game. Risks and uncertainties always seem to lurk unseen around the corner. Currently there is a consensus view that sulphur will be a surplus product in the coming years, but this depends on the energy road map unfolding as expected.

This presentation will describe this road map, outlining specifics such as how the increase in energy demand across the regions will be met, and from where. It will also consider the threats to this consensus posed by three main uncertainties – economy, environment and price:

- Economy – more than five years after the 2008/9 recession we are still talking in terms of recovery. Is trend growth now lower than in the past and what's going wrong in China? When will the next downturn be?
- Environment – on current trends there is no hope of limiting an increase in global temperatures to less than the 2C currently planned. Are governments getting ready to accelerate efforts to achieve this goal?
- Price – in November OPEC did the unthinkable and shifted the pricing paradigm. Are low crude prices the new norm and how do they change the energy landscape in the future?

Global sulphur & sulphuric acid supply & demand/market balance

Kimberly Gustin, CRU

This presentation will begin by covering the recent market developments, including the latest supply and demand side developments and the effect of the current balance on the traded market and recent price trends. After which a closer look at how recent global events are likely to shape the future of supply. What the impacts are of a changing macroeconomic environment and intensified political atmosphere are, and whether or not the sulphur market is undergoing a structural transformation.

Dry bulk freight market – a charterers' perspective

Derek Langston, Simpson, Spence & Young

The dry bulk freight market has entered 2015 at its one of its lowest points in the last 30 years. Not only are time charter rates near historical lows, but the collapse in oil prices and subsequent five-year low for bunker prices have further reduced voyage rate costs for charterers. Although last year's supply growth was constrained by the lowest new building deliveries growth (in percentage terms) since 2003 and trade was boosted by record iron ore, grain and steel cargo volumes, the first drop in annual seaborne coal trade since 1993 contributed to rate falls across all dry bulk carrier

sizes. In turn, declining expectations for 2015 have reduced longer-term period rates in the physical market in addition to leading to substantial falls in freight derivatives contracts.

This year's scheduled order book indicates a re-acceleration in new building deliveries is in prospect, while interest in demolition has been heightened by the negative outlook. Demand growth will be heavily reliant on China and India. Does this show structural oversupply in the freight market? Or, will there be more surprises ahead? This presentation aims to identify the ingredients required for an eventual freight market recovery as well as assessing the market from a charterers' perspective.

Caprolactam market review and implications

Leon Muijtjens, DSM Fibre Intermediates B.V.

Caprolactam's key outlet is polyamide 6 (PA6), of which approximately 5 million tons per year are produced worldwide. The global consumption of PA6 is expected to grow by approximately 3% annually. The applications of polyamide 6 are very diverse and covering many end-markets. Major outlets are bulked continuous fibers for application in carpets, textile fibers and yarns for garments, technical yarns for tire, engineering plastics for automotive and Electrical/Electronic (E&E) applications, and finally film for food packaging.

Recent years saw an impressive expansion of Chinese caprolactam capacity, and it will continue to grow in the coming years. The same applies to the downstream industries in China, e.g. PA6 and fibers. These investments are likely to result in significant overcapacity throughout the value chain. These Chinese investments will cause the loss of export opportunities for other caprolactam producing regions. This will inevitably result in local overcapacity in the various regions.

The presentation aims to inform the audience about, among other subjects, the caprolactam and PA6 value chains, about current and future demand/supply situation of the caprolactam markets, and the costs drivers of a caprolactam plant.

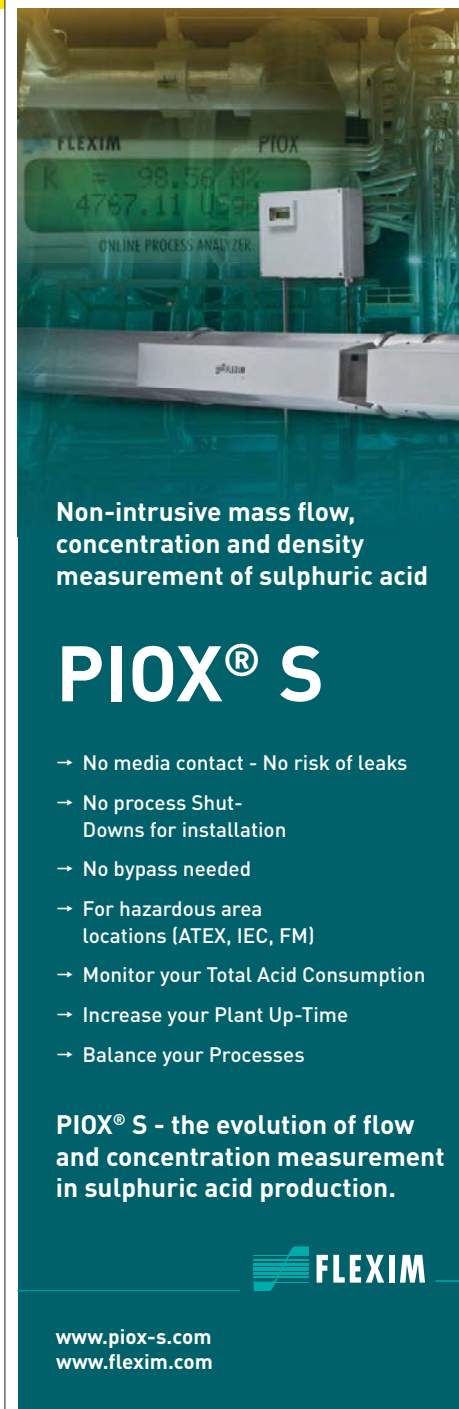
Sulphur Developments in Turkmenistan

Tom Smith, Sandvik Process Systems

Huge reserves of oil and gas have existed under central Asia for centuries and today, with global demand increasing, the greater CIS countries are investing more than ever to access the valuable natural resources that lie beneath their feet. Recent exploration in Turkmenistan has revealed gas reserves far greater than first expected, propelling the country up the list of countries with the largest gas reserves in the world.

However, these reserves contain sour gas with high levels of hydrogen sulphide that must be removed from the gas prior to use. As a result, sulphur production in Turkmenistan – currently in the region of half a million tonnes per annum – is expected to quadruple by 2019 as more of this trapped gas is extracted and processed. This increase will account for approximately 3% of the total global sulphur supply by 2019. With minimal domestic demand for its ever increasing sulphur supplies and changes to national stockpiling regulations, Turkmenistan's oil & gas producers will be compelled to solidify their sulphur for export.

This presentation will touch on the existing and future levels of sulphur from Turkmenistan and the logistical issues involved in trying to get it to end users around the world. It will then focus on a case study of a recent installation for forming, temporary storage and a sophisticated line of high speed bagging systems. ■



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The global market for sulphur

A survey of the latest developments which will affect the supply and demand for sulphur worldwide.

The total amount of elemental sulphur recovered in 2014 was about 56 million tonnes, according to CRU figures, of which 31 million tonnes were traded internationally. Over the next few years the total volume produced is expected to rise by 15 million t/a to 71 million t/a in 2019, with trade rising to 35 million t/a by that time. Much of the new supply is projected to come from sour gas projects, and the long-anticipated global market surplus that has been on the cards for several years now is expected to be finally upon us. This means that some sulphur producers are going to have to stockpile sulphur, and the questions then become; who and where?

New supply

Almost all elemental sulphur produced in the world comes from two main sources; refinery processing of crudes and other hydrocarbon feeds (oil sands, condensates) and processing of sour gas. These between them account for 99% of all elemental sulphur production.

Refinery sulphur

Recovered sulphur at refineries continues to grow due to three long-term trends. The first is growing demand for vehicle fuel, mainly in Asia, where refining capacity is due to rise from 30 million bbl/d to 36 million bbl/d by 2018. The second is tightening standards on sulphur content of fuels, again mainly in the developing world, and the third is rising content of sulphur in crudes.

In the OECD there is falling demand for refined products, and in places like Japan and Europe this has translated into refinery closures and falling sulphur output. In the US, refinery recovery of sulphur has actually increased, mainly due to additional processing of Canadian oil sands syncrude. In spite of continued delays and uncertainty over the Keystone XL project, and additional project delays in oil sands processing likely due to the falling oil price, US sulphur output may continue to increase slightly over the next few years. In addition, refiners in Europe and the US are now also having to cope with falling demand for marine heavy fuel oil and increased demand for low sulphur marine gasoil (MGO) due to the impact of IMO regulations on sulphur dioxide output from ships.

On top of this, regulations in sulphur content of fuels continue to tighten around

the world. Although most OECD countries have already moved to very low sulphur fuel standards (<15ppm sulphur), most countries in Asia, Africa and the FSU are moving from 50-500ppm sulphur fuel standards, and in South America and West Africa this is often higher still, over 2,000ppm permitted in some places, with the Middle East having some of the highest permitted sulphur standards in the world. However, regulation is moving quickly in some countries. China is moving to 'Euro-V' standards by 2017, with some key areas moving to 10ppm sulphur this year. In India a nationwide Euro-V standard will be adopted in 2016. In advance of this refineries are having to install desulphurisation capacity if they wish to serve these markets. Table 1 shows new desulphurisation capacity in major regions of the world over the next few years.

Table 1: New refinery desulphurisation capacity, '000 bbl/d

	2013	2014	2015	2016	2017	2018	Total
North America	240	85	60	0	0	0	385
OECD Europe	-200	0	35	-	114	0	-51
OECD Pacific	-89	-82	0	0	0	0	-170
FSU	114	160	50	35	0	0	359
Other Europe	0	45	20	0	0	0	65
China	799	139	331	850	0	164	2,283
Other Asia	284	104	-98	0	284	10	583
Latin America	90	111	160	0	70	245	676
Middle East	466	30	40	222	262	446	1,465
Africa	95	37	0	0	42	0	174
World	1,799	630	598	1,106	772	865	5,769

Source: IEA

Sour gas

New sour gas projects are treated in more detail in the article elsewhere in this issue. While North America pioneered the development of sour gas fields, production there continues to decline as cheaper shale gas undercuts more expensive sour gas production. However, the processing of sour gas is increasing markedly in three major regions; China, the Middle East, and Central Asia. In China and the Middle East, it is increasing demand for gas – especially, in the Middle East, non-associated gas – that is driving production. China is a sulphur deficit region and can absorb the new production, the Middle East tends to run a surplus and is looking to export much of the new sulphur. In Central Asia, the impetus is more for pumping of crude oil or recovery of condensates, where the sour gas tends to be an additional extra, and its processing merely an additional cost for oil producers.

Table 2 shows major recent and planned sour gas projects around the world. Most of the recent ones are still ramping up production, including Yuanba and Chuandongbei, Wasit, South Pars and Ras Laffan, with additional production beginning at Shah in Abu Dhabi and Ras Laffan in Qatar (via the Barzan project).

Other sources

Mined elemental sulphur remains very much a minority pursuit. Siarkopol in Poland was bought by Zakłady Azoty Tarnow in 2013, and now operates as Siarkopol Chemiczne Siarkopol. It produces 300,000 t/a of sulphur as a liquid which is sold into its parent company's fertilizer operations. Attempts to rejuvenate the Mishraq sulphur mine in Iraq by the Iraqi government got as far as shipping the sulphur plant modules, built by Decvo, out to the region, but they are currently in Jordan awaiting a resolution to the conflict in the north of Iraq with the Islamic State (IS). There has been some talk in the past few years about sulphur mining in Argentina, but no concrete interest.

However, on the metallurgical side, one interesting recent development is at Norilsk in Russia, where instead of sulphuric acid, the smelter plans to reduce metallurgical off-gases to hydrogen sulphide and recover it as sulphur in a Claus plant. This will add another 1 million t/a of sulphur production, though the plant is so remote that it seems unlikely that it will be anything other than stockpiled.

Table 2: Sour gas developments

Project	Location	Sulphur output (at capacity)	On-stream date
Middle East			
Habshan	Abu Dhabi	1.8 million t/a	2013
Shah	Abu Dhabi	3.1 million t/a	2015
Bab	Abu Dhabi	3.0 million t/a?	2020?
South Pars	Iran	+0.3 million t/a	2012-17
Ras Laffan	Qatar	+0.4 million t/a	2012-18
Karan	Saudi Arabia	0.3 million t/a	2012
Wasit	Saudi Arabia	0.8 million t/a	2015
Fadhili	Saudi Arabia	1.0 million t/a?	2018
Khuff/RHIP	Oman	0.3 million t/a	2019
Central Asia			
Orenburg	Russia	+0.4 million t/a	2015
Kashagan	Kazakhstan	1.1 million t/a	2016?
South Yolotan	Turkmenistan	1.8 million t/a	2013
China			
Puguang	Sichuan	3.0 million t/a	2011
Yuanba	Sichuan	0.3 million t/a	2014
Chuandongbei	Sichuan	1.5 million t/a	2015
Canada			
Varos	Alberta/BC	-0.5 million t/a	2014-2019
Total		15.6 million t/a	2014-2019

Demand

Sulphuric acid accounts for most elemental sulphur production – the sulphur market functions almost as a proxy market for sulphuric acid, as sulphur is far easier to store and transport than acid, for those who operate sulphur burning acid plants. Phosphate processing, mainly for agricultural uses, continues to predominate as the major use for sulphuric acid.

Phosphates

Just over half of all sulphur consumption (in all forms) depends upon phosphate fertilizer production. Phosphate consumption is dominated by four major economies; China, India, Brazil and the United States. Consumption was up in three of those during 2014, in each of China, the US and Brazil, offset slightly by a dip in India due to subsidy policy favouring urea fertilizers. Phosphate market fundamentals continue to be sound overall – grain prices are steady and global demand looks robust over the medium term, but pricing can very much affect short term fluctuations in the market.

The phosphate supply picture is pointing to a lot of over-building of capacity at the moment. China is at the tail end of a massive programme of DAP capacity build-

ing that has turned it into a net exporter. There are also projects which collectively amount to 20 million tonnes of additional phosphate rock from North Africa, 9 million from Jordan and Saudi Arabia, and 7 million t/a of rock from China in the period from 2013 to 2018. Phosphoric acid capacity could rise by 5.3 million t/a (tonnes P₂O₅) over the same period, according to IFA figures, with capacity additions in Morocco, Saudi Arabia, China and Brazil, by which time the total capacity of phosphoric acid is likely to be 61.5 million t/a, and demand is projected to be 47.7 million t/a. Last year alone, over 1.8 million t/a (P₂O₅) of phosphoric capacity was added, representing 5.5 million t/a of potential additional sulphuric acid demand. Morocco's expansion in its phosphate sector at Jorf Lasfar involves building four MAP/DAP complexes, each including 1.5 million t/a of sulphuric acid capacity, and the Saudi Ma'aden project at Waad al Shamal will include an extra 4.9 million t/a of acid capacity.

Metal leaching

Sulphur demand for sulphuric acid for metal leaching operations amounts to about 6.5 million tonnes S (in all forms) in 2013, representing about 19.6 million t/a of sulphuric acid consumption. There

is some zinc and uranium leaching (the latter especially in Kazakhstan), but copper leaching continues to predominate, particularly in Chile, although there are some new large-scale leaching projects under way in Peru as well. Chilean projects are suffering from declining mine production and falling ore grades and the country is switching to concentrate processing and away from SX/EW production. This will just about balance out the new consumption in Peru, and overall consumption in Latin America is relatively balanced, declining in the longer term. New production in Mexico and the US and especially central Africa will lead to slightly more acid consumption from the copper sector overall.

Nickel leaching has been the fastest growing segment of demand, but the high costs and temperamental nature of the high pressure acid leach process mean that the nickel market is now concentrating on other processes which are able to process cheaper, lower grade laterite ores such as ferronickel and "nickel pig iron" production. Some heap leaching projects in Turkey, the Philippines and Finland have run into difficulties and have not made much progress.

Sulphur surplus

Continued steady growth in phosphate capacity and the ramping up of some copper and nickel leaching projects are continuing to add demand for elemental sulphur, which could reach 66 million t/a by 2019, according to CRU. However, at the same time, the extra volumes coming from refineries and in particular sour

gas projects should – in theory – outweigh this to the tune of 5 million t/a. Figure 1 shows projections for sulphur supply and demand over the next few years, and as these charts generally do, they show a gradual move back into surplus by the sulphur market, beginning this year. The story of the past decade has been the story of the sulphur market surplus that never came, as sour gas projects in particular were delayed by engineering or financial difficulties, while a strong phosphate market continued to push demand, and there is no guarantee that this will not continue to happen, but the increasing oversupply in the phosphate market is more likely to lead to a slowdown in capacity building, while the fall in oil prices and the slowdown in the Chinese market have not – so far – changed the fundamentals of oil and gas markets sufficiently to affect ongoing sour gas projects.

Sulphur trade

The question then becomes where the sulphur will ultimately be sold from, and where it will be stockpiled. Looking a few years ahead, the major surplus regions/countries will be Canada, Kazakhstan/Russia and the Middle East (especially Abu Dhabi), and the major deficit nations still China, Morocco, Brazil, the US and to a lesser extent Australia, the Philippines and Madagascar (for leaching projects). Refiners tend not to stockpile sulphur to any major degree (outside of some of the oil sands producers in Canada) as they do not have the storage capacity and in any

event often see sulphur as peripheral to their main business of selling refined products, and are willing to take whatever the prevailing market price may be. It is among the sour gas producers, therefore, that we are most likely to see stockpiling.

China has a domestic sulphur deficit for the foreseeable future in spite of new sour gas production. Indeed, in spite of phosphate capacity overhang and additional smelting capacity, a continuing move away from making acid via pyrite roasting to sulphur burning, and more phosphate production could see China importing 10 million t/a of sulphur by 2019, and sour gas producers are not likely to need to stockpile.

Central Asia, conversely, is relatively logistically constrained for export to China and does not have easy access to ocean-going ports without a long and tortuous route. There is some local demand being developed, in particular for uranium leaching in Kazakhstan and some new fertilizer plants, but some sulphur may end up having to be stockpiled.

The Middle East is likely to have a large sulphur surplus in spite of some major phosphate projects being developed in Saudi Arabia and Jordan, and while Abu Dhabi and Qatar have worked hard on expanding their sulphur forming and export capacity, it is by no means certain that all of the sulphur produced there will find a market. The balance of storage and export may depend on the economics of production and prevailing market prices.

