

Enhanced Adsorbents for Mercury and Sulfur Removal from Refinery Process Facilities

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The global crude slate has changed rapidly in the last decade, as production areas such as the oil sands in Alberta and other shale oil formations throughout North America have developed. Throughout the world, more exploration opportunities and advanced recovery technologies have resulted in order to access crudes that weren't attainable in the past. Many of these crudes contain contaminants that can cause multiple corrosion issues during downstream processing. Catalyst poisoning and metallurgical issues result in millions of dollars in repairs and downtime, and health/safety issues for personnel in the refinery and in surrounding areas are also a concern. Two of the main contaminants present in crudes around the world are mercury (Hg) and sulfur (S), depending on the location and formation.

UOP is at the forefront of developing advanced alumina adsorbents solutions for removal of mercury and other contaminants such as sulfur, chlorides, fluorides and oxygenates from refinery process streams. The UOP series of GB-300 adsorbents provide a range of removal capacities to fit refineries' mercury removal needs. The latest commercial products (UOP GB-347 and GB-547) are able to provide long bed life with higher inlet concentrations of mercury than previous UOP adsorbent products. Other contaminants found throughout the world's crudes are sulfur compounds. Whether it is H₂S, COS, DMDS or other species, sulfur contaminants are detrimental to catalyst performance and contribute to equipment failures and health/safety issues if left in the system. UOP has multiple adsorbent solutions for refiners experiencing various types of sulfur contamination as well as varied refinery schemes.

With refineries aiming for higher profits and tighter environmental controls, adsorbents are playing a bigger part in keeping operations running smoothly, reducing costly shutdowns and keeping employees and the environment safe.

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Of these two contaminants, sulfur levels have increased in crudes that have been produced and processed over the last 50-60 years, so there are many more options for sulfur removal in both upstream and downstream applications. But as stated above, many newer crude formations that are being explored and processed contain mercury, so determining ways to remove this contaminant are essential to safe and efficient refinery operations. Initially we will discuss the impact of the presence of mercury and the options for removal, then review trace sulfur removal applications for refining.

Mercury in Crude Oils

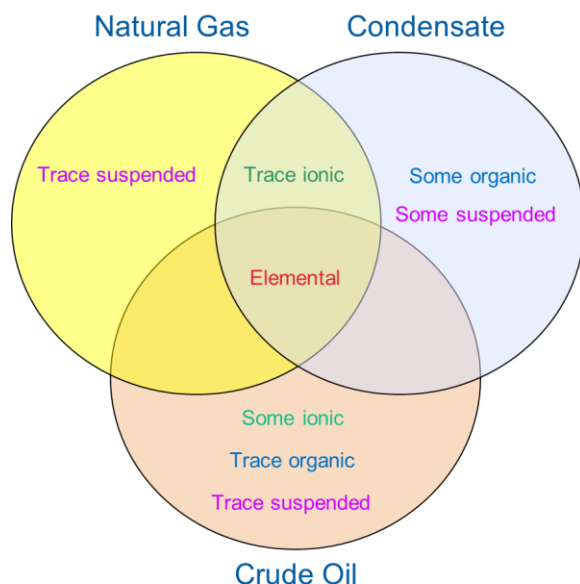
Understanding the properties of mercury is an important aspect of oil and gas production, processing and commercialization. Mercury is produced from hydrocarbon reservoirs along with crude oils, condensates and natural gas from the North Sea to offshore Australia. Major energy companies tend to have special task force teams dedicated to determining the effects that mercury will have on assets, hydrocarbon pricing, sales agreements and the environment.

Mercury is a naturally-occurring trace element that is found globally; mainly it is encountered in its most stable form known as mercury sulfide (HgS) or cinnabar. It is generally found in minor concentrations in fossil fuels however, even though Hg is a global issue, in certain geographical regions, Hg levels are elevated. Globally, the crudes with the highest concentrations of mercury typically come from Southeast Asia. Crudes from Thailand and Indonesia can run as high as 3,000 ppb_w¹, while crudes from the Austral basin in Argentina can have up to 2,000 ppb_w of mercury². Crudes from Argentina, Venezuela and Colombia also have been found to contain mercury; Even small amounts of mercury are considered hazardous to refinery operations. Therefore, refiners should be aware of their crude types and analyze for mercury to determine if it is present in their feed.

¹Oxford University - Dept of Earth Sciences – 2012

²Patent for Publication: US6537443 B1 – US Patent office

Figure 1. Forms of Mercury from Various Sources



There are various types of mercury ranging from elemental, organic, ionic and suspended. In refining applications the mercury will be in the elemental state (all forms of mercury that may be present in liquids are converted to elemental mercury after a hydroprocessing operation).

