Advanced Refining Technologies®

Delivering customized, environmentally friendly, catalytic solutions for the dynamic petroleum refining industry.
ART, the leading supplier of hydroprocessing catalysts, introduces three new catalysts for 2013.

No matter what system your refinery uses, Advanced Refining Technologies has the broadest, state-of-the-art catalyst portfolio, backed by the world-class R&D of Chevron and the manufacturing strength of Grace.

**Distillate Hydrotreating • 545DX** - enhanced ULSD run length

- Increased cycle length
- Maximized margins with opportunity feedstocks
- High versatility for ULSD applications
- Increased ability to process high endpoint and highly aromatic feeds

**Fixed Bed Resid Processing • ICR 173** - new deep MCR and S conversion catalyst

- Excellent sulfur, MCR and nitrogen conversion activity
- Superior metals tolerance
- Outstanding performance from middle to end of run
- Improved profitability with the use of cheaper, heavier crudes

**Ebullating Bed Resid Hydrocracking • HCRC™ (High Catalytic Resid Conversion) Technology** - higher catalytic and lower thermal conversion for EB RHC units

- Ultra-high activity
- Higher catalytic and lower thermal conversion
- Improved HDS, HDMCR, HDN and product API
- Flexibility to move to lower reaction severity, reducing sediment/coke formation
- A true breakthrough in catalyst technology

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Editorial

Dear Hydroprocessor:

Catalagram 112 SE (Special Edition) marks the fifth ART-only issue produced. We hope you have found the articles informative and the information contained helpful in meeting your challenges in hydroprocessing.

ART continues to grow and plan for the future. We’ve recently introduced four new catalysts. For distillate hydrotreating, there are 545DX for enhanced ULSD run length (article on page 2) and 425DX for enhanced ULSD at low pressure. For fixed bed resid hydrotreating units, ICR 173 is a new deep MCR and S conversion catalyst, and HCRC™ (High Catalytic Resid Conversion) Technology delivers higher catalytic and lower thermal conversion for ebulliating bed resid hydrocracking units.

In addition, we are expanding and upgrading our plants to produce these new catalysts and future ones that are currently under development. These products are being designed to help refiners meet the challenges of the expected growth in demand for diesel and other low-sulfur fuels, while continuing to further upgrade the bottom of the barrel.

Finally, we are expanding our staff to deliver better service to you. Our “People on the Move” on page 18 introduces you to recently added members of our team who will be supporting our customers in all three segments.

We at ART are committed to your success in hydrotreating and are actively growing our business to meet your current and future needs.

Sincerely,
Scott K. Purnell
Managing Director
Advanced Refining Technologies
Advanced Refining Technologies® continues to expand its line of ultra high activity DX® Catalyst Series in response to refiners’ demands for superior technology that delivers premium performance. This family of catalysts has exceeded expectations with its performance in demanding ULSD applications and the expansion of this line with 545DX will provide additional opportunities for refiners wanting additional capacity and increased yields without sacrificing cycle life. The ability to process difficult feed blends is one of the key advantages observed with this catalyst family. ART’s 420DX and NDXi have both demonstrated the benefits of this technology in ULSD units around the world, both as stand-alone catalysts and as part of a SmART® staged catalyst system.

545DX builds on this great success as shown in Figure 1, which compares the activity of several generations of ART NiMo catalysts. The figure shows that 545DX offers significant improvement in both HDS and HDN activity over NDXi. The feedstock used in this work contained 1.26 wt.% sulfur and 130 wppm nitrogen. 545DX has provided over a 25°F (14°C) advantage for HDS compared to NDXi as well as a 20°F (11°C) improvement in HDN activity.

Researchers at ART have been able to create a novel alumina support that was identified as a key property for improved catalytic performance. It is understood that there is a strong relationship between the role of increased surface acidity coupled with a tailored pore size distribution for improving the kinetic ability of the catalyst for reactions controlled through ring saturation, such as nitrogen and hard sulfur removal. This catalyst utilizes similar impregnation technology as NDXi using a chelate to bind to the nickel ions in the impregnation solution and reduce interactions with the alumina support. With some modifications to the manufacturing process, ART has been able to enhance this interaction allowing the chelate/ion complex to stay intact on the catalyst surface and promote the formation of significantly more Type II active sites.

In a concentrated effort to understand the improved activity of 545DX, ART has completed pilot plant testing over a wide variety of conditions and feedstocks, which clearly demonstrates the performance advantage available to refiners. Figure 2 shows the results of side-by-side testing of NDXi and 545DX at 775 psi hydrogen partial pressure and 2200 SCFB H₂/Oil ratio. At these conditions, 545DX clearly outperforms NDXi by over 20°F (11°C) at 10 ppm sulfur on a difficult feed containing 30% light cycle oil (LCO).
Additional benefits of 545DX are the improved HDN activity and increased aromatic saturation capabilities. This offers the flexibility for refiners to meet their HDS activity requirement while gaining additional volume swell. Figure 3 compares the additional API upgrade with 545DX relative to NDXi over a range of operating temperatures. As the chart shows, 545DX can provide upwards of one full number higher API upgrade when producing ULSD as compared to NDXi.

The need for refiners with lower pressure applications to be able to gain additional activity can present a kinetic challenge and is often addressed by utilizing predominantly CoMo type catalysts. The ability of NiMo catalyst to handle lower hydrogen pressure situations can be beneficial and can allow these refiners to increase throughput as well as see gains in cycle life. Figure 4 compares NDXi with 545DX at 580 psi hydrogen partial pressure over a range of LHSV, and it can be seen that, even as the process conditions become more severe, 545DX maintains its activity advantage over NDXi for ULSD.

The additional HDS activity combined with improved nitrogen removal and aromatic saturation allows refiners to utilize 545DX as a stand-alone catalyst for maximum upgrade in refinery markets demanding increased yields. 545DX can also be coupled with ART’s premium CoMo catalyst 420DX, in a SmartART® Catalyst System, which is ideal for hydrotreaters that need to operate with controlled or minimized hydrogen consumption. These units are able to benefit from a lower start of run temperature, as well as being able to gain some additional yield improvements that are not often gained in a hydrotreater system of 100% CoMo.