The North American market is continuing to change. Some contraction in US sulphur demand is expected, with Mississippi Phosphates now out of business and PotashCorp closing its Geismar acid plant and Suwannee River chemical plant in Florida. ICIS have calculated that up to 500,000 t/a of sulphur demand could go. Nevertheless, in spite of extra sulphur coming from Gulf refineries, the US could still be importing 2.1 million t/a by 2019. Most of this is likely to come from Canada, but a change in the US market dynamic has come from Mosaic's plan to start up a 1 million t/a capacity sulphur melter in Florida, allowing it to import offshore formed sulphur and remelt it for its own consumption, making it less dependent on molten sulphur cargoes. Some Canadian producers are also looking at ways of exporting solid sulphur rather than liquid as the US market – which has traditionally relied on liquid sulphur cargoes – contracts, in order to find other overseas markets.



ASRL REVIEW

A twice yearly review contributed by **Alberta Sulphur Research Ltd**

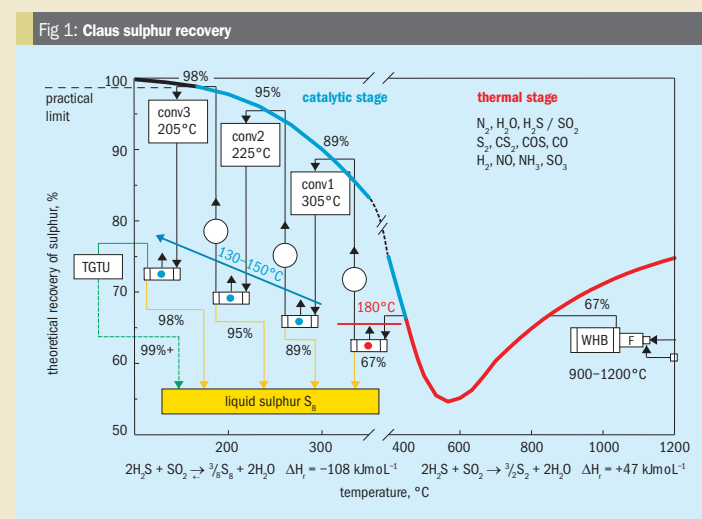
Taking advantage of existing equipment in a Claus sulphur recovery system

P.D. Clark, S.S. Bhella and N.I. Dowling, Alberta Sulphur Research Ltd.

Suggesting and developing new sulphur recovery technology is one thing, but having it implemented in practice is another matter. From a practical viewpoint, we already have working systems that can attain >99% conversion to sulphur, and this infrastructure has cost billions to put in place around the world. People are not about to abandon such investments just because a better gadget or two might become available. But, even more importantly, we know how to run a modified Claus sulphur recovery system reliably, so people do not want to risk unproven technologies. Nevertheless, lower costs with higher energy efficiency are the goals of modern, large scale industry, so improvements to existing systems are potentially useful. Recently, as part of the ASRL research program, we have examined one adaptation of a Claus plant which could improve it economically and utilise all of the 'pots and pans' already in place. Actually – some of the pots and pans can be discarded!

Modification of Claus sulphur condensers

The idea is simple: by incorporating some catalyst in a sulphur condenser, we can take advantage of the decreased



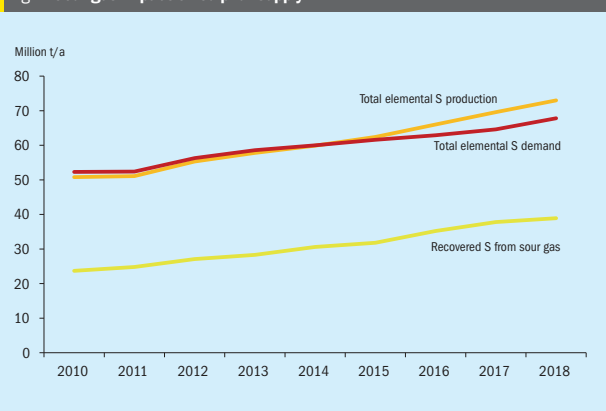
ing process gas temperature in that unit to push sulphur conversion to higher equilibrium levels, as is evident from the Gamson-Elkins plot (Figure 1). As shown in the schematic of Figure 2, Claus catalyst would be incorporated inside the condenser tube, allowing an incremen-

tal addition to sulphur recovery because of the lower temperature. Depending on how an individual plant would be run, we could expect higher conversion to sulphur and, if, configured to do so, manipulation of the H_2S/SO_2 ratio, so minimising the load on direct oxidation

or reducing tail gas-type units.

Process issues that would need to be taken into account are: the increased pressure drop throughout the plant that might result from having the catalyst inside the tubes; space velocity in a condenser tube in relation to the kinetics

Fig 1: Sour gas impact on sulphur supply



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Sour gas projects

COVER FEATURE 2

Sulphur markets

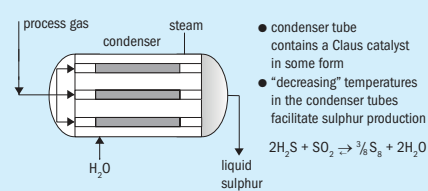
COVER FEATURE 3

ASRL review

COVER FEATURE 4

Smelter gas to sulphur

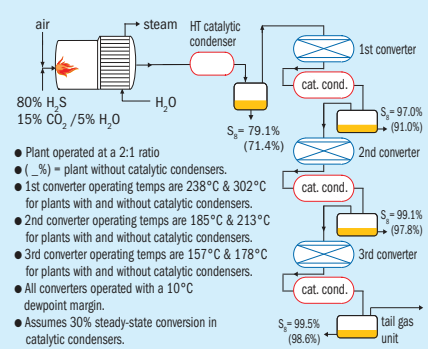
Fig 2: Hypothesis of Claus conversion in sulphur condenser



commercial potential:

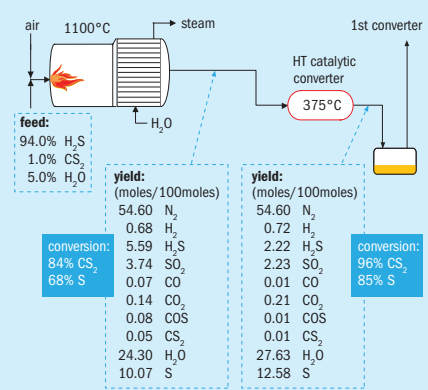
- increased conversion to sulphur in all condensers.
- continuous sub-dew point (130-150°C) reactor after first and second catalytic converter.
- H₂S / SO₂ ratio shift to off-load existing tail gas unit (direct oxidation reducing TG types).

Fig 3: Theoretical study on sulphur recovery in a sulphur plant using catalytic condensers



- Plant operated at a 2:1 ratio
- (%) = plant without catalytic condensers.
- 1st converter operating temps are 238°C & 302°C for plants with and without catalytic condensers.
- 2nd converter operating temps are 185°C & 213°C for plants with and without catalytic condensers.
- 3rd converter operating temps are 157°C & 178°C for plants with and without catalytic condensers.
- All converters operated with a 10°C dewpoint margin.
- Assumes 30% steady-state conversion in catalytic condensers.

Fig 4: HT catalytic converter for CS₂ & S conversion after a WHB



of the Claus reaction; and the potential for loss of catalytic activity as the liquid sulphur begins to form within the tube. If we consider a standard Claus converter operating at around 1,000 h⁻¹, we know that the major jump in temperature due to the exothermic Claus reaction occurs over the very top layer of catalyst, implying that the Claus reaction proceeds to equilibrium very quickly. Based on this temperature increase observation, only about 5% of the top part of the bed for the standard converter is actually necessary for the Claus reaction. This fact translates to a space velocity of around 15,000 h⁻¹ for the active part of the catalyst.

Of course, the rest of the bed may still be important for CS₂ conversion and for increasing the time a catalyst bed can be kept on-line. The space velocity inside a condenser tube is around 10,000 h⁻¹, so it should be possible to have some conversion to sulphur in a catalytic condenser tube (Figure 2). Of course, the main function of the condenser is to condense sulphur. Therefore, we can assume that at some point, the catalyst would become saturated with sulphur and lose activity because of drop in mass transfer of the reacting gases through a liquid film. Interestingly, calculations show that in a standard condenser, liquid sulphur starts to form around one third of the way down the tube, so a catalyst placed in the first third of the tube should stay largely in the gas phase.

What to expect from catalytic condensers

Assuming that a catalytic condenser could be designed, built and operated, how big is the prize in terms of extra conversion to sulphur? Equilibrium calculations show (Figure 3) that a catalytic condenser after the thermal stage and after the first real catalytic converter would have a profound effect on a plant, giving total sulphur recovery of 97% instead of 91% for a plant using standard condensers. These calculations were performed by adding 30% extra conversion to sulphur in the catalytic condensers, as this is the degree of conversion which has been found in laboratory trials. One advantage that accrues in a catalytic condenser plant is that the first converter can now be operated at a lower temperature because the extra

conversion to sulphur in the condenser after the WHB lowers the sulphur dew point in the first converter to 238 °C, so pushing conversion in the first converter over the Gamson-Elkins curve (Figure 1). Thus, if the situation shown in Figure 3 can be duplicated in commercial operation, the second catalytic converter and associated condenser can be dispensed with, removing the need to build or operate such units. Indeed, although we have not carried out pressure drop calculations, the increased pressure drop associated with putting some catalyst in the thermal and first converter condensers is probably negated by removal of the second catalytic converter and its condenser. Actually, as will be seen from the laboratory results, the situation could be much better than suggested in Figure 3. One point to note about Figure 3 is that the sulphur collection vessels after each catalytic condenser are shown for illustration only, and would not be additional vessels in a commercial plant.

Laboratory studies

In summary, experiments have been conducted which duplicate the situation depicted in Figure 3. Namely, tests were conducted taking a process gas composition from the Claus furnace at 380 °C, a typical temperature attained after passage through the WHB, and passing that gas through a Claus catalyst made at the ASRL laboratories which had demonstrated high rates of Claus conversion and also in destruction of CS₂. These experiments used a space velocity of 10,000 h⁻¹, so mimicking the residence time in a condenser tube. Also, it is important to note that at this temperature, the catalyst would be in a part of the tube which remains in the gas phase, so duplicating the situation inside the first section of a condenser tube. The results of these experiments are remarkable in that CS₂ conversion is close to 95%, and that the Claus reaction proceeds to equilibrium for the temperature of 380°C (Figure 4). Actually, these results should not really surprise us, as the tests were conducted about 60°C higher than normally used in a standard first catalytic converter. Thus, reaction rates should be approximately 26 times higher, which when translated to space velocity, suggests that we could actually operate a catalytic condenser

unit at 64,000 h⁻¹ and still observe the same conversion to sulphur and of CS₂. It should be noted that perfect H₂S/SO₂ ratio control was not achieved in the experiment shown in Figure 4, but if a 2:1 condition had been achieved, higher sulphur production would have been observed.

The serendipity of research

The catalyst used in this work is based on alumina onto which a layer of activated TiO₂ was deposited (Figure 5). The activating agent is Sc₂O₃. This catalyst actually arose from studies which were aimed at deduction of the mechanism of CS₂ conversion on TiO₂ Claus catalysts, a complex matter, but one which chemists like to take on. An important aspect of this mechanism appears to involve reduction of some Ti⁴⁺ to Ti³⁺. My colleague, Dr Minning Huang, decided that a more active catalyst could be made by doping TiO₂ with M³⁺ ions and correctly predicted that Sc³⁺ cations would be the most useful, because these cations have a very similar size to Ti³⁺ cations. So the first serendipitous feature of the current work is that we managed to produce a very active Claus catalyst with high CS₂ conversion activity simply because we wanted to know more about the surface mechanisms of TiO₂ catalysts. Dr Huang also showed that this active layer of TiO₂ and Sc₂O₃ could be deposited on standard alumina, making an affordable catalyst.

The second serendipitous feature of our work is, in fact, the realisation that we do not need to place this catalyst in a condenser downstream of the WHB as it operates in the gas phase. An ideal location would be in the piping after the WHB. Very little catalyst is required, approximately 1/10th of that used in the first catalytic converter (Figure 6). As can be seen, a very simple arrangement is suggested in which a dual pipe system is used so that the catalyst can be replaced without shutting the plant down. Thus the complication of placing a catalyst in a condenser tube is avoided, at least for the condenser after the WHB. The additional pressure drop for the in-pipe section (Figure 6) should be minimal because so little catalyst is needed. As is indicated in Figure 6, the sulphur plant to this point consists of the furnace, WHB, in-pipe catalyst and a conventional condenser.

Operation of first catalytic unit and downstream catalytic converter

As already mentioned, the increased conversion to sulphur accomplished with the in-pipe catalyst after the WHB allows operation for the first catalytic converter at <240 °C, at least for the acid gas chosen for this study. Besides allowing increased conversion to sulphur in that unit, advanced catalysts for CS₂ conversion in the first converter are no longer required, as the CS₂ has been dealt with in the in-pipe section. In addition, the degree of re-heat of the process gas needed for the first catalytic converter is reduced considerably, so saving energy and CO₂ emissions. The process gas leaving the first and only catalytic unit is now passed through a catalytic condenser. The laboratory data obtained show that ca. 30% extra conversion can be attained, which takes the overall conversion of sulphur to 97% (Figure 6). The tail gas can then be sent directly to the tail gas unit, which does not need any modification to accommodate the upgraded plant. Consideration of the way in which the experiments were done suggests that the 30% extra conversion is an under-estimate, because the laboratory catalytic condenser has been operated isothermally (Figure 7) so causing less of our catalyst to remain in the gas phase.

Fig 5: High activity Claus and CS₂ conversion catalyst

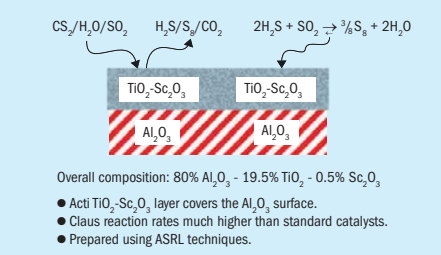


Fig 6: The re-modified Claus sulphur plant

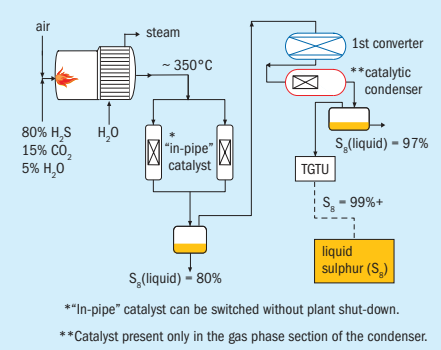
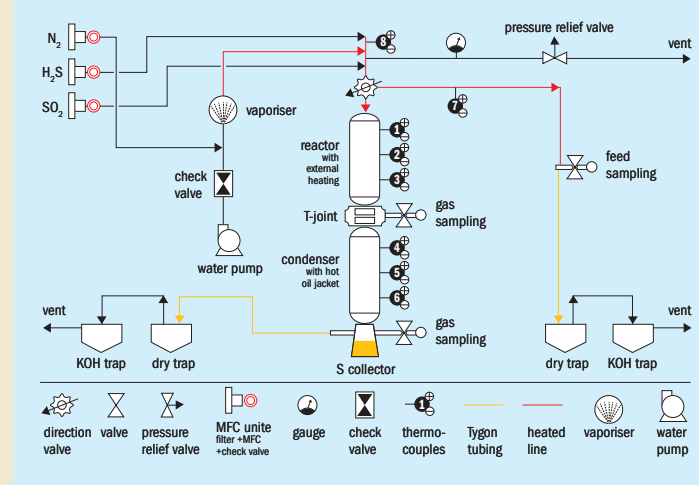


Fig 7: Bench scale catalytic reactor and condenser



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ISSUE 357
MARCH-APRIL 2015

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Practically, we suggest that the catalyst in the catalytic condenser would be in the form of an alumina monolith or foam placed in the initial part of each tube (Figure 6). Such materials are available, having been designed for the auto catalytic converter found in the tail pipe of all modern vehicles. These devices, made in the millions, have very little pressure drop, and so are ideal for the Claus application.

Manipulation of H₂S/SO₂ ratio

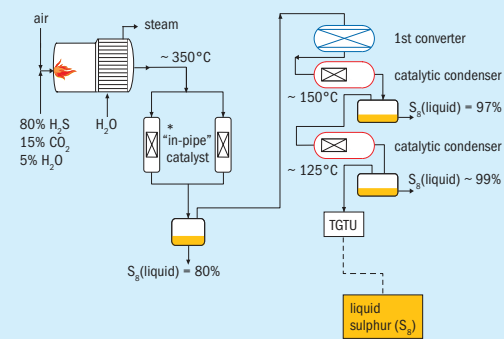
It was suggested earlier that the catalytic condenser can be used to manipulate H₂S/SO₂ ratio to higher values, an advantage of this strategy being that it minimizes the amount of SO₂ going to the tail gas unit. How could this adaptation be achieved? As seen in Figure 8, we have taken the existing condenser after the second catalytic converter and modified it to be a further catalytic condenser. The second catalytic converter is removed and replaced by the modified condenser, which to all purposes acts like an on-line sub-dewpoint catalytic bed. The calculations shown in Figure 3 suggest that 99% conversion to sulphur could be achieved at this point, assuming no kinetic limitations for the extra 30% conversion in catalytic condenser #2 (Figure 8). Such an adaptation has a very significant advantage for Claus

plants with either H₂S or SO₂ recycle-type tail gas units, as the solvent recirculation rates, and hence energy requirements, would be reduced.

