New Approaches to Deep Desulfurization for Ultra-Clean Gasoline and Diesel Fuels: An Overview

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1. Introduction
The U.S. Clean Air Act Amendments of 1990 and new regulations by the U.S. EPA [EPA Gasoline-RIA, 1999; EPA Diesel-RIA, 2000; EPA RFG, 1999] and government regulations in many countries call for the production and use of more environmentally friendly transportation fuels with lower contents of sulfur and aromatics. In the mean time, the demand for transportation fuels has been increasing in most countries for the past two decades. The total U.S. consumption of petroleum products reached 18.68 million barrels per day (MBPD) in 1998. Of the petroleum consumed, 8.20 MBPD was used as motor gasoline, 3.44 MBPD as distillate fuels (including diesel fuels and industrial fuels), 1.57 MBPD as jet fuels, 0.82 MBPD as residual fuel oil, and 1.93 MBPD as liquefied petroleum gas (LPG), and 2.72—for other uses [EIA/AER, 1999].

Clean fuels research including desulfurization and dearomatization has become an important subject of environmental catalysis studies worldwide. Figure 1 presents a qualitative relationship between the size and type of sulfur molecules in various distillate fuel fractions and their relative reactivities. The reactivity ranking in Figure 1 is based on our experimental observations and a large amount of literature information [Knudsen et al., 1999; Whitehurst et al., 1998; Song et al., 2000]. With the new EPA Tier II regulations to cut the diesel sulfur from current 500 ppmw down to 15 ppmw by June 2006 and sulfur reduction from current 350 ppm to 30 ppm by 2005-2006, refineries are facing major challenges to meet the fuel sulfur specification along with the required reduction of aromatics contents.

The problem of deep removal of sulfur has become more serious due to the lower and lower limit of sulfur content in finished gasoline and diesel fuel products by regulatory specifications, and the higher and higher sulfur contents in the crude oils. A survey of the data on crude oil sulfur content and API gravity for the past two decades reveals a trend that U.S. refining crude slates continue towards higher sulfur contents and heavier feeds [Swain, 1991, 1998]. The average sulfur contents of all the crude oils refined in the five regions of the U.S. known as five Petroleum Administration for Defense Districts (PADDs) increased from 0.89 wt% in 1981 to 1.25 wt% in 1997, while the corresponding API gravity decreased from 33.74° in 1981 to 31.07° in 1997. In 2000, the average crude feeds to US refineries has 1.35 wt% sulfur and 31.0° API gravity, whereas European refinery feed by comparison was sweeter at 1 wt% sulfur and 35 ° API gravity [Lawson, 2001].

2. Deep Desulfurization of Gasoline
Table 1 shows the typical gasoline pool compositions in the U.S. [Shorey et al., 1999] and in the Western Europe [Marcilly, 2001]. It is well known that naphtha from FCC makes up about 25-40% (average of 36% in the US) of gasoline blend stocks, but accounts for over 90% of the sulfur (up to 90-98%) and olefins in the entire gasoline pool. Therefore, the key to deep desulfurization of gasoline is sulfur removal from naphtha.

It is well known that sulfur removal from FCC naphtha can be achieved by catalytic hydrodesulfurization, but the accompanying decrease of octane number is a significant loss due to the saturation of olefins. Hydrotreating of FCC naphtha is an attractive process alternative, provided that octane losses are minimized. Because FCC naphtha also contains olefins which have higher octane number, selective sulfur removal without loss of octane number (loss of olefins) is desirable.

Approaches to reducing sulfur content in FCC naphtha include (1) post-treating product to remove sulfur from FCC naphtha [Sloley, 2001; O’Connor and Mayo, 2001]; (2) pretreating the FCC feed to remove sulfur [Shorey et al., 1999]; (3) increasing sulfur conversion in-situ to hydrogen sulfide during the FCC operation [O’Connor and Mayo, 2001].

There exist different active sites on hydrotreating catalysts (such as sulfided Co-Mo/Al2O3) for thiophene desulfurization and for olefin hydrogenation. Selective HDS could be achieved by passivating olefin hydrogenation sites. Some of the processes for gasoline desulfurization including selective HDS and alternate desulfurization options are briefly described below.