Many refiners have used the same crude slate for many years and do not test for mercury, as it has never been a recognized problem. But if the crude supply is changing due to more crude types being available on the market (and some cost savings by purchasing these crudes with more contaminants), they are exposing their process operations and personnel to possible

mercury issues.

Elemental Mercury

Due to its high density and tendency to agglomerate and bead, elemental mercury can be concentrated in drains and other low points of process plants. Upon breaching any mercury contaminated tank, pipe work or equipment, there is a possibility of worker airborne exposure, though there are few published studies in the oil industry. If hot work is involved, the 0.1 mg/m³ PEL and 0.025 mg/m³ TLV can be readily exceeded. In addition, mercury contaminated components may require classification as hazardous waste and may not be treatable as ordinary scrap iron.

At 20°C, elemental mercury has a vapor pressure of 1.2 µm Hg, which provides a surprising degree of volatility in the ppm range. Since a vapor pressure exists, it can be found in natural gas and distilled petroleum products. With this phase change, mercury concentration can also be modified. Just as any liquid evaporates, the partial pressure of mercury vapor in natural gas could make its concentration higher than that of the reservoir oil from which it came.

During refinery distillation, that segment of elemental mercury vapor is predominantly distributed in liquefied petroleum gas (LPG) and light distillate streams. But due to its weight, mercury vapor can also be found in the residual fraction, which also contains the majority of the insoluble and inorganic mercury salts. Little work has been carried out to assess the health impact arising from the mercury that is contained in fuels such as LPG and light distillates and used by consumers (cooking or transportation). Research is growing in these areas with reports confirming existing mercury emissions from common fuels³.

³Mercury in Crude Oils - 2013 MCA Spring Seminar Series -Tom Littlepage

Argentinean Crudes and Mercury Presence

Argentina is self-sufficient in crude oil reserves, but it still imports oil products (Figure 2). Oil and gas reservoirs could be as high as 2.5 billion barrels⁴ with the majority present in the Gulf of San Jorge Basin (Chubut and Santa Cruz provinces). The remaining reserves can be found in the Neuquén and Austral basins. In the last few years the shale oil formations that have been exploited have resulted in possibly 150 million barrels of shale oil. One of the impurities present in some of these formations is mercury.⁵

Figure 2

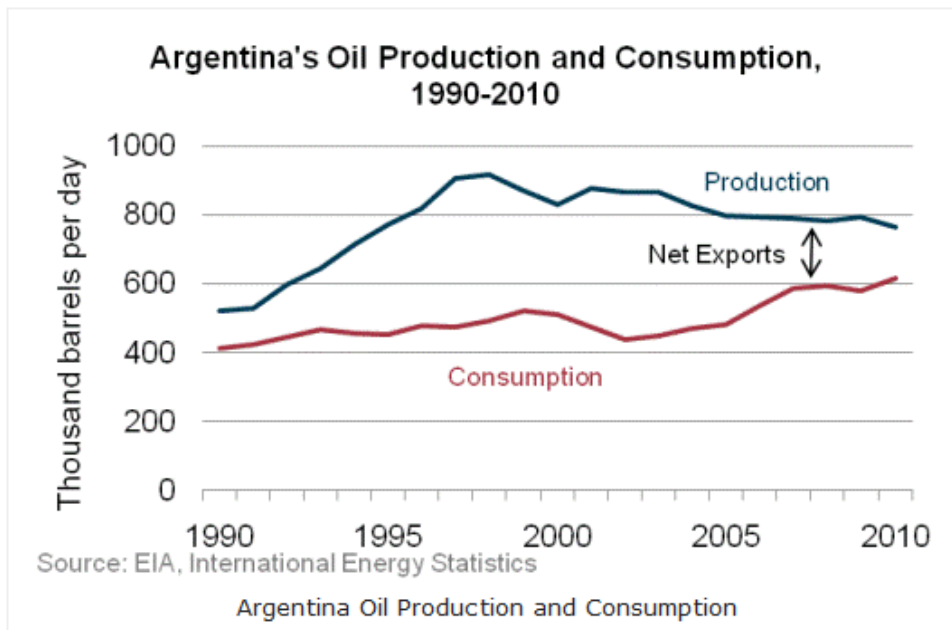


Figure 3 shows the variation of mercury content in crude oil from a reservoir in Argentina. The changes in mercury content varies widely even over just a month of analysis. As seen from the chart the mercury concentration changes between 1,500-8,000 ppm_w. The shale crudes from the Austral-Magellan's Basin is where the majority of mercury contamination comes from (See Figure 4 for Argentinean map).