Extensive pilot testing and expertise enable ART to provide the right catalyst system tailored for maximum refinery profit. 545DX will enable refiners to enhance their ULSD operation with either increased cycle length or additional use of opportunity feedstocks in order to maximize margin. The ability of 545DX to perform in different configurations provides a high level of versatility and makes it a top tier catalyst capable of exceeding refiners’ needs in demanding ULSD applications.
In an effort to lower costs and improve refining margins, refiners often look for opportunities to purchase lower cost crudes or other feedstocks. While these opportunity feeds can help improve profitability, there can be some unpleasant consequences. Many of these new feeds contain unknown levels of catalyst poisons such as silicon (Si) and arsenic (As). ART has seen an increase in the number of units experiencing poisoning from these contaminants in recent years as significantly more of these opportunity feeds are processed.

**Silicon**

Silicon is probably the most widespread catalyst poison encountered in hydrotreater feeds. The main source of silicon is from delayed coker operations which use an anti-foam agent based on polydimethylsiloxane to suppress foaming in the coker drums. The siloxane complex breaks down in the coking process to primarily cyclic methylsiloxane trimers. These species are volatile at coker temperatures with boiling points ranging from 270-475°F (132-246°C). As a result, these compounds tend to concentrate in the overhead products and, as a general rule of thumb, 70-80% of the silicon at the coker ends up in the coker naphtha fraction. More recently, even refineries that do not have cokers are experiencing silicon poisoning of hydrotreating catalysts once thought unlikely since their feed source comes directly from the refiner’s crude unit. These refineries have begun processing synthetic or other opportunity crudes and the process of making synthetic crude often involves a coking step. In addition, it is becoming more common to use silicon additives in the drilling process, and for pipeline companies to use them for both flow enhancing performance and foaming issues. It has also been found that silicon additives are sometimes used in barge unloading.

In the hydrotreater the silica fragments from the antifoam agent undergo a condensation reaction with the alumina surface of the catalyst forming a strong chemical bond. Once the silicon is bound to the alumina surface it cannot be removed by regeneration or other means. It is a more moderate poison compared to contaminants like sodium or arsenic, but it nonetheless results in activity loss of the order of 5-10°F (3-6°C) for each 1.0 wt.% Si deposited on the hydrotreating catalyst. The impact is worse for other catalysts which are used in processes downstream from the hydrotreater (i.e. reformers, isom units). Figure 1 shows the results of pilot plant testing which demonstrates the activity loss due to silicon contamination.
A variety of analytical techniques have been applied to silicon poisoned catalysts, and this confirms that the silicon is associated with the alumina support as opposed to the active metal sulfides of the catalyst. Furthermore, the silicon is dispersed throughout the available alumina surface as opposed to poisoning only the exterior of the catalyst pellet. As a consequence, the available alumina surface area of a catalyst has a significant impact on silicon capacity of a catalyst. This is shown in Figure 2 for a number of different ART catalysts. It is a plot of the relative silicon contamination observed on spent catalyst following a careful regeneration as a function of the fresh catalyst surface area. The capacity for silicon increases by a factor of 3 or more as the surface area per unit volume increases from roughly 100 m²/cc to over 200 m²/cc.

Another important aspect of silicon poisoning is that silicon pick up depends on unit operating temperature. Figure 3 shows the temperature dependence of silicon pick up for ART’s GSK-6A, an active ring used in top bed grading, and AT724G, ART’s premier silicon guard catalyst. The data represent spent catalysts and baskets samples from a variety of applications ranging from low temperature guard reactors to higher temperature applications like ULSD units.

The data clearly show that the operating temperature of the application must be considered when discussing silicon pickup capacity and when designing effective guard catalyst systems. The maximum capacity of the catalyst needs to be considered as well as the capacity at the operating temperatures of the specific unit in order to accurately predict the point at which silicon will break through into the next bed of catalyst or refinery unit.
Arsenic

Another poison which is becoming more common with the increase use of some synthetic and opportunity crudes is arsenic. Arsenic is also a permanent poison, so once it is on the catalyst it cannot be removed. It is a much more severe poison than silicon with roughly 60°F (33°C) of activity loss per 1.0 wt% arsenic on the catalyst. Figure 4 shows the results of pilot plant testing which clearly demonstrates the detrimental effects of arsenic on catalyst activity.

Unlike silicon, it is believed that arsenic reacts with the metal sulfide sites on the catalyst, and in particular, has an affinity for the nickel on the catalyst forming nickel arsenide. This suggests that catalysts containing high levels of nickel will pick up higher levels of arsenic. Figure 5 demonstrates this nicely. It shows the relative amount of arsenic pick up as a function of the nickel content on the fresh catalyst. This data represents a variety of ART NiMo catalysts which were retrieved from different hydrotreating applications which explains some of the scatter, but the trend is clear. Catalysts containing higher levels of nickel can pick up six times or more arsenic compared to catalysts with lower nickel levels.

Just like silicon, arsenic poisoning is an activated process, thus the arsenic capacity of a catalyst is strongly temperature dependent. This is demonstrated in Figure 6 which shows the arsenic pick up as a function of temperature for an ART NiMo catalyst. The arsenic pick up increases by a factor of 14 as temperature increases from 270-650°F (132-343°C).

Silicon Trapping

ART has long held a strong position in applications dealing with silicon contamination, and this was strengthened by technology advances like the START® (Silicon tolerance by ART) Catalyst System now with a decade of operating experience1. Figure 7 shows the results of basket testing for AT724G and AT535, key components of a
START system, against some competitive catalyst offerings. AT535 alone has essentially the same Si capacity as the competitor catalysts while AT724G has over 30% higher Si capacity; the data show the combination of AT724G and AT535 has the highest capacity and activity.

**Arsenic Trapping**

The START portfolio was further strengthened several years ago with the introduction of AT734G which is a combined silicon and arsenic guard. The new catalyst is built on the AT724G platform and exploits the different poisoning mechanisms for silicon and arsenic. It has very high alumina surface area for high silicon capacity and a higher nickel content imparting high arsenic capacity. AT734G has over four times the arsenic capacity of AT724G with similar silicon capacity. Figure 8 shows an example of the arsenic pick up capabilities observed in a basket test comparing AT724G, AT734G and AT535. The basket was installed in a cracked naphtha unit at a refinery processing synthetic crudes.