*Post treat FCC product to remove sulfur
- Apply conventional hydrodesulfurization without olefin preservation [Many companies]
- Convert organic sulfur to H2S by selective hydrodesulfurization while preserving olefins [ExxonMobil’s Scanfining; IFP Prime G+].
- Hydrodesulfurize organic sulfur, saturate olefins but convert paraffins for octane gain [ExxonMobil Octgain 125; UOP- INTEVEP ISAL].
- Catalytic distillation and hydrodesulfurization in the same vessel [CDTech’s CDHydro + CDHDS].
- Sulfur adsorption and capture by solid adsorbent at elevated temperatures under low H2 pressure [Phillips Petroleum S Zorb-Gasoline]
- Sulfur adsorption and capture by solid adsorbent at high temperatures [Research Triangle Institute TReND]
- Selective adsorption for removing sulfur (SARS) as organic compounds by solid adsorbent at ambient
temperature without using H\textsubscript{2} [Penn State University SARS process]

- Drop the organic sulfur to heavier fraction by alkylation of thiophenes [BP’s OATS process].
- Remove the organic sulfur by using caustic treatment [Merichem’s THIOLEX/REGEN process; EXOMER by Merichem and ExxonMobil] or extraction [GTC Technology’s GT-DeSulf].

*) Pretreat the FCC feed to remove sulfur
- Deep hydrodesulfurization of FCC feed before catalytic cracking in FCC reactor, which greatly reduces sulfur in FCC naphtha and in Light cycle oil [Akzo Nobel, IFP, etc.]

*) Increase sulfur conversion during FCC
- Convert more organic sulfur into H\textsubscript{2}S during FCC operation, which can moderately reduce sulfur in products [Akzo Nobel’s Resolve; Grace Davison’s Saturn (GSR-6.1)].

Figure 1. Reactivity of various organic sulfur compounds in hydrodesulfurization versus their ring sizes and positions of alkyl substitutions on the ring.
Compared to the diesel sulfur problem, it is not difficult to remove sulfur from gasoline. The challenges to the refinery for gasoline deep desulfurization are to meet the new EPA Tier-II regulations on sulfur contents and aromatic contents and still produce high-octane gasoline in a profitable manner.

In our laboratory, we are exploring a SARS (selective adsorption for removing sulfur) process for gasoline desulfurization at ambient temperatures without using H₂ [Ma et al., 2001, 2002]. The SARS can be applied for on-site or on-board organic sulfur removal before the reformer for fuel cells, and for new desulfurization system for producing ultralow-sulfur fuels in a future refinery. The SARS process is different from the Phillips S Zorb process that uses low-pressure H₂ at elevated temperatures [Gislason, 2001] and different from the TreND desulfurization process being developed by Research Triangle Institute that uses solid adsorbent at high temperatures [Turk and Gupta, 2001].

### Table 1. Typical Gasoline Pool Composition in U.S. and W. Europe

<table>
<thead>
<tr>
<th>Gasoline blend stocks</th>
<th>% Gasoline pool volume in US</th>
<th>% gasoline pool sulfur in US</th>
<th>% gasoline pool volume in W. Europe</th>
</tr>
</thead>
<tbody>
<tr>
<td>FCC naphtha</td>
<td>36</td>
<td>98</td>
<td>27</td>
</tr>
<tr>
<td>Reformate</td>
<td>34</td>
<td>---</td>
<td>40</td>
</tr>
<tr>
<td>Alkylate</td>
<td>12</td>
<td>---</td>
<td>9</td>
</tr>
<tr>
<td>Light straight-run naphtha</td>
<td>3</td>
<td>1</td>
<td>7.5</td>
</tr>
<tr>
<td>Coker naphtha</td>
<td>1</td>
<td>1</td>
<td>~0</td>
</tr>
<tr>
<td>Hydrocracked naphtha</td>
<td>2</td>
<td>---</td>
<td>~0</td>
</tr>
<tr>
<td>Isomerate</td>
<td>5</td>
<td>---</td>
<td>10</td>
</tr>
<tr>
<td>Butanes</td>
<td>5</td>
<td>---</td>
<td>5.5</td>
</tr>
<tr>
<td>MTBE</td>
<td>2</td>
<td>---</td>
<td>1.0</td>
</tr>
<tr>
<td>Total</td>
<td>100 %</td>
<td>100 %</td>
<td>100 %</td>
</tr>
</tbody>
</table>

