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ULSD IN REAL LIFE: COMMERCIAL PERFORMANCE OF STARS AND NEBULA TECHNOLOGY

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ABSTRACT

Tier 2 specifications for Ultra Low Sulfur Diesel (ULSD) are still 4 years away and most U.S. refiners are actively engaged in technology evaluation and capital investments. The time frame for ULSD in Europe is accelerated due to country specific specifications and tax incentives for producing clean fuels. This has lead to early application of new catalysts and technologies for producing clean diesel, particularly when it can be accomplished for little to no capital investment.

Akzo Nobel/Nippon Ketjen and BP have been on the forefront of development and application of new catalysts for clean transportation fuels. BP was one of the first users of catalysts utilizing STARS™ (Super Type II Active Reaction Sites) technology, demonstrating their high activity for reducing sulfur in diesel to very low levels. In 2001 Akzo Nobel introduced Nebula™ technology[1,2,3], a breakthrough in catalyst performance which, for the first time, offered the potential for a drop in hydrotreating solution to ultra low sulfur diesel. BP is again pioneering the application of a breakthrough catalyst technology by virtue of its fast-acting approach to implementation of new technology.

This paper discusses the latest insights into the kinetics of desulfurization to ultra low levels. The experiences of several BP refineries that have already moved into the ultra low sulfur diesel area are presented. The experience of these early adopters shows that producing ULSD is certainly not a simple matter but neither is it the capital intensive calamity feared by many refiners. Catalyst performance is key to the success of these projects both in terms of product properties and minimizing capital expenditures.

I INTRODUCTION

In early 2001 US EPA issued the Tier 2 regulations for on-road diesel fuel. Effective in mid 2006, diesel fuel must contain no more than 15 ppm sulfur as delivered from the pump. Though some legal challenges remain, the reality of ULSD in the United States in the not too distant future is a prominent strategic issue for nearly every refiner. Most of the action, however, has been in Western Europe, where the introduction of ultra low sulfur diesel is happening in two-stages – first to 50ppm and then to 10ppm.

Although the European specification for on-road diesel is still 350 ppm, retail sales in several countries (Belgium, Denmark, Finland, Germany, The Netherlands and the UK) have already moved substantially to 50 ppm sulfur because of tax incentives. Sweden already mandates that diesel sulfur content will be less than 10 ppm. Tax incentives for 10 ppm sulfur will begin in Germany in January 2003 and possibly also in the UK by late 2003. The European Union also is proposing that 10 ppm sulfur diesel be available throughout Europe by the end of 2005.

BP has been on the leading edge of clean fuels initiatives. A key component of BP's approach to breakthrough business performance is timely and appropriate application of technology from internal & external sources. BP has been particularly active in ultra low sulfur diesel:

- Ultra low sulfur diesel was required for the rollout of 50 ppm S diesel in the UK back in 1999 (Table 1). This was not mandated, but was encouraged by a tax-incentive from the UK government – within 18 months, nearly all on-road diesel met the new specification. BP was a key player by virtue of its refineries at Grangemouth (near Edinburgh, Scotland) and Coryton (near London, England).

- BP also launched its "Clean Cities" program in 1999,[4] when it committed to making cleaner fuels available in 40 cities worldwide. They exceeded their target by making clean fuels available in 59 cities by the end of 2000, and will make these fuels available in 90 cities throughout the world by the end of 2001.[5,6] In Western Europe, ultra low sulfur diesel has been a major factor.

- On the US West Coast, ARCO (a BP company) has developed a new ultra-low sulfur diesel fuel known as ECD-1, which was introduced to the California marketplace in late 1999. This contains less than 15 ppm sulfur.[7]

Table 1: UK On-Road Diesel Specification (1999)

Property	Unit	UK Specification
Sulfur	ppm	50 max
Cetane No.	cc	51 min
Cetane Index	cc	48 min
T95	°C	345 max
Density	kg/m^3	820-835

Thanks to tax incentives, some refiners in Western Europe have had the option of profitably pursuing ULSD in advance of mandatory regulations. While the incentives are usually not large enough to justify grassroots construction projects, they are often adequate to support unit

revamp and application of the latest catalyst technologies. Aside from the obvious financial benefit of captured tax incentives, early adoption offers these refiners the opportunity to test various strategies for producing ULSD. The power of commercial operation is substantial since it offers real world insights about producing ULSD, which are not possible to simulate in pilot plants or computer models.

