



New Haven Section American Chemical Society Newsletter

Volume 29, Number 1, Spring 2012 New Haven Section Chartered in 1912

Chair 2012 Jefferson Chin jrchin5@comcast.net

Chair-Elect 2012 Ken White charles.ken.white@gmail.com

Immediate Past Chair 2012 Dr. Dennis Jakiela dennis.jakiela@hotmail.com

Treasurer 2011-2012 Dr. Max Reeve maxreeve@comcast.net

Secretary 2012-2013 Dr. Walter Krol walter.krol@po.state.ct.us

Councilor 2010-2012 Dr. Olivier Nicaise olivier.nicaise@trincoll.edu

Councilor 2012-2014 Dr. Jerry Putterman jerry putterman@hotmail.com

Alternate Councilor 2012-2014 Kent Marshall Kent.Marshall@quinnipiac.edu

Alternate Councilor 2012-2013 Maria Parr <u>Maria.Parr@trincoll.edu</u>

Chemistry Olympiad Dr. Olivier Nicaise olivier.nicaise@trincoll.edu

Bulletin Editor Jefferson Chin jrchin5@comcast.net

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Website Revamped

As we transition away from our paper mailings, everyone can look for the updates in our emails or at our revamped website, <u>New Haven ACS</u> local section (http://www.newhaven.sites.acs.org/).

Message from the Chair

Jefferson Chin

Welcome to another edition of the New Haven section ACS newsletter. It has been a while since we last published and there have been a lot of things that have happened in the past year as well as things that are currently on going. Because of that, this newsletter is a blend, some articles recognizing accomplishments from last year and letting you know of things to look forward to. It has been exciting to see that at our annual elections last year, we were actually able to hold a real election for the two Alternative Councilor positions! For this race, there were four candidates, ready to volunteer and be a part of the local section. It was interesting to observe the votes roll in and contemplate the potential outcomes. In the end there can be only two Alternate Councilors and for this election it was Professors Maria Parr and Kent Marshall. The Executive Board welcomes the new Alternate Councilors.

The Executive Board does not want to forget about John Bayusik. John has been an Alternate Councilor since 2009 and we are grateful for his service. It has been enjoyable to work with him on a volunteer basis and get to know him over the past 5 years. *Thank You!* With that said, we are not saying good-bye, as we never truly let past board members disappear but only convince them that they first need to continue to attend the general meetings and second consider volunteering for another part of the local section activities.

Over the next 2 years the local section will be very active; this year we will be celebrating our 100th anniversary and next year we will be hosting the ACS's Northeast Regional Meeting (NERM) in downtown New Haven. You may be asking yourself – why aren't we hosting the NERM this to celebrate our 100th anniversary? The simple answer is because the Rochester local section is also celebrating their 100th anniversary this year and they put their NERM bid in 4 years ago! We are in the midst of planning both events and details will be forthcoming.

Please enjoy the newsletter!

NERM 2013 update

Although the New Haven section will be hosting the Northeast Regional meeting in 2013, planning for the meeting is in progress. The meeting will be October 23rd through 26th, 2013 at the Omni Hotel in downtown New Haven. We are anticipating that our NERM meeting will coincide with National Chemistry week so there should be lots of activities for all.

New Haven Science Fair

We will again be offering up two *Excellence in Chemistry* awards (\$100 for Grades K-8; \$100 for Grades 9-12) at this year's science fair.

The Science Fair will take place May 15 & 16, 2012 at University Commons, Yale University.

Tuesday, May 15, 2012, 5:00 to 8:00 pm: Judging **Wednesday, May 16, 2012** - 9am-1:00pm: Final Judging. 1-6pm: Public Viewing.

Thursday, May 17, 2012 - 9am-9pm: Public viewing and Awards

If you would like to judge and represent the New Haven Section, please contact Jeff Chin jrchin5@comcast.net.

If you would like to be a general judge, please contact Michelle Cirello, Project Manager, New Haven Science Fair Program, 203-782-4341. www.nhsciencefair.org

<u>A Salute to Longevity:</u> <u>Recognition of 50- & 60- Year Members</u>

The Officers of the Section are pleased to announce that the following Section members will reach the milestone of 50 or 60 years of membership in the American Chemical Society in 2012.

50- Year Honorees

Dr. John W. Faller Dr. Juan Haydu Dr. Aspet V. Merijanian Dr. John L. Morico Mr. Charles Paddock Mr. Donald Turecek Mr. Michael J. Uricheck

60- Year Honorees

Mr. Richard Alan Hagstrom Dr. Anthony N. Naglieri Dr. Owen York Jr. It is truly a pleasure to recognize these individuals and to congratulate them on their long record of ACS Membership, which has contributed to the success of the Society and the Local Section. These members have been invited to be the guests of the Section at a meeting of the General Membership, so that we can honor them more appropriately.

The Deepwater Horizon Oil Spill: Development and Implementation of Testing Methodology for the Reopening of Waters to Fishing.

Dr. Walter J. Krol

The Connecticut Agricultural Experiment Station, 123 Huntington Street, New Haven, CT 06504

Background

In 2005, The Department of Analytical Chemistry (DAC) at The Connecticut Agricultural Experiment Station (CAES) in New Haven was selected through a competitive grant process to become one of the original eight laboratories in the chemistry Cooperative Agreement Program (cCAP) in the Food Emergency Response Network (FERN). In 2010, the CAES was awarded five additional years of grant funding. There are currently fourteen cCAP FERN laboratories in the United States (US). The governing principle behind FERN is that cCAP network laboratories should have identical equipment and methodology to perform analysis in the event of a chemical threat to the food supply; thankfully, to date, there have been no such attacks on the nation's food supply. In return for the grant funding received, the DAC at the CAES 1) performs instrument and method evaluations, 2) participates in proficiency testing programs, 3) participates in surveillance exercises, 4) is partially responsible for Food and Drug Administration (FDA) method extension, development and improvement, 5) has hosted FDA technical meetings and training, and 6) has been activated by the FDA over concerns of melamine in food imported from China.