3. Deep Desulfurization of Diesel Fuels

The general chemistry of diesel fuel processing and hydrodesulfurization have been discussed in several recent reviews and books [Topsoe et al., 1996; gates and Topsoe, 1997; Whitehurst et al., 1998; Knudson et al., 1999; Song et al., 2000]. Table 2 shows the volume fraction of U.S. highway diesel pool, and Table 3 shows the corresponding sulfur levels of U.S. highway diesel blendstocks [EPA Diesel-RIA, 2000]. Among the diesel blend stocks, the light cycle oil (LCO) from fluid catalytic cracking (FCC) contains highest amount of sulfur and aromatics, and the LCO also tends to have the highest contents of refractory sulfur compounds, especially 4methyl dibenzothiophene and dibenzothiophene [EPA Diesel-RIA, 2000].

Deep desulfurization and ultra-deep desulfurization refers to processes to remove sulfur to below 500 ppmw and 15-50 ppmw, respectively. Described below are some of the processing approaches for ultra-deep desulfurization to produce ultra-low-sulfur (ultra-clean) diesel fuels. The key to ultra-deep desulfurization is the removal of refractory sulfur compounds, particularly 4,6-dimethyl dibenzothiophene, from diesel fuels. In addition to straight-run gas oil, the light cycle oil (LCO) from FCC of heavy oils is a major blend stock for diesel fuels in the US. LCO has higher sulfur contents, in which the content of 4,6-dimethyl dibenzothiophene is higher than other middle distillates in diesel pool. LCO also has higher aromatics contents, which makes the dearomatization necessary for producing low-aromatic diesel fuel.

The problem of deep hydrodesulfurization of diesel fuel is caused by the lower reactivity of 4,6-disubstituted dibenzothiophene, as represented by 4,6-dimethyl dibenzothiophene (4,6-DMDBT) which has much lower reactivity than any other sulfur compounds [Ma et al., 1994] in diesel blend stocks. The methyl groups at 4- and 6-positions create steric hindrance for the interaction between sulfur and active sites on the catalysts. The problem is exacerbated by the inhibiting effects of polyaromatics and nitrogen compounds in some diesel blend stocks for diesel as well as H₂S that exist in reaction system on deep HDS. Based on experimental results, polyaromatics compete with sulfur compounds on the surface of hydrotreating catalyst, perhaps more for the flat chemisorption thereby influencing the hydrogenation and subsequent hydrodesulfurization, whereas H₂S compete with sulfur compounds, affecting more of the direct C-S hydrogenolysis route.

Approaches to ultra-deep desulfurization include (1) improving catalytic activity by new catalyst formulation for HDS of 4,6-DMDBT; (2) tailoring reaction and process conditions; (3) designing new reactor configurations; and (4) developing new processes. One or more approaches may be employed by a refinery to meet the challenges of producing ultra-clean fuels at affordable cost.

Design approaches for improving catalytic activity for ultra deep hydrodesulfurization focus on how to remove 4,6-DMDBT more effectively, by modifying catalyst formulations to (1) enhance hydrogenation of aromatic ring in 4,6-DMDBT by increasing hydrogenating ability of the catalyst; (2)
incorporate acidic feature in catalyst to induce isomerization of methyl groups away from 4- and 6-positions; and (3) remove inhibiting substances (such as H\textsubscript{2}S) and tailoring the reaction conditions for specific catalytic functions. The catalytic materials formulations may be improved for better activity by using different supports (carbon, TiO\textsubscript{2}, TiO\textsubscript{2}-Al\textsubscript{2}O\textsubscript{3}, HY, MCM-41, etc.) for conventional alumina-supported CoMo, NiMo and NiW catalysts; by increasing loading level of active metal (Mo, W, etc.); by adding one more base metal (e.g., Ni to CoMo or Co to NiMo); and by incorporating a noble metal (Pt, Pd, Ru, etc.).

