



NEWSLETTER

INTERNATIONAL TUNGSTEN INDUSTRY ASSOCIATION

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17th Annual General Meeting

26-29 September, Marriott Hotel, Lisbon

The full programme, registration and hotel reservation forms are available from ITIA's office by email or may be downloaded from the ITIA website.

Delegates to the AGM will enjoy the city of Lisbon – its warm weather, the historical sights, the Tagus (that 'sea of straw' as it reflects the golden light of the afternoon), the centre of life which is the Baixa, and the mediaeval quarters (the Alfama and the Barrio Alto) where many of the best restaurants, taverns and night-clubs are to be found and where fado is said to have originated.

Apart from specialist Committee meetings on Sunday 26 and Monday 27 September, the event will begin on the evening of Monday 27 September with a barbecue and some unusual entertainment around the hotel pool.

The AGM will follow the successful format introduced in 2003 and take place during the mornings only of Tuesday 28 and Wednesday 29, leaving afternoons free for business discussions.

On Tuesday evening, there will be more fun and on this occasion it will be a visit to the Adega Regional de Colares, to taste some of the region's wines, dine in the cellars and listen to the songs and guitars of local musicians.

Presentations will include:

- ▼ HSE issues, including REACH
- ▼ "Coarse Grained Hardmetals for Mining and Road Reconstruction Tools" by Bernhard Szesny, Manager Application Engineering, H C Starck GmbH
- ▼ Promotional video for tungsten, to be presented by Wolf-Dieter Schubert, Technical Consultant to ITIA
- ▼ Market Report by Michael Maby, Secretary-General of ITIA
- ▼ "Tungsten Recycling – the Collection and Processing of Scrap" by Tom Blendulf, President and CEO of Martin Alloys Corp
- ▼ "King Island Scheelite - an Update" by Peter Gibson, Executive Chairman, Australia Tungsten Pty Ltd
- ▼ The Tungsten Market in China (details to follow)
- ▼ The Tungsten Market in Japan (details to follow)
- ▼ "The Tungsten Market in the USA" by Kim Shedd, Cobalt and Tungsten Specialist, US Geological Survey

New Members

Two companies have applied for membership this year:

- ▼ Amalgamated Metal Corporation Plc, a trading company based in London supplying concentrates, APT, WO₃ and FeW
- ▼ Chaozhou Xianglu Tungsten Industry Co Ltd, a company in Guangdong Province, producing and selling APT, WO₃, W and WC powders.

The ITIA's Technical Service is into its stride and an apposite article in these polluted times, entitled "Tungsten for a Cleaner Environment", is its second contribution in a series.

The article is in two parts and will be concluded in the next Newsletter in December. The Service is currently responsible for re-designing the ITIA website and revising the brochure "Tungsten", with the end of 2004 as a target date for completion of both exercises.

TUNGSTEN FOR A CLEANER ENVIRONMENT

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The worldwide preservation of our environment is, and will remain, one of the most important tasks for human beings. Unfortunately, still today a large percentage of the world's population and, interestingly, especially those bearing strong responsibilities, (like politicians) do not fully accept and act upon the above statement. Nevertheless, up to now a lot of effort has been undertaken to minimize or at least to decrease the release of hazardous materials to the atmosphere, to marine and fresh water and to the soil.

Tungsten has in different forms taken part in these efforts and these contributions are the aim of the present article.

There exist four applications where tungsten helps in cleaning products or exhausts from toxic compounds, in saving fossil fuels or in substituting a toxic metal:

1) Tungsten trioxide containing DeNO_x SCR catalysts which remove nitrogen oxides or specific organic compounds from exhaust gases (SCR stands for Selective Catalytic Reduction).

2) Hydrodesulfurization and Hydrodenitrogenation catalysts reducing the nitrogen and sulfur content of oil refinery streams and petroleum products, by restricting the formation of nitrogen oxides and sulfur dioxide during a subsequent combustion.