**Even Higher Capacity**

With the increasing incidence of silicon and arsenic poisoning, ART saw the need to make further improvements to the START system portfolio. This lead to the development of AT714G, a very high surface area guard with lower active metals content compared to AT724G. The new guard has extremely high silicon capacity, and the lower activity makes it ideal for activity grading. In addition, AT714G does not need to be activated in order for the silicon guard functionality to be effective. Figure 9 shows catalyst basket results comparing the silicon pick up of AT714G with AT724G, AT535 and a competitor catalyst. In this example AT714G picks up over 40% more silicon than AT724G which significantly increases the silicon capacity of the START system.

**Silicon Tolerant Catalyst**

The main component of the START system is AT535, a high activity NiMo catalyst. It has been widely recognized as one of the leading catalysts for naphtha and cracked naphtha applications as evidenced by over 300 units worldwide since its introduction. Even with this excellent track record, ART recognized a need for a high activity catalyst that is even more silicon tolerant than AT535. Utilizing the knowledge gained from numerous basket studies and spent catalyst analysis, some of which are outlined above, has allowed ART to design a new silicon tolerant catalyst, AT525. Figure 10 summarizes the results of a basket study comparing AT525, AT535 and a competitor catalyst. In this case both AT535 and AT525 outperform the competitor catalyst for silicon pick up by at least 15%, and it is readily apparent that AT525 significantly outperforms all catalysts in the test.
Advanced Refining Technologies has significantly enhanced its already strong position in naphtha and cracked naphtha applications, and has added another tool for silicon and arsenic trapping in ULSD and other applications. The StART system now includes an improved stand alone silicon tolerant catalyst AT525, and a new high-performance silicon guard material AT714G. These new additions combined with AT724G and AT734G offer unparalleled protection from silicon and arsenic allowing refiners to further extend unit cycle length under the most adverse conditions. This technology can be applied across a wide range of distillate hydrotreating applications enabling refiners to be better prepared to deal with the unexpected. Let the ART technical group custom fit these products into your process for improved reliability and performance.

**References**


**Introduction**

Advanced Refining Technologies (ART) is dedicated to the highest standards of health, safety and regulatory compliance. ART’s goals are to establish an outstanding record of product safety and to practice strong corporate citizenship. To achieve these goals ART has embraced Product Stewardship by implementing numerous significant refinements to its existing product stewardship program and is committed to continuous product stewardship improvement. Like many chemical suppliers, ART defines Product Stewardship (PS) as the responsible management of the health, safety, environmental and regulatory issues of a product throughout its life cycle. A robust product stewardship program provides ART a competitive advantage that should also benefit its customers.

The ART PS program is global in nature to support a portfolio of products that is manufactured at facilities worldwide operated by Grace and other partners. The ART program employs aspects from both partners of the joint venture. Grace provides the lead PS support to the joint venture, incorporating PS into all ART business decisions. Product stewardship is also a fundamental component of Chevron’s Operational Excellence (OE) framework, creating commitment by both partners in the ART venture to the PS program.

**New Product Development**

During new product development health, safety, and environmental considerations are integrated along with sustainability and other critical product stewardship elements into the process. From development through disposal ART applies PS focus to all steps shown in Figure 1. ART also works with other stakeholders such as customers, suppliers, and transporters to assure that our products can be handled safely, with acceptable risk and in compliance with all applicable regulations.

The ART PS program has been redesigned to improve the management of both today’s applicable chemical inventory laws and consideration of similar emerging chemical control regulations. A high level of focus is also placed on addressing other regulatory requirements such as the evolution of the Globally Harmonized System (GHS) for standardizing and harmonizing the classification and labeling of chemicals.

The foundation systems employed by ART are the stage gate product development PRISM (PRoduct Innovation and Strategic Marketing) process shown in Figure 2, and Management of Change (MOC).

The PRISM process is used to balance the business potential of a new product with the cost and technical risk associated with the development effort. As a new product advances through the stages, the benefits that it will bring to customers in improved performance...
FIGURE 2: PRISM Stage Gate Process

FIGURE 3: PRISM PS Deliverables
are weighed against the requirements needed to bring the product to market. As a result, the early stages of PRISM are used to evaluate the PS impact of the project both from a product composition and manufacturing process standpoint.

A detailed PS checklist is employed to specifically focus product introduction on the regulatory requirements and Environmental, Health and Safety (EHS) best practices that are addressed at each Stage in the PRISM process. During the Idea Evaluation Stage, a preliminary Risk Assessment is conducted. Often during this stage, a number of candidate technologies and possible formulations are explored simultaneously to determine if any of them would meet the product concept. The preliminary risk assessment allows the business leaders to understand if there are potentially higher PS or EHS risks associated with one formulation over another. Products or technologies with higher risk may be de-selected at this stage and would only advance if the hazards and risks can be adequately controlled.

Once the Risk Assessment has occurred, subsequent stages require additional PS actions as shown in Figure 3. Deliverables listed in multiple stages reflect escalating requirements as the product development process advances.

**Product Composition**

Product composition is the starting point for most product stewardship activities. It is also a major focus of ART’s PS program due to regulations such as Europe’s REACH (Registration, Evaluation, and Authorization of Chemicals), a newly created chemical inventory in Taiwan, on-going changes in China’s chemical management programs and expected changes to programs in Korea and other growing markets. These new or significantly revised regulations coupled with the expanding implementation of GHS require a heightened level of attention to the regulatory implications of product composition in order to minimize the potential for negative business impacts. If new substances are identified then they must be registered in those countries with chemical inventories (e.g., Canada, China, Japan, Korea) to assure compliance when products are imported into that country.

Proper registration of the substances contained in a product is both expensive and resource demanding. Approval of the registration application by the national agency assigned this responsibility can also be very lengthy, and there is always the potential for application rejection should the information provided not be complete or adequate. Recent applications submitted by ART are often over several hundred pages long, and full approval by the regulatory agency can take well in excess of nine months. For this reason, substance registration is performed early in the PRISM process; thereby also insuring that the potential for disruption of product supply, should a regulating agency question composition is diminished.

Substance registration typically requires submission of a significant amount of technical data on the substance, which must be generated through testing. The technical data requirement has resulted in large amounts of new data that ART has incorporated into MSDS’s and product information, affording our customers improved information on safe handling of ART catalysts and mitigating product risk.