Table 2 Volume Fraction of U.S. Highway Diesel Pool from each Fedstock Component

<table>
<thead>
<tr>
<th>Diesel Blendstock</th>
<th>Naphtha</th>
<th>Light Distillate</th>
<th>Heavy Distillate</th>
<th>Light Gas Oil</th>
<th>All Boiling Fractions Combined</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight Run</td>
<td>0.1</td>
<td>6.4</td>
<td>4.9</td>
<td>1.0</td>
<td>12.4</td>
</tr>
<tr>
<td>Hydrotreated Straight Run</td>
<td>0.3</td>
<td>8.1</td>
<td>41.2</td>
<td>2.3</td>
<td>51.9</td>
</tr>
<tr>
<td>Cracked Stock</td>
<td>-</td>
<td>0.1</td>
<td>0.8</td>
<td>2.2</td>
<td>3.1</td>
</tr>
<tr>
<td>Hydrotreated Cracked Stock</td>
<td>2.1</td>
<td>15.6</td>
<td>1.7</td>
<td>19.4</td>
<td></td>
</tr>
<tr>
<td>Coker Gas Oil</td>
<td>-</td>
<td>-</td>
<td>1.0</td>
<td>-</td>
<td>1.0</td>
</tr>
<tr>
<td>Hydrotreated Coker Gas Oil</td>
<td>0.1</td>
<td>2.1</td>
<td>3.7</td>
<td>2.3</td>
<td>8.2</td>
</tr>
<tr>
<td>Hydrocrackate</td>
<td>-</td>
<td>1.3</td>
<td>2.7</td>
<td>-</td>
<td>4.0</td>
</tr>
</tbody>
</table>


Described below are some of the processing approaches for ultra-deep desulfurization to produce ultra-low-sulfur diesel.

* Approaches for deep desulfurization to produce ultra-low-sulfur diesel
  - Apply conventional hydrotreatment (Many companies can apply simple hydrotreating)
  - Ultra-deep hydrotreatment of middle distillate [MAKFining premium Distillates technology (PDT) by Akzo Noble, Exxon Mobil, and Kellog Brown]
  - Ultra-deep desulfurization (SynHDS) and hydrodearomatization (SynSat) and cetane improvement by ring-opening (SynShift) of middle distillate [SynTechnology including new reactor design by SynAlliance including ABB Lummus, Criterion Catalyst, and Shell Global].
  - Two-stage hydrotreating for ultra-low-sulfur diesel fuel using industrially proven high-activity TK catalysts [Haldor Topsoe]
  - Ultra-deep hydrotreatment, hydrodenitrogenation and hydrogenation of distillate fuels [Unionfining by UOP]
  - Hydrodearomatization (HDAR) of middle distillate in the second stage [MAKFining premium Distillates technology (PDT) by Akzo Noble, Exxon Mobil, and Kellog Brown]
  - Sulfur adsorption and capture by solid sorbent at elevated temperatures under low-pressure H\textsubscript{2} [Phillips Petroleum S Zorb Diesel]
  - Selective adsorption of organic sulfur compounds in diesel fuels by solid adsorbent at ambient temperature without using H\textsubscript{2} [Penn State SARS process]
  - Oxidative desulfurization by chemical oxidation in liquid phase [Ultrasonic desulfurization process by SulphCo using H\textsubscript{2}O\textsubscript{2} under ultrasound radiatiation; chemical oxidation by Petrostar using peroxyacetic acid]
  - Biodesulfurization of distillate fuels using [microbial desulfurization process by Energy BioSystems Corp.]
  - FCC feed deep hydrotreating to remove sulfur before catalytic cracking which produces naphtha and LCO [Some companies can apply deep hydrotreating for FCC feed]
  - Undercutting of sulfur-rich narrow fraction of light cycle oil from FCC [to remove a narrow bp range that is rich in refractory sulfur compounds, and use it for off-road distillate fuels]

In our laboratory, we are developing new catalysts such as Co-Mo/MCM-41 and exploring effects of nitrogen on ultra-deep HDS for existing refinery processes [Song and Reddy, 1999; Reddy et al., 1998; Turaga and Song, 2001, 2002; Song et al., 2001]; exploring a novel SARS (selective adsorption for removing sulfur) process for ultra-deep desulfurization of diesel fuels at ambient temperatures without using H\textsubscript{2} [Ma et al., 2001, 2002; Sprague et al., 2002]; and further developing our proposed concept for design of sulfur-resistant noble metal catalyst for low-temperature hydrotreating [Song et al.,...]