3) Tungsten metal acting as a substitute for the toxic metal lead in fishery equipment and as shot grain.

4) Tungsten Electrodes used in gas discharge lamps and Tungsten trioxide-containing Smart Windows contributing in energy savings during lighting and air conditioning.

PART I: TUNGSTEN CONTAINING CATALYSTS FOR CLEANER EXHAUSTS AND FUELS

DeNO_x SCR CATALYSTS

Thermal power generation by diesel engines, gas turbines or boilers is always combined with the combustion of organic fuels (gaseous or liquid hydrocarbons, or solids like coal, lignite or waste). During combustion the organic compounds or carbon are oxidized to carbon dioxide and water. The oxidizing agent is air due to its 21% content of oxygen.

If the temperature during combustion is high enough, and this is an important prerequisite to attain a high yield in energy conversion, an unavoidable side reaction always takes place. A certain part of the nitrogen of the air is oxidized to NO and NO₂. The higher the temperature the more nitrogen oxides are generated. Nitrogen oxides (NO_x) is the generic term for a group of highly reactive gases, all of which contain nitrogen and oxygen in varying amounts and are environmentally hazardous.

Nitrogen oxides are dangerous in several regards:

They cause acute-lung irritation like chronic bronchitis (persistent inflammation of bronchial tubes). Concentrations of less than 1 ppm are enough to disable persons with chronic respiratory diseases. They contribute sulfur oxides to acid rain and they can combine with volatile organic compounds in the lower region of the atmosphere in sunlight to form ozone. Ground-level ozone itself is harmful to human health and damaging to forests and crops.

Nitrogen oxides in the stratosphere react with ozone forming oxygen, thus interfering with the natural process of ozone formation by UV rays. That means they contribute to the destruction of the ozone layer, which protects us from dangerous, short wave length UV rays.

Power plants emit about one quarter of the total human made contribution of nitrogen oxides to the atmosphere. This contribution is controlled today, due to DeNO_x catalysts and technology, which reduce both NO_x and particulate emissions.

Diesel-powered cars, trucks and buses, but also home-heating gas stoves, on the other hand, generate more than half of all nitrogen oxides in Europe and the US and, although new regulations have been set to reduce the impact that vehicles exhaust has on human health and the environment, these small but numerous "emitters" are responsible for today's still increasing overall NO_x emissions, in particular in urban areas. This situation will require new fuel formulations as well as new emission standards for combustion motor vehicles, especially for diesel-powered cars, as the implementation of clean-burning fuels and improved exhaust gas treatment is as beneficial to public health as it is to the environment. Also in this regard DeNO_x technology can contribute in reducing air pollution from combustion engines.

The Catalyst

Titanium dioxide in the *anatas* modification acts as the high specific surface carrier (approx. 90 m²/g) which contains the catalytic active components (vanadium pentoxide: V₂O₅ and tungsten trioxide: WO₃). Vanadium pentoxide is an oxidation catalyst and tungsten trioxide improves the selectivity of the catalyst, suppressing the oxidation of sulfur dioxide to sulfur trioxide, by minimizing the risk of corrosion of downstream equipment. Tungsten trioxide moreover is responsible for the *anatas* stabilization and for improving the titanium dioxide activity (specific surface).

A typical composition of commercial catalysts is:

TiO₂ 78%; WO₃ 9%; V₂O₅ 0-5%; SiO₂ 7.5%; Al₂O₃ 1.5%; CaO 1%; SO₄²⁻ 0.5-2%

For special applications the catalyst composition can be varied between certain limits to be adopted accordingly.

There are several production methods. The first is to suspend precipitated titanium dioxide in *anatas* form in water, which is doped with tungstate in ammoniacal solution. A thin layer of H_2WO_4 is then formed by precipitation onto the TiO_2 particles, which act as crystallization nuclei. The layer is converted to WO_3 in a succeeding calcination step. Now vanadium pentoxide is added together with the other components, which act as mechanical promoters. After mixing with binder, plastifiers and glass fiber the mass is extruded to honey-comb-structures, dried and calcined. In this catalyst type, the whole mass is catalytically active.

