Impact of sulphiding agents on ULSD catalyst performance

A research programme demonstrated the comparative performances of a conventional sulphiding agent and an agent that offers safer, cleaner catalyst activation

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The purpose of this research was to compare the hydroprocessing performance of three different commercially available cobalt-molybdenum (CoMo) catalysts in two sets of circumstances:
(a) after activation with Lubrizol's SulfrZol 54 (SZ54) and
(b) after activation with dimethyl disulphide (DMDS).

SZ54 is a sulphiding agent commercialised by Lubrizol Corporation for the activation of hydroprocessing catalysts. This alternative to traditional sulphiding agents offers a higher flash point, low odour, and emissions improving technology, which results in safer, cleaner, and effective catalyst activation to refineries.

CoMo hydrotreating catalysts and input on procedures were provided by the participating catalyst vendors. Two samples of each catalyst were activated, one with DMDS and the other with SZ54.

The feed was a straight-run gasoil (SRGO) with 1.444 wt% sulphur, 173 wtppm nitrogen, 28.2 wt% aromatics, and SimDist distillation range (0.5% to 95%) of 92°C to 391.4°C.

A common range of operating conditions for the tests was selected after consultation with the catalyst vendors. Testing was performed by Avantium using a combination of advanced high-throughput testing equipment, robust experimental design, and extensive technical expertise. Performances were measured simultaneously using Avantium's multi-reactor Flowrence parallel testing technology. During the activity test, pressures and flows were the same for all catalysts while reaction temperatures were varied.

Evaluation of the differences in performance between catalysts is not part of this study and therefore not reported here. For each of the catalysts evaluated, results indicated that sulphiding with SZ54 delivered the same level of HDS as sulphiding with DMDS. For achieving 10 wtppm sulphur, activity differences between catalysts activated with the two sulphiding agents were <0.3°C, which is within the typical repeatability for hydroprocessing catalyst activity tests (usually about 1°C).

In addition to product sulphur, other important performance indicators were evaluated: product nitrogen, hydrogen consumption, liquid product density, and light end yields. In each case, results confirmed that sulphiding with SZ54 is technically equivalent to sulphiding with DMDS for hydrotreating middle distillates in order to produce ultra low sulphur diesel (ULSD). Some aspects of this research may lead the way to improved sulphiding technology.

Background

Lubrizol sought independent confirmation of previous comparisons of DMDS vs SZ54 performance for commercial hydrotreating catalysts activation. In addition to seeing benchmarks for sulphiding agent behaviour, catalyst and technology suppliers saw how independent testing matched their catalyst performance expectations.

Project participation

This unique and challenging project required cooperation and simultaneous alignment of interests among multiple world-leading vendors:
• Lubrizol[1] provided expertise, sulphiding agents, and funding
• Catalyst vendors provided catalyst and process expertise, catalyst samples, and approval of the testing protocol. The catalysts samples were supplied by world leader catalysts manufacturers from Europe and the US
• Avantium[2] provided project management, high-throughput catalyst testing technology, testing expertise, and funding.

Positive relationships developed during Avantium's previous experience with all parties proved to be crucial during the alignment of project participants.

The sulphiding step

Commercial hydrodesulphurisation (HDS) employs catalysts in which the active species are molybdenum or tungsten sulphides promoted by cobalt or nickel sulphides. Production of ULSD typically is accomplished by HDS over CoMo or NiMo catalysts. During HDS, hydrodenitrogenation (HDN) and saturation of olefins and aromatics also occurs.[3,4]

Most often, the catalysts are delivered as oxides and activated by reductive sulphidation (sulphiding) in the presence of hydrogen at elevated pressure. Sulphur can be supplied in different ways: as H_2S, with the native sulphur in a feedstock, or as a sulphur spiking agent. Spiking agents include DMDS, dimethylsulphoxide (DMSO) and SZ54, which contains di-t-butylpolysulphide (DBPS).[5]

Comparison of DBPS and DMDS[5]		
Property	DBPS	DMDS
Hydrocarbon from decomposition	i-butane + H_2S	Methane + H_2S
Destination of decomposition hydrocarbon	Alkylation unit	Fuel gas
Sulphur content, wt/wt%	54	68
Initial decomposition temperature	320°F (160°C)	392°F (200°C)
Flash point (closed cup)	212°F (100°C)	61°F (16°C)
Odour description (vendor)	Low odour (like diesel)	Unpleasant garlic-like odour

Table 1

(There are noteworthy exceptions to the need for in situ sulphiding. Presulphurised catalysts include the actiCAT products offered by Criterion Catalysts & Technologies,[6] and catalysts that are presulphided with the Totsucat technology offered by Eurecat.[7])

Sulphiding is one of the most critical aspects of fixed bed commercial hydroprocessing. Process upsets during catalyst sulphiding can have deleterious consequences.[8]

The sulphur in feedstocks and spiking agents is converted into H_2S, which in turn reacts with the catalytic oxides. Relevant chemical reactions include the following:

Sulphur-containing gasoil + H_2 → sulphur-lean gasoil + H_2S
Spiking agent + H_2 → hydrocarbon + H_2S
1. CH_3-S-S-CH_3 + 3 H_2 → 2 CH_4 + 2 H_2S
2. C_4H_9-S-S-C_4H_9 + 3 H_2 → t-C_4H_{10} + 2 H_2S

Sulphiding reactions[1]
3. MoO_3 + 3 H_2S + H_2 → MoS_2 + 3 H_2O
4. WO_3 + 3 H_2S + H_2 → WS_2 + 3 H_2O
5. 3 NiO + 2 H_2S + H_2 → Ni_3S_2 + 3 H_2O
6. 9 CoO + 8 H_2S + H_2 → Co_9S_8 + 9 H_2O

Unwanted reduction of nickel oxide
7. NiO + H_2 → $Ni(m)$ + H_2O

Questions exist about the exact stoichiometry of Reactions 5 and 6.[7,9] Regardless, successful activation requires excess H_2S, excess H_2, and close adherence to procedures developed by catalyst manufacturers.

Properties of DBPS and DMDS
DMDS is the most widely used sulphur spiking agent for activating hydroprocessing catalysts. **Table 1** compares DMDS with an approved alternative: DBPS, the main constituent of Lubrizol's SZ45. Note that DBPS has a higher flash point, lower odour, lower initial decomposition temperature, and lower emissions.

Advantages of SZ54
When compared to DMDS, SZ54 has the following advantages[5]:
• **Safety**. Due to the higher flash point of SZ54 – 212°F (100°C) vs 61°F (16°C) for DMDS – it presents less of a fire hazard. SZ54 requires no special packaging, transportation, or storage requirements. In contrast, DMDS usually is stored under nitrogen pressure in closed containers.
• **Co-produced hydrocarbon**. SZ54 has a major economic plus: in addition to H_2S, it yields primarily isobutane, which is a high-value refinery intermediate stream required for alkylation. As sulphiding with SZ54 proceeds in a hydroprocessing unit, the isobutane is easily isolated from the hydrogen rich recycle gas.

In contrast, DMDS yields methane, which accumulates in the recycle gas, decreasing hydrogen partial pressure (ppH_2). Often, the ppH_2 gets so low that the methane content must be decreased by purg-

Straight-run gasoil feed for sulphiding and testing	
Property	SRGO
Sulphur, wt%	1.444
Nitrogen, wtppm	173.2
Hydrogen, wt%	13.07
SG 60/60°F, g/ml	0.8538
Aromatics, wt%	28.2
T5, °C	181.2
T30, °C	273.0
T50, °C	306.6
T70,°C	335.0
T95, °C	391.4

Table 2

ing recycle gas. During a purge, the methane, along with valuable hydrogen and even sometimes H_2S, is routed to the flare. (Usually, purges of sour recycle gas are amine scrubbed before they reach the flare. If not, a purge can generate undesired SOx.)
• **Odour**. DMDS has an unpleasant odour. Even small leaks can be a nuisance to refinery workers and the surrounding community. In contrast, SZ54 is less volatile and possesses a far less offensive, diesel-like smell.
• **Transport regulations**. Due to its low flash point, shipment of DMDS is regulated by the US Department of Transportation (DOT) and similar organisations in other jurisdictions. Often, DMDS trucks must be continuously supervised. In contrast, the US DOT classifies SZ54 as non-hazardous for transportation.

Experimental programme
The experimental programme compared the performance of three different commercial CoMo ULSD catalysts after sulphiding with SZ54 (which is comprised mostly of DBPS) and after sulphiding with DMDS. Avantium received catalyst samples from each vendor in the form of extrudates with diameters between 1.6mm and 1.8mm.

Feedstock
The straight-run gasoil (SRGO) used for sulphiding and for the test came from a commercial refinery. SRGO properties are presented in **Table 2**. Note that the sulphur content is 1.44 wt% and the nitrogen content is 173 wtppm. For sulphiding, the SRGO was spiked with SZ54 or DMDS to reach a total sulphur of 3 wt%.