* Fuel Chemistry Division Preprints 2002, 47(2), 441*
Table 3  Sulfur levels of U.S. Highway Diesel Blendstocks (CA Excluded)

<table>
<thead>
<tr>
<th>Diesel Blendstock</th>
<th>Naphtha</th>
<th>Light Distillate</th>
<th>Heavy Distillate</th>
<th>Light gas Oil</th>
<th>All Boiling Fractions Combined</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight Run</td>
<td>827</td>
<td>1770</td>
<td>2269</td>
<td>4980</td>
<td>2218</td>
</tr>
<tr>
<td>Hydrotreated</td>
<td>362</td>
<td>119</td>
<td>394</td>
<td>548</td>
<td>358</td>
</tr>
<tr>
<td>Cracked Stock</td>
<td>-</td>
<td>2219</td>
<td>2892</td>
<td>6347&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5322</td>
</tr>
<tr>
<td>Hydrotreated</td>
<td>18</td>
<td>37</td>
<td>939</td>
<td>1306&lt;sup&gt;a&lt;/sup&gt;</td>
<td>874</td>
</tr>
<tr>
<td>Coker Gas Oil</td>
<td>540</td>
<td>1800</td>
<td>3419</td>
<td>-</td>
<td>3419 (?)</td>
</tr>
<tr>
<td>Hydrocrackate</td>
<td>8</td>
<td>25</td>
<td>310</td>
<td>400</td>
<td>258</td>
</tr>
<tr>
<td>Hydrocrackate</td>
<td>-</td>
<td>1.3</td>
<td>2.7</td>
<td>-</td>
<td>4.0</td>
</tr>
<tr>
<td></td>
<td>-</td>
<td>12</td>
<td>120</td>
<td>-</td>
<td>85</td>
</tr>
</tbody>
</table>

<sup>a</sup>: Indicate properties that were not reported in the refiner survey. These values were calculated by EPA using the reported sulfur contents of like boiling fractions in other diesel blend stocks by assuming the same relative sulfur levels between boiling fractions.


4. Deep Hydrogenation of Diesel Fuels

High aromatic content in distillate fuels lowers the fuel quality and contributes significantly to the formation of environmentally harmful emissions [Stanislaus and Cooper, 1994; Cooper and Donnis, 1996]. Reducing aromatic content along with sulfur content is generally desirable with respect to diesel fuel quality, as aromatic reductions increase cetane levels and generally improve combustion characteristics. California Air Resources Board (CARB) passed legislative measures to limit the sulfur and aromatic contents of diesel fuel to 0.05 wt% and 10 vol%, respectively, effective October 1993. However, reducing aromatics consumes hydrogen and increases the cost of desulfurization relative to a case where only sulfur was being removed.

Currently, conventional hydrotreating technology is adapted for dearomatization by aromatics saturation [Stanislaus and Cooper, 1994]. Typical conventional catalysts for fuel hydrosnrocessing are sulfided Co-Mo and Ni-Mo supported on alumina. Some studies have shown that complete hydrogenation of aromatics is not possible owing to equilibrium limitations under typical hydrotreating conditions. Conventional middle distillate hydrotreaters designed to reduce sulfur and nitrogen levels would lower the diesel aromatics only marginally [Stanislaus and Cooper, 1994; Cooper and Donnis, 1996]. For example, Ali and Siddiqui [1997] compared 3 types of hydrotreating catalysts, CoMo/Al₂O₃, NiMo/Al₂O₃ and NiW/Al₂O₃, for dearomatization of light cycle oil. They observed that the type of catalyst has a critical influence on the composition and properties of the product. Their data show that it was possible to obtain a diesel product that meets stringent specifications using one type of catalyst in a single-stage reactor even under severe operating conditions [Ali and Siddiqui, 1997].

While noble metals are active for hydrogenation at low temperatures, their use is limited because of their sensitivity to sulfur poison. In current processing schemes involving noble metal catalysts, two or more stages with multiple catalyst beds are used to achieve deep desulfurization and deep hydrogenation. Hydrodesulfurization occurs in the first stage over a Ni-Mo or Co-Mo catalyst, followed by intermediate byproduct gas removal. Finally, hydrogenation over the noble metal catalyst operates in the last stage or bottom bed where the concentrations of catalyst poisons (organosulfur and H₂S) are extremely low [Stork, 1996; Maxwell, 1997]. Commercial examples of two-stage or multi-stage hydrosnrocessing technology include the Shell Middle Distillate Hydrogenation process by Shell [Lucien et al., 1994; Stork, 1996], the Dual-Stage Process by Haldor-Topsoe [Cooper et al., 1994], and hydrotreating process by IFP [Marchal et al., 1994], and the SynSat process developed by Criterion/Lummus [Suchanek, 1996; Maxwell, 1997]. There are no reports of noble metal catalysts that can operate without such intermediate H₂S removal [Stork, 1996].