⁴(Oil & Gas Journal Jan 2011)

⁵Reference - Oil Peak Argentina Energy Report 2012

Figure 3 – Argentinean Crude Mercury Concentration Variations

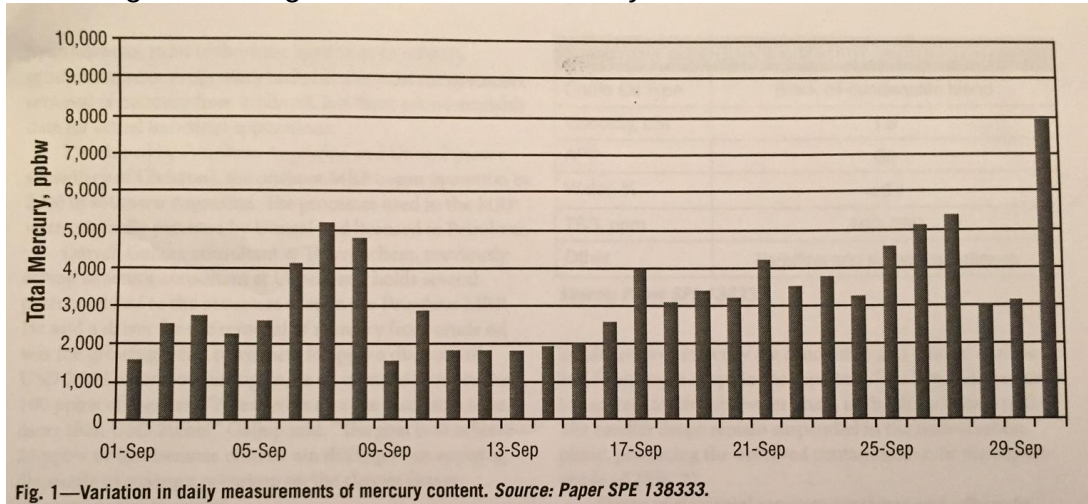


Figure 4 – Location of Oil & Gas Reserves in Argentina



Elevated mercury levels raise a number of crucial concerns for the fossil fuel related industry, from LNG, refineries, and petrochemicals to coal-fired power stations. The presence of mercury in natural gas can have catastrophic effects on gas plants, where

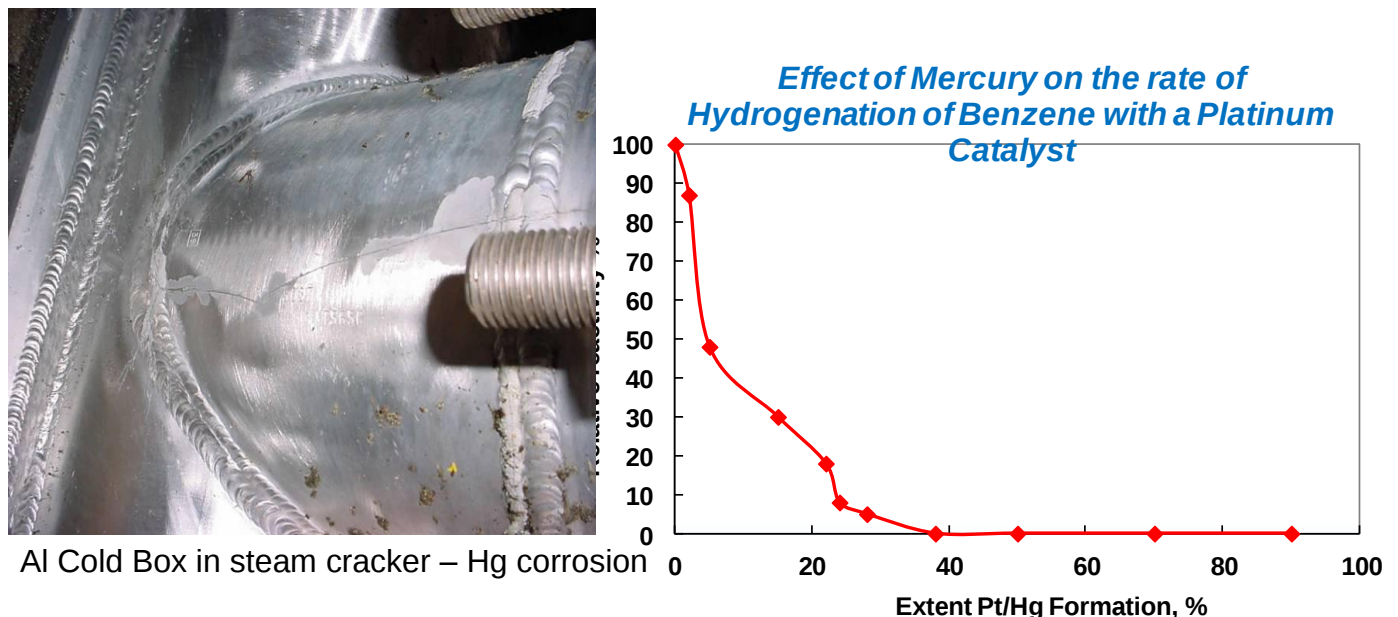
mercury attacks aluminum welds leading to cracking and eventually failure, resulting in uncontrolled loss of hydrocarbon containment, loss of process plant availability, and unscheduled shutdowns costing millions of dollars.