Testing equipment
For this evaluation, Avantium used a testing unit with 16 parallel single pellet string reactors (SPSR). The equipment layout is illustrated by the schematic diagrams shown in **Figures 1** and **2** (one or more patents apply).[10] The unit employs Flowrence technology, which provides tight control of process conditions – temperature, flow rates, and pressure. The system provides
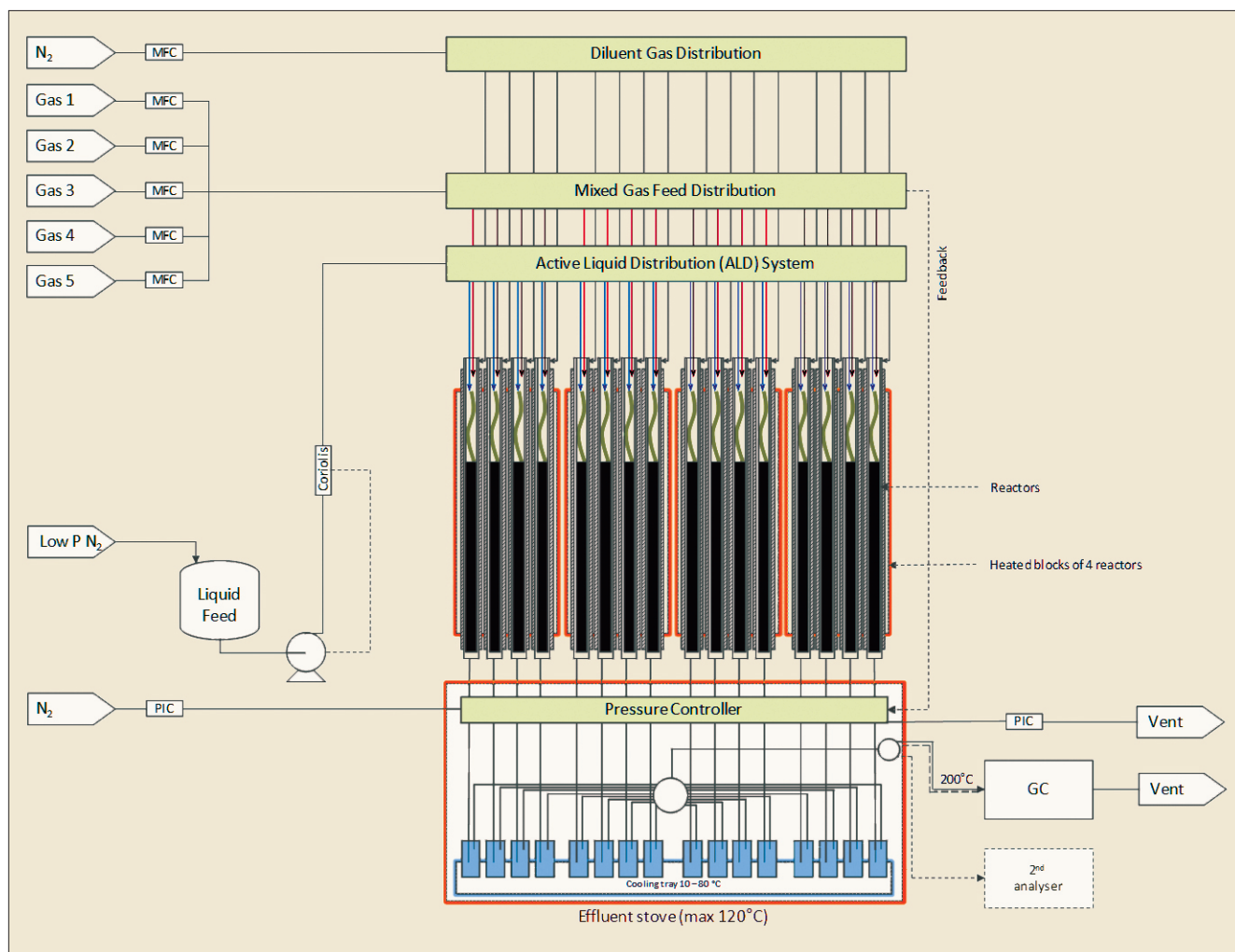
Figure 1 Schematic representation of the Avantium pilot plant with 16 parallel SPSR

for accurate measurement of yields, hydrogen consumption, and product properties.

Moonen *et al* (2017)[11] found that for gasoil hydroprocessing, there is excellent correspondence between results from an Avantium SPSR unit and those from a bench-scale unit with a catalyst volume of 225 ml. Their experiments confirmed that SPSR are no more susceptible to wall effects, channelling, and back-mixing than the much larger reactors. The authors explained the similarity with rigorous modelling of the corresponding hydrodynamics.