Liquid sulphur degassing consequences

Research at ASRL has shown that most of the H₂Sx found in liquid sulphur is produced in the condenser downstream of the WHB. H₂Sx will not have been present in the furnace chamber or in the WHB, because the temperatures are too high for the relatively unstable -S-S- bond and probably because it forms by reaction of H₂S with Sx radicals as the liquid sulphur begins to condense. Also, the amount of H₂Sx increases with H₂S partial pressure. So in the process scheme shown in Figures 6 and 8, further conversion to sulphur in the in-pipe catalyst section lowers the H₂S partial pressure and hence the amount of H₂Sx that can be formed. This effect would also be true for the sulphur condensed after the first catalytic converter, so a sulphur plant, as shown in

Fig 8: The re-modified Claus sulphur plant



Figures 6 or 8, should produce sulphur with considerably less H₂Sx, so off loading the amount of degassing required in the downstream unit. Hence further energy savings would accrue in terms of less air flow requirement for a typical air-blown degassing system.

Concluding comments

Research on the concept of a catalytic Claus condenser has shown that it may be possible to improve the efficiency of a Claus plant with only minor modi-

fications to an existing layout. Possible benefits include lower requirement of dedicated CS₂ conversion catalysts, energy savings throughout the plant and, most importantly, use of less equipment. As is often the case, benefits not foreseen during planning of the research came to light as results presented opportunities for new designs. Just in case someone wishes to try it out we have a name for the adaptation: the Re-modified Claus Process, a linguistic nightmare if ever there was one!



Jeffrey J. Bolebruch Senior Market Manager

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Apollo Environmental Systems Ltd.	EnCana Corporation	OMV Exploration and Production GmbH	Sulfur Recovery Engineering (SRE)
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Enviro-Industries Ltd.	IPAC Chemicals Limited	Sandvik Process Systems, Inc./Brimrock Group Inc.	Total S.A.
CB&I	Jacobs Canada Inc. / Jacobs Nederland B.V.	Saudi Arabian Oil Company (Saudi Aramco)ASC	UniverSUL Consulting
Cenovus Energy	KT - Kinetics Technology S.p.A.	SemCAMS ULC	Virtual Materials Group Inc.
Chevron Energy Technology Company	Linde Gas and Engineering	Shell Canada ULC	Weatherford International LLC/ICTC
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Sulphur recovery plant



Sulphur's annual survey of recent, current and future sulphur recovery unit construction projects maps the developing shape of brimstone production from fuel and gas processing plants worldwide.

PHOTO: IATE

Sulphur recovery

OPERATING COMPANY	OPERATING SITE	PROCESS TYPE	TOTAL CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
AZERBAIJAN							
SOCAR	Garadagh	H ₂ S, CO ₂ , amine	n.a.	Amec Foster Wheeler	Amec Foster Wheeler	New	2018
BAHRAIN							
Bapco	Sitra	Claus, NH ₃ , amine, SWS, AGRU	3 x 250 t/d	WorleyParsons	n.a.	New	2017
BELARUS							
JSC Mozyr	Mozyr Refinery	Claus, TGTU	240 t/d	Silrtec Nigj	n.a.	New	2015
BELGIUM							
ExxonMobil	Antwerp Refinery	SWS	n.a.	Fluor	Amec Foster Wheeler	New	2017
ExxonMobil	Antwerp Refinery	O ₂ enrich, amine TGT	325 t/d	WorleyParsons	Amec Foster Wheeler	Revamp	2016
BRAZIL							
Petrobras	Premium I	SuperClaus	2 x 240 t/d	Jacobs	n.a.	New	2017
Petrobras	Premium II	SuperClaus	240 t/d	Jacobs	n.a.	New	2017
Petrobras	RPBC II	Claus, NH ₃ , TGT	42 t/d	WorleyParsons	n.a.	New	2015
Petrobras	REDUC	SuperClaus	2 x 62 t/d	Jacobs	n.a.	Revamp	2017
Petrobras	RNEST	SWS, Claus, NH ₃ , amine TGT	2 x 250 t/d	WorleyParsons	n.a.	New	On hold
Petrobras	Maranhao Premium 1	2 x Claus, NH ₃ , H ₂ , amine TGT	238 t/d	Amec Foster Wheeler	n.a.	New	n.a.

KEY
 BTX = BTX destruction
 Fuel = Fuel gas supplemental burning
 O₂ = Oxygen enrichment
 NH₃ = Ammonia destruction
 H₂ = Hydrogenation
 SRU = Sulphur recovery unit
 SWS = Sour water strip
 TGT = Tail gas treatment unit
 n.a. = Information not available

Sulphur recovery continued

OPERATING COMPANY	OPERATING SITE	PROCESS TYPE	TOTAL CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
CAMEROON							
SoNaRa	Limbe	SRU, SWS	17 t/d	Amec Foster Wheeler	Amec Foster Wheeler	New	n.a.
CANADA							
NAOS	Kai Kos Dehseh	Claus, NH ₃ , amine, SWS	2 x 390 t/d	WorleyParsons	n.a.	New	On hold
Irving Oil	Eider Rock Refinery	Claus, NH ₃ , amine	2 x 375 t/d	WorleyParsons	n.a.	New	On hold
PetroCanada	Fort Hills Upgrader	Claus, NH ₃ , amine	2 x 700 t/d	WorleyParsons	n.a.	New	On hold
Suncor Energy	Montreal	Claus, SCOT	2 x 100 t/d	Jacobs	n.a.	New	2016
CHINA							
CNPC/Chevron	Chuadongbei	Claus, SCOT	2 x 687 t/d	WorleyParsons	n.a.	New	2015
Guo Tai	Ordos City	O ₂ , H ₂ , amine TGT, D'GAASS	10 t/d	Fluor	n.a.	New	2015
HuaJin	Xilingol	O ₂ enrich	10 t/d	Fluor	n.a.	New	2014
Inner Mongolia Manshi	Ordos	SRU	51 t/d	Jacobs	n.a.	New	2017
Jiutai Energy	Linyi, Shangdong	EuroClaus	32 t/d	Jacobs	n.a.	New	2016
Pucheng Clean Energy	Weibei, Shaanxi	SRU, degas, TGT	105 t/d	KPS Tech & Eng	Tianchen	New	2015
Shijiazhuang Yingding Gases	Shijiazhuang, Hebei	EuroClaus	12 t/d	Jacobs	n.a.	New	2015
Wulan	Xinganmeng	O ₂ , H ₂ , amine TGT, D'GAASS	26 t/d	Fluor	n.a.	New	2015
Yitai	Hanginqi	O ₂ , NH ₃ , S degas	2 x 59 t/d	Fluor	n.a.	New	2015
COLOMBIA							
Ecopetrol	Barrancabermeja	Claus, NH ₃ , TGT	2 x 130 t/d	WorleyParsons	Amec Foster Wheeler	New	On hold
CUBA							
CuvenPetrol	Cienfuegos	Claus, TGT, Aquisulf	2 x 185 t/d	Lurgi	n.a.	New	2015
ECUADOR							
Petroecuador	Esmeraldas	Claus	50 t/d	Prosemat	n.a.	New	2015
FRANCE							
Chevron	Gonfreville	Claus	25 t/d	Prosemat	n.a.	New	2015
Total	Gonfreville	LPG treatment	n.a.	Amec Foster Wheeler	Amec Foster Wheeler	Revamp	2015
INDIA							
HPCL	Visakh Refinery	Claus, NH ₃ , amine	300 t/d	WorleyParsons	n.a.	New	2015
Reliance	Jamnagar	O ₂ , NH ₃ , amine TGT	4 x 1,300 t/d	WorleyParsons	n.a.	New	2016
INDONESIA							
Pertamina	Balongan	Claus, NH ₃ , H ₂ , amine TGT	1,100 t/d	Amec Foster Wheeler	n.a.	New	n.a.

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 SWS = Sour water strip
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Sulphur recovery continued

OPERATING COMPANY	OPERATING SITE	PROCESS TYPE	TOTAL CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
IRAQ							
Midland Ref. Co	Daura	Claus, TGT, Aquisulf	2 x 125 t/d	Lurgi	n.a.	New	2015
North Ref. Co	Kirkuk	Claus, TGT, Aquisulf	3 x 135 t/d	Lurgi	n.a.	New	2015
South Ref. Co	Maissan	Claus, TGT, Aquisulf	3 x 272 t/d	Siirtec Nigi, Lurgi	Lukoil	New	2015
Gazprom Neft	Basra	SRU, LT-SCOT, amine	2 x 157 t/d	Jacobs	n.a.	New	2015
Petrochina	Halfaya	Claus, amine	3 x 60 t/d	WorleyParsons	n.a.	New	2017
Lukoil	Yamana	Claus, SCOT	n.a.	WorleyParsons	n.a.	New	2017
KAZAKHSTAN							
Agip KCO	Kashagan	Claus, TGT	2 x 1,900 t/d	WorleyParsons	Black & Veatch, Petrofac	New	2015
Pavlodar Oil Chem	Pavlodar Refinery	Claus, TGTU	180 + 260 t/d	Siirtec Nigi	Rominserv, Technip	New	2015
KUWAIT							
Chevron	Wafra	Claus, amine	2 x 218 t/d	WorleyParsons	n.a.	New	2018
CGUP	Wafra	Claus, amine	2 x 400 t/d	WorleyParsons	n.a.	New	2018
KNPC	Mina al Ahmadi	Amine	400 t/d	WorleyParsons	n.a.	New	2015
MALAYSIA							
Petronas	Johor	SuperClaus	3 x 470 t/d	Jacobs	n.a.	New	2015
MEXICO							
PEMEX	Duba	SRU	n.a.	Amec Foster Wheeler	n.a.	New	n.a.
PEMEX	Cadareyta	SMARTSULF, NH ₃	132 t/d	WorleyParsons	n.a.	New	2016
NIGERIA							
Dangote Oil	Lekki Refinery	SuperClaus	2 x 115 t/d	Jacobs	n.a.	New	2017
OMAN							
OOC	Duqm Refinery	NH ₃ , H ₂ , amine TGT	3 x 355 t/d, S degas	Fluor	n.a.	New	2015
PDO	Yibal Khuff Sudair	Claus, TGT	250 t/d	WorleyParsons	n.a.	New	2015
PERU							
Repsol	La Pampilla	2 x Claus, NH ₃ , O ₂ , H ₂ , amine, TGT	83 t/d	Amec Foster Wheeler	SAINC	New	2016
QATAR							
Qatar Petroleum	Mesaieed	Sour gas, AGE, Claus, TGT	310 t/d	Worley Parsons, Black&Veatch	Petrofac/Prosemat	Revamp	2015

KEY

BTX = BTX destruction
 Fuel = Fuel gas supplemental burning
 O₂ = Oxygen enrichment
 NH₃ = Ammonia destruction
 H₂ = Hydrogenation
 SRU = Sulphur recovery unit
 SWS = Sour water strip
 TGT = Tail gas treatment unit
 n.a. = Information not available



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Sulphur recovery continued

OPERATING COMPANY	OPERATING SITE	PROCESS TYPE	TOTAL CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
RUSSIA							
Angarsk Refinery	Angarsk	Claus, NH ₃ , TGT	2 x 57 t/d	WorleyParsons	n.a.	New	2015
Bashneft	Ufa	Amine, SWS	n.a.	Amec Foster Wheeler	n.a.	New	2018
Gazpromneft	Moscow	LPG treat, amine	n.a.	Amec Foster Wheeler	Amec Foster Wheeler	New	2020
Rosneft	Novokubishevsk	Claus, NH ₃ , TGT	2 x 192 t/d	WorleyParsons	n.a.	New	2017
Lukoil	Volgograd	NH ₃ , H ₂ , amine TGT, D'GAASS	2 x 76 t/d	Fluor	n.a.	New	2015
Lukoil	Kstovo Refinery	Claus, TGTU	2 x 290 t/d	Siirtec Nigi	Tecnicas Reunidas	New	2015
Mariiskiy	Mari El Republic	SRU, TGT, amine	n.a.	Shell	Amec Foster Wheeler	New	n.a.
Orsknefteorg	Orsk	EuroClaus	2 x 99 t/d	Jacobs	n.a.	New	2016
OOO Ilkii NPZ	Krasnodar Krai	EuroClaus	86 t/d	Jacobs	n.a.	New	2016
Varino Refinery	Varino	Smartsulf	15 t/d	WorleyParsons	n.a.	New	2017
SAUDI ARABIA							
Luberef	Yanbu	SRU, SCOT	2 x 81 t/d	Jacobs	n.a.	New	2015
Aramco/Mobil	Yanbu	EuroClaus	3 x 220 t/d	Jacobs	n.a.	Revamp	2015
Yasref	Yanbu	SRU, TGT	3 x 630 t/d	WorleyParsons	n.a.	New	2015
SINGAPORE							
Singapore Refining	Singapore Refinery	SuperClaus	80 t/d	Jacobs	n.a.	Revamp	2015
SPAIN							
Petronor	Muskiz	EuroClaus	86 t/d	Jacobs	n.a.	Revamp	2016
TURKEY							
STRAS	Aliaga/Izmir	SRU, TGT, amine, SWS	463 t/d	Tecnimont KT	FosterWheeler	New	2017
Turkish Petroleum	Mansuriya	Claus, amine	230 t/d	WorleyParsons	n.a.	New	2016
UNITED ARAB EMIRATES							
Gasco	Ruwais Refinery	Claus, amine	30 t/d	WorleyParsons	n.a.	New	2015
Gasco	Shah, Abu Dhabi	BTX, H ₂ , Flexorb TGT Acid enrich, D'GAASS	4 x 2,500 t/d	Fluor	Fluor	New	2015
IPIIC	Fujairah	SRU, SWS, amine TGT	330 t/d	Amec Foster Wheeler	n.a.	New	2018
UNITED KINGDOM							
ConocoPhillips	Humberside	Claus, NH ₃ , TGT	132 t/d	WorleyParsons	n.a.	New	2015
ExxonMobil	Southampton	EuroClaus	2 x 125 t/d	Jacobs	n.a.	Revamp	2015

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Sulphur recovery continued

OPERATING COMPANY	OPERATING SITE	PROCESS TYPE	TOTAL CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
UNITED STATES							
Chevron	Richmond, CA	O ₂ enrich	580 t/d	WorleyParsons	n.a.	On hold	n.a.
Hydrogen Energy California	Kern County, CA	O ₂ enrich, NH ₃ , H ₂ , Amine TGT, D'GAASS	100 t/d	Fluor	n.a.	New	On Hold
Leucadia	Chicago, IL	Claus, TGT	2 x 215 t/d	Black & Veatch	n.a.	New	On hold
NCRA	McPherson, KS	D'GAASS	194 t/d	Fluor	n.a.	Revamp	n.a.
Sinclair Oil	Sinclair, WY	Claus	n.a.	Amec Foster Wheeler	Amec Foster Wheeler	Revamp	2016
UZBEKISTAN							
Lukoil	Bukhara, Karasul	SuperClaus, TGT	2 x 405 t/d	Jacobs	n.a.	New	2016
VENEZUELA							
PDVSA	El Palito	SRU, amine TGTU, SWS	250 t/d	Shell	Amec Foster Wheeler	New	2018
PDVSA	Monagas	Amine regen, SWS	54 t/d	Amec Foster Wheeler	Amec Foster Wheeler	New	2016
PDVSA	Puerto La Cruz	Claus, NH ₃ , amine	2 x 225 t/d	WorleyParsons	n.a.	New	2017



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A change in focus



David Hayes reports from Japan, where Sumitomo's main acid trading company Acids says that it is switching its sulphuric acid export focus to the Philippines.

Acids Co. Ltd is a sulphuric acid and sodium sales company founded in 2003 and jointly owned by Sumitomo Metal Mining Co, which owns three smelters in Japan, and Dowa Mining Holdings Co, with another two smelters. The company says that it is planning to increase sulphuric acids sales over the next 18 months to the Philippines, where demand is growing due to planned expansions of nickel leaching and copper smelting activities at various locations across the country.

Acids' owners, Sumitomo Metal Mining Co and Dowa Mining Holdings Co, are two of Japan's largest sulphuric acid producers and together supply about 90% of Acids' sulphuric acid sales volume. The remaining sales volume is purchased from other sulphuric acid producers. "Sumitomo produces 1.3 million t/a, it's almost always the same level," explained Toshio Gunji, Sales Department General Manager at Acids Co Ltd. "We have a one month turnaround every two years. The last time was October/November 2013, so the production that year was 1.2 million t/a."

Dowa produces 500,000 t/a at the company's two metal refineries. The firm's

Akita zinc refinery produces 260,000 t/a, while Dowa's Onahama copper refinery produces 240,000 t/a. "Sumitomo and Dowa produce 1.8 million t/a and we buy in about 200-300,000 t/a," Gunji said. "Coral Bay is using 600,000 t/a a year, so we need to buy in from outside sources. The Philippines' share is 1.4 million t/a and our domestic sales are about 600,000 t/a a year. We also have Taiwan and Southeast Asia customer exports of about 200,000 t/a, so we buy in from other Japanese and South Korean smelters."

In Japan, Acids normally buys in sulphuric acid from four suppliers, including Nippon Phosphoric Acid Co, a fertilizer manufacturer. "The amount we buy from them depends on their surplus as they use sulphuric acid for fertilizer production," Gunji said. "Towa Gosie, a chemical manufacturer, supplies us with some; others include Ube Industries which produces gypsum and gypsum board. Other Japanese smelters supply us as well, not a fixed amount as it depends on the supply situation."

According to figures released by the Ministry of Economy, Trade and Industry (METI), production of sulphuric acid in Japan was 6.35 million t/a in the financial

year ending March 31, 2014 (FY2014), registering a 5.8% decrease compared with production of 6.74 million t/a the previous year. Almost 5 million t/a of sulphuric acid is obtained from copper and zinc smelting in Japan, where copper smelters are the largest single source of acid.

"Total production in 2013 was 6.6 million t/a of sulphuric acid; demand in Japan is stable but decreasing," Gunji explained. "Demand now is about 3.8 million t/a, so Japan exports about 2.8 million t/a."

In Taiwan, Acids supplies sulphuric acid to four fertilizer manufacturers, of which three export part of their fertilizer production. Taiwan is a small market, however, and demand for sulphuric acid is not expected to grow in future.

"The Philippines is our biggest market. Until 2012 the China market was rather big, but now we send most acid to the Philippines. We stopped supplying China in 2014. We mostly supplied fertilizer customers," Gunji said.