The mercury also attacks cryogenic components of LNG plants; it poisons catalysts and will cause reduction in quality of refined products. The brazed aluminum heat exchangers commonly found in LNG plants, cryogenic hydrocarbon recovery plants and petrochemical plants are particularly susceptible to liquid-metal embrittlement (LME) caused by mercury. LME is a corrosive attack that can cause crack initiation and propagation within equipment. Over the past several decades, LME has been responsible for a number of failures of brazed aluminum heat exchangers, resulting in unscheduled plant shutdowns, costly repairs and even fires.

Additional consequences of mercury include:

- Mobility – Mercury is mobile and will adsorb onto pipeline surfaces and other equipment; it can then desorb back into gas streams, contaminating the product for transportation
- Mercury can concentrate and drop out as liquid in colder sections of the plant, making subsequent maintenance difficult
- Catalyst poison - Precious metal catalysts operating at low temperatures (e.g. SHP) are susceptible to Hg poisoning. 1:1 Hg amalgams with platinum and Pd (palladium) are stable at temperatures $<150^{\circ}\text{C}$. See Figure 5 below of Pt Catalyst deactivation due to mercury.
- Equipment protection - Stress cracking of aluminum in the presence of mercury can cause costly repairs and downtime for refinery operations. The photo below shows an alumina cold box in a steam cracker with corrosion due the presence of mercury.

Figure 5



The other risk mercury poses is in maintenance and servicing. For example, it has been reported that during routine maintenance and service operations personnel have come across silvery metallic-like crude deposits in various fittings and crevices. Initial mercury investigation did not realize the affinity of mercury to form amalgamation with metallic surfaces. Mercury forms amalgams with a number of metals, including commonly-used metals such as aluminum, brass, copper, iron, nickel, zinc and precious metals. Environmental pollution by mercury has meant that even the polar snow/ice caps now have mercury deposits so environmental control and protection is becoming more important. In addition, the recent global mercury mitigation acts/publications like UNEP, Global Mercury Assessment 2014 are also bringing more scrutiny to mercury-related matters. Due to mercury becoming more prevalent and problematic, there is a demand for technologies to mitigate against the adverse effects of mercury presence.

Mercury Speciation in Hydrocarbon Streams

Mercury in samples is usually reported and thought of as total mercury, however, it is now appreciated that mercury exists in a number of chemical forms (species/compounds), each with its own unique characteristic properties. It has been demonstrated in literature that different mercury species are present in crude oil and their distribution varies across the whole spectrum of petroleum products, from natural gas, LPG, NLG, cracked naphtha, gas condensates and feedstock, etc. We will focus on refining. In Figure 6, the red dots show where you can treat for mercury if equipment and/or catalyst protection or exposure is a concern. The blue dots indicate where you can remove Hg to meet product specs and if mercury isn't a potential poison for the catalyst.

When considering the difference in treating natural gas versus crudes, natural gas traditionally contains over 90% elemental mercury, while crude oil and unprocessed gas condensates contain in addition to elemental mercury, various dissolved and insoluble species including mercury sulfide. In addition to this, certain external factors like sample source, history, chemical interaction of species (either with sample containment systems or degradation of certain species) and production/processing parameters have an influence on the species found.

However, due to difficulty and restrictions in quality analytical measurements of mercury speciation, there is a limited amount of data available in the petroleum industry. It has now become paramount that all the mercury species are determined in order to provide the best solutions for efficient plant operations and mercury removal systems. Unlike all other trace metals, mercury is a liquid at room temperature (25°C) and atmospheric pressure (1 atm). Its melting and boiling points are significantly lower than other trace metals, thus it is abundantly found in gaseous streams.

Mercury species found in hydrocarbons:

- Elemental
- Organic (methyl, ethyl mercury)
- Ionic (mercury chloride, Hg^{2+})
- Particulate
- Suspended (weakly adsorbed)
- Insoluble/Complexed (mercury sulfide, selenide)