Catalysts are key when trying to produce ULSD in existing hydroprocessing units. Akzo Nobel/Nippon Ketjen have been at the forefront of these early applications with its STARS and most recently, Nebula, technology. STARS technology catalysts have 100% Type II active sites, distinguished by the low interaction between active site and support substrate.[11] This leads to a much higher intrinsic activity for Type II active sites compared to the Type I sites present on conventional catalysts. High intrinsic activity is particularly beneficial when it is necessary to remove sulfur and nitrogen to very low levels as is the case with clean fuels. In 2001 Akzo introduced Nebula technology, a breakthrough in catalyst activity achieved by dramatically increasing active site density compared to conventional catalysts.

BP and Akzo Nobel have been working closely on pioneering applications for new catalyst technology. In 1998, BP was one of the first oil companies to recognize the value of STARS catalysts for the production of low sulfur diesel in Europe. On the strength of pilot tests, BP decided to use KF757 in two diesel HDS units – one each at the BP Grangemouth and Coryton refineries. The new catalysts were installed in March 1999 and September 1999 respectively, and have performed very well. BP now uses Akzo Nobel/Nippon Ketjen STARS catalysts, both KF-757 and KF-848, at several sites across Europe.

Having developed a good technological relationship, and enjoyed the success of working together to implement new catalyst technology, BP and Akzo Nobel started discussions in mid-2000 about applying the emerging Nebula technology in a BP hydrotreater. The opportunity was evaluated though BP's Refining Technology and Technology Management Networks, and a decision taken to make a detailed performance/risk assessment for a diesel hydrotreater at a refinery in Europe. The timing was excellent, as the diesel hydrotreater was due for turn-around. With this target, BP and Akzo Nobel started a program of testing and technical evaluation, which successfully lead to the installation and start-up of Nebula in the second half of 2001.

II   PRODUCING ULTRA LOW SULFUR DIESEL

The complexity of producing ULSD has been well established. Up to now most attention has focused on the very low reactivity of sterically hindered dibenzothiophenes (SH-DBTs) in the diesel fraction. These particular molecules follow a hydrogenation rather than hydrogenolysis (direct) pathway to desulfurization.[3,8,9] The two-step and relatively slow path to desulfurization of these molecules is the root cause of the difficulty of producing ULSD. More recently it has been established that, in addition to the concentration of SH-DBTs in the feed, a number of other factors have important influences on the desulfurization of diesel to 10 ppm. These include:

- Organic nitrogen content
- Hydrogen consumption / availability
- Aromatic equilibrium / sulfur floor

Effect of Nitrogen in ULSD

Organic nitrogen is known to adsorb on catalytically active sites, essentially blocking that site from the ability to perform hydroprocessing reactions. Eventually the organic nitrogen molecule will react and desorb from the active site but the relative slow rate of reaction at typical diesel hydrotreating conditions ensures that a high percentage of active sites will be blocked. When it is only necessary to remove sulfur molecules following the direct path of desulfurization, the relatively high rate of reaction for these molecules is not severely influenced by the concentration of nitrogen. Reactions involving aromatic saturation, however, are much slower and benefit greatly from increased active site availability. Since hydrogenation is required to remove SH-DBTs, the concentration of organic nitrogen has a very large effect on the apparent activity and performance of a catalyst system. Figure 1 shows the effect of nitrogen on HDS activity for KF-848, NiMo STARS catalyst.
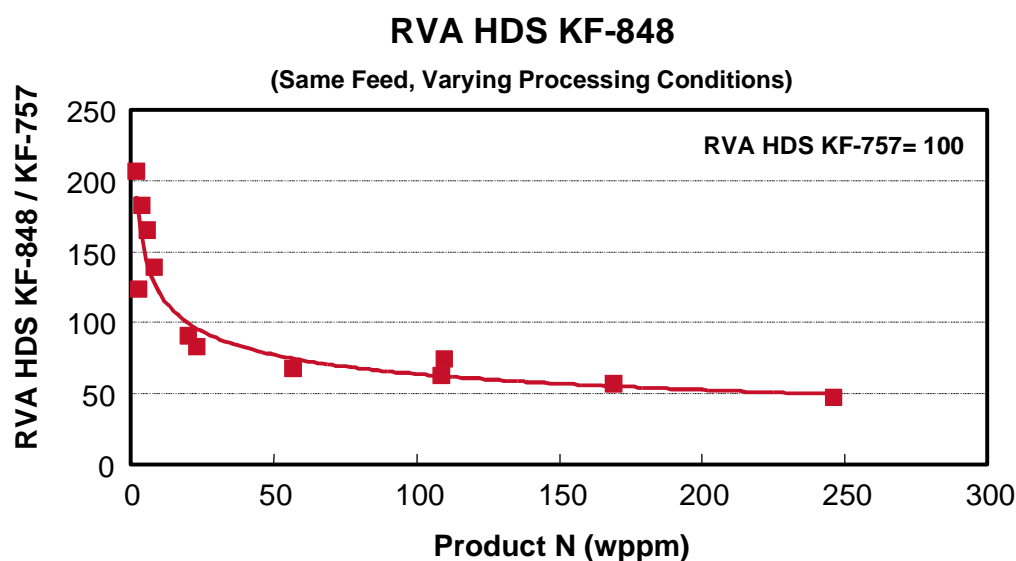
RVA HDS KF-848
(Same Feed, Varying Processing Conditions)