A New Approach for Catalyst Design.

Because of its importance, sulfur resistance of noble metal catalysts has been the subject of a number of studies [Cooper and Donnis, 1996; Absi-Halabi et al., 1997; Lin and Song, 1996; Schmitz and Song, 1997; Song et al., 1999a, 1999b]. More recently, a new approach has been proposed for the design of sulfur-resistant noble metal catalysts for low-temperature hydrotreating of sulfur-containing distillates to
produce clean distillate fuels [Song, 1998, 1999a] such as diesel fuels and jet fuels. The proposed design concept [Song, 1999b] invokes some unique zeolites as supports for noble metals and utilizes (1) shape-selective exclusion, (2) hydrogen spillover, and (3) two types of sulfur resistance. Unique zeolite supports can be used to prepare bimodal distributions of noble metal particles. Some metals are located in small pores (Sm: pore opening less than about 5 Å); whereas, others will be contained in large pores (La: pore opening larger than 6 Å). Preferably, the two pore systems inter-connect, or are at least uniformly distributed so that they are in close proximity. Diffusion of organosulfur compounds such as thiophenic molecules into the small-pores would be inhibited by size (shape-selective exclusion). The large pores (large micropore or mesopore range) would preferably allow fast diffusion and reaction of bulky polycyclic aromatic and sulfur compounds.

The thiophenic molecules could enter the large pores, but not the small pores. However, H₂ molecules can readily enter both types of pores, dissociatively adsorb on metal contained within, and be transported between pore systems by spillover. When the metal in the large pores becomes inactivated by adsorbed sulfur, spillover hydrogen could recover the poisoned metal sites by elimination of R-S-R and R-S-H. It is also of interest to classify sulfur resistance as either type I, resistance to organic sulfur compounds, or type II, resistance to inorganic H₂S (5). The metal species, particularly those in small pores, should have higher type II sulfur resistance. Figure 2 shows a simplified representation of the proposed new concept [Song 1999a, 1999b].

![Figure 2](image-url)

**Figure 2.** The proposed new concept for catalyst design based on shape-selective exclusion, hydrogen spillover, and two types of sulfur resistance. The black dots indicate metal particles on internal surface.

5. Conclusions

Heightened concerns for cleaner air and increasingly more stringent regulations on sulfur contents in transportation fuels will make desulfurization more and more important. The sulfur problem is becoming more serious in general, particularly for diesel fuels, as the regulated sulfur content is getting an order of magnitude lower, while the sulfur contents of crude oils refined in the U.S. are becoming higher.

The challenge for deep desulfurization of diesel fuels is the difficulty of removing refractory sulfur compounds, particularly 4,6-dimethyldibenzothiophene, with conventional hydrosulfurization processes. The problem is exacerbated by the inhibiting effects of polyaromatics and nitrogen compounds as well as H₂S on deep HDS.

The challenge for gasoline deep desulfurization is the selective HDS of thiophenic compounds without a significant loss of octane number. Octane loss can be avoided by preserving olefinic components in FCC naphtha, or by isomerizing paraffinic components of naphtha.

The chemistries of gasoline and diesel fuel processing have evolved significantly around the central issue of how to produce cleaner fuels in a more efficient, environmentally friendly and affordable fashion. New design approaches are necessary for making affordable ultra-clean fuels.

Acknowledgments

I am grateful to Prof. Harold H. Schobert of PSU for many helpful general discussions on fuel chemistry, and to my coworkers including Dr. Xiaoliang Ma, Mr. Uday Turaga, Mr. Gang Wang and Mr. Michael Sprague of PSU for helpful discussions on diesel fuel desulfurization. I would like to acknowledge US Department of Energy, US Air Force Office of Scientific Research, and US Department of Defense for partial financial support of various portions of our research.

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