Practical advantages of SPSR

Traditional hydroprocessing pilot plant studies employ relatively large reactors, typically with an inside diameter (ID) ranging from 1.2-2.5cm and a catalyst bed length of 30-80cm. Consequently, within a limited budget and tight timeframe, it becomes impractical to evaluate
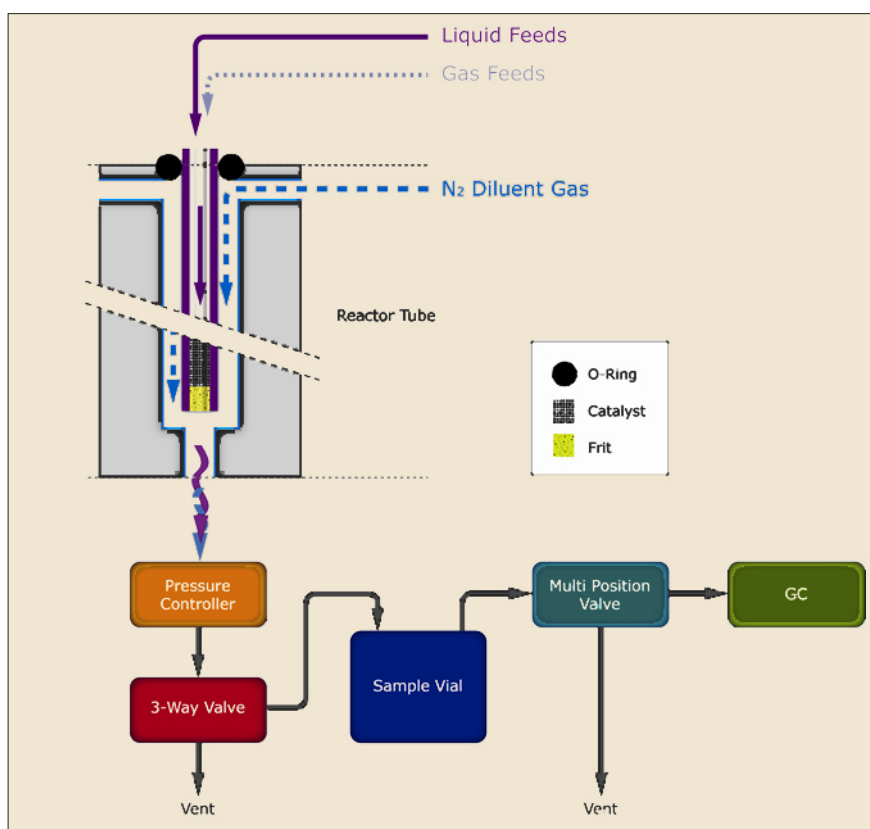

Figure 2 Schematic diagram of reactor and effluent set-up

more than a few alternative catalysts or sets of process conditions – in this case, alternative sulphiding agents.

Associated with series tests are inherent uncertainties, for example, due to differences in catalyst loading. Replicate tests, which can minimise such uncertainties, are seldom even considered. In a conventional pilot plant, the research described in this article would have required several barrels of feed and 32 separate month-long runs.

In contrast, parallel small-scale testing in SPSR significantly reduces feed and catalyst requirements. It also reduces labour and utilities costs. Most significantly, it provides a way to accomplish more in a given amount of time. Reactor loading is more reproducible because the diameter of the extrudates is slightly smaller than the reactor diameter; extrudates automatically line up in strings (see **Figure 3**). The narrowness of the reactors decreases maldistribution of gas and liquid over the catalysts, thereby preventing catalyst bed channelling and incomplete catalyst wetting. An inert diluent can be introduced after catalyst pellets are loaded to further increase flow distribution and wetting.
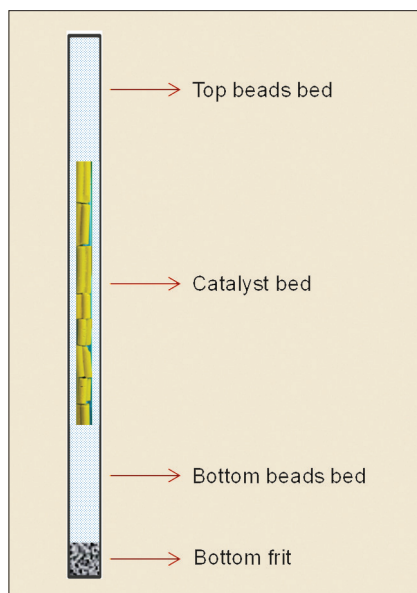

Figure 3 Reactor loading schematic

Reactor loading
The weights of catalyst for this study were calculated using the compact bulk density (CBD) provided by the vendors. A bottom section of ceramic beads (50-200μm diameter) was supported by a small frit (20μm pore diameter). Above the support beads, stacks of catalyst extrudates were loaded into the isothermal section, which has the following dimensions:
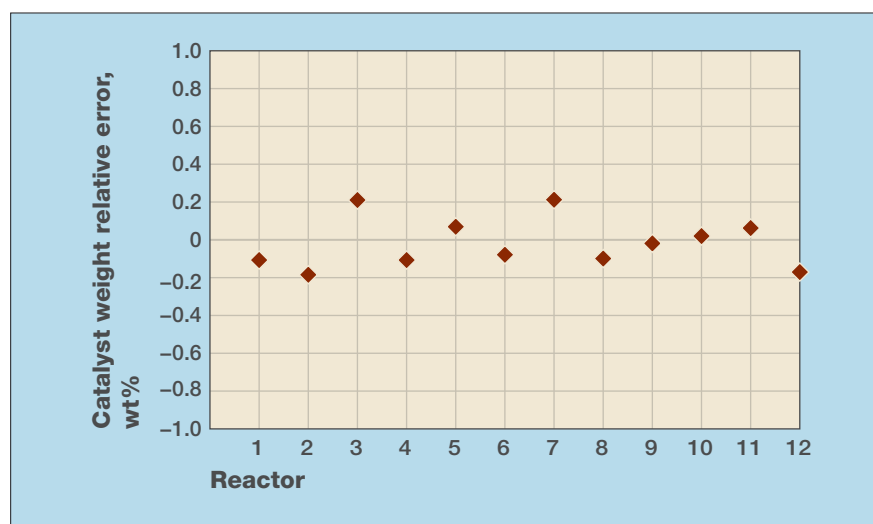
300mm length and 2mm internal diameter. The void volume between the catalysts was filled with the ceramic beads to prevent channelling, wall effects, and back-mixing. A second bed of ceramic beads was added at the top until the reactor was completely filled.

