Exclusive 4-Page R & D Issue

DCG PARTNERSHIP I, LTD.

The Premier Manufacturer of Calibration Standards for the Hydrocarbon and Biofuels Industries

Volume 3, Issue 6

November-December 2010

STANDARD ISSUE

YOU'RE INVITED TO DCG'S HOLIDAY LUNCHEON

"SEE PAGE 3"

Reporting Advances

in Catalytic nPentane Isomerizaion

Since the 1930's, the isomerization of light paraffins has been a key process for the production of high-octane-number gasoline. From the classic Friedel-Craft reaction to the modern advances in optimization of heterogeneous catalyst, the engineering of the process has developed incredibly, and the evolution has not stopped yet.

In the last couple of years, the research department of DCG Partnership, has been collaborating with the Department of Chemical and Biomolecular Engineering, of Rice University in Houston, TX, USA, in a combined effort with the Department of Materials Science & Engineering of Lehigh University in Bethlehem, PA, USA, and the Center of Research and Technology Hellas, in Thessaloniki, Greece, in order to optimize the preparation of supported metal oxide isomerization catalysts, focusing on tungstated zirconina. The idea is to control the nanostructure of the catalyst, in order to increase the activity and selectivity.

Zirconia-supported tungsten oxide (WO_x/ZrO_2) is considered an important supported metal oxide model acid catalyst, for which structure-property relationships have been studied for numerous acid-catalyzed reactions. The catalytic activity for xylene isomerization, alcohol dehydration, and aromatic akylation follows what is known as a volcano-shape dependence on tungsten surface density, this means that the activity increases up to a point and then drops.

However, WO_x/ZrO_2 has not been studied for *n*-pentane isomerization, with regard to surface density dependence. In this work, WO_x/ZrO_2 was synthesized using commercially available amorphous $ZrO_x(OH)_{4-2x}$ and model crystalline ZrO_2 as support precursors. The project aimed to investigate the structure properties of the most active catalyst in order to identify the active sites.

All catalysts were synthesized by incipient wetness impregnation of an aqueous solution of ammonium metatungstate into amorphous zirconium oxyhydroxide and model crystalline zirconium oxide support. Catalysts were prepared by adjusting the tungsten oxide loading and by selecting the desired calcination temperature. The exact morphology of each catalyst (surface nanostructure, Lewis (L) and Brønsted (B) acidity) was analyzed using scanning transmission electron microscopy (STEM), X-ray diffraction, Nitrogen physisorption and Pyridine FTIR.

The catalysts were analyzed for *n*-pentane isomerization activity and selectivity as a function of tungsten surface density, catalyst support type, and calcination temperature. These studies were performed on an isothermal downflow reactor at 523 K. The temperature along the catalytic bed was successfully controlled with minor temperature fluctuations ±1 K (Figure 1).

(cont. on page 2)

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(cont. from page 1)

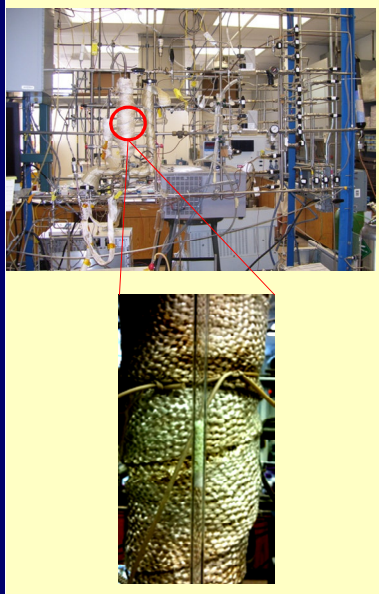


Figure 1: Downflow reactor setup used to determine nC_5 isomerization activity of WO_x/ZrO_2.

The preheating zone was kept at 523 K and the downflow line leading to the GC was kept at 343 K to prevent any condensation of heavier hydrocarbon fractions (>C6). The reaction took place at 1.04 atm, almost atmospheric conditions, with no pressure drop observed along the catalytic bed. The GC had a valve with a dual channel configuration (Figure 2), one with an alumina plot column to a FID, and the other with a packed Hayesep Q column to a TCD. The preheating zone was kept at 523 K and the downflow line leading to the GC was kept at 343 K to prevent any condensation of heavier hydrocarbon fractions (>C6). to a TCD.