For example, the European CHemicals Agency (ECHA) announced in February 2012 that boron oxide, a substance that would be contained in any commercial boron based hydrotreating catalyst, would be classified as a Substance of Very High Concern (SVHC) based on toxic impact on reproduction. This classification likely will mean that the substance will be incorporated into the Authorization process, which is a supplemental regulatory obligation that is both costly and rigorous in terms of reporting requirements for users. ART is phasing out products containing boron in anticipation of other countries adopting a similar position on boron oxide. This proactive approach is an advantage to ART customers who can be assured that our product portfolio is routinely adjusted to reduce regulatory risk.

**Product Packaging**

In the Development stage of PRISM, manufacturing process consideration such as product labeling and packaging are evaluated, and any special handling of the packaged material is also finalized. The vast majority of ART Fixed Bed Resid (FBR) and Distillate HydroTreating (DHT) products are delivered in bulk bags, which ART recommends not be reused. The bags are UN approved Intermediate Bulk Containers (IBCs – see Figure 4) which are specifically required for hazardous materials such as cobalt when shipped internationally.
ART recommends that used bags be shredded and discarded according to all state and local environmental regulations. Bags that are not shredded should be disposed of according to local environmental regulations and in a manner such that the bags cannot be reused for any other purpose. If products are packaged in drums, labels should be removed and drums should be reclaimed, recycled or reused. If drums are reused, ART recommends that all labels be removed and those that contained nickel, cobalt or molybdenum products be triple rinsed before reuse.

**PS Legacy Product Review**

Recognizing the product stewardship benefits resulting from the PRISM process, ART products commercialized prior to the adoption of this program were reviewed using the PS elements defined in PRISM. Legacy products were reviewed to confirm that they are also managed safely and in compliance with all regulatory requirements. This PS review was conducted across all 3 segments (Fixed Bed Resid, Ebulating Bed Resid, Distillate Hydrotreating) with significant focus downstream of the manufacturing process as shown in Figure 5.

This analysis considered potential hazards and risks of existing products, along with control measures such as MSDS, labels, customer communications, regulatory and other documents. Priorities for additional control measures were identified as part of a continuous improvement plan.

**Customer Communications**

A significant portion of PS information generated during the PRISM process is only useful if there are systems in place to effectively distribute the information to the users of ART products. Customer communications have been enhanced through a comprehensive program recently introduced to provide product regulatory conformance information to all current and new ART customers. This program includes annual PS reviews with customers to ensure that the latest versions of MSDS’s are available for all ART products that are at the customer site. A PS folder was created to contain the MSDS’s supplied to the customer during the review, highlight ART’s commitment to the program, and provide handling guidelines for ART products. This folder, along with other key PS documentation, is available on the www.e-catalysts.com website in the Technical Service/Product Stewardship section.
ART sales and service personnel also encourage customers to distribute MSDS’s for ART products to any 3rd party facilities or personnel who may store or handle the products. This is often relevant when products are purchased and require storage well in advance of planned turnarounds and when outside personnel are used to load the materials. Technical guidelines provided to customers by ART such as startup or loading procedures are vetted by PS personnel to ensure they contain recommendations that are consistent with the information provided in the MSDS.

**Additional Advantages**

The focus of ART’s PS program is now centered on increased customer involvement. By incorporating customer PS objectives into the program, customers who purchase products from ART will have greater confidence that they will receive the support necessary to help address their PS and associated requirements. ART is achieving its commitment to global regulatory requirements with support from dedicated Grace PS personnel located around the world. These personnel review new country requirements and incorporate them into existing programs to maintain global regulatory compliance. Some examples of the ways that customers can use ART PS resources include: to increase safety awareness for non-refinery personnel and new employees, to support refinery MOC programs, to resolve shelf life questions, to provide current MSDS’s for various products on site, and to support customer reporting obligations for REACH or other regulatory programs.

**Conclusion**

Our customers and neighboring communities can be confident that ART follows established environmental, health and safety (EHS) programs and that we are continuously working to reduce the impact our facilities and products have on the global environment. For additional information please call your ART sales and service personnel.
As the distillate markets allow for increased distribution worldwide, refiners need to be more aware of the additional product specifications that can be present at the various locations. One product property that is difficult to modify with general hydrotreating is the ability to improve (lower) the cloud point and cold filter plugging point of the diesel. Cold flow properties are determined by the wax or crystals that are formed as the diesel is cooled. The formation of these crystals can plug filters and lead to poor engine performance.

There are various approaches to meeting cold flow targets, the simplest of which is the blending of lighter material (kerosene or jet) into the fuel. Other options include the use of additives, solvent dewaxing, or adding a separate isomerization reactor. All of these options have disadvantages, including high costs or yield losses. Diluting with blending stocks, such as kerosene, has the added complication that the blending stocks must separately meet all of the same requirements, such as sulfur, of the finished ultra low sulfur diesel (ULSD) and could require that the blending stock undergo additional hydrotreating. This also involves taking a higher value fuel, degrading its value by blending it into diesel, and will have volume limits in order to stay within distillation and flash point specifications for diesel fuel.

The ability to modify the cold flow properties of the diesel in the hydrotreater can have significant economic advantages that the other options do not provide. Use of a specialty catalyst is required in order to do catalytic or hydro-dewaxing (HDW) to improve the cold flow product properties within the ULSD hydrotreater complex, and to avoid making changes to naphthenes or iso-paraffins that already have acceptable cold flow properties. Understanding the cold flow requirements first is necessary to create an individually tailored process and avoid the pitfalls associated with inappropriate quantities of HDS catalyst such as yield losses and not having the flexibility to meet market demands. Figure 1 shows some simple reactions that can effectively improve the cold flow properties of the diesel product. The resultant products contain some olefinic material due to the cracking mechanism and require proper catalyst staging to achieve process goals.
The typical process of dewaxing utilizes a ZSM-5 type catalyst. The structure of ZSM-5 is such that only straight chained hydrocarbon molecules (normal paraffins or n-paraffins) fit inside the cage structure and are cracked into smaller, lighter molecules. These molecules have significantly lower cloud and pour point characteristics. Figure 2 shows some of the various n-paraffins present in a typical diesel boiling range. The melting point is what influences the cloud point of the diesel if left unconverted, and of course, the higher the carbon content, the higher the boiling point.

Due to the nature and structure of the zeolite, the catalysis choice is important as these structures can easily be poisoned by nitrogen and olefins present in the feed. Even in high-pressure applications hydrotreating has only a small impact on product cloud point. Figure 3 shows the effect of hydrotreating on cloud point at 1400 Psi hydrogen partial pressure and with a feed containing 50% cracked material. This figure examines the cloud point improvement across a wide range of product sulfur and operating temperatures. Even at high temperatures, well beyond that required to produce ULSD, there is little change in product cloud point.