Exceptionally precise loadings can be achieved. In this study, four reactors were loaded with each one of the three catalysts for a total of 12 reactors loaded into the unit. For the catalyst weights, the average deviation was just 0.1 wt% (see **Figure 4**).

Catalyst activation (sulphiding)
Catalyst activation (sulphiding) was accomplished in two campaigns. In the first campaign, half of the reactors were sulphided using DMDS and in the second using SZ54. The same activation procedure was followed for sulphiding each catalysts with both agents. Same pressure, gas/oil ratio, and space velocity were used for the activation of all catalysts (see **Table 3**), however different temperature ramps were used for the activation of each catalyst following the vendors' guidelines. Temperature programmes for the catalysts activation are not presented for confidentiality reasons, however these were representative of common activation procedures followed in refineries.

The catalysts were dried at 50°C under flowing nitrogen for one hour before the introduction of feed. After drying, all catalysts were soaked with the sulphiding feed for four hours at space velocity 3 h^{-1}.

The catalysts were sulphided with the SRGO+DMDS feed using the operating conditions presented in **Table 3**. At each sulphiding stage, the temperature of each heating block was controlled independently. This allowed the simultaneous use of different temperature programs for sulphiding the three different catalysts. After sulphiding, the unit was cooled to 30°C and flushed with un-spiked SRGO and then N$_2$ to remove any remaining traces of H$_2$S. Then the reactors were removed from the unit and stored safely while minimising any contact with air.

Figure 4 Relative error during catalysts loading

Operating conditions for sulphiding and the evaluation test			
Operating condition	Soaking	Sulphiding	Evaluation tests
H$_2$ pressure, barg	45	45	55
LHSV, h^{-1}	3	2	1.5
H$_2$/oil ratio, Nl/l	50	300	300

Table 3

Catalysts loading matrix		
Catalyst	DMDS	SZ54
A	R1 & R3	R2 & R4
B	R5 & R7	R6 & R8
C	R9 & R11	R10 & R12

Table 4

Test schedule and analysis per condition			
Test condition	Duration (days)	Offline analysis	Mass balance check
Line out	10	1 S, 1 N per day	2 /day
Condition 1: Target = 50 wtppm S	4	Same as above	2 /day
Condition 2: Target = 100 wtppm S	4	Same as above	2 /day
Condition 3: Target = 10 wtppm S	4	Same as above	2 /day
Condition 4: Back check Condition 1	3	Same as above	2 /day

Table 5

In the second campaign, sulphiding was accomplished with the SRGO+SZ54 blend following the exact same procedure. Again, after sulphiding, the reactors were cooled, flushed, and removed from the unit avoiding exposure to air. Special measures were applied to minimise contact with air during the storage and subsequent re-installation of reactors containing sulphided catalysts.

Catalyst performance tests

Six reactors from each sulphiding batch were loaded into the unit according to the matrix shown in **Table 4**. That is, each heating block contained two identically loaded and sulphided reactors. The duplication provided a back check to minimise concerns about whether or not results from any particular reactor might be spurious.

Immediately after reinstallation, the reactors were flushed with N_2 to remove any traces of air or moisture picked up by the catalysts during the reactors installation procedure. The operating pressure was adjusted to 55 barg at a rate of 0.5 barg/min and then the reactors were soaked with the testing feed (SRGO) at the same conditions used previously for sulphiding.