RVA HDS KF-757= 100

Figure 1: Effect of Nitrogen on Relative HDS Activity

The exponential increase in HDS activity at low product nitrogen content is due to the dramatic increase in the number of active sites available for hydrogenation. With a product nitrogen content of over 20 ppm it has been estimated that over 90% of total active sites are blocked by organic nitrogen. When operating conditions and sufficient catalyst activity are present in the reactor to reduce organic nitrogen, the activity of the catalyst system becomes much higher. As Figure 1 shows, the NiMo catalyst, KF-848, with a much higher hydrogenation and hydrodenitrogenation activity accelerates faster than the CoMo catalyst, KF-757.

Hydrogen Consumption in ULSD

Hydrogen consumption increases significantly when producing ultra low sulfur diesel compared to low sulfur diesel operation. As shown in Figure 2, hydrogen consumption will rise 20-40% when making 10 ppm S diesel as compared to 500 ppm S diesel. Though the hydrogenation route is required for removal of SH-DBTs, the relatively small amount of these molecules in the feed results in a negligible increase in hydrogen consumption. The main contributor

to the increased hydrogen consumption is aromatic saturation since the conditions necessary for removal of SH-DBTs are also favorable for aromatic saturation.

For the same level of sulfur removal, there is very little difference in hydrogen consumption between different catalysts. Even differences in composition, for instance NiMo and CoMo, show nearly the same hydrogen consumption for a given level of sulfur removal. This is not really surprising since, for the same sulfur content, the same number of SH-DBTs must be removed. The conditions in the reactor necessary to reach 10 ppm S will be different, depending on the catalyst, but the hydrogen consumption for a given sulfur content will be quite similar.
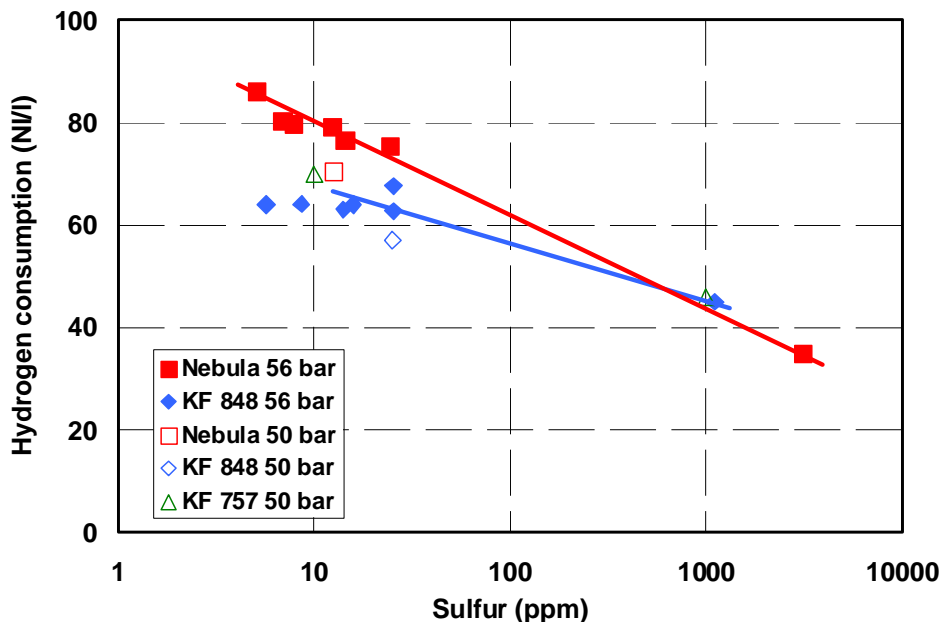

Figure 2: Hydrogen Consumption vs. Product Sulfur

One caveat to the preceding explanation occurs at high temperature. As shown in Figure 3 one must compensate for the aromatic equilibrium curve when comparing hydrogen consumption with different catalysts and reactor conditions. When temperatures are below the equilibrium point hydrogen consumption can be compared on an equal sulfur basis. However, once temperatures are higher than the equilibrium point, hydrogen consumption between catalysts cannot be reliably compared.