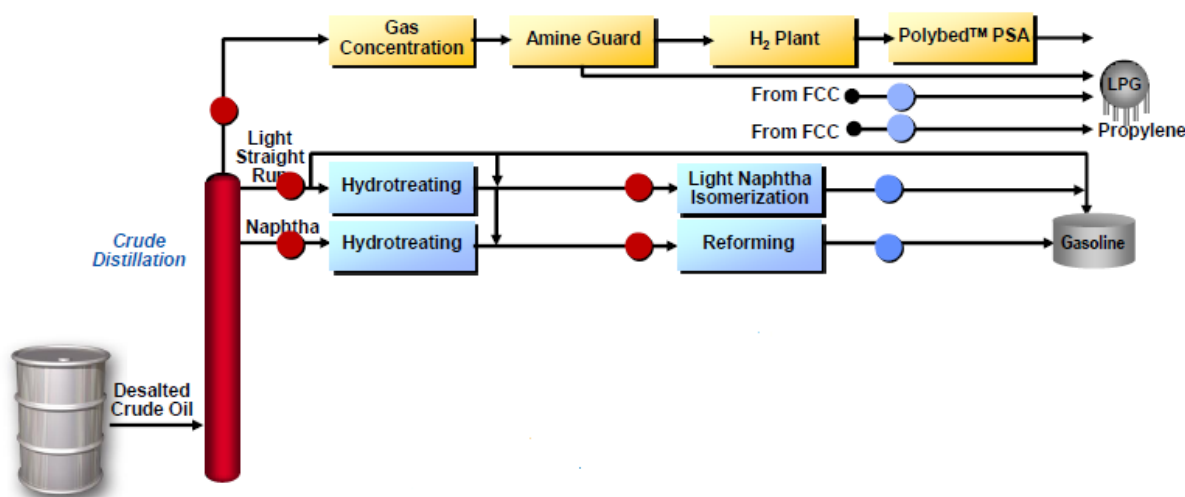
Options for Hg Removal in Refinery Applications

Mercury compounds segregate into naphtha streams and light products from an FCC. The location of mercury removal units (MRU) is dictated by the goal of the refiner:

Options to locate the MRU:

- After process unit to meet product spec and if not a poison to catalyst
- Upstream for equipment/catalyst protection or if exposure is a concern
- The farther upstream the unit, the greater the potential for other forms of Hg that will not be removed

Figure 6 – Typical Locations to Find Mercury in Refinery Operations



Advanced Solutions for Mercury Removal

Metal sulfides are optimal for Hg removal. Oil and gas typically contain sulfur and the removal of mercury requires the presence of sulfur. Metal-containing adsorbents and catalysts are part of oil and petrochemical processes and are heavily utilized for mercury removal. Several metal sulfides are powerful oxidizers for Hg. These reactions have favorable thermodynamics with low residual Hg with some metal-sulfide (MeS). Mercury is removed as HgS, the most stable mercury compound. Both Hg and the metal (Me) can be recovered if needed.

UOP understands and appreciates that mercury determination is a very complex procedure that involves many external factors. This amalgamation can not only lead to classification of plant materials as dangerous, toxic, contaminated waste but it also can possess a crucial health risk to maintenance and servicing personnel. Furthermore, mercury is now universally recognized as a corrosive agent. By amalgamating to metallic surfaces, it can reduce the integrity of plant materials, in particular high pressure transportation lines, tankers and cryogenic heat exchangers (aluminum parts). This can lead to incomprehensible damage to an organization's confidence, human health and the environment by causing potential explosions and leakage/spillages into the environment. As such, it is imperative that the mercury be determined accurately

and precisely. This danger was realized in 1973 at the Skikda LNG plant (Algeria) when the cryogenic heat exchangers underwent a catastrophic failure due to mercury-induced corrosion.

UOP Mercury Removal Adsorbents

With the advent of better analytical techniques, it is possible to measure and remove low levels of mercury before it threatens pipeline assets, gas plant equipment and the environment. UOP has several effective fixed-bed solutions for removing mercury from natural gas and hydrocarbon liquids, providing a total mercury management solution from dynamic regeneration (UOP HgSIV™ adsorbent i.e. regenerable) process to static permanent (UOP GB series i.e. non-regenerable) process.

The uniqueness of UOP HgSIV adsorbent makes it an ideal drop-in solution that can be added to existing dehydration units to remove mercury while not reducing the bed's ability to remove the water. Thus, no additional vessels are required, as you use one vessel for both water and mercury removal. UOP also can offer non-sulfided and pre-sulfided GB products, which can be directly related to the stream composition or a client's obstacles. A final solution segment is compound bed or mixed bed solutions, where a single GB bed can remove not only Hg but also H₂S co-currently. UOP is able to offer an extensive array of solutions for refineries needing multiple contaminant removal:

- UOP HgSIV™ molecular sieve regenerative adsorbents
- UOP GB copper-based non-regenerative adsorbents
- UOP H₂S copper-based non-regenerative adsorbents

The basis of removal is very simple. As the gas or liquid flows through the fixed bed, mercury and H₂S is retained by the adsorbent and remains in the vessel, resulting in effluent gas or liquid hydrocarbon that has mercury and H₂S levels within typical industry targets. However, the technology and science behind the solution is far more complex, and all UOP adsorbent solutions are designed and delivered with the following beneficial parameters:

Beneficial Parameters	Benefits
Advanced active metal component results in high elemental mercury removal performance	Have smaller bed size, help reducing capital investment for new units and total adsorbent costs (purchase and disposal)
Large pore volume combined with custom designed pore distribution results in good resistance to liquid-carry over upsets	Help reduce unplanned shutdowns due to liquid carry over upsets
High surface area substrate results in high metal utilization	Provide options for user to improve throughput or adsorbent bed life.
Particle size and shape result in low and stable pressure drop across bed	Help reduce unplanned shutdown due to high pressure drop

Proven crush strength, attrition, and abrasion resistance	Help reduce unplanned shutdowns due to dust leakage to downstream equipments
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UOP provides a range of mercury removal adsorbents that cater to a variety of customer needs.

UOP Product	Application
GB-346 / GB-562	Liquid-Phase / Gas-Phase Hydrocarbon Streams
GB-346HP / 562HP	High Capacity Liquid-Phase / Gas Phase Hydrocarbon Stream
GB-347 / GB-567	Highest Capacity Liquid Phase / Gas Phase Hydrocarbon Stream (Extruded - Trilobe)

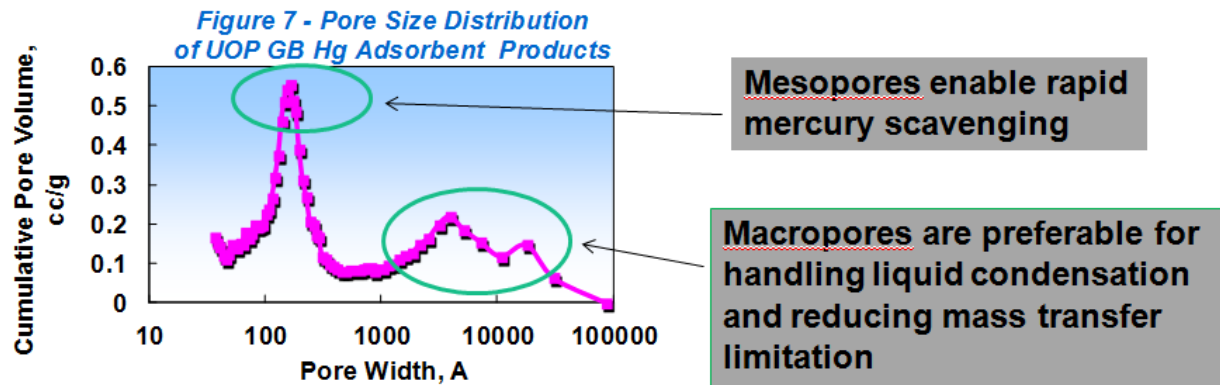
All products are available in sulfide and un-sulfided (oxide) form.

UOP GB-347 and GB-567 products are the latest premier UOP Hg-removal adsorbents that are able to provide long bed life with higher inlet mercury concentrations than previous UOP adsorbent products. The extruded shape of the adsorbent enhances its capacity for mercury removal, along with the unique controlled pore structure that comes from UOP's state-of-the-art manufacturing process. From the chart below you can see the jump in capacity between the highest capacity extruded GB-567 and the high capacity beaded GB-562HP. Customers can choose from any of the three products depending on their needs (mercury concentration in their refinery, economics for their reloads, and operating conditions in their mercury removal unit). One of the things that makes UOP products unique is the manufacturing process used; one of the results of this manufacturing process is a much more varied pore structure in the adsorbent products. But why is pore size and distribution important?

Pore Structure

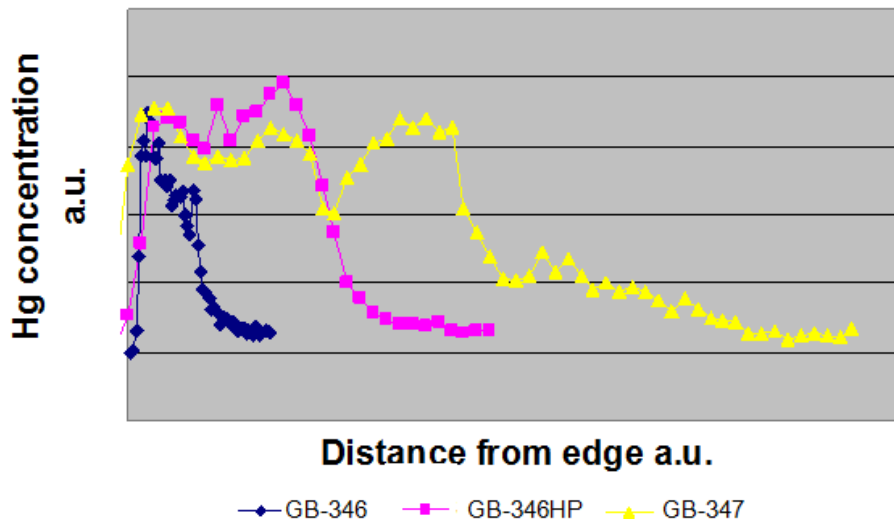
Porous inorganic matrices support the sulfide phase for practical use. UOP adsorbents contain modified alumina as a support, which has large macroporosity. These adsorbents are designed to operate effectively at short contact times and handle liquid hydrocarbon streams. The custom design of the active component and porosity is the key to highly efficient mercury guard adsorbent systems. UOP's manufacturing process creates a range of pore size (macro/meso/micro) that allow for high-performance contaminant removal. As seen in Figure 7 – UOP adsorbents have a high percentage of mesopores that allow for rapid adsorption of the contaminant into the adsorbent, while the macropores assist with handling liquid carry-over incidents and reducing mass