Another production scheme uses a corrugated fiber reinforced titanium dioxide carrier, which is then impregnated with the active components and afterwards calcined.

All types of catalysts have a structure with a large number of parallel channels. Channel diameter can be varied typically between 3 and 9 mm, by that covering applications ranging from dust free off gases to highly dust laden flue gases with more than 30 g/Nm³ dust (**Fig.1**).

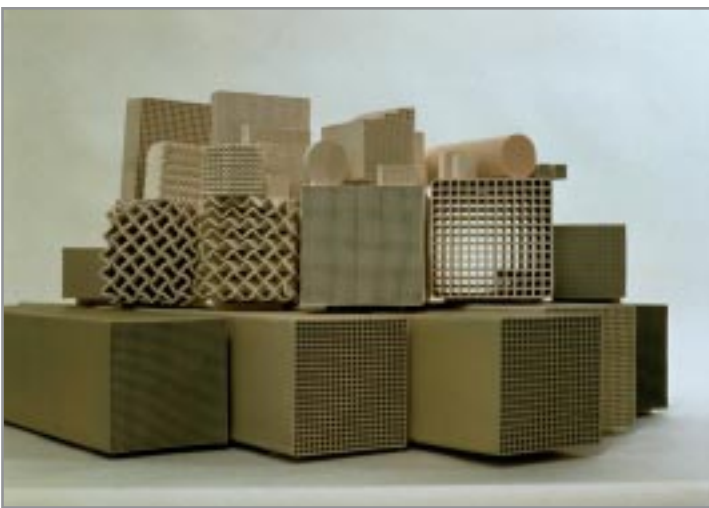
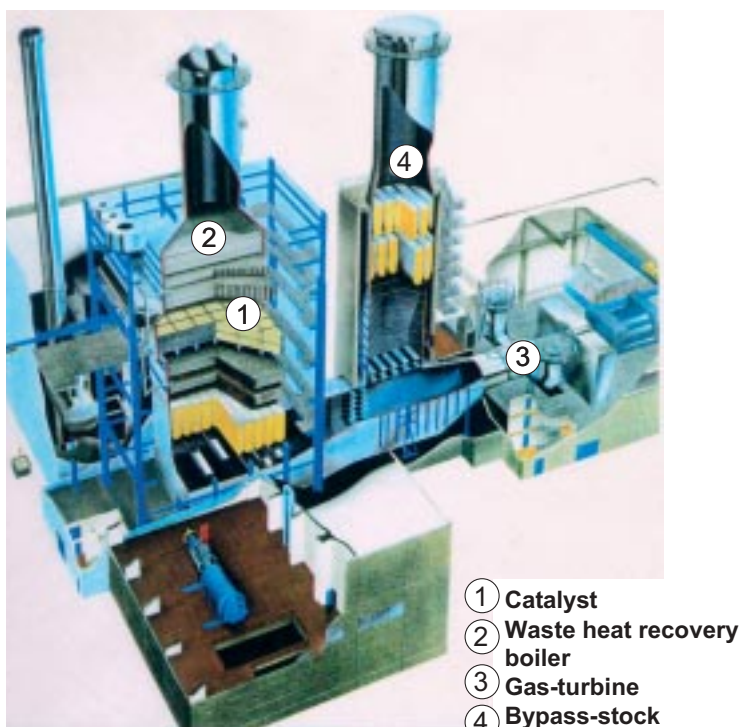


Fig. 1: DeNOx catalyst; different profiles. The catalyst elements exhibit a honey-comb-like structure, formed by extrusion. By courtesy of Porzellanfabrik FRAUENTHAL GmbH (Ceram), Austria.



The catalyst blocks are manufactured in steel cassettes of different size. Single cassettes are normally used in small installations. For larger installations single cassettes are assembled into modules (**Fig.2**).