Product analysis

Reactor outlet gas compositions were analysed by online gas chromatography (GC). Helium (the internal standard) and H_2 were measured with a thermal conductivity detector (TCD). Hydrocarbons were quantified with a flame ionisation detector (FID). The liquid effluent was collected continuously for eight hours twice per day. The samples were weighed offline, and the weight results were automatically coupled with the online GC results to determine material balance.
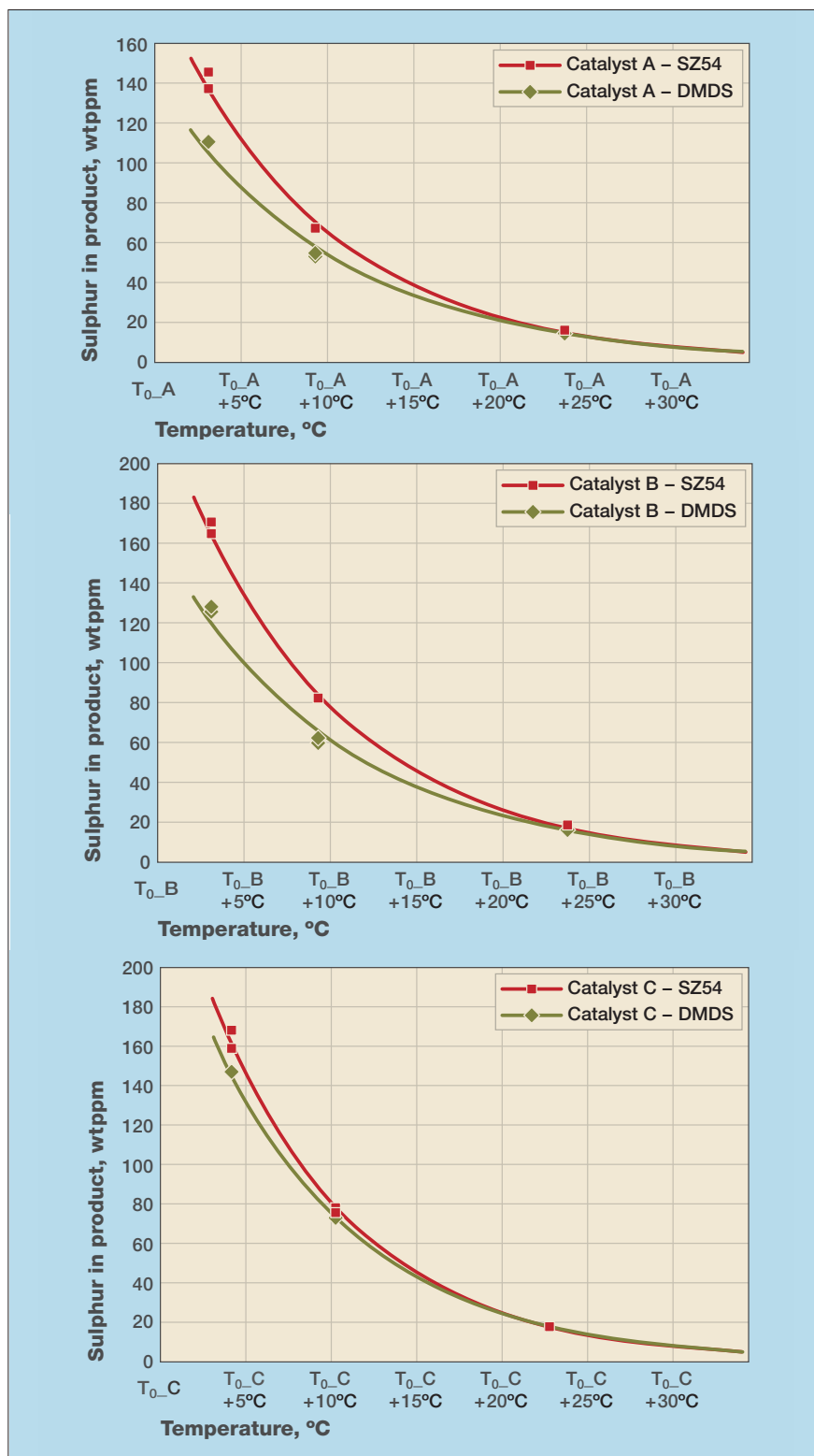
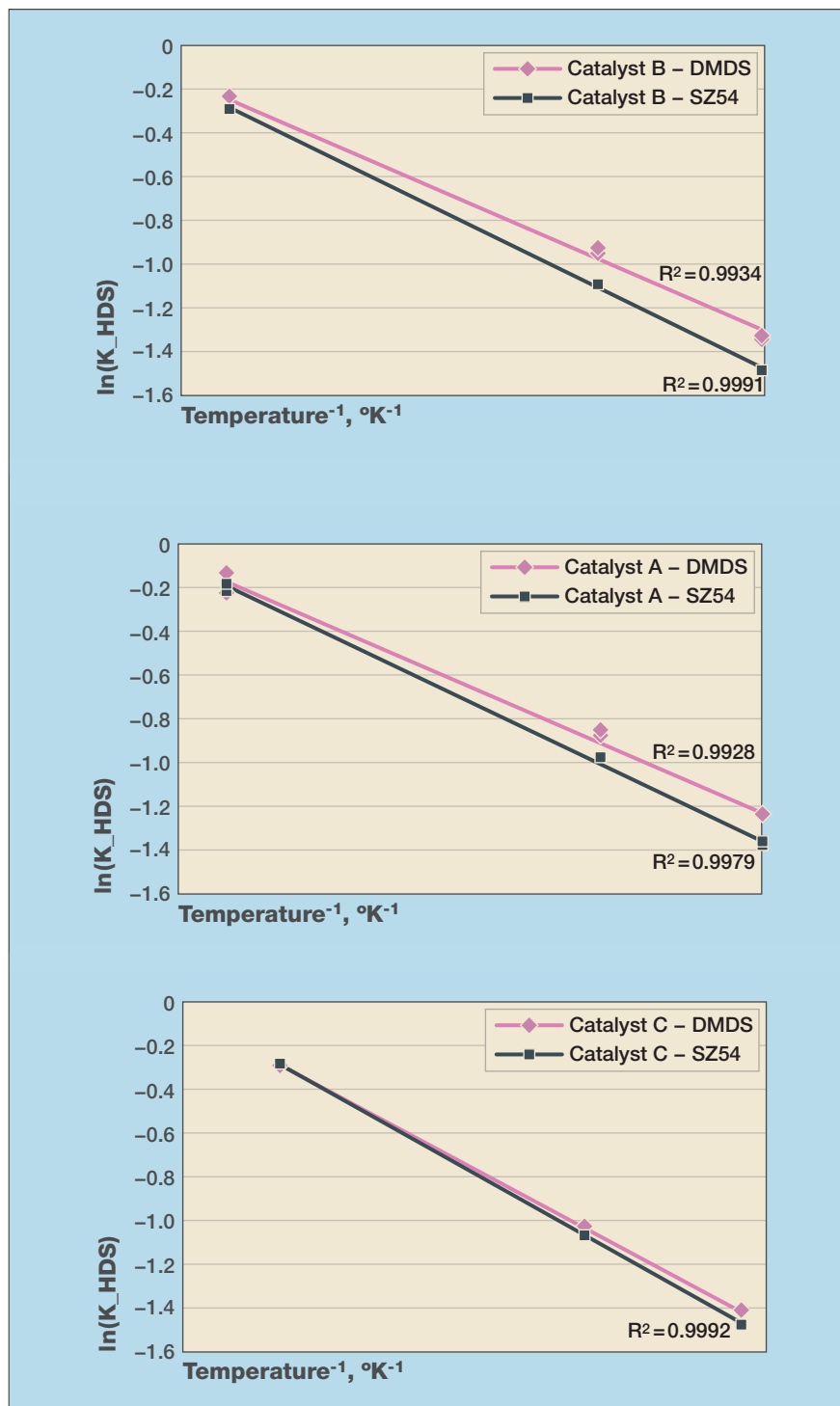Total sulphur and nitrogen were