Hydrogen is a significant cost issue for refiners and when hydrotreating to make ULSD there is no doubt that hydrogen consumption will increase. However, refiners should not lose sight of the positive aspects of increased hydrogen content in terms of diesel volume and cetane. The economics of increased hydrogen consumption will vary by refinery, but in most cases the benefits of higher diesel hydrogen content will partially, if not fully, compensate for the cost of the extra hydrogen.
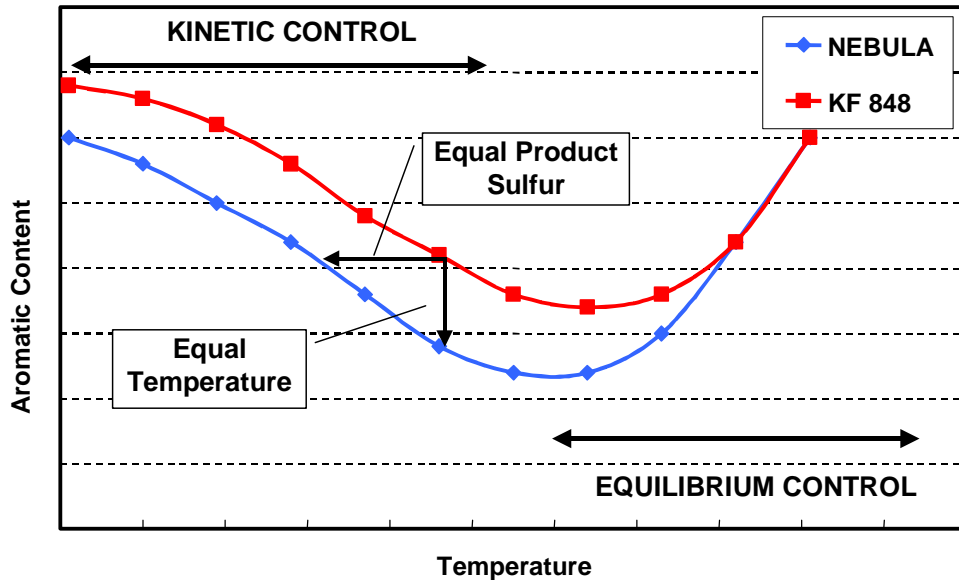
Figure 3: Effect of Temperature on Aromatic Content (Hydrogen Consumption)

Aromatic Equilibrium and the Sulfur Floor

Figure 3 demonstrates the high dependence of temperature on aromatic hydrogenation and thus overall hydrogen consumption. Since the first step of the hydrogenation route for SH-DBT removal is hydrogenation, one would expect the desulfurization rate to fall with the hydrogenation rate when producing ULSD. The result of this equilibrium control of hydrogenation rate is the sulfur floor effect, the point at which further increasing temperature has little to no effect on decreasing product sulfur. In practice, the effect is not so dramatic because the reaction rate for the second step in the SH-DBT removal process and for hydrodenitrogenation increases with rising temperatures. Nevertheless, at some temperature sulfur will no longer fall and as the temperature is increased beyond this point product sulfur will actually begin to increase. The exact product sulfur content at which the floor occurs is difficult to estimate because it is dependent on so many interrelated factors such as:

- concentration of SH-DBTs
- feed and product nitrogen content
- hydrogen pressure and circulation rate
- space velocity
- catalyst

The consequences of a sulfur floor are very significant for making ULSD. If the sulfur floor is too high, the refiner has few options other than decreasing feed endpoint or space velocity to meet the sulfur target. Operating philosophy changes will be needed to make sure that the first response to a high product sulfur content is not to immediately raise temperature. Upsets in the refinery, crude changes, etc., even if short-lived, will likely have serious implications on the ability of the diesel hydrotreater to produce ULSD. Feed distillation becomes a critical issue and possibly even the main control point for the diesel hydrotreater nearing end of run.

The sulfur floor also has a very significant impact on reactor design. The sulfur floor will likely set end of run temperature constraints lower than the product color or reactor metallurgical

limits of the past and this will significantly reduce the operating temperature window. Start of run operating temperatures in excess of the aromatic equilibrium temperature will clearly result in very short cycles. As a consequence, multiple catalyst beds with hydrogen quench in between becomes very advantageous.

III   COMMERCIAL EXPERIENCE WITH STARS TECHNOLOGY IN ULSD

BP, Grangemouth

BP first used a STARS catalyst in its diesel HDS unit in Grangemouth, near Edinburgh.[8] This unit, built in 1995, was originally designed for 500 ppm sulfur at 35,000 bpsd. It has a two-bed reactor, without quench, and operates at 45 bar (650 psi) ppH_2. KF-757 catalyst was dense loaded in March 1999. The unit currently runs at 42,000 bpsd (mainly North Sea LGO) and a product sulfur level below 50 ppm. The rate of deactivation during the first 400 days of operation was low.