DeNOx Process

This process is considered the most efficient commercially proven method to remove nitrogen oxides from off gases. It was invented by Japanese scientists in the 1960s.

The NO/NO₂ ratio in power plant off gases is about 95/5, but can be different in other emission requirements.

The nitrogen oxides react with ammonia at the catalyst surface to form nitrogen and water vapor. The temperature for the reaction should range between 150 and 450 °C. The following main reactions occur within the catalyst:



The side reaction



which is 100% when using $TiO_2-V_2O_5$ catalysts, is less than 1% in the case of the DeNOx ($TiO_2-V_2O_5-WO_3$) catalyst. The nitrogen oxide removal is typically around 80%, but may go up as far as 95%. The ammonia slip is below 2 ppm.

The process is schematically shown in **Fig.3**, and consists basically of a catalytic reactor and an ammonia storage and injection system. The reducing agent can be either liquid pressurized ammonia, or an aqueous ammonia solution. An aqueous 32,5 wt% solution of urea (Ad Blue) can be used as well.

Applications

In principle the process can be applied to any flue gas containing nitrogen oxides and oxygen. Typical examples are:

Boilers fired by: coal, oil, lignite, pet coke or gas

**Diesel Engines (both 2-stroke and 4-stroke):
in trucks, marine vessels, emergency power
generating sets**

Gas Engines

Gas Turbines

Waste Incineration Plants

Chemical Plants

Cement Plants

Fig.2: Cut through of a gas-turbine unit showing DeNOx catalyst modules for flue-gas treatment. By courtesy of WIENSTROM GmbH, Austria.

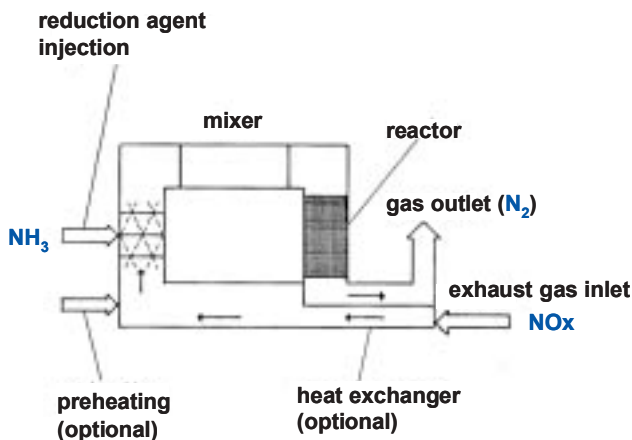


Fig3: Schematic drawing of a DeNOx-SCR process unit; the catalyst contains tungsten oxide; NOx in exhaust gases are transformed catalytically into harmless nitrogen.

Each application has its own characteristic demand to the catalyst and the technology. For example, in a coal fired boiler the catalyst is placed immediately after the boiler and upstream of the electrostatic precipitator. So the catalyst is exposed to a heavily dust-laden flue gas and must be resistant to erosion and poisons from the fly ash.

Catalysts in marine vessels and trucks not only remove nitrogen oxides, but also provide excellent noise attenuation. Especially for trucks, designs are in use, combining different catalysts in order to reduce also the amount of unburned hydrocarbons (**Figs.4,5**).



Fig.4: SCR catalyst for automotive application; each monolith consists of about 8000 parallel channels. By courtesy of Porzellanfabrik FRAUENTHAL GmbH (Ceram), Austria.

Due to the avalanche-like increase of the percentage of diesel engine driven passenger cars there are intentions within the European Union to install the same rigid regulations for the exhaust of diesel engines in passenger cars as for gasoline driven ones. This would imply the installation of respective catalysts especially also in regard to NOx emission. In this regard SCR-systems can also contribute in saving fuels as the diesel engines can then be tuned to their economic optimum.



Fig.5: "WINTERTEST" of a catalyst for automotive application; the catalyst is part of the silencer system, together with a urea tank and an injection control system. By courtesy of M. Signer, IVECO Switzerland.