Typically, the target market for these products requires more than several degrees decrease in cloud point below the value of the feed. This suggests the use of hydro dewaxing in conjunction with the ULSD unit is desirable.

As mentioned previously, for HDW catalyst to perform most efficiently requires some hydrotreating first since it is susceptible to poisoning from the organic sulfur and nitrogen present in the feed. Figure 4 shows pilot plant results from operating a system using untreated feed over dewax catalyst at 5.75 LHSV to simulate the feed rate over a dewax bed in a hydrotreater. This work was completed over the entire range of temperatures for a ULSD hydrotreater from start of run to end of run. As the figure shows, there is very little change in product values. The cloud point of the product is improved only slightly and this corresponds with a small increase in bromine number, which is expected based on the reactions listed in Figure 1. It is important to note that it also shows that there is very little sulfur and nitrogen removal, and interestingly enough there is actually a slight increase in the volume percent for mono and poly aromatic species in the product, as indicated by a negative percent change.

Decreasing the LHSV over the dewax catalyst bed produces similar trends for the total and poly aromatics conversion as well as the HDN and HDS conversions. There are only a few degrees change in cloud point and almost no change in the bromine number from the higher LHSV. This clearly indicates that it is important to provide some level of hydrotreating in advance of the HDW catalyst in order to be able to utilize the zeolitic acid function. It is also important to note that there needs to be enough hydrotreating catalyst available in the system in order to meet the other product specifications such as sulfur and aromatics, since the HDW catalyst provides no sulfur or nitrogen removal.
One of the keys to successfully combining a dewaxing catalyst with an HDS system is an understanding of the tradeoffs between dewaxing and HDS activity as the amount of dewax catalyst is changed. ART completed a number of pilot plant tests with two different amounts of dewax catalyst and ART’s NDXi, a premium nickel molybdenum catalyst for ULSD applications. The pilot plant work consisted of testing loadings of 10% and 20% dewax.

The first set of data examines the ability of the system to meet 10 wppm sulfur in the diesel at both low pressure (500 Psi hydrogen partial pressure) and at a higher pressure (975 Psi hydrogen pressure). Figure 6 compares the two systems and the base case is the 10% dewax system at low pressure. This base condition is for producing 10 wppm product sulfur and is the zero point on the temperature axis. As expected, the higher pressure system outperforms the low pressure application by almost 30°F (~17˚C). At lower pressure, the difference between the systems shows a 10°F (~6˚C) higher temp required with the increased dewax catalyst volume. The additional temperature required to meet ULSD also needs to be considered as this could mean a debit of 4-8 months in cycle life if too much HDW catalyst is loaded into the hydrotreater.

The ability to determine product cloud point and how it is expected to change over time is also important. Similar to a hydrocracking reactor, as the temperatures are increased over the bed of HDW catalyst, the ability to break the n-paraffins increases. There is a clear difference in the ability to make a cloud point change based on not only LHSV over the dewax bed, but also the operating pressure of the unit, much like that for producing ULSD. In Figure 7, the base case is again the 10% dewax bed, and the zero point on the chart is the point at which 10 ppm sulfur is produced. Moving from left to right is changing the WABT relative to the expected SOR temperature for 10 wppm sulfur.

Once the temperature is high enough to begin dewaxing, the kinetic response for converting the n-paraffins is linear for both catalyst systems. In these cases, with the combination of hydrotreating catalyst and dewax catalyst (HDT-HDW), there is still a requirement for the removal of sulfur and nitrogen in order for the system to perform well. At 20°F (~11˚C) below the expected SOR temperatures, the system is still capable of decreasing the cloud point 5-7 numbers with the remaining sulfur to the HDW catalyst between 60-100 wppm. Increasing the residence time over the HDW catalyst has a significant impact on the product cloud point. At the temperature required to produce 10 ppm product sulfur the 20% HDW case results in 8-10 degrees more cloud point reduction compared to the 10% HDW case.

Similar trends are observed when the two catalyst systems are operated at higher pressure, as shown in Figure 8 for both 10% and 20% HDW catalyst systems. The zero point on the x-axis is where the 10% HDW system is able to produce 10 ppm product sulfur. The important item to note is that the total cloud point improvement is much greater in this application than at lower pressures. In this case, the
20% HDW case 20-25°F (−11-14°C) of cloud point improvement is achieved at the temperature required for 10 ppm sulfur. Similar cloud point reduction was achieved for the 20% HDW case at 10 ppm sulfur at low pressure, but as shown in Figure 6, there is 20-30°F (−11-17°C) difference in WABT for 10 ppm sulfur between low and high pressure. The impact of residence time in the dewax bed is a little larger at high pressure as well. The 20% dewax system gives about 10-12°F (5.5-6.6°C) more cloud point reduction compared to the 10% case at 10 ppm product sulfur.

As discussed earlier, the HDW function is to break the n-paraffins into smaller molecules in order create less waxy molecules in the finished diesel product. This chain breaking reaction, although somewhat selective, does have the potential disadvantage that it can convert diesel boiling range material into naphtha or possibly lighter materials. These materials, if the refinery is able to tolerate and utilize them, can be considered quite valuable as well. One concern is that if a significant volume of diesel is converted to lighter products, the downstream equipment might not be capable of handling excess light materials.

Figure 9 compares the two loadings and the two pressures in terms of liquid volume yields of 250°F (121°C) plus boiling material. There is a clear importance to understanding the limits of placing too much HDW catalyst into a hydrotreater. Tailoring a system to be able to control the activity of the HDW catalyst is important so that as EOR temperatures are reached, the system is capable of being controlled so that the yield losses are within expectations. Note that the high pressure system, regardless of the percent of HDW catalyst, is able to maintain better liquid yields benefiting from an environment in the hydrotreater that allows it to be highly selective in what molecules it will work on. This is also due to the ability of the NiMo catalyst to saturate and modify the hydrocarbon molecules before it has a chance to be converted by the HDW catalyst. In the lower pressure system, ring saturation does not occur as easily, and the HDW catalyst is unable to be as selective in which molecules it is converting and results in a greater liquid yield loss.