Figure 5 Sulphur product results

Figure 6 Basis for kinetic parameters estimation for HDS

analysed with a TN/TS 3000 S/N analyser, which has a detection limit of around 2 ppm ±0.2 ppm.

Density measurements were performed with a Mettler Toledo DM50 unit.

Operating conditions

Catalyst vendors estimated reaction temperatures for reaching product sulphur contents of 10, 50 and 100 wtppm for their respective catalyst.

The reactors pressure was controlled to 55.0 barg. The flows of liquid and gas to the reactors were tightly controlled at the specified set points, allowing an average mass balance closure of 99.94±0.4%.

Calculated flow based parameters were LHSV = 1.5 h^{-1} and H$_2$/oil ratio = 300 Nl/l.

Testing protocol

A summary of the testing protocol is shown in **Table 5**. The test began

with a six-day stabilisation (line out) period to ensure a proper stabilisation of the active sites on the catalysts. After this, three different temperature conditions were evaluated with a duration of 3-4 days each. Finally, a back check or return point was performed at the end of the run in order to verify the relative activity of each catalyst.

The sulphur and nitrogen contents of liquid products from each reactor were measured daily. With online GC, each product gas stream was monitored continuously. For material balance calculations, liquids from each reactor were accumulated twice per day for eight hours each time.

Test results

Product sulphur

Figure 5 shows the product sulphur results determined during the performance test for each catalyst. Recall that the runs were made in duplicate: two different reactors were loaded with the same catalyst in all cases in order to evaluate the reproducibility of the results. The reproducibility was superb: differences between all duplicates were less than 5% and most differences at ULSD conditions were less than 1 wtppm.

The level of desulphurisation reached during the evaluation was between 99% and 99.9%. This implies that the identification of differences in catalysts HDS performance as presented in **Figure 5** required a very high accuracy from the experimental set-up.

Even though the catalysts were provided by different vendors, all of them showed similar comparative performance when sulphided with SZ54 and DMDS. All catalysts sulphided with SZ54 showed a slightly lower HDS capacity (compared with the same catalyst sulphided with DMDS) when operated at the 100 ppm sulphur target condition. This difference was even smaller during the 50 ppm sulphur target condition and basically none when the catalysts were operated at ULSD conditions (<20 ppm sulphur product). The difference in response observed for all catalysts in **Figure 5** seems to indicate
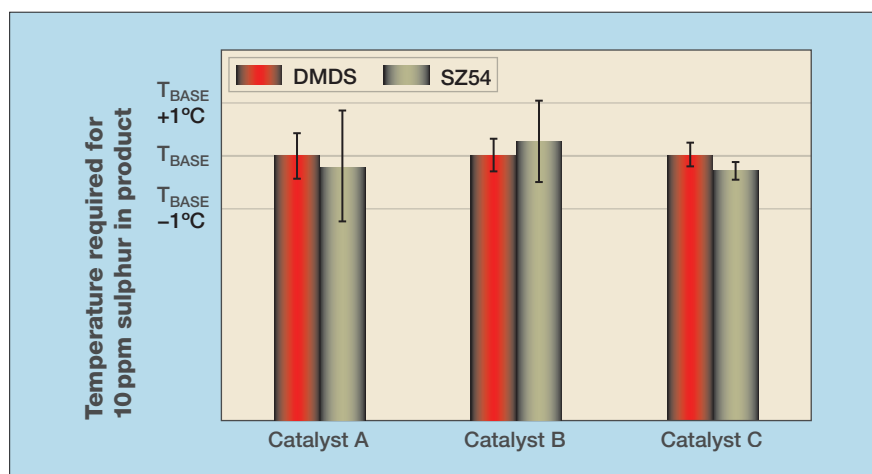
Figure 7 Temperatures required for 10 wtppm product sulphur