Taganito

The Sumitomo Metal Mining (SMM) share of Acids' export business has grown quickly during the past three years due to the start up of Sumitomo's Taganito nickel project. Exports to SMM's Philippines projects currently account for about 70% of Acids' total sulphuric acid sales, compared with around 33% in 2012 when Coral Bay accounted for one third of the firm's total sulphuric acid business, exports to other customers another one third, and domestic sales in Japan the final third. Currently the Philippines accounts for over 85% of Acids' exports while remaining exports are supplied to customers in Taiwan and Southeast Asia.

Increased shipments to the Philippines have resulted in smaller exports to other countries during the past three years, and plans to expand supplies of sulphuric acid to major customers in the Philippines follows a major shift in Acids' sales focus over the past three years, reflecting the continuing slowdown in demand in Japan and the start up of the Taganito nickel mining project in Mindanao in the southern Philippines in 2013, where production is due to be expanded this year due to forecast stronger global nickel prices and the possibility of selling to a copper smelting project in the Philippines. "Taganito uses 800,000 t/a of sulphuric acid a year now but they will expand production by

20%, so they will need more acid," Gunji said. "They will expand in 2016. They are expanding because nickel prices are good. They will need another 160,000 t/a of sulphuric acid a year."

"Acids is dealing about 2.2 million metric tonnes of sulphuric acid a year. Our domestic sales are 600,000 t/a, so we export about 1.6 million t/a," said Gunji. "Our main export markets used to be China, Taiwan and Chile where the market price is good and it's used by the copper refining industry. Sumitomo started nickel leaching at Coral Bay in Palawan in the Philippines in 2005. There was a ferro-nickel mine there, and now they are processing the old spoil. The new customer in the Philippines is the Taganito mining project in Mindanao in the Philippines. It's also a nickel mine. They started in June 2013. They are using 800,000 t/a of sulphuric acid a year, which is half of our exports."

Consumption

Demand for sulphuric acid in Japan has been in long-term decline as major consuming industries continue to relocate production to lower cost production bases elsewhere in Asia. Japan's consumption of sulphuric acid was about 7 million t/a in the early 1990s. Annual demand in Japan is half that figure today and is forecast to see a further decline in future. "Demand from steel and paper factories is down in Japan as these factories are moving to China and Southeast Asian countries," Gunji said. Domestic demand for sulphuric acid in Japan has fallen by around 25% or about 1.2 million t/a since FY2007, immediately prior to the economic downturn, when domestic demand stood at 5 million t/a. It dropped to 4.1 million t/a in FY2009, immediately following the 2008 economic downturn, and has continued to slip by 100,000 to 200,000 t/a most years since then.

"After the economy recovered from the 2008 Lehman shock, Japanese sulphuric acid users started moving their factories out-

side Japan, they started investing in other Asian countries," Gunji explained. "Also, caprolactam manufacturers abandoned their production as Chinese caprolactam producers were increasing their output. Japanese manufacturers had a difficult time to continue caprolactam output and other businesses that consume acids."

Almost every major consumer of sulphuric acid in Japan has seen demand slip during the past decade.

Fertilizer companies in Japan, for example, used 373,000 t/a of sulphuric acid in FY2013, down 38% from the 604,000 t/a employed in FY2007. Other industries also have seen demand for sulphuric acid decline in recent years. "Titanium oxide producers also are decreasing their consumption in Japan because they are losing their competitiveness with China," Gunji said. "Titanium oxide is used to make industrial paint. Chinese titanium oxide manufacturers are selling at a low price so Japanese paint manufacturers are now importing their supplies."

Synthetic fibre production used 448,000 t/a of sulphuric acid in FY2013, according to METI, down from 638,000 t/a in FY2007, while aluminium sulphate production required 277,000 t/a of sulphuric acid in FY2013, compared with 375,000 t/a in FY2007.

Prices

Meanwhile, sulphuric acid prices seem unlikely to change in the immediate future with the current period of price stability expected to continue for a while. "Acid prices are very stable, they have risen 35% since 2006 and we don't expect to see them change this financial year," Gunji said. "The yen depreciation has not affected us as the foreign acids market has been very tight for the past five to six years; that's why we export at high value."

"We ask domestic customers to accept higher prices, otherwise they will have nothing." ■



Toshio Gunji,
Sales Department General
Manager, Acids Co. Ltd.

Above: Nickel processing at Taganito, Philippines.

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Sulphuric acid projects 2015

Recent and planned construction projects in the sulphuric acid industry

OPERATING COMPANY	OPERATING SITE	APPLICATION	CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
AUSTRALIA							
Nyrstar	Port Pirie	Smelter off-gas	n.a.	Outotec	Outotec	Revamp	2015
BRAZIL							
MBAC	Itafos	Sulphur burning	600 t/d	Haldor Topsoe	n.a.	New	2014
CANADA							
Vale	Sudbury	Nickel smelter off gas	1,400 t/d	Jacobs	Jacobs	New	2015
CHINA							
Henan Zhongyuan	n.a.	Smelter off-gas	4,820 t/d	MECS	n.a.	New	2015
Hubei Huaqiang	n.a.	Sulphur burning	667 t/d	MECS	n.a.	New	2015
Jiangsu Sailboat	Lianyungang	Spent acid regeneration	n.a.	Jacobs	n.a.	New	2014
Jinchuan	Fangchenggang	Smelter off-gas	2 x 2,550 t/d	MECS	n.a.	New	2014
Wytton Dazhou	Dazhou, Sichuan	Sulphur burning	1,200 t/d	MECS	n.a.	New	2014
CHILE							
Codelco	Calama	Smelter off-gas	880 t/d	Outotec	Outotec	New	2014
Codelco	Mejillones	Smelter off-gas	100 t/d	n.a.	Foster Wheeler	New	2015
Codelco	Potrerillos	Smelter off-gas	n.a.	Outotec	Outotec	Revamp	2016
GERMANY							
Grillo Werke	Duisberg	Spent acid regeneration	+120 t/d	Grillo Werke	n.a.	Revamp	2015
INDIA							
FACT	Kochi	Sulphur burning	2,000 t/d	n.a.	n.a.	New	2016
Paradeep Phosphates	Paradeep	Sulphur burning	2,000 t/d	MECS	Jacobs	New	2015
Rourkela Steel	Odisha	Sulphur burning	125 t/d	n.a.	n.a.	New	n.a.
JORDAN							
JIFCO	Ei Eshidiya	Sulphur burning	4,500 t/d	MECS	SNC Lavalin	New	2014
KAZAKHSTAN							
Kazatomprom	Stepanogorsk	Sulphur burning	450 t/d	MECS	Desmet Ballestra	New	2015
MEXICO							
Baja Mining	Santa Rosalita	Sulphur burning	2,650 t/d	MECS	SNC Lavalin	New	2014
MOROCCO							
OCP	Jorf Lasfar	Sulphur burning	4,200 t/d	MECS	n.a.	New	2016
NAMIBIA							
Namibia Custom Smelter	Tsumeb	Smelter off-gas	1,000 t/d	Outotec	Outotec	New	2015
PERU							
PetroPeru	Talara	WSA	460 t/d	Haldor Topsoe	n.a.	New	2014
Rio Seco	Rio Seco	WSA	60 t/d	Haldor Topsoe	n.a.	New	2014
SCC	Tia Maria	Smelter off-gas	1,640 t/d	n.a.	n.a.	New	2017
Votorantim	Cajamarquilla	Smelter off-gas	n.a.	Outotec	Outotec	New	2016
RUSSIA							
Ural Mining	Svyatogot	Smelter off-gas	n.a.	Outotec	Outotec	Revamp	2018
Norilsk	Nadezhda	Smelter off-gas	n.a.	Outotec	Outotec	Revamp	2015
SAUDI ARABIA							
Ma'aden	Umm Wual	Sulphur burning	3 x 5,050 t/d	MECS	SNC Lavalin	New	2016

Recent and planned construction projects in the sulphuric acid industry

OPERATING COMPANY	OPERATING SITE	APPLICATION	CAPACITY	LICENSOR(S)	LEAD CONTRACTOR	TYPE	START-UP
SERBIA							
RTB Bor	Bor	Smelter off-gas	1,820 t/d	MECS	SNC Lavalin	New	2015
SOUTH KOREA							
Korea Western Power	Taeon	WSA	96 t/d	Haldor Topsoe	n.a.	New	2015
TAIWAN							
Heng-I	n.a.	Sulphur burning	500 t/d	MECS	n.a.	New	2014
TUNISIA							
Groupe Chimique TunisienGafsa		Sulphur burning	1,800 t/d	MECS	n.a.	New	2015
TURKEY							
Cengiz Group	Samsun	Smelter off-gas	n.a.	Outotec	n.a.	Expansion	2016
Toros Tarim	n.a.	Sulphur burning	2,200 t/d	MECS/Ballestra	n.a.	New	2015
TURKMENISTAN							
Turkmenchimia	n.a.	Sulphur burning	1,500 t/d	MECS	n.a.	New	2015
UGANDA							
Sukuru Phosphate	Tororo	Sulphur burning	600 t/d	n.a.	n.a.	New	2016
UZBEKISTAN							
JSC AMMC	Almalyk	Smelter off-gas	1,500 t/d	Outotec	Outotec	New	2014
ZAMBIA							
Kansanshi Mining	Solwezi	Smelter off-gas	4,400 t/d	Outotec	Outotec	New	2014
Mopani Copper	Mulifera	Smelter off-gas	1,150 t/d	n.a.	n.a.	New	2014



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Boosting sulphur recovery with sub-dewpoint processes

Over the years, improvements to sub-dewpoint processes have allowed sulphur recoveries as high as 99.9% to be reached. More recently, the development of internally cooled catalytic reactors has also opened the door for sub-dewpoint operation in the Claus unit itself.

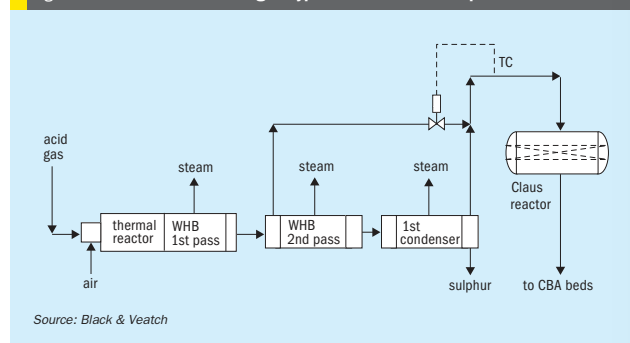
The overall sulphur recovery achieved in conventional Claus units is primarily dictated by the thermodynamic equilibrium of the Claus reaction at the outlet of the final catalytic converter. This thermodynamic equilibrium allows increased sulphur conversions when the temperature is lowered. Conventional Claus reactors are operated at temperatures above the dewpoint of liquid sulphur which limits the achievable conversion to 95-98% (depending on the number of catalytic stages) for typical refinery acid gases. Where the goal is a sulphur recovery efficiency (SRE) of at least 98%, a level exceeding what can be obtained with the conventional modified-Claus process, this limitation can be overcome by operating a Claus catalytic reactor at sulphur sub-dewpoint temperatures and allowing liquid sulphur to accumulate on the catalyst. This has been the basis of the so called dry bed sub-dewpoint processes like Sulfreen™, CBA and MCRC for many years. These sub-dewpoint processes are all variations of the same basic concept, but differ in the method used for regeneration of the sub-dewpoint reactor. Sub-dewpoint processes are generally classified as tail gas treatment (TGT) processes given that the process follows a conventional modified-Claus thermal stage consisting of a thermal reactor, waste heat exchanger, sulphur condenser, reactor preheat step, and catalytic reactor.

Sub-dewpoint Claus reactor operation improves the Claus reaction equilibrium, converting more H₂S and SO₂ to product sulphur. The sub-dewpoint reactor operation also takes advantage of adsorption of product sulphur vapour from the gas onto the catalyst surface, removing sulphur vapour from the gas. The process is cyclical. While in adsorption mode a catalyst bed operates in a low temperature range where the produced sulphur adsorbs onto the catalyst surface. Before the adsorbed sulphur accumulates to a level deactivating the catalyst bed, the bed is heated to desorb the product sulphur. Switching valves rotate process flow through the catalyst beds and condensers, sequencing the beds through adsorption/desorption cycles over optimised time intervals. By allowing the beds to run at cooler temperatures for longer intervals, overall sulphur recovery levels can be made to increase. When very high sulphur recoveries are required a hydrogenation/quench/amine tail gas treating unit is typically installed. However, this type of unit has much higher capital and operating costs than sub-dewpoint technology.

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Fig 1: Claus front end with hot gas bypass for Claus reactor preheat



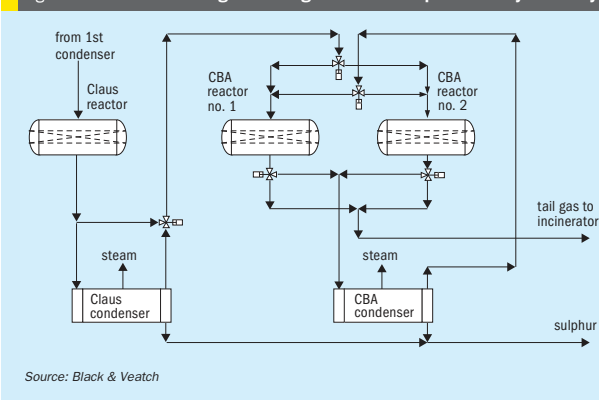
Source: Black & Veatch

CBA process

Cold bed adsorption (CBA) technology is a type of sub-dewpoint Claus process, originally invented by Amoco, for the recovery of elemental sulphur from H₂S-containing acid gases produced as a byproduct of gas production and oil refining. CBA sulphur recovery technology has been around since the 1970s. To date, about 35 cold bed adsorption (CBA) sulphur recovery units of varying configurations have been commissioned throughout the world to achieve higher sulphur recovery efficiencies than that provided by conventional modified-Claus units.

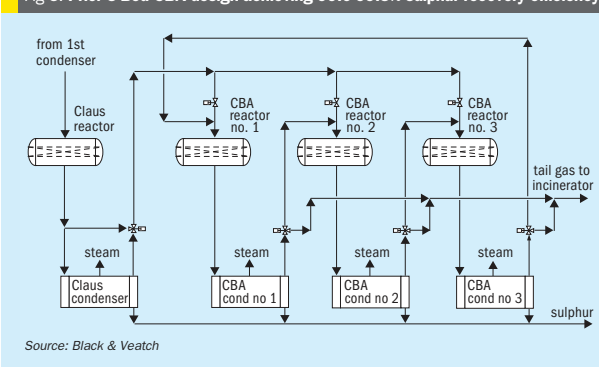
Past application of CBA technology has proven to offer competitive costs and good performance in 2-bed designs up to 99% and in 3-bed designs up to 99.2% sulphur recovery efficiency.

Fig 2: Prior 2-bed CBA design achieving 98.5-99.0% sulphur recovery efficiency



Source: Black & Veatch

Fig 3: Prior 3-Bed CBA design achieving 99.0-99.3% sulphur recovery efficiency



Source: Black & Veatch

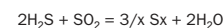
Improvements to the CBA process may now allow for competitive designs which achieve average daily sulphur recoveries at or above 99.5% for a 3-bed CBA operating on a rich acid gas without the use of special equipment or catalysts¹.

Basics of CBA

A CBA unit is identical to the modified-Claus process through the first catalytic stage (Fig. 1). Hot gas bypass is typically used for preheat of the first catalytic Claus reactor to ensure it has sufficient effluent temperature for the CBA regeneration step. In the CBA process, subsequent catalytic Claus reactors, termed CBA reactors, downstream of the initial hot Claus reactor, operate at temperatures below the sulphur dewpoint of the gas. The elemental sulphur produced from the Claus reaction

in the CBA reactors condenses on the catalyst in the reactors. Two key performance advantages are obtained using these cool, sub-dewpoint CBA reactors, both of which are responsible for CBA's enhanced sulphur recovery efficiency compared with conventional sulphur plants:

- Increased Claus conversion of H₂S and SO₂ to elemental sulphur because of improved thermodynamic equilibrium of the Claus reaction at lower temperature:



- Adsorption of elemental sulphur vapour onto the catalyst surface as a condensed phase, reducing the sulphur concentration in the gas to levels significantly below the vapour-liquid equilibrium saturation value achieved in a sulphur condenser.

The CBA reactor, in its cool, sub-dewpoint operating mode, is an unsteady-state process, with elemental sulphur continually accumulating inside the catalyst bed. After a period of time, the bed is heated using hot effluent gas from a hot upstream conventional Claus reactor to drive off the accumulated sulphur and regenerate the reactor. After regeneration, the reactor is cooled and placed back into the sub-dewpoint operating condition, and another CBA reactor then undergoes regeneration. Other sub-dewpoint processes exist, such as the Sulfreen and MCRC processes, but differ from CBA in the method used for regeneration of the sub-dewpoint reactor^{2,3}.

The CBA process features the same basic equipment as a modified-Claus unit. Aside from the use of switching valves and certain metallurgy upgrades, there is no difference in equipment used for the CBA process compared with equipment in the conventional modified-Claus process. The CBA reactor beds use the same activated alumina catalyst. The CBA condensers generate lower-pressure steam than modified-Claus condensers, enabling them to cool to lower temperatures for sub-dewpoint reactor operation. There is no reheat step between CBA reactors. Either two-way or three-way switching valves are used to cycle the process gas flow through the hot and cool reactor steps in much the same way a molecular-sieve unit is operated for dehydration. To regenerate a CBA bed, the condenser downstream of the conventional Claus reactor is bypassed, sending hot gas to the first CBA bed, which heats and regenerates the bed. After regeneration is complete, the Claus condenser is again placed in service to cool the first CBA bed to operating temperature.

Previous CBA Designs

There are several different CBA flow schemes which have been considered in the past⁴. A flowsheet like that shown in Fig. 2 has proven to be an optimum configuration considering both capital and operating costs to achieve sulphur recovery efficiencies ranging from 98.5-99.0%. This flowsheet uses two CBA reactors.