ART’s extensive pilot testing has shown that there is a complex interaction between dewaxing and hydrotreating in ULSD applications. There is a balance between dewaxing activity and HDS activity, which needs to be understood when designing a catalyst system. Furthermore, the liquid yield needs to be considered at both SOR and EOR as this is highly dependent on the amount of dewax catalyst in the system. ARTs technical services staff can work with refiners in order to provide the right catalyst system tailored for maximum refinery profit.
People on the Move

Mike Zehender has been named Global Segment Director, Distillate. Mike, who most recently served as National Technical Sales Manager for Grace Refining Technologies (RT), originally joined Grace in 1997 and worked in sales and technical service roles in ART and RT. He will be focused on the overall global growth of the Distillate segment, particularly driving new products and growth in the emerging regions. Mike has a BS in Chemical Engineering from the University of Cincinnati and will complete his MBA at Thunderbird University next year.

Eboni Adams will be joining Advanced Refining Technologies (ART) as Sales Operations Manager, ART and will report to Scott Purnell. Prior to joining ART, Eboni held an Operations role, where she served as Transactional Lean Six Sigma Black Belt. Prior to her Black Belt role, Eboni was a Customer Service supervisor, originally joining Grace in 1997. Eboni has a B.S. in Chemical Engineering from University of Illinois in Chicago. In addition, she has completed her MBA in Marketing from Georgia State University in Atlanta, Georgia.

Ben Koenigsknecht has joined the Advanced Refining Technologies (ART) Technical Service group as Sr. Technical Service Engineer at the Technical Center reporting to Darryl Klein. Ben comes to us from UOP, where he worked as a Development Specialist and Technical Advisor. He earned a B.S. in Chemical Engineering and a B.A. in Philosophy from Michigan State University.

Brian Slem is joining Grace as Technical Service Manager in Advanced Refining Technologies (ART) reporting to Chuck Olsen and is based in our Chicago office. Brian brings 24 years of refining experience with a background in Process Design, Strategic Planning, Operation Engineering, Planning and Economics, and Engineering Management. Brian was most recently responsible for setting the direction for optimization and monitoring of a Midwest refinery’s Hydroprocessing and Petrochemical Process Units. Brian holds a BSChE from the University of Illinois at Chicago.

Carrie Constantine recently joined Grace as an ART Tech Service Engineer, based at our Chicago location and reports directly to Chuck Olsen. Carrie previously worked as a technical service engineer for Johnson Matthey Catalysts. Prior to that, she spent 5 years with LyondellBasell in quality management and process engineering positions. Carrie has a BS in Chemical Engineering from Purdue University.

Henry Saternus has been named Senior R&D Engineer in Chicago, reporting to Chuck Olsen. Henry, who received a B.S. degree and a Masters degree from the Illinois Institute of Technology, joined Grace in 1998 as a Process Engineer and later was named Production Manager. During his time in production, Henry was involved in several R&D scale ups and expansions and played a major role in increasing rates and productivity at the Chicago site.
Hydroprocessing Cracked Materials

2. What are the operating constraints in co-processing coker naphtha in a ULSD (ultra low sulfur diesel) and/or gas oil hydrotreater unit?

Meredith Lansdown / Brian Watkins / Brian Slemp
Advanced Refining Technologies, Chicago, IL

Co-processing coker naphtha in ULSD service can have several undesirable effects on the performance of the hydrotreater and the catalyst if the system was not properly designed to handle it. In general, coker stocks have a higher level of olefins present from the coking process. Once in the hydrotreater these olefins will quickly be saturated consuming additional hydrogen and generating heat. As a general rule of thumb, 1 mole of hydrogen is required per mole of carbon-carbon double bond, or between 5-10 times the bromine number reduction in standard cubic feet of hydrogen per barrel (SCFB). This additional heat (130-160 Btu/SCF Hydrogen consumed), if not spread out through a decent portion of the catalyst bed, will initiate the subsequent reactions creating a much higher temperature rise than expected. This excess temperature can also speed up the coking or polymerization mechanism which will lead to an increase in pressure drop. This can set an upper limit as to how much coker naphtha can be processed either by a need to limit the heat rise, or from too much hydrogen consumption near the reactor inlet that could starve the downstream catalysts.

A system that is properly size and activity graded will be extremely important when co-processing coker naphtha in a diesel unit. ART utilizes a grading system to help mitigate pressure drop build-up. ART’s GSK-19 is a 19 mm inert ring with a very high void fraction used for trapping large particulates and is placed at the top of the reactor. GSK-9 is loaded next and is a 9 mm macro-porous ring that traps iron as well as other finer particulates that can increase pressure drop. ART also utilizes two other types of active grading, GSK-6A and GSK-3A, which are smaller rings with a small amount of active metals present in order to begin any olefin saturation reaction as well as provide additional void space at the top of the reactor. Underneath the grading options, it is recommended to use a layer of ART’s AT724G or AT734G, which can provide olefin saturation, additional void fraction for pressure drop mitigation, as well as a trapping mechanism for silicon...
Another major concern is that coker naphtha can also bring silicon into the unit which is a permanent poison for hydrotreating catalyst. A silicon guard, such as ART’s AT724G or AT734G should be loaded in the reactor to mitigate silicon poisoning. Silicon pickup is temperature dependent, and at the higher temperatures ULSD units are operating at, silicon pickup in the order of 16-25 wt% could be expected with AT724G or AT734G. If arsenic is present in the coker stocks the use of AT734G is preferred as it will have the same silicon pickup as AT724G but will also protect the active catalyst against arsenic poisoning.

A third concern is the high degree of vaporization of the coker naphtha. ULSD hydrotreaters are typically designed such that their feed distribution system will contain liquid, and the additional gas present from the coker naphtha may cause some systems to perform poorly giving rise to maldistribution. In order to minimize feed vaporization and poor distribution tray utilization, the coker naphtha should be mixed with the other feed streams at a temperature where it is still liquid before feeding to the charge heater. The recovery system should also be evaluated for the increase in naphtha that will be present so that the downstream equipment is not overloaded.

A final consideration would be that additional coker naphtha in a diesel can generate incremental dry gas products such as methane and ethane. These products will increase in concentration in the recycle gas loop, causing a decrease in the hydrogen partial pressure for the hydrotreater. It will also increase the molecular weight of the recycle gas, which can lead to compressor capacity limitations. These additional products can also lead to incremental stripper offgas and related problems.