Activation energy estimates		
Catalyst	Ea (kJ/mol) Sulphided w/ DMDS	Ea (kJ/mol) Sulphided w/ SZ54
Catalyst A	Base	Base + 18.44
Catalyst B	Base	Base + 21.53
Catalyst C	Base	Base + 11.03

Table 6

Other product properties comparison (@ 10 ppm sulphur target conditions)				
	Average difference @ 10 ppm sulphur target			
Property	Range	Cat A	Cat B	Cat C
Nitrogen, wtppm	0.3-0.6	-0.03	0.03	0.02
Density, g/ml	0.8326-0.83334	-0.0001	-0.0006	0.0000
H_2 consumption, m^3 H_2/m^3 feed	55-63	4	2	0
Light end yield, wt%	0.64-0.87	-0.02	0.01	-0.03

Table 7

slightly different HDS activation energies depending on the sulphiding agent used for the catalysts activation.

In any case, the results presented in **Figure 5** clearly demonstrate that catalysts sulphided with SZ54 show the same level of desulphurisation as catalysts sulphided with DMDS when compared at ULSD production operating conditions.

Kinetic parameters
Kinetic parameter estimates were based on plots developed from the product sulphur values obtained during Condition 1 (50 ppm sulphur target), Condition 2 (100 ppm sulphur target) and Condition 3 (100 ppm sulphur target) as previously presented in **Figure 5**. In pseudo first order kinetics, the natural logarithm of the rate constant is line-

arly and inversely proportional to the absolute temperature. The slope of the line is Ea/R, where Ea is the activation energy and R is the ideal gas constant. **Figure 6** presents the graphs from which activation energies (Ea) were determined. For each catalyst, the coefficient of determination (r^2) is excellent: all were above 0.99. The relative comparison of the Ea values is given in **Table 6**.

Activation energy is a measure of sensitivity to temperature. Therefore, this research indicates that catalysts sulphided with SZ54 seem to be more temperature sensitive than those sulphided with DMDS. This result does not have any direct implication for the performance expected from the catalyst (independently of the sulphiding agent used for the activation) when it is operating in an ULSD produc-

tion commercial unit, as the sulphiding agent does not seems to affect the performance of the catalyst at high HDS conditions.

On the other hand, the differences observed in the activation energy for catalysts sulphided with SZ54 may lead to further studies by Lubrizol and catalysts vendors in order to optimise the operating conditions for the catalysts activation. Usual hydroprocessing catalysis activation procedures are based on the use of DMDS which present a higher decomposition temperature and different physical properties.

Temperature required for 10 wtppm product sulphur
Figure 7, which is based on the kinetic parameters, shows temperatures required to achieve 10 wtppm product sulphur. The difference between sulphiding agents is very low: <0.3°C for all catalysts. As can be seen in **Figure 7**, the error bar estimated from the measurement of repeated experiments (duplicated reactors) is in most cases higher than the difference in performance between the catalysts sulphided with different agents. This enables the conclusion that each catalyst sulphided with the different agents will reach 10 wtppm product sulphur at a similar SOR temperature with an uncertainty (95% confidence level) of less than ±1°C.

Other product properties
Table 7 shows a comparison of all other product properties measured during the tests for the catalysts sulphided with each agent. In general, no significant differences were observed for nitrogen, liquid density, hydrogen consumption, and light end yields production independently of the sulphiding agent used.

Conclusions
This research compared the hydroprocessing performance of three commercial CoMo catalysts from three different vendors in two sets of circumstances: (a) after activation with Lubrizol's SZ5 and (b) after activation with a conventional sulphiding agent. For each of the three catalysts, results indicated that for making products with <20 wtppm

sulphur, sulphiding with SZ54 delivered the same level of HDS as sulphiding with DMDS.

For achieving 10 wtppm sulphur, there were no differences in HDS capacity between catalysts activated with the two sulphiding agents considering the experimental error (approximately ±1°C with a confidence level of 95%).

In addition to product sulphur, other important performance indicators were evaluated: product nitrogen, hydrogen consumption, liquid product density, and light end yields. In each case, results confirmed that SZ54 can be used in commercial applications as a substitute for DMDS without impairing the overall performance of the tested catalysts. All catalysts suppliers participating in this study acknowledged the results, thereby validating the commercial relevance of the study.

Some aspects of this research may lead to new developments in hydroprocessing catalyst sulphiding procedures. The difference in activation energies for catalysts sulphided with different additives is unexpected and may lead the way to improved sulphiding technology.

Flowrence is a trademark of Avantium.

References

1 Lubrizol Corporation is a Berkshire Hathaway company providing complex specialty chemicals to optimise the quality, performance and value of our customers' products while reducing their environmental impact.
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