The variety of materials being incinerated today represents a special challenge to the DeNOx catalysts, because the off gases show a similar large variation in composition. The catalyst does not only remove the nitrogen oxides but also dioxin and sulfur and must have a very high dust tolerance. Dioxin tolerances below 0.1 ng/Nm³ are attainable, being far below the limits prescribed by law.

During use, dust particles accumulate on the surfaces of the catalyst and can make it inactive. Now and then, therefore, the modules have to be disassembled and regenerated by washing operations (where possible), or disposed under environmentally friendly conditions.

TUNGSTEN NICKEL CATALYSTS FOR HYDROTREATING

The importance of hydrotreating

With the world production of more than three and a half billion tons of crude oil per year (about 77-78 million barrels a day), the hydrodesulfurization (HDS) and hydrodenitrogenation (HDN) of petroleum feedstock on molybdenum and tungsten containing catalysts is one of the largest heterogeneous catalytic processes employed in the world industry. The importance of the process is even increasing as a result of growing requirements of environmental protection and the tendency in the refineries towards processing of crude oils with even higher sulfur levels. And 120 million barrels a day are forecast by the US Energy Information Administration for the year 2020.

Hydrodesulfurization is the reaction of organosulfur compounds with hydrogen to yield hydrocarbons and H₂S, whereas hydrodenitrogenation gives hydrocarbons and ammonia. Both processes are part of hydrotreating. Hydrocracking operations contribute to increasing the desirable gasoline and diesel yields, while reducing the volume of heavy oil that is produced. A simplified flow sheet of the different stages of product formation in a refinery is presented in **Fig.6**.

The reasons for sulfur and nitrogen removal during hydrotreating crude oil or various refinery products are:

- To minimize the amount of sulfur and nitrogen oxides which form during the combustion process in order to reduce air pollution. New environmental regulations require lower sulfur concentrations in fuels in general ("clean fuels").

- To convert aromatics to saturated hydrocarbons (ring opening) thereby decreasing the concentration of aromatic hydrocarbons present in the fuel (ultra-low aromatics levels in distillates). This process, called Hydrodearomatization (HDA), reduces aromatic content and subsequently soot and particulate emissions during combustion of fuels and decreases the amount of carcinogenic residual aromatics in the exhaust gas.

- To prevent the poisoning of catalysts used in the subsequent steps of product fuel preparation, such as reforming and cracking. Nitrogen compounds are a poison for the zeolite cracking catalyst and large sulfur amounts are poisonous for Pt/Re reforming and isomerisation catalysts.

Chemically bound sulfur and nitrogen can be found in petroleum feedstocks as thiols, organic sulfides, thiophenes, especially dibenzothiophenes, which are difficult to remove, as well as amines, pyridine, and quinoline compounds. The sulfur content of crude oil ranges between 0.2 and 4 %, whereas nitrogen is contained between 0.1 and 0.9 %. More recently, modern oil drilling techniques made it possible to obtain heavier crude oil, which is even higher in sulfur and nitrogen containing compounds.

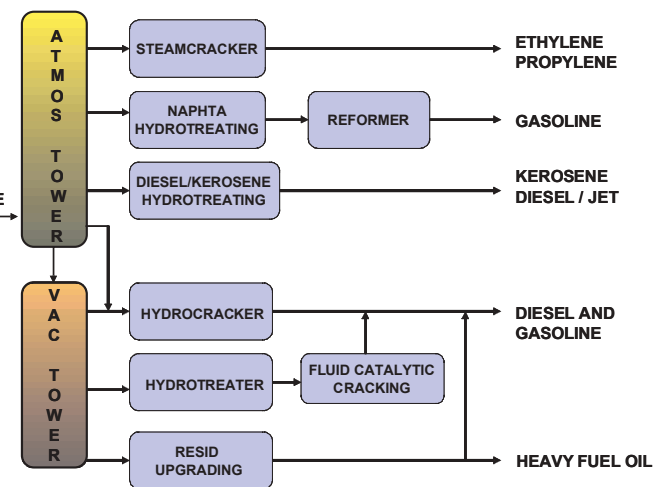


Fig.6: Simplified process flow sheet to separate, treat and convert crude oil into valuable products; molybdenum and tungsten-containing catalysts are used in hydrotreating and hydrocracking (HDS, HDN, HDA).