To reach sulphur recovery efficiencies in the 99.0-99.3% range, past CBA projects have applied a flowsheet like that shown in Fig. 3 as an optimum approach⁵. This flowsheet uses three CBA reactors. Flow is always sequential through all three CBA reactors, with their sequence rotated

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ISSUE 357
MARCH-APRIL 2015

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to bring each CBA reactor to the lead position downstream of the Claus reactor for regeneration. The order in which the CBA reactors are sequenced is important for optimum sulphur recovery.

Regardless of the chosen CBA flowsheet arrangement, each CBA reactor cycles through the following modes of adsorption and desorption (in order):

- **Adsorption:** The CBA reactor is cool, below the sulphur dewpoint, allowing liquid sulphur to condense directly onto the catalyst as it is produced.
- **Heat-up:** The CBA reactor is placed in the first position downstream of the Claus reactor. The Claus condenser is bypassed, sending hot gas to the CBA reactor, heating it from sub-dewpoint to regeneration temperature. The Claus reaction diminishes with increasing temperature, eventually ceasing as temperatures rise.

- **Plateau:** The catalyst bed approaches an intermediate bulk temperature where remaining sulphur is steadily vaporised and immediately recondensed in the downstream condenser.
- **Heat soak:** The catalyst bed is brought to a near uniform temperature from top to bottom ensuring complete regeneration.

- **Pre-cool:** The Claus condenser is placed back into service, sending cool inlet gas to the CBA reactor. The freshly regenerated catalyst bed begins cooling. As cooling progresses, the exothermic Claus reaction is initiated which places a lower limit on the ultimate temperature reached while the reactor is cooling in the 1st reactor position. It is impossible to completely cool a CBA reactor to a low sub-dewpoint operating temperature while it is in the first position; the Claus reaction heat prevents cooling the latter part of the bed below about 177°C.

- **Final cool:** To reach a low sub-dewpoint temperature throughout the catalyst bed of about 127-149°C, the bed is switched to a position behind another CBA reactor, where it completely cools for adsorption service.

Improved 3-bed CBA switching sequence

As mentioned, in the pre-cool step it is not possible to completely cool a CBA reactor bed to sub-dewpoint operating temperature because of the Claus reaction. There is a significant amount of H₂S and SO₂ in the gas leaving the upstream Claus reactor. As the reactor cools, the Claus reaction equilibrium becomes favoured toward elemental sulphur, so the progress of the exothermic Claus reaction increases the catalyst bed temperature. Therefore, the CBA reactor must be placed behind another CBA reactor for it to cool to optimum sub-dewpoint operating temperature.

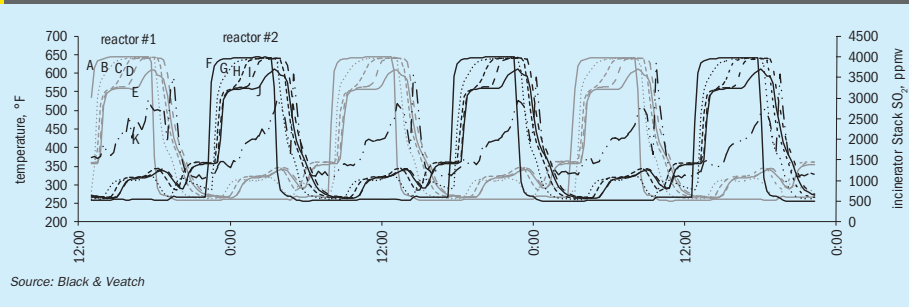
In previous 3-bed CBA designs⁶, a freshly-regenerated CBA reactor, after the pre-cool step, would be placed in the back/final position after regeneration. The back reactor would be moved up to the middle position, and the middle reactor moved up to the first position, and the freshly-regenerated but incompletely cool reactor placed in the back position where it could completely cool. Then the reactor now in the first position would undergo its regeneration cycle. These reactor sequencing steps for these previous designs are given in Table 1 below.

On previous projects, spikes in emissions were observed when a precooled, but still warm, CBA reactor was switched to the final position. After the last CBA reactor became completely cool, emissions returned to normal. The temperature of the final CBA reactor greatly influences stack

Table 1: Prior 3 bed switching sequence

Step no.	CBA reactor no.1	CBA reactor no.2	CBA reactor no.3	Typical duration
1	FRONT – Precool	MIDDLE – Adsorb	BACK – Adsorb	3 hrs
2	BACK – Final Cool	FRONT – Adsorb	MIDDLE – Adsorb	3 hrs
3	BACK – Adsorb	FRONT – Heat Up	MIDDLE – Adsorb	9 hrs
4	BACK – Adsorb	FRONT – Plateau	MIDDLE – Adsorb	
5	BACK – Adsorb	FRONT – Heat Soak	MIDDLE – Adsorb	
6	BACK – Adsorb	FRONT – Precool	MIDDLE – Adsorb	3 hrs
7	MIDDLE – Adsorb	BACK – Final Cool	FRONT – Adsorb	3 hrs
8	MIDDLE – Adsorb	BACK – Adsorb	FRONT – Heat Up	9 hrs
9	MIDDLE – Adsorb	BACK – Adsorb	FRONT – Plateau	
10	MIDDLE – Adsorb	BACK – Adsorb	FRONT – Heat Soak	
11	MIDDLE – Adsorb	BACK – Adsorb	FRONT – Precool	3 hrs
12	FRONT – Adsorb	MIDDLE – Adsorb	BACK – Final Cool	3 hrs
13	FRONT – Heat Up	MIDDLE – Adsorb	BACK – Adsorb	9 hrs
14	FRONT – Plateau	MIDDLE – Adsorb	BACK – Adsorb	
15	FRONT – Heat Soak	MIDDLE – Adsorb	BACK – Adsorb	

Fig 4: CBA reactor temperature profiles and stack SO₂, two-bed flowsheet



Source: Black & Veatch



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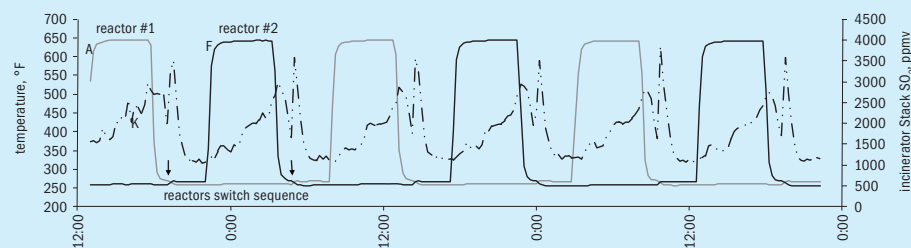
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Fig 5: CBA reactor inlet temperatures and stack SO₂, selected curves from Figure 4



Source: Black & Veatch

emissions due to the favoured Claus reaction and sulphur adsorption equilibriums at lower temperatures. Operating data from existing CBA plants are presented in Figs 4 and 5 to illustrate the sulphur recovery efficiency penalty by not having a completely cooled bed in the final position. These data are from a two reactor configuration, but the same trends also apply to a three reactor configuration following a switch in bed sequence, when a precooled but still-warm CBA reactor is switched to the final position.

Figure 4 presents actual temperature profile data for the two CBA reactor configuration. The grey curves A-E represent CBA reactor no.1. The black curves F-J represent CBA reactor no. 2. Curves A and F represent the reactor inlet temperatures, curves E and J represent the reactor outlet temperatures, and the broken curves B, C, D and G, H, I represent intermediate bed temperatures partially down the beds. Curve K represents the incinerator stack SO₂ concentration. In the first hump in Fig. 4, reactor no. 1 is in the lead position, reactor no. 2 in the lag position. In the second hump, reactor no. 2 is in the lead position, reactor no. 1 in the lag position. In the third hump, reactor no. 1 is again in the lead position, reactor no. 2 in the lag position. Reactor positions continue to alternate.

Figure 5 shows the same curves A, F, and K from Fig. 4. The points where curves A and F, the reactor inlet temperatures, cross each other represent points in time a few minutes after a switch in lead/lag reactor position occurred. Notice that curve K, the incinerator stack SO₂ ppmv value, has a spike which occurs immediately after each time the reactors switch position. This spike is caused by the still-warm reactor which is placed in the final position

Table 2: Calculated equilibrium tail gas sulphur content over alumina catalyst

Final CBA reactor outlet temperature °F/°C	Equilibrium elemental sulphur vapour in CBA tail gas at saturation, lbmoles/hr	Incinerator SO ₂ , ppm
260 / 127	0.13	31
300 / 149	0.68	162
350 / 177	2.94	700
400 / 204	10.3	2,451
450 / 232	35.3	8,408

Table 3: Improved 3-Bed switching sequence

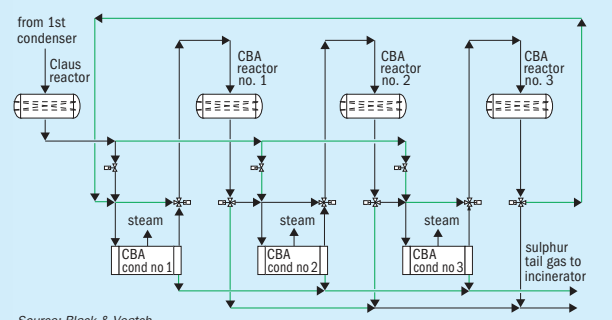
Step no.	CBA reactor no.1	CBA reactor no.2	CBA reactor no.3	Typical duration
1	FRONT – Precool	MIDDLE – Adsorb	BACK – Adsorb	3 hrs
2	MIDDLE – Final Cool	BACK – Adsorb	FRONT – Adsorb	3 hrs
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14	FRONT – Plateau	MIDDLE – Adsorb	BACK – Adsorb	
15	FRONT – Heat Soak	MIDDLE – Adsorb	BACK – Adsorb	

after the switch. The emissions spike tails off as the lag reactor cools completely to optimum sub-dewpoint temperature.

We do not know the exact nature of the cause of these emissions spikes, we only know that they occur. The cause could be the reverse of the Claus reaction, where adsorbed sulphur on the still-warm, latter portion of the bed converts back into H₂S

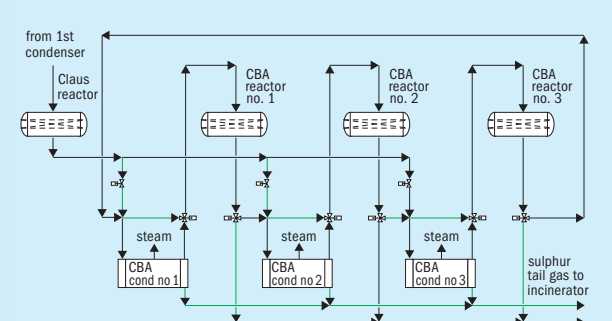
and SO₂. Or, it could be residual adsorbed sulphur being stripped from the still-warm, latter portion of the regenerated bed by the gas which has already had its sulphur vapour content reduced in passing through the cooler, first portion of the bed. If the plant has a limit in permitted SO₂ emissions in any one-hour period, these spikes can be troublesome.

Fig 6: First sequence of beds in improved 3-bed CBA configuration, with reactor 1 in front, reactor 2 in middle, and reactor 3 in back position



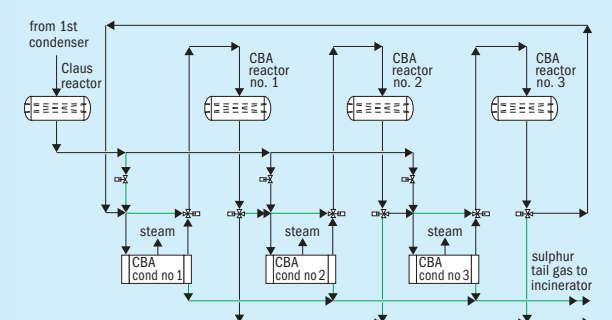
Source: Black & Veatch

Fig 7: Second sequence of beds in improved 3-bed CBA configuration, with reactor 3 in Front, reactor 1 in Middle, and reactor 2 in back position



Source: Black & Veatch

Fig 8: Third sequence of beds in improved 3-bed CBA configuration, with reactor 2 in front, reactor 3 in middle, and reactor 1 in back position



Source: Black & Veatch

Calculated adsorption equilibrium amounts of sulphur vapour at various temperatures over catalyst are shown in Table 2 and illustrate the limitations of warm catalyst in adsorbing elemental sulphur. In Table 2 the tail gas rate is assumed constant at a flow consistent with a ~650 t/d CBA plant. The pre-cool step brings the outlet temperature of the cooling, freshly-regenerated CBA reactor to the range of 204°C-232°C. As can be seen from Table 2, the catalyst cannot adsorb sulphur vapour to low levels in this temperature range.

A key advantage of the 3-bed CBA flowsheet, and one that has not yet been taken, is to place the freshly-regenerated, but incompletely cooled CBA reactor in the middle position instead of the final position after the pre-cool step. In the middle position the CBA reactor will cool to sub-dewpoint temperature, like it does in the final position, because in the middle position it is also placed behind another CBA reactor. By switching the pre-cooled CBA reactor to the middle position and not the final position, we avoid placing a still-warm reactor in the final position and causing a spike in emissions. Sulphur recovery efficiency is improved. Table 3 is a revision to Table 2, showing the reactor sequencing steps of the improved 3-bed switching sequence.

Improved 3-bed condenser arrangement

A 1991 Amoco patent describes an arrangement of CBA reactors and condensers where a CBA reactor is paired with the condenser immediately upstream, the pair switched as a unit to alternately sequence the CBA reactors in the flow path⁷. One advantage of this arrangement described in reference 7 is that one condenser may be deleted from the previous 3-bed flowsheet shown in Fig. 3. In Fig. 3 the condenser downstream of the last CBA reactor flowing to the incinerator condenses no sulphur. Sulphur vapour concentration in the gas leaving a sub-dewpoint CBA reactor is below what will condense at the temperature of the condenser.

Figures 6-8 below show a 3-bed CBA scheme implementing both the condenser arrangement in reference 7 and the improved 3-bed switching sequence described in Table 3. The bed switching sequence is from Fig. 6, to Fig. 7, to Fig. 8, back to Fig. 6, and so on. This condenser arrangement, together with the improved 3-bed switching sequence, allows for a further improvement to sulphur recovery efficiency.

Improved condenser operation

To further improve the overall sulphur recovery efficiency, the CBA reactor in the final position can be operated at an inlet temperature approaching the sulphur freezing temperature of ~116°C, which improves Claus conversion and the adsorption of product sulphur on the catalyst. Previous CBA designs have used condensers which were kept at some margin above 116°C to avoid the deposition of solid sulphur in the condenser. If the temperature of the last condenser's tubes is reduced to below 116°C, the gas can be cooled to very near 116°C and the last CBA reactor operated at a temperature significantly below previous designs for improved sulphur recovery efficiency.

The potential drawback to reducing the last condenser's temperature below the sulphur freezing point is that solid sulphur will accumulate in the condenser tubes. However, the condenser-reactor unit switching scheme described in Figs 6-8 provides for quickly warming this condenser when the last CBA reactor-condenser unit is switched to the first position for regeneration. In the first position hot Claus reactor effluent gas flows to the cool condenser, quickly melting any accumulated sulphur. While the condenser is at a sub-freezing temperature in the last position, the accumulation of sulphur in the condenser will be an amount insufficient to cause significant plugging, because the gas entering the last condenser has very little sulphur vapour present since it is entering the final conversion stage and the majority of the sulphur has already been recovered upstream.

In this scheme the shell side of the condensers in the first two positions are operated at a typical CBA condenser shell-side temperature of about 118°C, and the condenser in the last position is operated at a shell-side temperature below 116°C, sufficient to produce an outlet gas temperature of 116°C. At the cool condition the condenser sees in the last position, it has a large excess of surface area, because it is designed to condense a lot of sulphur from the effluent of a hot regenerating reactor in the first position. Accumulated sulphur fouls the last condenser's heat transfer surface, but the large excess surface area still allows the condenser to operate effectively.

Lowering the temperature of the final reactor improves sulphur recovery efficiency in two ways. First, the Claus reaction equilibrium is more favoured by the lower tempera-

Fig 9: Profile of last CBA reactor outlet temperature

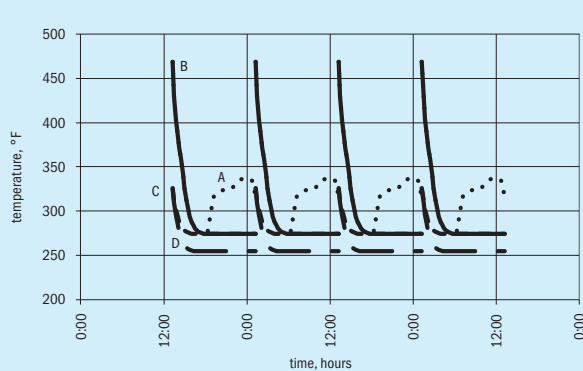


Table 4: Comparison of calculated step-wise average sulphur recovery efficiency for improved 3-bed CBA configuration vs. previous 3-bed design

Step	Duration (hours)	Sulphur recovery efficiency revised 3-bed CBA	Sulphur recovery efficiency prior 3-bed CBA
Precool	3.0	99.52%	99.36%
Final cool	3.0	99.55%	99.43%
Heat up	4.1	99.55%	99.43%
Plateau	1.4	99.60%	99.35%
Heat soak	1.9	99.64%	99.45%
Overall average for cycle	13.4	99.56%	99.41%

Table 5: Acid gas feed composition basis used for Table 4

Component	lbmoles/hour
Carbon dioxide	135.91
Hydrogen sulphide	100.47
Methane	0.86
Water	10.31
Total	247.55

ture, allowing a greater conversion of H₂S and SO₂ to elemental sulphur. Second, the catalyst bed will adsorb a greater amount of elemental sulphur vapour at the lower temperature.

Performance comparison with previous and alternative designs

Figure 9 illustrates the envisioned outlet temperature profile for the final CBA reactor for the proposed changes:

- Curve A is the final reactor outlet temperature profile for the 2-bed design shown in Fig. 2. The first part of curve A is the same path traced by curve B.
- Curve B is the final reactor outlet temperature profile for the previous CBA 3-bed design shown in Fig. 3.
- Curve C is the final reactor outlet temperature profile for the improved 3-bed switching sequence of Figs 6-8, but with conventional CBA reactor inlet temperature of about 127°C.
- Curve D is the final reactor outlet temperature profile for the improved 3-bed switching sequence of Figs 6-8, using a colder final reactor inlet temperature of 116°C.