transfer limits. Products that are designed with mesopores and have less macroporosity are less able to recover from liquid carry-over incidents.



An Extruded shape provides a higher capacity for removal than a beaded product for a variety of reasons, including better dispersion of the active component throughout the adsorbent and specifically-designed porosity distribution for Hg to penetrate into the adsorbent. As seen from Figure 8 the trilobe adsorbent (yellow) allows for deeper penetration into the extrudate and results in much higher capacity for mercury removal than beaded adsorbents.

Figure 8 –Variance of Hg Penetration in UOP GB Adsorbents



UOP solutions can handle customer needs from low to high capacity mercury removal.

Sulfur Removal in Refining

Along with mercury, sulfur is a strong contaminant that is present in crudes like West Texas Sour, and also some shale oil and bitumen crudes. Sulfur removal adsorbents have been in the industry for many decades, and progressively have become more robust and able to capture more sulfur species than in the past. UOP continues to work with refiners and petrochemical customers to understand their current contaminant issues and develop new products that meet and exceed their needs.

There are many species of sulfur that need to be removed from process streams, with just a few listed below:

- H_2S
- Simple and complex mercaptans
- Disulfides
- Thiophenes

Some may be in conjunction with mercury removal, while most will be individual sulfur guard beds (SGB) with the sole purpose to remove the sulfur species in the process stream. Usual reasons for the removal of sulfur are:

- Provide ultra-low sulfur concentration in effluent streams to assure protection of downstream catalyst
- Meet local environmental regulations
- Assure attainment of sulfur specifications for refinery product streams

Typically for process streams of C4 or lighter hydrocarbons, regenerable sulfur removal is cost-effective. Sulfur concentrations >10ppmw will best be treated with a regenerable adsorbent that will be utilized through multiple adsorption and regeneration steps until the adsorbent is spent (typically 250-300 cycles). For C5+ hydrocarbons requiring sulfur removal, regenerable options are usually not cost-effective and a non-regenerable adsorbent solution is typically best.

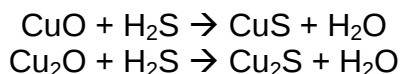
In many refinery process operations there is hydrotreating, Claus units and amine systems that can remove high concentrations of sulfur compounds (tons of sulfur per day), so it is important to remember that adsorbents are for trace contaminant removal only. Most common treatment areas for these non-regenerable applications are in isomerization process units (like Penex and BenSat) or downstream of naphtha hydrotreaters in Cat Reforming units. Typical operating temperatures range between 120°C-175°C at pressures to keep the process stream in the liquid phase.

UOP Sulfur Removal Adsorbents

As with mercury removal adsorbents, UOP has multiple sulfur removal adsorbents to cater to the many needs refiners have for different sulfur species removal. What a refiner needs to consider is what species of sulfur to remove at what concentration, is there a benefit to using a Cu-based or Ni-based or ZnO-based adsorbent for their specific condition, is their isomerization unit susceptible to high temperatures, and how tight a sulfur specification they need to reach.

Cu-based Sulfur-Removal Adsorbents

The most common adsorbent for ambient or low temperature sulfur removal (H_2S and mercaptan) is a Cu-based alumina. For a refiner with these sulfur species and temperatures between 100°C - 200°C , a Cu-based adsorbent is typically the most cost-effective along with sufficient capacity to provide many months of sulfur removal protection. Typically a CuO or Cu_2O is the metal chosen, and when reacted with H_2S forms CuS or Cu_2S respectively along with water:



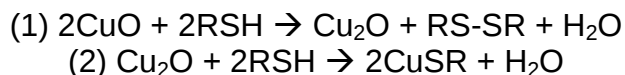
Cu-based adsorbents will usually cost less per kg than other metal adsorbents, and provide the highest amount of H_2S removed per U.S. dollar spent for a sulfur guard bed adsorbent. They are also easier to load and unload than Ni-based adsorbents, which require more specific handling requirements due to the hazards of nickel. Nickel adsorbents are more costly to purchase and dispose of but do have specific benefits that will be discussed later. At the typical lower temperatures in isomerization sulfur guard bed operations, Cu is a more active metal for H_2S and mercaptan removal than Ni or ZnO

UOP's prime H_2S adsorbent is ADS-120, a Cu-based alumina adsorbent that is resistant to reduction and reduces water evolution (water being a poison for isomerization catalyst). In general ADS-120 reduces water evolution approximately 50% compared to standard Cu-adsorbents.