SOx and NOx emissions

The negative effect of nitrogen oxides to the environment and livings was already discussed above. Sulfur dioxide (SO₂) formed during combustion of fossil fuels causes a wide variety of health and environmental impacts because of the way it reacts with other substances in the air. Sulfur dioxide contributes to respiratory illness and can be particularly problematic for people with asthma and heart diseases. Moreover it causes acid rain, which damages plants and water life and corrodes metallic and concrete materials.

Since 1993, the US Environmental Protection Agency (EPA) has restricted diesel sulfur to 500 ppm, whereas in California and the EU diesel fuels may not exceed sulfur levels of 350 ppm. According to the European Directive 2003/17/EC, the sulfur level in fuels has to be reduced to less than 50 ppm, while at the same time the introduction and widespread availability of zero sulfur fuels (=less than 10 ppm by wt) will be mandated. From January 1st

2009 onwards all the available petrol and diesel have to comply with a sulfur specification of less than 10 ppm by wt. However, fuels with sulfur contents < 10 ppm are available today already in several European countries and the US. They are named "sulfur free" or *ultra low sulfur diesel*. Tax incentives are made in several countries for the refiners as part of a push for clean air by governments.

New diesel standards also mean a demand for low aromatic fuels, which requires a considerable capital investment for the refiners and has led to the development of a new generation of HDS and HDA catalysts.

HDS and HDN Catalysts

Catalysts in use are Mo-Ni, Mo-Co and W-Ni in the sulfided form (MoS₂, WS₂). Sometimes also a mixture of Mo and W is used. The catalyst is a build-up of the catalytically active phases (Mo, W), promoters (Co, Ni), modifiers (to further improve activity and selectivity), the support (mostly γ -Al₂O₃ or zeolite, but also alumina-silica or activated carbon) and binders which create the necessary physical strength of the extrudate.

Moly-based catalysts are the work horses for HDS (Mo-Co) and HDN (Mo-Ni) today and are mainly used for the pretreatment stages during hydrotreating, as they proved superior to W-Ni-based catalysts. The latter are used in a series of specific feedstock treatments, in particular during mild hydrocracking operations for lower aromatic contents and controlled lube oil hydrocracking.

Commercial W-Ni catalysts have compositions such as 4-16% Ni and 16-20% W on a γ -Al₂O₃ or zeolite support. Examples for hydrotreating and hydrocracking catalysts are shown in **Fig.7**.

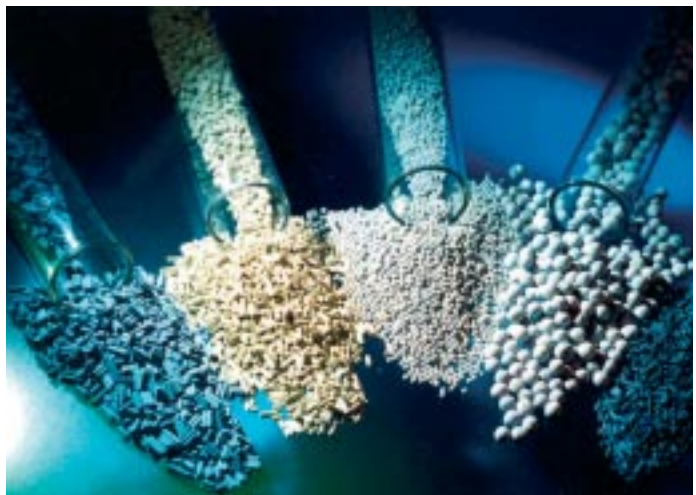


Fig.7: Picture of a series of HDS, HDN and HDA catalysts. By courtesy of F. Danzinger, OMV, Austria.