Minimising the final reactor outlet temperature over the complete cycle is important

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to achieve optimum sulphur recovery efficiency. In Fig. 9, curve D is the coolest through the overall cycle, and hence obtains the highest sulphur recovery efficiency.

The combined effect of the improved bed sequence and cooler final CBA reactor operating temperature for the 3-bed CBA flowsheet, represented by Curve D in Fig. 9, is shown in Table 4 below. Both cases in Table 4 consider a typical acid gas feed for a gas plant application, given in Table 5. Note that the case study feed is not particularly rich in H₂S and contains a fair amount of CO₂. The figures in Table 4 consider a Claus reactor having only activated alumina catalyst, so the overall level of recovery in this instance could be further improved by using either a promoted alumina catalyst or titania in the Claus reactor. Improvement in Claus reactor COS/CS₂ conversion can be particularly advantageous for CBA applications given the reduced COS/CS₂ hydrolysis conversion across CBA reactors, because of their low temperature, and the sizable contribution of unconverted COS/CS₂ to SO₂ emissions at high levels of recovery.

All equipment sizes for the results in Table 4 are identical. Table 4 only considers the changes illustrated with respect to bed sequencing and final bed inlet temperature. These calculations do not alter the bed sequence durations, which impact the rate at which the CBA beds load up with sulphur. Bed sequence durations are typically optimised in the field based on actual conditions. Overall sulphur recovery is calculated based on the length of time for each step in the sequence and the average recovery calculated for that step. The improvement in this case amounts to an increase of 0.15% in average sulphur recovery efficiency, which equates to a reduction in SO₂ emissions of 25% for the improved 3-bed design compared with the previous 3-bed design.

The results are of a theoretical and calculated nature, based on observed performance of past CBA units. Factors which affect sulphur recovery, such as combustion air control to achieve optimum H₂S:SO₂ ratio, possible leaks in switching valves, and the health of the catalyst in the reactors, also influence the achieved sulphur recovery efficiency. Field testing of these improvements can be accomplished in any 3-bed CBA unit to establish the reduction in emissions which can be achieved.

Fig 10: Sulfree™ process scheme

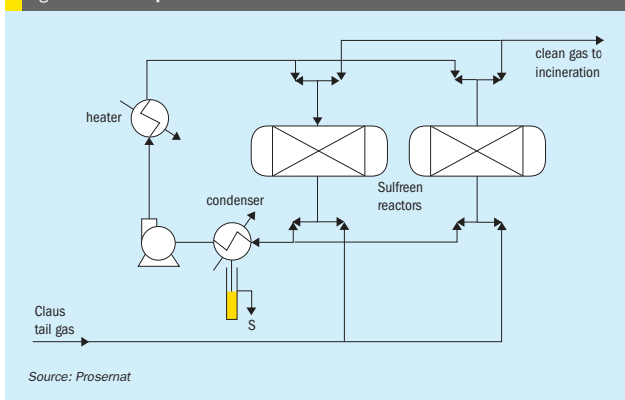


Fig 11: Sulfree™ unit at Lacq natural gas field (France)



Sulfree™: A pioneer in dry sub-dewpoint processes

Sulfree™ was the first sub-dewpoint process to be introduced. Invented in the late 1960s, the aim of the process was to providing overall sulphur recovery from Claus plants through the implementation of tail gas treatment systems to decrease SO₂ emissions to the atmosphere. The technology was originally developed by SNPA (now Total) in association with Lurgi. The first Sulfree™ commercial units were started in 1970 in Lacq, France (1,200 t/d, see Fig. 10) and Ram River, Canada (2,200 t/d) gas plants. The concept of the sub-dewpoint process has since been widely used in many tail gas treatment plants worldwide.

The original idea of the technology can basically be seen as an extension of the Claus process, at an operating temperature below the sulphur dewpoint (approximately 130°C). These specific operating conditions resulted in the substantial enhancement of sulphur yield due to the combined effect of:

- the low temperature which thermodynamically favours the yield of the Claus reaction, and thus the formation of elemental sulphur;
- the constant removal of sulphur from the reacting gas through adsorption within the porous media of catalyst and the minimisation of the sulphur vapour losses even below equilibrium.

This resulted in a multi-sequence process design with a minimum of two reactors

working alternatively to produce and accumulate sulphur during an adsorption reaction phase, and subsequently release the accumulated sulphur during a regeneration phase. This regeneration is achieved by circulating in close loop hot tail gas over the reactor being regenerated. The hot gas vaporises the accumulated liquid sulphur which is then recovered in an external condenser. Figure 11 shows the basic process scheme.

Over the years, the Sulfree™ process has been improved to avoid problems such as SO₂ peak during regeneration, sulphate deposits in case of O₂ breakthrough and sequential valve failure, experienced in some early designs.

Today, Sulfree™ units can be designed for reliable operation achieving 99 to 99.9% recovery.

Up to 99.9% recovery with HydroSulfree™ and DoxoSulfree™

The technological breakthrough accomplished by the basic Sulfree™ process further benefited from significant developments in the field of catalysis, and subsequent process developments such as HydroSulfree™ (enhanced COS/CS₂ hydrolysis), which was able to reach a specification of 99.5% recovery.

However, these recovery levels are close to the thermodynamic limits of the Claus reaction.

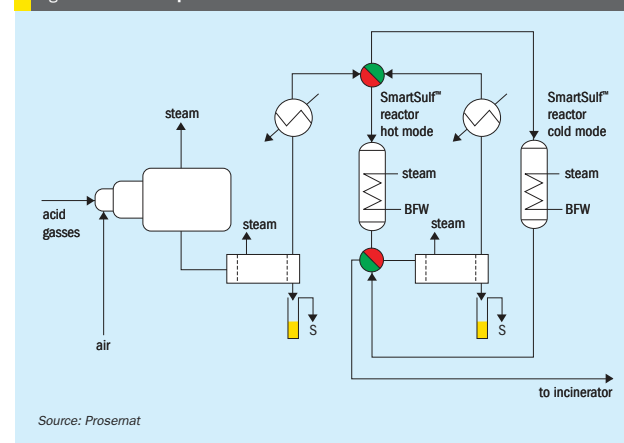
A promising new sub-dewpoint process was commercially launched by Elf/Lurgi in the early 2000s with the target was to increase the recovery up to 99.9%. This required a new chemical approach for the production of sulphur from H₂S to be considered.

From the experience acquired with Sulfree™, it seemed sensible to maintain the basic concept of totally catalytic operation below sulphur dewpoint. This has the following advantages:

- production of pure sulphur;
- absence of side effluent streams, thus no side treatment required;
- an operation similar to the Sulfree™ cyclic process using a proven technology which is easy to operate.

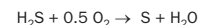
A new route was investigated to achieve a 99.9% recovery by adding a final direct oxidation step after the sub-dewpoint Claus reaction in order to convert the residual H₂S into sulphur in the presence of air. This process scheme has been patented and named DoxoSulfree™. In the first step, the tail gas from the Claus plant has an H₂S/SO₂ ratio controlled to obtain a slight

Fig 12: SmartSulf™ process scheme



Source: Prosermat

excess of H₂S compared to the required quantity to maintain the Claus ratio. After cooling to 125°C, the tail gas is fed to a conventional Sulfree™ catalyst bed where the Claus reaction takes place and converts most of the SO₂ due to the excess of H₂S. The gas is then cooled to 90-100°C and fed to a second catalytic stage together with a stream of air so that the direct oxidation of the residual H₂S can take place according to the following equation:



Both beds are regenerated through a common regeneration loop.

A 99.9% recovery performance is equivalent to 250 to 300 ppmv of total sulphur at the outlet of the oxidation bed. The key to the development of the DoxoSulfree™ process was the specific oxidation catalyst. The formulation was able of achieving the necessary specifications of the process:

- high conversion efficiency (> 95%) of H₂S with no trace of SO₂ emission (almost 100% selectivity) throughout a 24 hour period of adsorption reaction within a relatively low range of temperature (90 to 130°C);
- good flexibility with the possibility to work at O₂/H₂S ratio as high as 3 to 4 mol/mol, thus avoiding constraints of sharp ratio adjustments such as for Claus base systems;
- maintenance of the selectivity over a wide range of residence times, adding to the flexibility of the process.

Catalyst ageing was tested on the laboratory scale under the conditions relevant to the process for periods as long as six months on-stream, including under high severity conditions, without deactivation.

The first pilot plant was installed in Lacq, France, to assess the performance level on an industrial gas stream. The work carried out during the project was aimed at demonstrating the effectiveness of the technology to recover up to 99.9% sulphur on the Elf site of Lacq. The pilot was tested from September 1998 to April 1999 confirming the excellent performance of the process.

The SmartSulf™ process: lowest capex/opex sub-dewpoint process

The concept of using internally cooled Claus catalytic reactors in order to achieve high sulphur recoveries in a 2-stage Claus unit was originally developed by Linde under the name ClinSulf® and this process was then further developed by its inventor under the tradename of SmartSulf™ in its own founded company: ITS Reaktor Technik.

In 2014, Prosermat completed its technology portfolio with the acquisition of ITS Reaktor Technik, owner of the SmartSulf™ technology. This technology has industrially proved its reliability after continuous developments and many reliable industrial references in refineries as well as in a direct oxidation mode for lean acid gases.

The SmartSulf™ process employs unique, identical isothermal catalytic reactors to

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achieve up to 99.7% sulphur recovery in a 2-stage Claus train, without a tail gas treating unit. Figure 12 shows a typical process diagram.

In essence, sulphur conversion efficiency is maximised by operating the lower bed of the No.2 reactor at sub-dewpoint.

The isothermicity of the reactors is achieved by installation, in the lower part of the reactor, of a thermoplates heat transfer bundle, which removes the heat of reaction, to maintain the catalytic bed at its optimum temperature:

- Operation of the No.1 Reactor in a conventional Claus unit is always a compromise between higher temperatures favorable to COS/CS₂ conversion (kinetics), and lower temperatures favorable to Claus reaction equilibrium (thermodynamic – Le Chatelier’s principle). Furthermore because the Claus reaction is exothermic, the temperature increases toward the end of the reactor, which limits the outlet equilibrium conversion.
- In the SmartSulf™ reactor, the upper bed (containing no heat removal provisions) is operated hot enough (300-350°C at the outlet) to achieve high COS and CS₂ conversion, while the lower is cooled to slightly above the sulphur dew point (250-270°C) for maximum sulphur conversion. The combination of those two temperature zones is unique to SmartSulf™ design and allows higher sulphur conversions than in a conventional Claus reactor.

Boiler water is circulated in the thermoplates, where heat is removed by water vaporisation. Waste steam is then disengaged and re-condensed in a closed-loop steam condenser. During steady-state, the No.1 reactor steam drum pressure is typically 40 kg/cm²g. In order to optimise the lower bed temperatures, the loop pressure is automatically adjusted by throttling condensate flow from the steam condenser.

Sulphur conversion efficiency at the No.1 Reactor outlet is already greater than 90%. The converted sulphur is then condensed in the sulphur condenser. The gas temperature is then increased in a reheater to ensure operation above the sulphur dew point in the top section of the No.2 reactor.

Once Claus reaction equilibrium has been achieved in the upper bed, the thermoplates heat exchanger cools the lower

bed to 100-125°C by controlling the closed-loop pressure at approximately 1 kg/cm²g. The lower temperatures further shift the Claus reaction equilibrium which, combined with lower sulphur vapour losses, results in much higher recovery efficiencies than other traditional sub-dewpoint processes.

Sub-dew-point operation in the lower bed of the 2nd Reactor gradually fills the catalyst pores with liquid, and potentially solid, sulphur. Before the catalyst activity is lost by sulphur loading depending on plant capacity, regeneration is accomplished by simply reversing the sequence of the two reactors. The original No.1 Reactor is free of sulphur and is switched to cooler No.2 reactor service. The sulphur-loaded No.2 reactor then becomes the No.1 Reactor, where the higher operating temperatures drive the sulphur out of the catalyst pores. Switching is accomplished by proprietary 4-way valves especially developed for this application with the aim to minimise the number of sequential valves compared to other traditional processes.

Like any Claus based processes, the performance is related to the accuracy on the H₂S/SO₂ ratio and sub-dewpoint processes are particularly sensitive to any off-ratio. To overcome this issue, Prosernat now features a proprietary and specific air control system which allows guaranteeing recoveries up to 99.7% in an industrial environment where H₂S/SO₂ ratio fluctuations are common.

Prosernat believes that SmartSulf™ solution compared to other technologies, offers some key advantages, in particular:

- By using internally cooled reactors, higher sulphur recovery can be achieved than in any other similar sub-dewpoint reactors.
- Total number of equipment items is minimised and accordingly capex are reduced.
- Plot area requirement is the smallest in the market.
- Fully automatic process: maintenance is similar to conventional Claus units.
- No effluent or by-products are produced such as sour water.
- Easy retrofit of conventional Claus configuration to SmartSulf™ configuration.

The SmartSulf™ technology is backed-up by 13 industrial references sold over 20 years, among which:

- 9 have been put into operation, the last one came on stream in 2014;

- 4 are under construction, with two units awarded in 2014;
- 6 have been sold to repeat customers.

The future for sub-dewpoint processes

The dry sub-dewpoint processes have many advantages: higher sulphur recoveries due to the low operating temperatures, in the Claus reaction or direct oxidation mode, no side streams, and, despite their cyclic nature, easy operation which can be fully automatised.

From the original late 1960s concept of operating a Claus reactor at sub-dewpoint temperatures many improvements have been developed both for higher sulphur recoveries and reduced number of equipment with lower capex/opex. Considering the past and ongoing developments it is expected that in the future the use of sub-dewpoint operation shall continue to play an increasingly important role in refinery and gas plants operators’ strategies to reduce SO₂ emissions, even for the most stringent emission standards. ■

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SULPHUR
ISSUE 357
MARCH-APRIL 2015

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Direct reduction of SO₂ to elemental sulphur

Le Gaz Intégral (LGI) has developed a process to recover elemental sulphur from the SO₂ present in the flue gas of nickel and copper ore production facilities. **Frank Cross** of LGI reports on the process, which involves reduction of sulphur dioxide by methane. This had been attempted in the past without much success, primarily due to the formation of soot. LGI performed extensive laboratory and pilot plant tests to identify in which conditions SO₂ and CH₄ would react without soot formation. The tests were successful and allowed the identification of soot free operating conditions, but also revealed a number of new issues, related to the introduction of fluids into the reaction furnace, as well as side reactions over the catalyst. LGI has performed extensive research and tests to come up with a solution to these issues.

The ore reserves at mines in the Taimyr Peninsula in the Russian Arctic region near the city of Norilsk are very rich in sulphur. Processing the ores in the smelter works creates, as by-product, large amounts of atmospheric emissions of sulphur dioxide (SO₂).

Emissions of SO₂ lead to acid rain, affecting vegetation, soils, rivers, flora and fauna. SO₂ is harmful to human life and can lead to respiratory illness.

Norilsk Nickel decided to cut these emissions by recovering elemental sulphur from the flue gases. Elemental sulphur will be recovered as a solid and either stored locally or shipped (see Fig. 1).

The existing plant flash smelters flue gas treatment comprises waste-heat boilers for gas cooling followed by dust separation. The gases are then sent to stack.

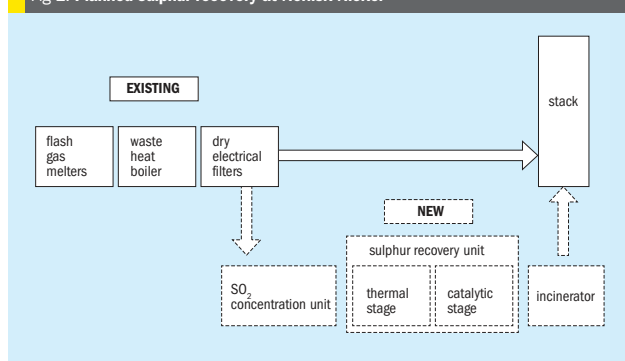
A SO₂ concentration unit followed by a sulphur recovery unit will be added.

Sulphur will be recovered in a two stage process: high temperature reaction of SO₂ and CH₄, followed by hydrolysis of COS and CS₂ and reaction of SO₂ and H₂S (Claus reaction) to form sulphur over a catalyst.

The first stage of the sulphur recovery unit has been attempted numerous times in history.

In principle SO₂ and methane can react directly to make sulphur and catalysts have been used to promote this reaction.

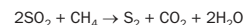
Fig 1: Planned sulphur recovery at Norilsk Nickel



However, the high temperatures generated by the process if used with a concentrated feed limit its practical application.

Attempts made by first producing reducing gas (CO+H₂) from methane, then reacting the reducing gas with SO₂ have been hampered by soot formation.

LGI developed an innovative method for carrying out the direct thermal reduction of SO by natural gas, without going through the intermediate reducing gas step, summarised in the chemical reaction:

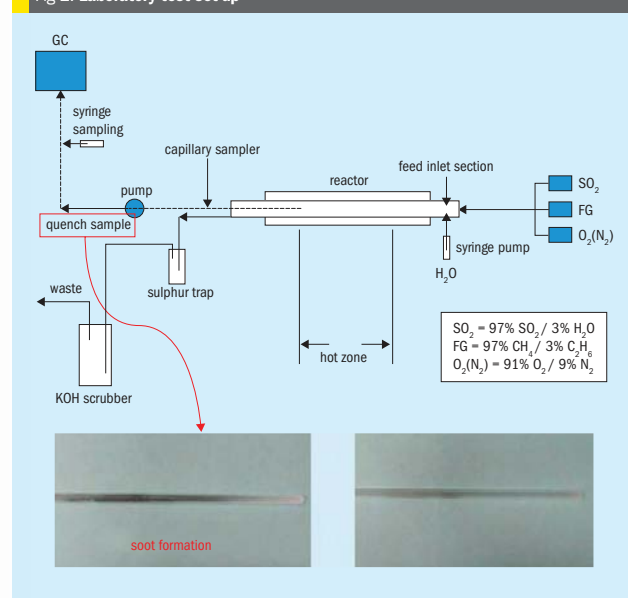


Historical information indicates that the above reaction should take place at high temperature, around 1,300°C if soot is to be avoided.