**Process Fouling**

4. What are the hydrotreating operating issues when processing shale-derived light, sweet and highly paraffinic crudes such as Bakken, Eagle Ford, and Utica? What hydrotreating/catalyst strategies can offset any negative effects? What options are available to optimize the distillate hydrotreater(s) with these light, sweet crudes?

Greg Rosinski / Chuck Olsen / Brian Watkins
Advanced Refining Technologies, Chicago, IL

The processing of highly paraffinic crudes can pose difficulties with various product grades meeting specifications such as cloud and pour point as well as cold filter plugging point. In these cases where the refiners market demands for meeting a more stringent specification, changes to the hydrotreating operation may require the combined system of a catalytic dewax catalyst in ULSD/Jet/Kero hydrotreating or some form of mild hydrocracking in heavier applications in order to limit the longer paraffinic chains.

In refineries designed with higher hydrogen pressures and low space velocities for dealing with more refractory feedstocks, the introduction or switching to lighter paraffinic crudes can experience incremental light end generation. The high horsepower of these hydrotreaters can cause the undesirable reaction of eliminating some of the paraffin chains once the remaining reactions have gone to near completion.

Some crudes from these areas have been known to contain higher quantities of iron than typical, and processing the heavier fractions will require the use of adequate feed filtration in order to prevent fouling and plugging in equipment. The use of additional top-bed particulate trapping materials are also recommended in order to avoid an unexpected skim or turnaround.

The processing of light, sweet crudes can have benefits to a refinery as well, as the demand on hydrotreating performance can be lessened at similar processing rates. It can also allow for additional upgrading of barrels by increasing throughput or, if the process conditions warrant, provide the ability to place additional hydrogen into the feed, allowing for higher distillate yields at the current processing rates.

5. What is the panel’s experience with hydrotreater fouling/poisoning issues arising from processing of synthetic or bitumen-derived crudes? How can the impact be mitigated?

Greg Rosinski / Chuck Olsen / Brian Watkins
Advanced Refining Technologies, Chicago, IL

There is large variation in the quality and types of synthetic crudes, and there can be problems with trying to process many of them. In general, synthetic crudes can be classified into two basic types.

One is a blend of upgraded naphtha, distillate and gas oil range “material” and with no resid or bottoms material included. Fully upgraded synthetic crudes are more difficult to treat than might be expected when comparing typical feed characterizations. The upgrading process typically includes several hydrotreating and hydrocracking steps as well as coking processes to tighten up the material to make its flow characteristics easier to handle. The molecules left in the oil are the more refractory and difficult to remove species. The fractions that are pulled from the refinery’s crude processing units however typically have a high API along with the lower sulfur and nitrogen, masking these difficulties. These crudes can also pose a dif-
ficulty with the presence of silicon which can be introduced from the coking process or through flow enhancers added after processing, and, if left undetected, will poison downstream hydrotreaters.

The other crude type utilizes the whole bitumen including bottoms which has been blended with other sweet synthetic or more conventional crudes. With synthetic crudes that do contain bottoms there can be significant problems with poisoning. Since these types of synthetic crudes contain bitumen, they can have higher levels of Ni+V in the HVGO compared to similar boiling range materials from conventional crude oils. This can be dealt with in the hydrotreater using an appropriately sized bed of demet catalysts such as ICR132 and ICR161. Some of these crudes may also contain silicon, from upstream coking processes, and they may also contain arsenic which is present in shale and bitumen derived oils. In these cases a combined silicon/arsenic guard such as AT734G from ART can be used to help mitigate the impact of these poisons.

Many of the synthetic crudes may also contain very fine particulates of clay or sand that are associated with asphaltenes or other heavy polycyclic molecules. These particulates can be removed through proper use of the desalter to avoid crude tower fouling. However, these small particulates, typically <5 microns, when not properly removed, can pass through feed filters into the catalyst beds resulting in pressure drop issues. Often this pressure drop can occur in lower catalyst beds due to the denaturing effect of hydrotreating on the heavy molecules. The removal of sulfur, nitrogen and aromatic saturation as well as the heat and hydrogen present, can cause the fine particulates to precipitate out further down in the catalyst bed. Use of larger void spaced catalyst can help to alleviate this problem to some level, however, we are not aware of anyone who has found a solution that completely alleviates this problem.

**Process Upset Response**

6. Following an emergency shutdown which includes the loss of the recycle gas compressor, is it possible to quantify the effect on catalyst deactivation? What are the best practices to minimize catalyst deactivation?

Robert Wade
Advanced Refining Technologies, Worms, Germany

Reducing reactor temperature and sweeping with H₂ will help minimize deactivation due to coke formation. ART recommends that for loss of the recycle gas compressor cold feed should be used to reduce reactor temperature below 400°F. In addition, make up hydrogen should be used to sweep the reactors. If the recycle gas compressor cannot be recovered within an acceptable period, then the feed should be pulled once the reactor temperatures are reduced below 400°F (~204°C), and the reactor should be swept with make up H₂ to remove residual liquid. This sweep should continue until the recycle gas compressor is recovered and feed is reintroduced. If the shutdown will be prolonged then the reactor may be held under N₂ below 200°F (~93°C).

A comparison of reactor temperature at the same conditions and severity using the same feed should be performed to quantify the amount of catalyst deactivation. If this is not possible, then previously established normalization parameters may be used to make a reasonable comparison of reactor temperatures before and after the emergency shutdown.

A hot H₂ strip may also be performed once the recycle gas compressor is restarted to recover activity. However, this has a limited ability to recover all of the catalyst activity and is highly dependent on the current and prior operation conditions and stability.

**Hydrocracking**

7. What must a refiner consider when evaluating mild HC as a way to increase diesel production in their gas oil hydrotreater?

Robert Wade & Charles Olsen
Advanced Refining Technologies, Worms, Germany, Chicago, IL

There are a number of considerations when designing a catalyst system for mild hydrocracking service. This mode of operation tends to have higher fouling rates than conventional high pressure hydrocracking (HCU) service, and also has 2 competing constraints compared to a single constraint for the typical high pressure service. In a mild hydrocracking operation cracking conversion is controlling at SOR and, as the hydrotreating (HDT) catalyst deactivates through the run, the HDS and HDN conversion will start to become controlling. This means it is critical to understand how the H₂ partial pressure varies throughout the run and the impact this has on the expected fouling rate of both the HDT and HCU catalysts. In addi-
tion, it is important to have a good understanding of the nitrogen slip to the HCU catalyst so that the activity of the HCU catalyst is properly balanced against desired selectivity.