BP, Coryton

Following the successful trial at Grangemouth, BP next used KF-757 at its Coryton refinery, which manufactures ultra low sulfur diesel in a 50,000 bpsd unit, revamped to 500 ppm sulfur in 1991. This has a two-bed reactor and product liquid quench between the beds. The unit runs under rather severe conditions: only 28 bar (405 psi) ppH_2 at reactor inlet. Test run results reported in this case are based on 100% LGO, although the feed can contain up to 20% LCO.

KF-757 catalyst was loaded in September 1999,[8] replacing KF-752. Though STARS catalysts can be presulfided with feed sulfur alone, in this case the catalyst presulfiding was done with DMDS due the low sulfur content of the feed. Product sulfur during the first 6 months of operation was <50 ppm, corresponding to >98% HDS as shown in Figure 4. Hydrotreating severity increased during the period because feed sulfur increased from about 0.2 wt% to about 0.5 wt%.
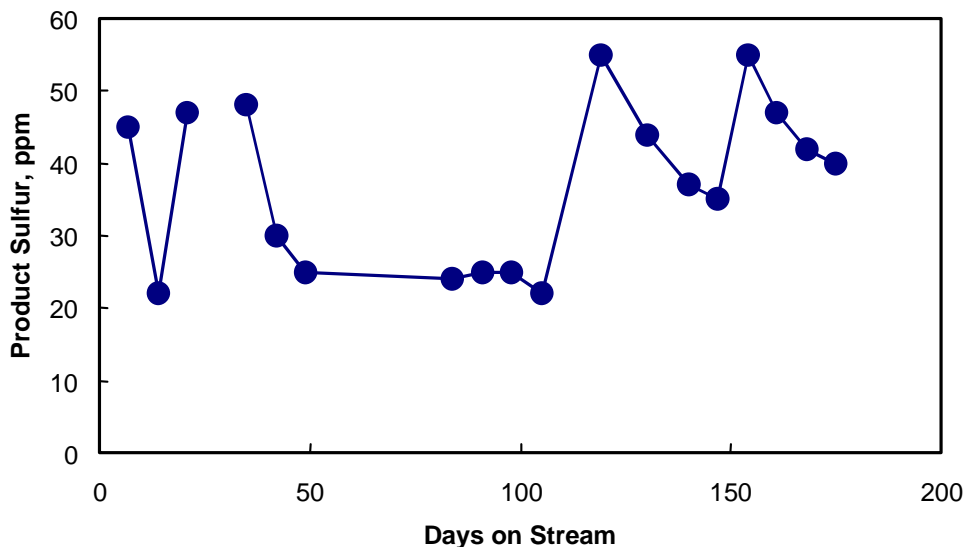

Figure 4: Coryton Product Sulfur

Figure 5 compares the normalized operating temperatures (WABT) for KF-757 and the previous catalyst, KF-752. This shows that the STARS catalyst operates at about 20°C (36°F) lower operating temperature. Catalyst deactivation is the difference between actual required operating temperature and the temperature calculated from Akzo Nobel's proprietary diesel HDS model for start-of-run conditions. This shows that the low operating temperature results in a very stable operation with less than 1°C (1.8°F) deactivation per month.
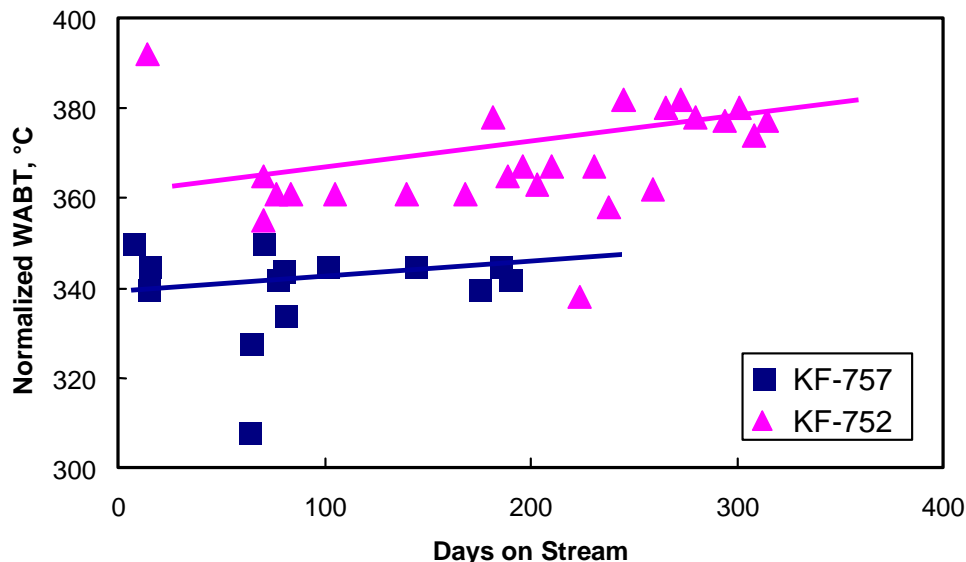