The needed high temperature is achieved partly by the reaction of SO₂ and CH₄ and partly by reacting CH₄ in the CH₄/SO₂ mixture with oxygen. The reaction of SO₂ with methane to form sulphur is only partial due to the thermodynamic equilibrium. In addition, other reactions take place and H₂, CO, COS, CS₂ and H₂S are formed.

A second reaction step is therefore provided in the process, where the remaining

Fig 2: Laboratory test set-up



SO₂ is reacted with H₂S in conventional Claus catalytic converters and COS and CS₂ are hydrolysed.

As the catalyst would be poisoned by the presence of soot, it is critical to establish conditions in the thermal stage of the process in which soot will not form.

For the total reaction mixture (O₂ + SO₂ + CH₄ and heavier hydrocarbons from natural gas) soot is not an equilibrium product, but its formation is possible depending on the mixing of the reactants.

The combustion of CH₄ with oxygen to make heat takes place with a deficiency of oxygen in order to only use part of the CH₄ present in the SO₂/CH₄ mixture.

Soot is likely to form in these sub-stoichiometric conditions, due to dissociation of CH₄ and other hydrocarbons into C and H₂, or by the introduction of cold gas into flame zones.

Soot formation can be suppressed by injection of steam but this increases the volume of process gas, reduces the reaction temperature and adversely affects the Claus reaction equilibrium.

Soot can also be avoided by providing correct gas mixing and enough residence time for soot to react with other components. The available thermodynamic simu-

lators do not, however, allow to model soot formation or removal, or the effect of mixing the feed gases.

Thermodynamic simulators only approximately model the thermal reactor, the waste heat boiler and (as was discovered during the test runs) the catalytic reactors.

This prompted LGI to perform tests both in the lab (Fig. 2) and at a pilot unit (Fig. 3).

The first set of tests was done at the lab by Alberta Sulphur Research Ltd (ASRL). As soot formation was thought to be affected by the way the reactants were introduced in the reactor and their residence time, different schemes were tested.

In a first series of test (single section furnace), all reactants were introduced at the inlet of the reactor. In a second series of tests (two-section furnace) part of the reactants were introduced in the inlet of the reactor and part further downstream. Different residence times were tried.

The presence of soot in the reactor effluent could not be detected by analysis of the effluent by gas chromatography, as for other components, since soot is not a gaseous component. It was detected by visual observations of the quench tube leaving the reactor which corroborated

the results of a carbon balance calculated from the gas analysis.

Testing at increasing residence time clearly showed that adequate selection of the residence time avoided soot.

Test results also provided effluent composition which was compared to the one given by the computer simulator (Sulsim from Aspen Technology, Inc.). The two were quite close which gave confidence in the model, although the test results showed less H₂ and CO, more COS in the effluent and more sulphur conversion than the model predicted.

The mechanism for the formation of soot is complex and affected by mixing patterns of the reactants (natural gas, oxygen and SO₂). This will be different in a laboratory scale apparatus and at industrial scale. The design of the burner of the industrial furnace is likely to have a significant role in the way reactants are mixed.

This prompted LGI to perform tests at a pilot unit. The pilot unit was an adaptation of a commercial 10 t/d two stage Claus unit using oxygen enrichment, which had been operated for about 20 years.

The unit was adapted to the furnace residence time established during the laboratory tests. Purposely made burners were designed and manufactured (Fig. 4). The selected catalysts were loaded in the reactors in the quantity required to achieve the chosen space velocity.

As the feed stock to the sulphur recovery unit of the industrial application would contain very concentrated SO₂, pure SO₂ could be used to model the feedstock. SO₂ was supplied in liquid form and fed to the unit through a vaporiser and pre-heater.

Fig 3: Fit out of a tested burner



Fig 4: Pilot unit



The natural gas used was the refinery fuel gas whose composition was also very close to the one that will be available at the industrial site.

Filter papers were used to detect the presence of soot in gas samples. The produced sulphur was analysed for ash content in the laboratory. Methane break through the thermal reactor was monitored as it is a precursor of soot formation.

Tests at various temperatures in the thermal reactor were carried out. The minimum temperature to prevent soot formation and the corresponding oxygen flow were identified. It was found that a higher oxygen flow than foreseen in the design would be required, perhaps due to the relatively high heat losses from the pilot plant.

The robustness of the process was tested, including operation with an excess or deficiency of one of the reactants (CH_4 , SO_2 , O_2), as will happen during process upsets in an operating plant. The process control concept was validated by operating the unit under full automatic control using a tail gas analyser to maintain the normal 2:1 $\text{H}_2\text{S}:\text{SO}_2$ ratio. Transient operation during change-over from stand-by (burning natural gas) to SO_2 operation was tested to verify the start up and shutdown procedures. Figure 5 shows an operator's view of the process control system of the thermal reactor during the pilot tests.

As process models are very poor at predicting the formation of CS_2 , which happens at low furnace temperature, various furnace temperatures were tested to find the lowest acceptable furnace temperature prior to formation of dark sulphur.

The tests showed that sulphur conversion in the thermal reactor was higher than predicted by the model.

Thermal stage effluent compositions were obtained, that could be fed back into the simulator as inlet to the catalytic stage.

Specific burner design

The tests showed that the thermal reactor burner was a challenge. The minimum SRU high intensity burners were found inadequate. This was thought to be due to the large flow of inert gas (SO_2) entering the burner which made the flame unstable.

Several tests were necessary to select a suitable burner and the way to introduce the natural gas, SO_2 and oxygen streams.

Catalytic reactions

The pilot plant contained two catalytic stages, which allowed testing of the performance of the Claus reactors.

The first catalytic converter has a significant duty to react COS in this application compared to a normal Claus application. The thermal reactor effluent indeed contains 2-3% COS, compared to around 0.1% in a normal Claus process.

Typical Claus catalysts were used to hydrolyse COS and CS_2 and carry out the Claus reaction between H_2S and SO_2 . A higher than usual temperature rise in the reactor was expected due to the COS hydrolysis.

The actual temperature measured in the reactor was, nevertheless, much higher than expected. It meant that some reactions, unseen in a standard Claus unit, were taking place.

The composition of the feed to the first Claus reactor is significantly different from that of a standard Claus unit with a very

Fig 5: View of the thermal reactor during the pilot tests



high CO content and higher than usual H_2 and water contents.

A high temperature rise in the reactor adversely affects the Claus reaction equilibrium and reduces the sulphur conversion. There is also the potential to damage catalyst and equipment.

This prompted LGI to perform further tests of the catalytic stage. These tests were done at Euro Support BV test premises. The test facilities included multiple small reactors which allowed a large number of test conditions and catalysts to be tried (Fig. 6).

Elemental balance derived from the compositions of the reactor inlet and outlet streams, stoichiometry and thermodynamics allowed to make assumptions about the reaction mechanisms.

This was complicated by the number of species involved and possible combinations. Two unexpected reactions could nevertheless be characterised and their yield mapped against space velocity, catalyst type and reactor temperature (Fig. 7).

The unexpected reactions were found to be very sensitive to the temperature in the reactor and highly exothermic.

Accurate prediction of the temperature in the reactor was essential to confirm sulphur conversion and equipment design. As the test reactor is isothermal and the industrial reactor adiabatic, the heat of reaction had to be accounted for in setting the industrial reactor design temperature.

In the end, the tests allowed to select the catalyst, predict the industrial reactor temperature and effluent composition, and confirm that the required conversion level was achieved.

These results were implemented in the industrial plant design.

Fig 6: Reactors used for catalyst stage tests



Due to the side reactions, the temperature in the first catalytic reactor is significantly higher than in a normal Claus plant, and could affect the activity of the catalyst over its required life.

A very severe ageing process was applied to the catalyst in order to simulate its conditions after three years.

The activity of this severely aged catalyst was then tested.

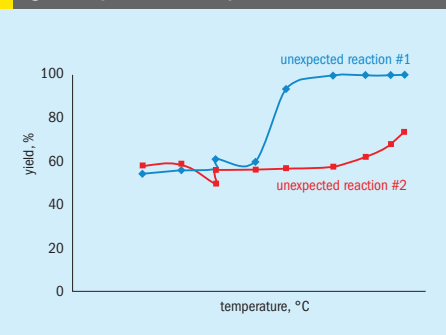
Conclusion

LGI performed extensive testing to design and validate the process of direct reduction of SO_2 by natural gas. Although successful, the tests revealed a number of specific design requirements, including reaction furnace and burner design, catalyst selection and catalyst stage equipment design.

LGI is in the process of patenting this new technology as well as the catalyst.

This technology, selected by Norilsk Nickel for its ore production facilities in the city of Norilsk, will ultimately allow the recovery of one million tons of sulphur every year. This will be a major contribution to the protection of the environment in the Arctic zone.

Fig 7: Unexpected reactions yield



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ISSUE 357
MARCH-APRIL 2015

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Desulphurisation of coke oven gas

New desulphurisation technologies have been developed and implemented to meet the demands of new environmental regulations in steel and power plants. RATE has been involved in the licensing of several grass root sulphur recovery projects including the design of a special acid gas removal process named Coke S-MAX. **M. Rameshni** and **S. Santo** of RATE discuss the Coke S-MAX technology and how it has been implemented in steel and power plants for the processing of coke oven gas to achieve zero emissions.

Coke oven gas treatment

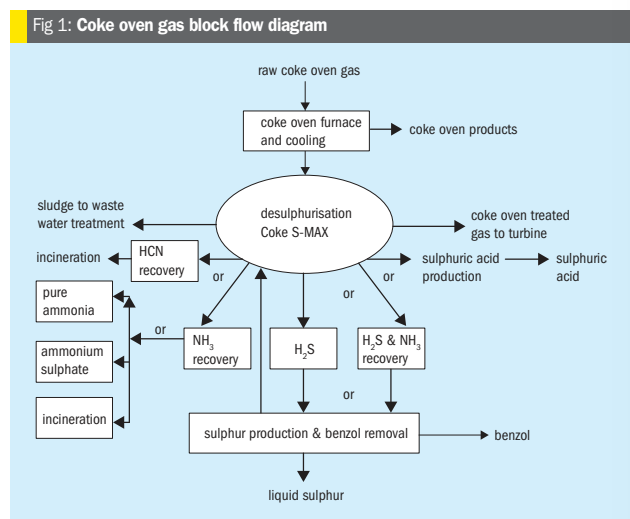
The significance of coke oven gas and its constituents has changed substantially over the past decade. Today, the primary task of a modern coke oven gas treatment facility is to convert crude gas into an environmentally compatible fuel as economically as possible.

Crude gas is cooled, compressed and freed from constituents that might be hazardous to the environment or plant. Apart from the cleaned coke oven gas, the only by-products produced in modern plants are crude tar, crude benzol, and sulphur. The necessary process steps and equipment are chosen to eliminate gaseous emissions and to minimise the level of contaminants in the waste water.

Coke oven furnaces and heaters have improved over the years. Furnaces are available from different companies e.g. ThyssenKrupp Industrial Solutions. In addition, coke oven gas has been treated by different technologies such as Diomax, physical solvents, and many other absorbents but none of them meet the new environmental regulations in a cost effective manner.

Depending upon the characteristics of the coke oven gas different processes can be applied:

- process for desulphurisation of coke oven gas to remove H₂S, ammonia and HCN;
- process for the desulphurisation of ammonia-lean coke oven gas;
- processes for the production of elemental sulphur or sulphuric acid, in simultaneous combination with ammonia cracking in the SRU or incinerator.



In steel plants, after the removal of impurities, coke oven gas is used as a fuel in the integrated steel plant as well as for heating the coke oven battery. The removal of ammonia and hydrogen sulphide from coke oven gas is an environmental necessity. The main components from the coke oven are varying amounts of ammonia, HCN, H₂S and CO₂, which must be processed in the most feasible and economical way to ultimately produce sulphur.

Some power plants fire semi-coke gas and shale gasoline produced as by-products in the production of fuel oil from oil shale to generate electricity and increase

the revenue earning potential of the shale oil co-product semi-coke gas. Units are constructed to treat and combust sour semi-coke gas and shale gasoline from the retorting units using similar components to those in steel plants.

For these specific applications, RATE has developed Coke S-MAX, technologies which remove the H₂S and ammonia from coke oven gas to provide a treated coke oven gas suitable for power or energy use.

Figure 1 represents a simplified block flow diagram for the various options when treating coke oven gas from steel plants.

Coke S-MAX

The Coke S-MAX process is particularly suitable for the removal of H₂S from gases containing ammonia since ammonia is used as the absorbent in the Coke S-MAX solution. Ammonia in the raw gas is absorbed to generate aqueous ammonia; no additional chemicals are required for the process.

Once the Coke S-MAX solution is saturated with ammonia, any additional ammonia will not be absorbed but instead will pass through with the treated gas. Additional ammonia scrubbers or towers must be added if ammonia has to be removed from the treated gas.

A large part of the proprietary design of the Coke S-MAX process is embodied in the design and operation of the absorber and stripping towers, the temperature and the solution concentrations maintained throughout the system.

H₂S removal of up to 100% is achievable compared to 98% H₂S removal for other processes. The organic sulphur compounds cannot be removed. The gases produced by Coke S-MAX are processed in the sulphur recovery unit (SRU) using air or oxygen to produce 99.9% pure high quality sulphur. The SRU is followed by the tail gas unit with low temperature hydrogenation catalyst and the quench system. The overhead of the quench system is recycled to Coke S-MAX so there is no need for an amine section in the tail gas unit.

The treated gas from Coke S-MAX is mostly fuel/hydrocarbons that can be used in the coke oven furnace or incinerator.

Two schemes have been developed; the first for rich H₂S gas containing some ammonia where both H₂S and NH₃ are processed in the sulphur recovery unit (Coke S-MAX1) and the second for lean H₂S gas containing high ammonia where H₂S and NH₃ will be separated (Coke S-MAX2): H₂S is processed in the sulphur recovery unit and the ammonia can be destructured in a special incinerator or sent to a fertilizer unit.

Coke S-MAX1 process description

In the Coke S-MAX1 process, coke oven gas from the coke oven unit is cooled to 52°C by direct contact in the wash tower, where most of the ammonia will stay with the cooled gas.

The cooled gas enters the coke oven gas absorber where it counter-currently contacts with the freshly stripped lean

ammonia solution to remove both H₂S and NH₃. Nearly all of the H₂S, ammonia and a portion of the CO₂ are absorbed before the treated gas exits from the top of the absorber. The rich solution enters the acid gas stripper where the absorbent solution is heated to strip the absorbed acid gases. The lean absorbent is then cooled and returned to the absorber. The stripped acid gas is sent to the sulphur recovery unit to recover the sulphur.

The bottom of the absorber contains rich absorbent solution (containing H₂S and ammonia) which is pumped and heated prior to entering the acid gas stripper. The rich absorbent is processed in the acid gas stripper using a LP steam reboiler. Ammonia is very soluble in water and requires high reboiler duty to be stripped. The reboiler duty has to be carefully calculated to strip all of the H₂S plus some ammonia but to leave enough ammonia in the stripped water so that the solution can be used as an absorbent to absorb the H₂S in the coke oven absorber. The acid gas stripper overhead flows to the wash drum using steam condensate from the reboiler.

The liquid from the wash drum is added to the stripped water from the stripper as a lean ammonia absorbent to wash the coke oven gas in the coke oven absorber. To ensure the absorbent contains enough ammonia to remove the H₂S in the coke oven absorber, a slip stream of the stripped water containing ammonia is added to the absorbent stream to the coke oven absorber.

Since both H₂S and ammonia are processed in the sulphur recovery unit, the purity of the overhead stream is not a factor. This scheme is used where both H₂S and ammonia can be processed in the sulphur recovery unit with or without oxygen to achieve stable combustion temperature, and basically H₂S is relatively rich.

The remaining stripped water enters the ammonia stripper, where LP steam is injected to strip the remaining ammonia and the overhead is sent to the sulphur recovery unit. The water from the ammonia stripper is sent to the water treatment system.

A small amount of solution is purged from the re-circulating absorbent solution to control the build-up of impurities and is then sent to the water treatment system.

The goal of obtaining a high degree of sulphur removal conflicts with that of simultaneously obtaining high H₂S selectivity; one goal must be compromised to

obtain the other. To obtain a high degree of desulphurisation, the partial pressure of H₂S in the absorbent must be low and the acid gas concentration in the regenerated lean solution must be at the lowest possible level. Thus large amounts of solution must be circulated through the absorber and greater amount of utilities consumed in regenerating and recirculating the solution.

The system proposed for a model oil-shale gas desulphurisation plant is based on a spray tower absorber with six absorption stages.

High CO₂ content results in increased CO₂ absorption. Calculations indicate that the H₂S content of the treated gas will be about 63 ppmv and the acid gas stripped from the rich Coke S-MAX1 solution in the acid gas stripper contains about 6 mol-% H₂S. This equates to an H₂S removal of at least 98% and with the gas assuming COS in the range of 10-50 ppmv which will be hydrolysed in the sulphur recovery unit, then followed by the tail gas low temperature hydrogenation catalyst and the quench system.

The quench overhead will be recycled back to the front of Coke S-MAX1. The overall sulphur recovery of 99.99% will be achieved basically "zero emission".

While suitable for treating coke-oven gas containing large amounts of ammonia, carbon dioxide, and hydrogen cyanide, the Coke S-MAX1 process, is only marginally capable of processing oil-shale gases because the ratio of CO₂ to H₂S in the gas is very high. For some direct-fired oil-shale gases the process may not be capable of producing an acid gas of sufficient H₂S content for effective operation of the Claus system, therefore, for those cases that require more effective H₂S removal Coke S-MAX2 is applied.

Coke S-MAX2 process description

Coke S-MAX2 is more comprehensive compared to Coke S-MAX1 because the ammonia is not processed in the sulphur recovery unit, therefore the stream has to be H₂S free otherwise H₂S will violate emission regulations. The ammonia stripper is therefore designed with an additional absorber to assure that all the H₂S is washed and separated from the ammonia. In addition there is a significant amount of cyanide in the stream; cyanide is very soluble in water and requires a separate stripper to remove the cyanide. The ammonia and cyanide are both sent to the incineration system as separate streams.