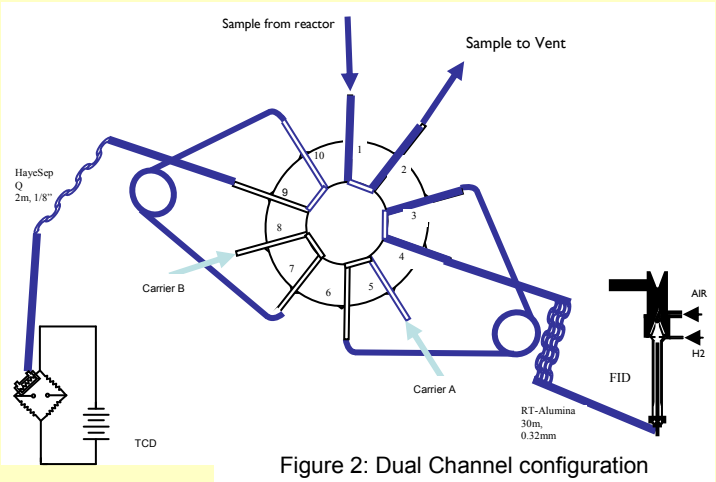


Figure 2: Dual Channel configuration

The chromatographic conditions of this configuration were designed to perform a complete analysis of each sample, considering all possible byproducts in the reactions, in a run under 30 min, to allow a sequence of multiple analysis in a continuous flow test of 10 hours.

The chromatographic conditions of this configuration were designed to perform a complete analysis of each sample, considering all possible byproducts in the reactions, in a run under 30 min, to allow a sequence of multiple analyses in a continuous flow test of 10 hours. All the reactor feeding gas blends where prepared gravimetrically with very close screening of the possible contaminations in the raw products, like the presence of olefins and the levels of moisture. The cylinders and gas flow controllers where chosen to avoid any possible metal contamination in the flowing gas, which are known to have an effect on the overall catalytic activity.

The experimental results have shown a strong dependence between pentane isomerization activity of WO_x/ZrO_2 and the nature of the support, the calcination temperature and tungsten oxide surface density. Samples prepared using the amorphous support demonstrated a volcano-shape dependence on tungsten surface density with maximum activity at 5.2 $W \cdot nm^{-2}$, just above ML coverage (completed coverage of ZrO_2 with a monoatomic layer of WO_x) and at the onset of the WO_3 crystallization, in contrast to samples prepared on crystalline support that were completely inactive.

The calcination temperature of 973 K, not 773 K, favored the formation of the active sites (which were identified as sub-nm Zr-WO_x clusters) by increasing the overall activity and selectivity, without promoting their surface *ex situ* acidic properties. The induction period which is critical for the activation of the highly selective Zr-WO_x clusters was responsible for the increased catalytic activity and selectivity. The reducible nature of the Zr-WO_x clusters resulted to the formation of very active and selective sites *in situ* that promote the bimolecular isomerization mechanism. The complete details of this work have been published in the Journal of American Chemical Society 2010, 132, 13462-13471.

By:

Nikolaos Soultanidis Ms., PhD candidate at the CHBE Department at Rice University.

Alejandro Gonzalez, Director of Research at DCG Partnership

DCG's Holiday Luncheon

Please join us for lunch with all the holiday trimmings!

We would love to see you, provide you a tour of our facilities and have you spend some time with our Chemist Consultants.

When: Tuesday, December 14, 2010

Time: 12:00 noon to 2:00 pm

Where: DCG Partnership 4170A Main, Pearland, Texas 77581

Please R.S.V.P.

(Call and/or e-mail attendance for luncheon by December 10, 2010 to Lori @ 281-648-1894 x 206 or email: lori@dcgpartnership.com)

We value working with you, and look forward to seeing you Here!

CUSTOM STANDARDS

We specialize in creating custom standards for the unique specifications of individual clients. Allow us to work with you to meet your needs. No task is too great or too small

DCG's Laboratory Proficiency Testing Program

What is a Laboratory Proficiency Test?

A proficiency test is one of the most important things you can do assess the reliability of your measurement data within your laboratory or between two or more laboratories or measurement sites. Regardless of your laboratory's quality system, a proficiency test is an excellent way to validate your measurement processes and ensure that all aspects of the laboratory measurement practices are sound. Obtaining this regular feedback, which includes analyses of the quality control data from the test sites, is critical. It will allow you to correct any problems and to prevent incorrect results from being reported. It will also take away any uncertainty of quality on the individual laboratory / test sites.

Our standards are shipped to the sites of your choice as a blind sample and the results are reported directly to the coordinator of the project. DCG can tailor the proficiency test to meet your individual needs. As always with DCG's calibration standards, our proficiency standards are Primary Reference Standards, +/-1% or less gravimetric uncertainty per component at the 99.5% confidence interval. NIST Traceable by weight with the gravimetric values verified by one or more analytical techniques - NIST Certificate #'s: 822/266926-02, 822/272801-06, 822/274081-06. Where applicable, this Reference Standard meets or exceeds the following guidelines: ISO 6142, ISO 6143, GPA 2198, API 14.1.6.2 and API 14.1.6.3.