Figure 5: Coryton Normalized Bed Temperature

In a controlled series of test runs, Coryton examined the effect of operating temperature from 330 to 353°C (625-667°F) on product sulfur, holding operating conditions and feed quality constant. As shown in Figure 6, the test runs showed that beyond a certain operating temperature, the rate of sulfur removal hardly changed. This is a clear indication of the sulfur floor effect – under ultra deep HDS conditions, the molecules that remain in the product are mainly the SH-DBTs, which are preferably removed via pre-hydrogenation of one of the aromatic rings adjacent to the thiophene. Due to aromatic equilibrium constraints, this hydrogenation step is inhibited at higher operating temperatures. The best way to avoid this limitation is through the use of a high activity catalyst, which permits operation at lower temperatures.
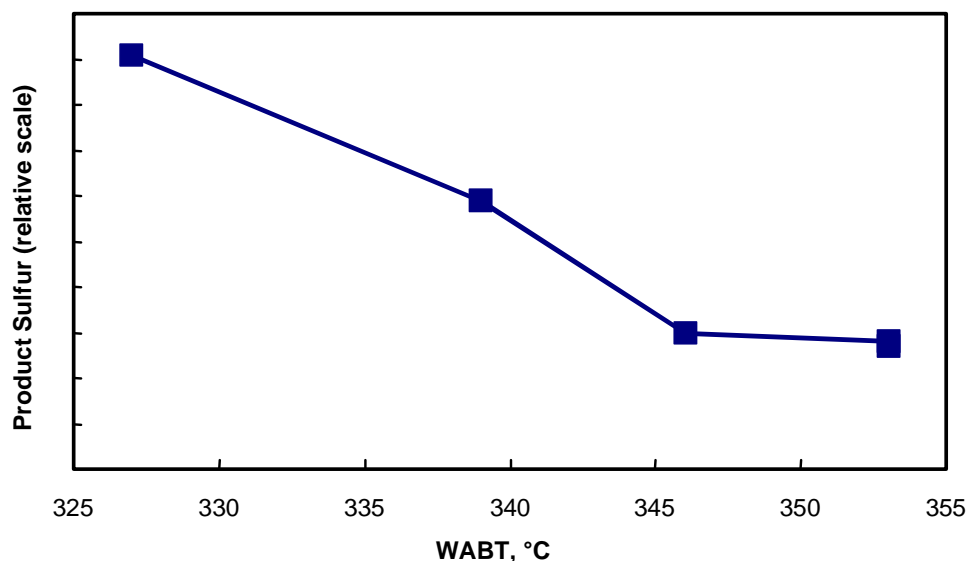
Figure 6: Coryton Test Runs – Product Sulfur vs. Reactor Bed Temperature

IV   COMMERCIAL EXPERIENCE WITH NEBULA TECHNOLOGY IN ULSD

As can be seen from the previous section, Akzo Nobel's STARS technology meant that BP was able to make <50 ppm S diesel using existing diesel HDS units without major revamp. However, even STARS did not suit all sites, particularly if 10 ppm S diesel was to be considered.

The commercial trial was made on a large hydrotreater dedicated to producing ultralow sulphur diesel for domestic use and for export. It was thus of economic benefit to be able to produce the tightest diesel specifications because many parts of Europe have tax incentives for 50ppm S diesel and are considering incentives for 10ppm S.

Prior to this turnaround, the refinery had used KF-756, a high activity CoMo catalyst for diesel HDS. For three years a stable and satisfactory performance had been achieved. With the coming catalyst change, test runs were done in late 2000 to investigate what product sulfur levels might be obtained and the impact on H_2 consumption. The main conclusions were as follows:

- >10 ppm product S could be produced at close to EOR conditions only by processing a light feed at reduced throughput
- H_2 consumption increased significantly depending on the process conditions

The test runs confirmed that to produce any significant amount of 10 ppm S operation would not be practical even with a STARS catalyst, due to excessively short cycle length. To be able to produce near sulfur free diesel on a continuous basis, the refinery considered increasing reactor volume. As an alternative, the discussion with Akzo Nobel about the Nebula technology were focused specifically on this unit because the catalyst change might avoid a considerable capital expenditure.