A small amount of solution is purged from the re-circulating absorbent solution to control the build-up of impurities and is then sent to the water treatment system.

The goal of obtaining a high degree of sulphur removal conflicts with that of simultaneously obtaining high H₂S selectivity; one goal must be compromised to

Table 1: Major equipment function in Coke S-MAX2

Description	Function	Product
Coke oven gas absorber	absorb H ₂ S & ammonia by absorbent	coke oven treated gas
Acid gas stripper	to remove H ₂ S	rich H ₂ S stream to SRU
Acid gas washer	to remove any residual NH ₃ in H ₂ S stream	rich H ₂ S Stream free of NH ₃ to SRU
Ammonia stripper	to remove NH ₃	rich NH ₃ to incineration
H ₂ S absorber	to remove any residual of H ₂ S in ammonia stream	rich NH ₃ free of H ₂ S to incineration
Cyanide stripper	to remove cyanide	rich cyanide to incineration
H ₂ S reboiler	provide heat to strip H ₂ S	refer to acid gas stripper
Ammonia reboiler	provide heat to strip NH ₃	refer to ammonia stripper
Cyanide reboiler	provide heat to strip HCN	refer to cyanide stripper

Ammonia and H₂S are produced in many processes applied to carbon oils, shale oil, coke oven gas and tar sands which means processes contain sulphur compounds, nitrogen and hydrocarbons. In this scheme, H₂S and NH₃ are separated and 100% recovered in two stripping columns as defined below:

- The H₂S-NH₃ mixture is stripped in the first stripping distillation column to obtain rich H₂S vapour which flows to the sulphur recovery unit. The bottom of the stripping column contains rich NH₃ which is processed in the second tower.

- Rich NH₃ is stripped in a second stripping distillation column to obtain rich NH₃ vapour which purified further in the H₂S absorber to remove any residual of H₂S before sending it to the ammonia burning incinerator or to the fertilizer unit.
- The overhead of the H₂S absorber through the knockout drum is pure rich NH₃. The bottom of the H₂S absorber and the knockout drum containing H₂S will be recycled to the first distillation tower H₂S stripper through the feed tank.
- Due to the variation of feed compositions, various features are considered to achieve stable operation, such as adding a pump around cooling system to the H₂S stripper and the H₂S stripper, plus recycling the H₂S absorber bottom to the feed tank upstream of the H₂S stripper.
- In some cases, for very lean acid gas, a concentrator is added to improve tower stability. In so doing, a constant feed flows to the stripper and will provide much higher stability. In addition the second and the third tower will be much smaller.

Fig 2: Coke S-MAX2 process flow diagram

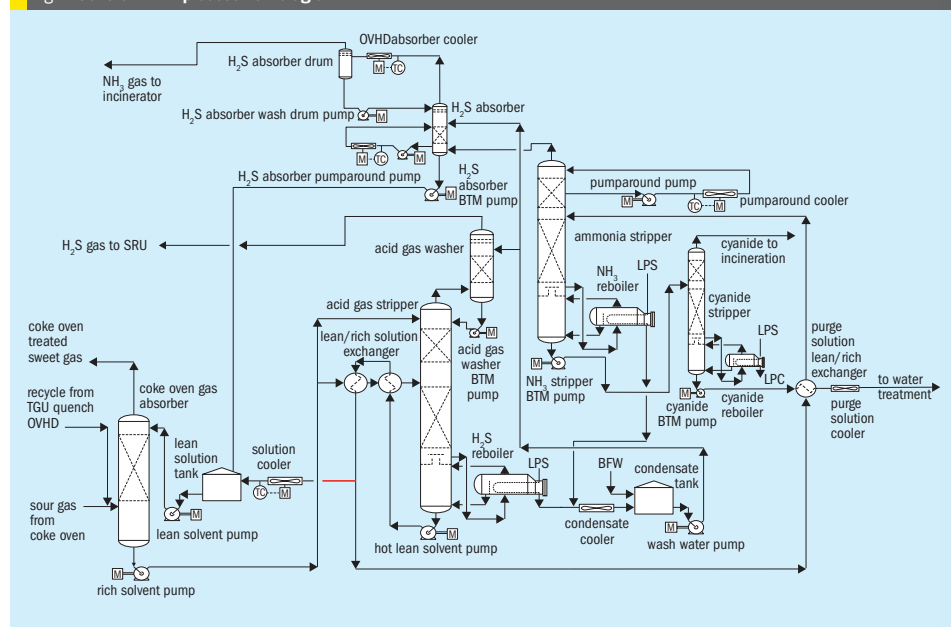


Table 1 lists the major equipment functions for Coke S-MAX2. Other equipment such as vessel, tanks, other heat exchangers and pumps are not listed in this table.

In the Coke S-MAX2 process (Fig. 2), the ammonia stripper overhead is sent to the H₂S absorber to wash all the H₂S using steam condensate water. The H₂S absorber is equipped with a pump around cooler and pump to keep the absorber cool and maintain tower stability. The absorber overhead flows to the overhead cooler and then the overhead knockout drum where pure ammonia is produced and sent to incineration, or another facility. The ammonia-free H₂S stream with possible BTEX is sent to the sulphur recovery unit and, depending on the H₂S concentration and impurities, can operate on air or oxygen.

It is expected that cyanide is removed and processed in the incinerator, where HCN is converted to ammonia and ammonia is burned in the incinerator. Fig. 2 shows the third stripper, where cyanide is stripped as a separate stream to the incinerator. Alternatively, a catalytic reactor can be provided upstream of Coke S-MAX for hydrolysis of HCN cyanide to NH₃, in which case the HCN stripper section can be eliminated.

In cases of high HCN present in the coke oven gas, 32 wt-% free S ammonium polysulphide (Tessenderlo Kerley APS KC-2040) is injected to neutralise HCN to harmless S₂Cn (S + Cn => S₂Cn); three times the material balance amount is used. A typical injection is a retractable spray device.

The main purpose of Coke S-MAX is to recover coke oven gas as a treated gas for power consumption, i.e. to recover the energy that could otherwise be wasted due to contaminants and emission restrictions. It is completely different from a two-stage desulphurization where the goal is only to separate H₂S from ammonia.

Sulphur recovery design criteria

The Claus unit operates with air or oxygen operation based on the H₂S concentration and the level of impurities. The sulphur recovery unit can operate with air mode operation as long as the combustion temperature is adequate for destruction of ammonia and impurities. If the combustion temperature is not adequate an oxygen line is provided. Oxygen is available in coke oven plants and it is not considered as an additional investment

Oxygen enrichment raises the reaction furnace temperature which ensures complete destruction of heavy hydrocarbons and ammonia, reduces the formation of Cn, COS, BTEX and CS₂, and shortens the gas residence time requirements for contaminants destruction.

In addition a well-designed burner and reaction furnace, which promotes good mixing of the reactants, is essential for complete destruction of undesirable feed contaminants.

Since the coke oven gas is dealing with lean gases with high ammonia content and impurities a single chamber without any recycle is normally used as long as the acid gas burner is designed for handling oxygen enrichment and 94% alumina refractory is used in the reaction furnace.

If ammonia is not processed in the SRU then the ammonia stream will be eliminated and processed in the incinerator or any other facilities.

The thermal section of the Claus unit is followed by two Claus catalytic stages using conventional alumina Claus catalyst alumina and titanium catalyst in the first reactor for COS and CS₂ hydrolysis.

The liquid sulphur flows to the sulphur pit, where liquid sulphur degassing takes place. The liquid sulphur degassing can be internal pit degassing or external outside pit degassing according to customer preference considering the residence time required for each option and plot space limitation.

The tail gas unit is designed using low temperature hydrogenation catalyst. The tail gas from the final condenser of the sulphur recovery unit enters the hydrogenation section through the steam heater. The steam heater, using high pressure steam, heats the tail gas up to a temperature that will permit the desired hydrogenation in the hydrogenation reactor. Sulphur compounds are converted to H₂S by the hydrogenation and hydrolysis reactions described above. These reactions are exothermic creating a temperature rise across the catalyst bed. The gas exiting the hydrogenation reactor is cooled in the reactor effluent cooler by producing low-pressure steam.

The gas is further cooled, and water is condensed in the quench column. The quench column is a one stage column in which the gas is first desuperheated by contact with circulating water stream and then further cooled. Most of the water in the gas is condensed, by contact with a

circulating stream of cooled water in a packed bed.

A pH analyser with a low-pH alarm is installed in the circulation line and will indicate when the pH of the quench water is reducing, from either a breakthrough of SO₂, or incomplete reduction of the sulphur compounds in the gas stream from the hydrogenation reactor.

Water is evaporated as the gas is desuperheated. Water is returned to the desuperheater section from the upper quench column section under level control.

In the upper packed section of the column, most of the water vapour in the tail gas is condensed by direct contact with a circulating stream of cooled water pumped by the quench column circulating pump. The water must be removed from the gas to prevent water balance problems in the amine treating section. The circulating water is cooled first in the quench column air cooler and further cooled in the quench column water cooler, which uses cooling water as the cooling medium. As the gas cools, water is condensed.

The condensed water is purged from the column to the sour water system on level control. During start-up, about 33% of the design quench column overhead is recycled to the tail gas heater by the start-up blower. This provides the process gas flow required to heat up the hydrogenation reactor.

The quench overhead is recycled to the Coke S-MAX unit through a recycle blower for zero emission operation.

The treated tail gas from the absorber is sent to the Incinerator. The residual H₂S and other sulphur compounds in this gas stream are oxidized to convert all the sulphur compounds to SO₂.

The flue gas is cooled in a waste heat boiler by generating high-pressure steam. Part of the high-pressure steam is used for the tail gas heater. The rest of the high-pressure steam along with the excess high pressure steam from the SRU is superheated in the superheater coil of the incinerator waste heat boiler before exporting to the high-pressure steam header. The incinerated flue gas is routed to the stack.

The incineration section consists of a forced draft incinerator with heat recovery. If the ammonia is not processed in the sulphur recovery unit then pure ammonia can be destructed in the ammonia burning incinerator e.g. using Duiker technology to destruct the ammonia to nitrogen and water with low NOx burner incineration.

Table 2: Feed compositions of coke oven gas and products

Composition Mole fraction	Coke oven feed gas	Treated coke oven gas	Solvent absorbent
H ₂	0.55175	0.54077	0.00000
CH ₄	0.22661	0.22209	0.00000
CO	0.06109	0.05987	0.00000
CO ₂	0.02217	0.01579	0.00000
C ₂ H ₆	0.02956	0.02897	0.00000
O ₂	0.00542	0.00531	0.00000
N ₂	0.04926	0.04828	0.00000
Water	0.03941	0.07880	0.98789
NH ₃	0.00987	0.00001	0.01120
H ₂ S	0.00362	0.00005	0.00032
NaOH	0.00000	0.00000	0.00000
HCN	0.00124	0.00005	0.00059
COS	0.00000	0.00000	0.00000
CS ₂	0.00000	0.00000	0.00000
Property			
Molar flow, kmol/h	3569.20	3641.37	8259.65
Mass flow, kg/h	38946	39478	148795
Temperature, °C	39.98	47.75	40.00
Pressure, kgf/cm ² (g)	0.50	0.40	6.00

Coke oven gas project

RATE was recently awarded a coke oven gas project including the construction of a semi-coke gas utilisation unit (SCGU) to increase the revenue earning potential of the shale oil co-product semi-coke gas (SCG). The purpose of the unit is to combust SCG for power production.

SCGU will be constructed to treat and combust sour semi-coke gas and shale gasoline from the retorting units. The scope of the SCGU includes utilities and off sites. To support the power plants, the installation of various water, steam, air, and fuel utilities, and off sites such as the tanks and gas cleaning facilities, are envisioned. The customer is considering generating electricity by firing semi coke gas and shale gasoline produced as byproducts in the production of fuel oil from oil shale.

The main objectives of the SCGU are:

- combustion of semi-coke gas;
- combustion of shale gasoline;
- sulphur removal from semi coke gas before combustion and/or from flue gas after combustion;
- production of electrical power.

Sour coke oven gas and semi coke oven gas compositions

The feed compositions for the sour coke oven gas contains H₂S, NH₃, HCN, BTEX,

plus H₂, N₂, CO, O₂, H₂O, CO₂, other hydrocarbons, naphthalene, tars and particles. The H₂S content varies from 5 g/m³ to 500 g/m³ (about 5%) and NH₃ varies from 7 g/m³ to 1000 g/m³. Since the gas volume is very high, the amount of sulphur is highly significant, therefore, even though oxygen enrichment is used to provide the stability of combustion temperature, using oxygen for such large capacity will also help to reduce equipment size and will reduce the capital cost.

For the semi-coke oven gas the feed composition of H₂S is 5 mol-% to the desulphurisation unit based on light shale naphtha balance where the H₂S concentration to the sulphur plant becomes much richer compared to table 2.

Table 2 represents the feed compositions for the raw coke oven gas, treated gas to the turbine, and the feed compositions to the sulphur recovery unit; ammonia will be processed in other units e.g. such as incineration or fertilizer units.

The gas temperature to the desulphurisation unit is 43°C and the treated coke oven gas is about 30°C. The treated gas composition and the feed to the sulphur recovery are shown in Table 2. An overall sulphur recovery of 99.99+ is requested. In addition, the liquid sulphur quality shall meet more than 99.5% purity, less than 0.2% moisture, density 1.93 g/cm³ and finally ferro content 0.003% in solid form.

Table 3: Feed compositions of coke oven gas and products

Composition Mole fraction	Rich H ₂ S to SRU	Rich NH ₃ to Incineration	Rich CN to incineration
H ₂	0.00302	0.00000	0.00000
CH ₄	0.00205	0.00000	0.00000
CO	0.00036	0.00000	0.00000
CO ₂	0.61413	0.00000	0.00000
C ₂ H ₆	0.00033	0.00000	0.00000
O ₂	0.00005	0.00000	0.00000
N ₂	0.00018	0.00000	0.00000
Water	0.01852	0.07794	0.48233
NH ₃	0.00000	0.92206	0.25022
H ₂ S	0.36135	0.00000	0.00000
NaOH	0.00000	0.00000	0.00000
HCN	0.00002	0.00000	0.26745
COS	0.00000	0.00000	0.00000
CS ₂	0.00000	0.00000	0.00000
Property			
Molar flow, kmol/h	35.22	33.86	15.77
Mass flow, kg/h	1400	579	318
Temperature, °C	50.85	50.00	104.73
Pressure, kgf/cm ² (g)	6.50	0.12	1.00

Tables 2 and 3 represent the coke oven gas feed composition and the product, where for the purpose of this paper the capacity has been changed. The sulphur capacity for this paper is 12 t/d.

As shown in Table 1, the coke oven feed gas composition contains high CO₂ with lean H₂S gas and significant NH₃ and cyanide. Due to the lean H₂S gas, the absorbent solvent rate is higher than normal.

Since the treated coke oven gas is used in a turbine to generate electricity, it is required to meet environmental regulations for NO_x, SO_x, CO, and dust. For this project H₂S, NH₃, and HCN has to be fully removed.

Considering the characteristic of the feed compositions, Coke S-MAX2 technology was selected to remove all of the H₂S, NH₃ and HCN, and possible BTEX and to separate NH₃ from the acid gas to the sulphur recovery unit.

The sulphur recovery is designed using oxygen enrichment to assure the destruction of any other components. The tail gas is designed using low temperature catalyst followed by the quench system where the quench overhead is recycled back to the desulphurisation unit for zero emission. The incineration system is designed based on ammonia burning technology.

Table 3 represents three separate streams from three different strippers where H₂S, NH₃ and cyanide are separated and

each stream can be processed in different units.

The feed to the SRU contains about 36% H₂S plus other components, therefore, the sulphur recovery is designed using 100% oxygen enrichment. The combustion temperature in the reaction furnace is 1,022°C.

For each tonne of sulphur (1000 kg), the oxygen consumption is 0.35 tonne (350 kg).

The quench system in the tail gas unit is designed for about 18 m³ of water circulation. The quench overhead is recycled to the Coke S-MAX absorber through a blower.

The treated water is used as makeup water in the Coke S-MAX due to water loss from purging strippers.

The high ammonia stream could be burned in an incinerator or used in a fertilizer unit. Due to the low H₂S concentration, ammonia cannot be processed in the sulphur recovery unit. The sulphur recovery has low H₂S concentration and it is more cost effective not to make sulphur plant larger.

The cyanide stream could also be

Table 4: Cost estimate

Coke S-MAX sections	Million US\$ (Gulf Coast)
Coke oven absorber and H ₂ S Stripping Section	47.0
NH ₃ stripping section	31.4
HCN stripping section	30.9
SRU 100% oxygen enrichment	4.6
Tail gas hydrogenation	1.9
Total installed cost	115.8

burned in an incinerator to ammonia and then to nitrogen and water.

Cost estimate

According to different costs estimates conducted, eliminating the ammonia from the sulphur recovery unit will result in cost savings of around 30%, even though the incineration cost increases by around 15%, the reduced operating cost for fuel and electricity consumption is very significant and more steam will be produced.

The capital cost for the combination of Coke S-MAX, the sulphur recovery and the tail gas treating unit is shown in Table 4.

As mentioned earlier, the HCN can be eliminated by providing a catalytic stage upstream of Coke S-MAX to hydrolyse HCN to NH₃ as well as COS.

Table 4 represents the cost estimate for this application based on Coke S-MAX2 and 100% oxygen enrichment. The saving in capital cost is at least 30-35% compare to other current technologies in the market. The operating cost is significantly lower because no solvent or absorbent is required, the tail gas amine unit is eliminated, the quench overhead is recycled back to the Coke S-MAX, and most importantly H₂S, NH₃ and HCN are removed from the gas as a separate stream. ■

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ISSN: 0039-4890

Design and production:
JOHN CREEK, DANI HART



Printed in England by:
Buxton Press Ltd
Palace Road, Buxton, Derbyshire,
SK17 6AE

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Published by: BCInsight Ltd
Southbank House, Black Prince Road
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