The addition of HCU catalyst to an existing hydrotreater will come at the cost of decreased HDS/HDN catalyst volume. This will reduce the activity and life of the HDT section. The HCU catalyst does have some level of HDT activity, but not enough to overcome the decreased volume of HDT catalyst. Figure 1 below summarizes pilot plant data which demonstrates how the HDS activity of the catalyst system is impacted by the addition of HCU catalyst. The addition of a small amount of HCU catalyst has little or no impact on HDS activity, but higher volumes of HCU catalyst result in decreasing HDS activity.

The changes in HDT activity need to be balanced against the cracking activity of the catalyst system as shown in Figure 2. Not surprisingly, increasing the amount of HCU catalyst results in decreasing HDS activity.

Achieving the proper balance of HDS/HDN activity and cracking activity is a challenge and the system is best customized on a case by case basis. The volume and type of HCU catalyst needs careful consideration in order to provide the desired conversion and minimize production of excess gas and naphtha. This is especially important as the cycle progresses and the reactor temperatures approach EOR.

A mild hydrocracking system will make more light products so the product recovery section needs to be evaluated to ensure it can handle the increase in lighter products. In addition, there will be an increase H₂ consumption and corresponding increase in heat release (higher exotherms). Consideration needs to be given to make up H₂ requirements and temperature control scheme to make sure they are adequate for the expected changes.

9. What are the possible causes of high pressure drop in lower beds of HT and HCU’s? What techniques are used to diagnose the causes prior to shutdown? Are there any mitigation techniques or strategies to extend the cycle?

Greg Rosinski & Charles Olsen
Advanced Refining Technologies, Chicago, IL

There are several causes of pressure drop related to a poor turnaround or poor catalyst loading. A damaged or dirty outlet collector, catalyst support screens or improper size grading at the bottom of a catalyst bed can all lead to high pressure drop in a lower bed. It is important that the reactor and screens be cleaned before loading any catalyst. Proper size grading of catalyst at the bottom of each bed is also important. If the catalyst size difference between grading layers is too large, smaller diameter catalyst can migrate through subsequent layers and ultimately plug the support screens or outlet collector. It is also critical to make sure nothing (tools, hardhats etc.) gets left in the reactor during a loading.

Pressure drop in a lower bed can also be caused by the gradual accumulation of iron sulfide or other fines. These fines have small enough particles that they can be carried through upper beds. The particulates can then drop out in a lower bed. Some synthetic crudes may have fine particulates of clay or sand which can deposit in lower beds as discussed in our answer to question 5.

Excessive coke formation due to hydrogen starvation from poor gas distribution or low H₂/Oil ratio combined with higher temperatures has also been identified as a cause of lower bed pressure drop. It is important to keep the H₂/Oil ratio above a specific minimum and, if reduced H₂ availability is anticipated, the charge rate should be reduced or cracked stocks removed for the period, to ensure that the minimum H₂/Oil ratio is maintained.

Another cause for lower bed pressure drop which we have seen involves debris or water introduced though the quench line. Quench systems should be drained and/or exercised while the catalyst is cold to prevent sudden water vaporization and breaking of catalyst pellets.

10. Does hydrotreated product recycle count the same as fresh virgin feed for the catalyst break-in period?

Meredith Lansdown / Brian Watkins
Advanced Refining Technologies, Chicago, IL

Hydrotreated product recycle can be supplemented for feed during the catalyst break-in period after sulfiding, however there are several issues that need to be considered when doing so. The initial break-in period is designed so that the catalyst does not see elevated initial temperatures along with materials that could prematurely place a...
high level of coke down on the catalyst surface. This allows the hyper-active sites on the catalyst that are present just after sulfiding to be tempered in order to prevent this. Use of product recycle has the advantage that the material will be very low in coke precursors present in other stocks which will help prevent rapid coke lay-down. It also has the advantage that, if this is done to supplement a shortfall in total feed to the unit due to a limited quantity of available straight run stocks, the unit can be maintained at maximum rates, ensuring that good distribution is maintained and minimizing pre-mature fouling.

The downfall would be that if product recycle is the sole source of feed to the unit, that over a short period of time, all of the sulfur will be removed from the feed. This will increase the hydrogen partial pressure at the catalyst surface and could begin to reduce the active metals if H₂S presence is not maintained to keep the catalyst sulfided. In this case it is recommended that minimum H₂S levels on the order of 5,000 wppm are maintained in order to keep the catalyst sulfided. It is also recommended that temperatures no higher than the final sulfiding temperature be used to also minimize any possible reduction or premature coking.

Hydroprocessing

14. What are cycle life limiting factors in low pressure jet/kero hydrotreaters?

Greg Rosinski / Brian Watkins
Advanced Refining Technologies, Chicago, IL

For most jet and kerosene hydrotreaters, the end of run condition limitation is typically the product color or smoke point. Aromatics and poly-nuclear aromatic (PNA) compounds are a problem for both smoke point and color. Color bodies are PNA’s that form as the reactor temperature is increased. Several of these types of molecules are green/blue and fluorescent in color, and the color is apparent at very low concentrations of these species. Certain nitrogen (and other polar) compounds have also been implicated as problems for distillate product color. These species can polymerize to form various color bodies and can also form sediment via oxidation and free radical reactions. At end of run conditions when the outlet temperatures are high enough, PNA saturation reactions become equilibrium limited, and the formation of aromatic compounds is more favorable.

If the unit is having difficulties with color, several steps can be taken to extend the cycle length. Increasing the H₂/Oil ratio to the hydrotreater will increase the reactor outlet H₂ partial pressure helping to slow down the reverse equilibrium reaction and can help in prolonging cycle life. Feeds such as light cycle oils and coker stocks typically have higher H₂ consumption than straight run materials, which reduce the outlet H₂ partial pressure. Increasing the available hydrogen to these units will help in maintaining the kinetic reaction. If unable to add additional hydrogen, minimizing cracked stocks will also help with color.

If the unit has enough quench available, operating with a descending temperature profile for the last bed can also assist with increasing cycle life. This also has the added benefit of improving the outlet hydrogen partial pressure which as mentioned above is beneficial to the PNA saturation reactions.
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