BP and Akzo Nobel undertook a detailed risk assessment to evaluate unit performance with the new technology. This involved close cooperation between the staff at the refinery, Akzo Nobel, BP Refining Technology and the BP Hydrotreating Network. In parallel with extensive pilot tests at Akzo Nobel, BP also carried out confirmatory pilot testing which broadly confirmed

Akzo Nobel's claims that Nebula was significantly more active than STARS catalyst (Figure 7). BP's process model suggested that the activity of the new catalyst in the pilot plant was at least twice as active as KF-757.
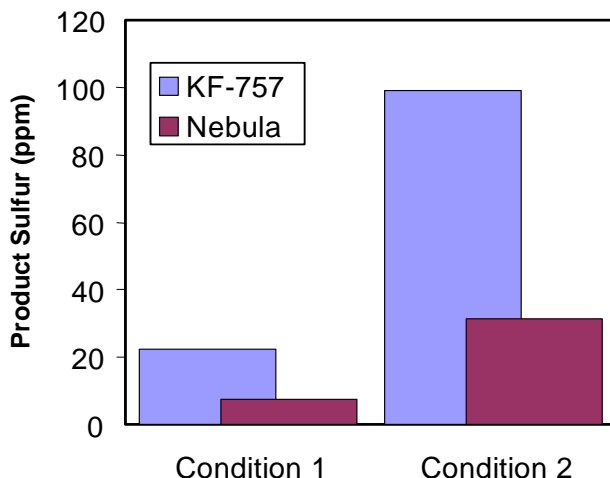

Figure7: BP Pilot Runs – Nebula vs. KF-757 (Feed = 0.857 SG, 0.78 wt% S)

In the economic evaluation, the impact of H_2 consumption was taken into account. Refineries running their DHTs using low purity hydrogen from the reformer may have to make up any shortfall through imports of high purity hydrogen. The latter is incrementally quite expensive and should be as low as possible to minimize production costs. Akzo Nobel's pilot plant test program was developed to check:

- activity of KF-848 and Nebula at the commercial conditions
- capability for removal of SH-DBTs
- feasibility of applying very high activity catalysts
- impact of LCO in the feed
- impact of H_2 purity

KF-848 and Nebula-1 were loaded in separate pilot plant reactors with independent heating, allowing the reactors to be operated at constant product sulfur. The WABT was adapted to reach the target sulfur so that the H_2 consumption at constant product sulfur could be evaluated rather than at constant reactor temperature. This is important in order to get a good comparison of H_2 consumption at actual operating conditions. Table 2 and Table 3 show the pilot plant feed properties and test conditions, respectively. Note that when switching to 10-20% LCO in feed, the LHSV was reduced for KF-848 because at high LHSV KF-848 could not reach the target sulfur level without exceeding the maximum WABT.

Table 2: Pilot Plant Test Feed Properties

Feed LCO content, %	0	10	20
SG	0.855	0.866	0.878
Sulfur, wt%	0.86	1.0	1.2
Nitrogen, ppm	119	206	293
Total aromatics, %	32.1	37.3	42.7
D86 distillation, °C (°F)			
IBP	199 (390)	204 (399)	207 (405)
90%	350 (662)	353 (667)	355 (671)
95%	363 (685)	365 (689)	370 (698)
FBP	370 (698)	374 (705)	378 (712)

Table 3: Pilot Plant Test Operating Conditions

LHSV, hr^{-1}	2.3
H$_2$/Oil, Nl/l (SCFB)	200 (1120)
Pressure, bar (psi)	50 (725)

As shown in Table 4, we concluded the following from the test work:

<u>On SR LGO</u>

- <10 ppm S is achieved at 349°C (660°F) with 100% Nebula
- the activity difference between Nebula and KF-848 is ~30°C (50°F)

<u>On 10% LCO</u>

- with Nebula, WABT needs to be increased ~15°C (27°F) compared to the LGO case
- ULSD cannot be produced with KF-848 at LHSV of >2 hr^{-1}

Table 4: Pilot Plant Test Results

Condition	1	2	3
%LCO	0	10	20
Pressure, bar (psi)	50 (725)	50 (725)	50 (725)
WABT, °C (°F)			
Nebula-1	349 (660)	360 (680)	360 (680)
KF-848	378 (712)	383 (721)*	383 (721)*
Product S, ppm			
Nebula-1	9	17	70
KF-848	14	14*	41*
H$_2$ Cons., Nl/l (SCFB)			
Nebula-1	50 (300)	53 (315)	54 (321)
KF-848	33 (196)	36 (214)*	41 (244)*

*LHSV reduced from 2.3 to 1.0, as KF-848 reached EOR WABT

The difference in H_2 consumption between Nebula and KF-848 is high, but it must taken into account that:

- KF-848 is deactivating more quickly than Nebula because it is operated at a high temperature
- At a high temperature, aromatic saturation drops because of thermodynamic restrictions. If KF-848 had been tested at the same WABT as Nebula, but different LHSV to achieve the same product sulfur, the difference in H_2 consumption would have been much smaller. The latter would have been very relevant in case the refinery decided to increase reactor volume.