As with the UOP Hg adsorbents, the ADS-products are manufactured to produce higher surface areas and better pore distribution which both enhance metals utilization. These adsorbents also have lower reactivity in olefinic streams which reduces by-product formation and coking effects if there are temperature excursions. Coking covers up the active surface area and reduces the adsorbent's ability to remove sulfur species and shortens the bed-life of the SGB. ADS-120 has shown to be unreactive towards olefins up to 260°C , so the formation of oligomerized by-products is almost eliminated.

For more specialized refinery needs in trace sulfur removal, there is a product called ADS-130. It is a reduced version of ADS-120 and provides the refiner with a sulfur adsorbent that does not have any water evolution, does not form disulfides while removing mercaptans, is not self-heating, and also removes some thiophenes. Water generation not only impacts the isomerization catalyst, but it can also overload the feed dryers with excess water and end up costing the operator expensive molecular sieve change-out.

Sometimes there is a risk for forming disulfides while removing mercaptans (1), but the ADS-130 doesn't do this – it converts the mercaptan to a Cu-mercaptan for removal (2).



This reduces additional sulfur contaminants that need to be removed from the process stream and saves on multiple adsorbent costs for a single system. Being a Cu-based adsorbent, these products are again lower fill costs versus Ni-based products for similar applications. There are over 100 units around the world using ADS-120/ADS-130 products that are meeting or exceeding customer requirements.

Ni-Based Adsorbents

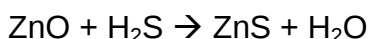
As stated above, Ni-based adsorbents are usually less favorable than Cu-based adsorbents for the more stringent loading, unloading and disposal requirements and associated costs. However, Ni-adsorbents provide some benefit to certain SGB applications, as they have slightly higher capacity for H₂S, mercaptan and Thiophene removal than a standard Cu-based alumina adsorbent. Frequently, Ni-adsorbents are chosen as they have a higher capacity for thiophene removal than a Cu-adsorbent (even higher than the ADS-130). ADS-11L is the UOP Ni-based product that has been commercialized for many years with successful operations.

ZnO-based Adsorbents

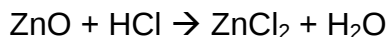
As has been discussed, Cu- and Ni-based adsorbents are widely used for isomerization SGB applications at temperatures ranging from 120°C-175°C. But there are other SGB operations that are at much higher temperatures and need an adsorbent that can perform at these conditions. This is when ZnO-based adsorbents are best utilized.

Standard ZnO alumina adsorbents only remove H₂S; they are not for removing mercaptan or other sulfur species. However, UOP's ADS-280 product also has the capability to remove a small percentage of COS. The alumina component of ADS-280 provides sufficient capacity for the COS removal through the hydrolysis of the COS to CO₂ and H₂S. ADS-280 has a high surface area compared to some other commercially available ZnO products, which results in longer bed life and savings for the refinery in down-time and change-outs. Operating temperatures for H₂S removal using ZnO range between 150°C-450°C and can apply in H₂ generation plants along with isomerization plants.

As with other metals, the ZnO forms a ZnS and water after reaction:



Typical process streams for using ZnO adsorbents are natural gas, naphtha, and LPG streams. But a refinery must confirm if chlorides are present in the stream, as chlorides can form ZnCl₂, which is detrimental to downstream equipment.



ZnCl₂ has a low melting point (292°C) and can be volatilized at low temperatures. If chlorides are present, a guard bed is needed to remove them.

Sulfur compounds will continue to be an issue for refineries around the world. With the above products, UOP can help refiners meet and exceed their product specifications and operational reliability.

Conclusion

With the changing trends in the oil industry, refineries must be more aware of contaminants in their crudes and know what products are the best for their overall operation. As more shale oil and bitumen crudes come into the market, and cheaper oil prices have refineries shopping around for lower-quality, less-costly feedstock, there will be more issues with mercury and sulfur contamination of process streams. High capacity adsorbents are available to reduce change-outs and downtime that also remove multiple contaminants in a single bed to save on capital and operational costs. There are multiple products that can help refiners with these new challenges, and UOP is there for customers around the world and with offices in South America to support refining and petrochemical plants in the region.