We wish you a DCG Holiday

(Sung to the tune of "We wish you a Merry Christmas")

We wish you a DCG Holiday, We wish you a DCG Holiday,
We wish you a DCG Holiday, and a Happy New Year…
Glad tidings we bring, from DCG to you,
Glad tidings for the Holidays,
And a Happy New Year!,

As we fill your standard orders
As we fill your standard orders
As we fill your standard orders,
We inspect twice for perfection!
Glad tidings we bring from DCG to you,
Glad tidings for the Holidays, And a Happy New Year

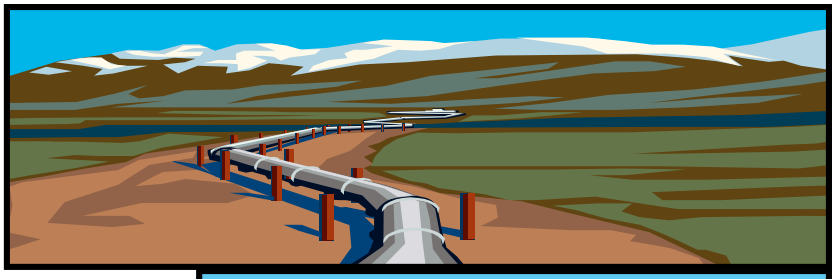
We'll ship our Ultra Protection Heat Blankets,
We'll ship our Ultra Protection Heat Blankets,
We'll ship our Ultra Protection Heat Blankets,
Year-round and with just-in-time delivery!
Glad tidings we bring from DCG to you,
Glad tidings for the Holidays, And a Happy New Year!

Employee Spotlight:

Alejandro Gonzalez

The Research and Development Director for DCG, Alejandro Gonzalez, attended the Central University of Venezuela. It was there his career path began. His thesis on developing a method for taking and preserving samples for the analysis of Sulfur species in Natural Gas replaced some traditional GPA (Gas Processors Association) methods, and it is still used today with the name SULFTRAP (Patent No. US 5109713. Dec., 1993). After he graduated, Alejandro went to work for PDVSA Intevep taking samples and analyzing Sulfur and Hydrocarbon content for the characterization of natural Gas in new oil and gas wells. His work again helped his company convert from GPA2261 Backflush to GPA2286 Natural Gas Extended Analysis. While working on a special project, Alejandro and two colleagues developed a method to quantify the amount of Hydrogen Sulfide present in asphalt, transformer oil, Orimulsion, lubricants, residual fuel oil and other viscous petroleum substances.

Alejandro installed this method in the Shell Westhollow research facility in Houston, TX in 1995 for testing by experts for ASTM (American Society for Testing and Materials). It became a standard method for the industry in 1996 (ASTM D6021-96). As part of the same work, Alejandro designed an apparatus and an instrument for taking, preserving and processing this kind of sample, which resulted in two more US patents: US Patent No. 5390551 (Feb,21, 1995) and US Patent No. 5379654 (Jan. 10, 1995). Alejandro accepted a job offer from DCG Partnership in 2003 and moved his family to Texas. For nearly eight years Alejandro has headed Research and Development of new products. He has worked to expand the DCG product line to include a standard for total acid number in lubricant oil, liquid fuels and biofuels, standards for Paraffins and Aromatics for DHA and SimDis, a standard for Sulfur compounds for Sulfur SimDis and a standard for Glycerine and Glycerides in Biodiesel. He is an active member in three areas of the American Chemical Society Analytical, Fuel and Petroleum Divisions. Alejandro also sits on the D02, D16 and E48 committees for ASTM International , and is the Chairman for the D02.D.05 subcommittee. His most recent new developments in analytical methods include a purity of Benzene (for ASTM International D16 committee) and Methanol in Biodiesel. He is working in Research and Development with Rice University and Lehigh University involving advances in catalysis (featured in this newsletter). With the new Research and Development Lab at DCG, Alejandro and DCG plan to work with more research institutions and colleges to take research to a new level.

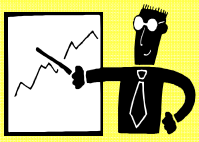


DCG: Meet and Greet & School Schedule

November:

Nov. 8th-12th Understanding Chromatography, DCG office

Nov. 15th-19th Method Development, DCG office



December:

Dec. 6th-10th, ASTM, Jacksonville, FL

Dec. 13th–17th, Understanding Chromatography—DCG office

December 14th—DCG HOLIDAY PARTY– DCG OFFICE

DCG HEADS FOR ALASKA!

Congratulations to one of our own.

At the recent Gulf Coast Conference in Galveston, TX; Roy Rodriguez (Client Liaison for DCG Partnership) was selected as the new Chair for the Rocky Mountain Exchange Group. Watch for DCG's next newsletter which will have more information on this Interlaboratory Crosscheck Program and on the meeting to take place in May 2011 in Kenai, Alaska.