The impact of temperature on H_2 consumption is shown in Figure 8. The data included in this graph were measured on SR LGO without LCO.
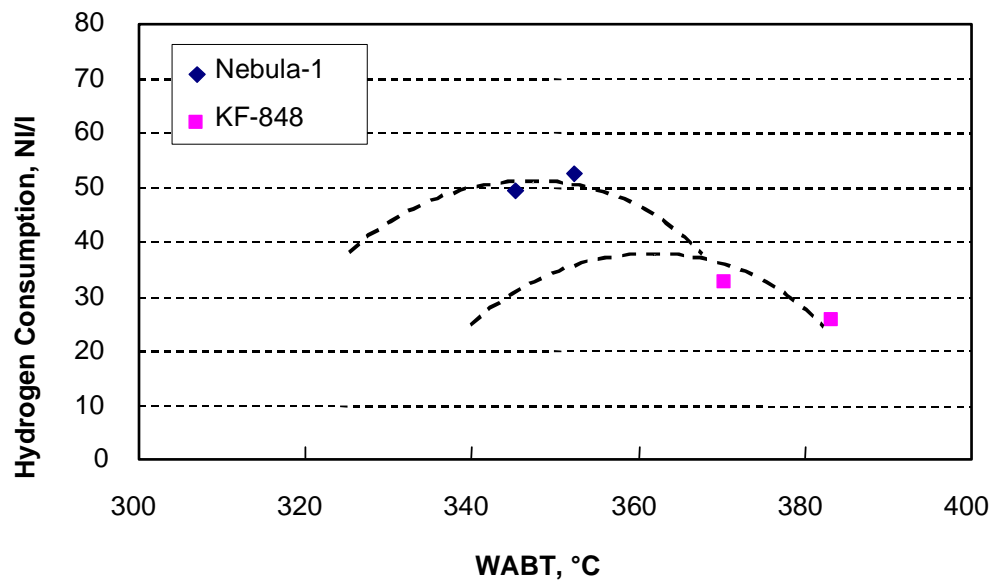

Figure 8: Effect of Reactor Temperature on H_2 Consumption

Based on the test work, Akzo Nobel made a recommendation and forecast for a system with 50% KF-848 and 50% Nebula-1. Based on this information, an economic comparison was made between the catalyst change and installation of extra reactor volume. This analysis showed a benefit for changing to the KF-848/Nebula-1 catalyst system. From start to finish, the evaluation took less than 3 months. KF-848 and Nebula-1 were loaded into the unit and started up in the second half of 2001.

The refinery hydrotreater has two beds – one was filled with Nebula, the other with KF-848. Procedures for catalyst loading and activation were straightforward. Because of the high catalyst activity, the feed quality and operating severity were increased gradually to break-in the catalyst. Since start-up, the refinery has operated the unit mainly to produce ultra low sulfur diesel at 50 ppm S, but test runs at full feed rate showed that <10 ppm S operation could be achieved with KF-848/Nebula-1 at substantially lower reactor temperature than predicted for a conventional catalyst. Process modeling by BP suggests that Nebula-1 is operating with at least twice the activity of alternative catalysts.

V   CONCLUSIONS

The drive for lower sulfur fuels has triggered an acceleration in catalyst development over the last few years. With STARS and Nebula technology Akzo Nobel Catalysts and Nippon Ketjen have provided breakthrough solutions for the refining industry to help reach the new targets. STARS has become the workhorse technology for production of ULSD and Nebula has been proven to be a "drop-in" solution to clean fuels in some cases. As important as the catalyst developments themselves is understanding the application of the catalysts. Knowledge and understanding of the kinetics of ultra low sulfur diesel continues to improve.

BP has achieved breakthrough business performance through use of Akzo Nobel's latest generation catalysts to make ultra low sulfur diesel. BP was the first major user of STARS technology for 50 ppm S diesel in the UK and of Nebula technology to make 10 ppm S diesel in Europe. BP believes that catalysts using Nebula technology have exceptional activity, possibly a factor of 2-3 above other catalysts available in the industry.

A key success factor to implementing these technologies has been BP's business approach. This allows early and rapid implementation of the most valuable technologies, thus creating maximum value and a differentiated position in the industry. Excellent supplier relationships, active internal networks and robust management processes allow fast decision-making encompassing risk analysis and performance assurance, so that technology roll out is faster than that of competitors. BP concentrates on early awareness, access and implementation of the best technologies whatever the source. Where appropriate, BP creates new technologies ahead of the market, followed by extremely fast deployment at commercial scale.

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