In the process of impregnation, the ceramic support is contacted with aqueous solutions of the catalyst components (W, Ni). Ammonium metatungstate is commonly used for the introduction of tungsten but ammonium paratungstate, tungstic acid, phosphotungstic and silicotungstic acid are used as well. Nickel nitrate is used for nickel. Impregnation results in a thin layer on the surface of the support. This step in the preparation is a crucial factor in attaining a high degree of dispersion of the oxides, which is a prerequisite of high activity and selectivity of the catalyst.

Before use, the catalyst is treated with hydrogen and a sulfur-containing feed, and the surface oxides are converted to sulfides that are the catalytically active species.

Catalyst suppliers advise on the activation treatment in order to carry out the activation properly. Some commercial catalysts are also available in a presulfided or fluorided form. Because of the high economic importance of hydrotreating the details of manufacturing industrial catalysts are closely held secrets by the producers.

During extended use, the catalysts are deactivated by deposition of coke but also by materials present from the feedstock, as for example, organovanadium or organonickel compounds in heavy petroleum fractions and residues. Such compounds form sulfides on the surfaces of the catalyst, thereby "blocking" the catalytic sites. Arsenic is a further catalyst poison forming nickel arsenide. Deposits on the catalyst can be regenerated several times by controlled combustion either "in-situ" or "ex-situ" and can be used for multiple cycles.

Spent hydroprocessing catalyst may be used as a source for molybdenum or tungsten, but usually this is uneconomical. Environmentally friendly disposal is then the only alternative.

Hydrotreating units commonly are large down-flow fixed-bed reactors (**Fig. 8**). Multibed operation permits use of different catalysts and temperatures in the layer, and thereby reactor performance is improved. Catalyst choice is influenced by the nature of feedstock to be hydrotreated.



Fig. 8: Down-flow fixed-bed reactor for HDS, HDN and HDA diesel treatment (right).

By courtesy of F. Danzinger, OMV, Austria.

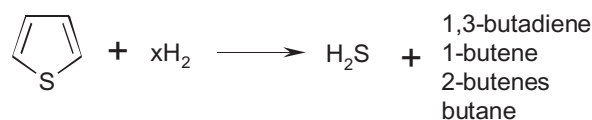
Besides the well known fixed bed reactor technology, also so-called ebullating bed technologies are in use for unique residue upgrading processes (LC-Fining, H-Oil). The residue fraction of crude oils contains significant amounts of metals (Ni, V, Fe etc) as well as higher carbon residues (known as Conradson (CCR) or Ramsbottom (RCR) carbon content). Higher percentages of carbon residues and asphaltenes in the feedstocks lead to an accelerated deactivation of the catalyst in use and require therefore a more improved catalyst change out method. In the above mentioned LC-Fining and H-Oil processes the catalyst is withdrawn on a semicontinuous basis.

Chemical Reactions:

Any sulfur bound in organic molecules is converted to hydrogen sulfide according to equ.(1)



In the case of heteroaromatic compounds, such as, for example, thiophenes, the ring is opened and hydrogenated (equ.2).



Typical working conditions are 40 to 100 bar at 300–400 °C, but can go up to 300 bar and 450°C in the case of residue upgrading. Feed rates per unit are between 2,000 and 10,000 tons per day. The catalyst lifetime is depending on heavy (1/2 – 1 year) or light (10 years) distillate.

The analogous reaction for HDN is given in equ. (3):



The hydrogen sulfide formed is separated and converted to elemental sulfur by the *Claus process* ($2 H_2S + O_2 \rightarrow S_2 + 2 H_2O$). For example, 70 % of the sulfur produced in the US stems from **HDS**.

The paper will be continued in the December 2004 Newsletter. PART 11 deals with the substitution of lead through tungsten and the use of tungsten in energy saving devices.

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