On April 20, 2010 at 10:45 am EST, an explosion occurred on the Deepwater Horizon oil rig in the Gulf of Mexico in which eleven

workers were killed. The rig burned for two days before sinking. Concomitant with the sinking, the first oil slick was sighted. Late on the night of April 29th, an oil spill estimated to cover 3850 square miles (sq. mi.), made its first landfall in Louisiana. On May 2nd, the NOAA (National Oceanic and Atmospheric Administration) closed 6814 sq. mi. of waters to fishing. At the height of the spill on June 21, 2010, about 88,000 sq. mi. of State and Federal Waters, almost 40% of the entire Gulf of Mexico, was closed to fishing.

Efforts to stop the leak were complicated by numerous factors, including the fact that the well-head was over 5000 feet below the surface. By July 30, 2010 over 1.8 million gallons of dispersant (mostly Corexit® 9500) had been used at the surface and 1.1 million gallons used at the well-head. Drs. Christina Robb and Brian Eitzer in the DAC at the CAES assisted the FDA in developing methodology for the analysis of the primary ingredient of Corexit®, 2-butoxyethanol. There continues to be a debate over the toxicity of these chemicals; however it is known 1) that their toxicity is less than most of the constituents in the oil and 2) dispersants have never been used at this magnitude in the past.

The well-head was capped on July 15th, and permanently sealed on September 19, 2010. Approximately 205,800,000 gallons (4.9 million barrels) of crude were released, making it the 5th largest oil spill on record. As of October 2010, it is estimated that 17% of the oil was recovered directly from the well; 5% was burned at the surface; 3% was skimmed from the surface; 8% was chemically dispersed; 16% was naturally dispersed and 25% evaporated or dissolved. The remaining 26% of the oil was not accounted for. Twenty six percent is 54 million gallons; twice the amount spilled by the Exxon Valdez.

Re-Opening Protocols

Generally, states have jurisdiction over ocean waters out to about three nautical miles (it is nine nautical miles for some states). States must work with the FDA to ensure seafood from these waters is safe. Ocean waters beyond state control are under federal control through the NOAA. By mid-May, a reopening protocol for the waters closed to fishing had been established employing 'The NOAA Method' of analysis. This method was developed to test seafood after the 1989 Valdez spill in Alaska. It is a rigorous and definitive Gas Chromatographic Mass Spectral (GC/MS) analysis for Poly Aromatic Hydrocarbons (PAHs) and oil related contaminants in seafood. The NOAA method is described in a sixty-four page document. It involves accelerated solvent extraction (ACE) with dichloromethane, followed by clean-up by 1) silica/alumina columns and 2) size exclusion HPLC (high performance liquid chromatography). The final analysis is performed by GC/MS. The results obtained from analyses performed by the NOAA method following the Valdez oil spill survived scrutiny in a court of law.

The original protocol for reopening waters for fishing was envisioned as a tiered system. It included the analysis of samples of shrimp, oysters, crab and over a dozen species of finfish. Affected states could only collect samples from waters that were visibly free of oil. All samples collected were delivered to the NOAA laboratory in Pascagoula Mississippi for organoleptic (sensory) analysis. There were three levels of organoleptic analysis conducted by two expert 8-member teams. It was estimated that members of these teams could detect oil taint in seafood at approximately five parts per million (ppm). The sensory/olfactory analysis initially occurred on raw seafood samples, then on cooked samples, and, finally, the samples were tasted. If five of the eight team members passed the sample, it was then submitted for chemical analysis employing the NOAA method.

Roadblock to Testing

On May 27, 2010, during a national teleconference phone call involving all of NOAA, FDA, representatives of the impacted states, and personnel in the cCAP FERN state labs, it was established that 1) only three labs had the necessary equipment on hand to perform the required analysis of seafood, 2) the cost to purchase the required equipment was estimated to be approximately \$100,000 per lab, 3) it would take a minimum of two to three months to receive, install and validate the equipment and the method and 4) that the NOAA method has a very low throughput with about seventeen samples per week per lab. The NOAA administrator predicted up to 90,000 samples may have been needed to reopen all closed waters. Additionally, added pressure was coming from the Governors of some Gulf States who began holding daily press conferences accusing NOAA and FDA of dragging their feet on seafood testing. Given all of these pressures, having a back-log of untested seafood due to NOAA method shortcomings was clearly not an option.

The Role of Seafood Testing in the DAC at the CAES

In early June, 2010, a decision was made that the FDA forensic chemistry center (FCC) in Cincinnati, Ohio should take the technical lead in the development of new chemical screen. The FDA FCC was to utilize the FERN cCAP laboratories to develop a screen that could be implemented after samples pass the organoleptic analysis but prior to analysis by the NOAA method. It was hoped that this intermediate screen could rule out false negative samples. Samples that failed the screen would then be subjected to further analysis by the NOAA method.

On June 9th, the DAC at The CAES and the Minnesota Department of Agriculture received messages from the FDA FCC requesting an immediate teleconference call. On that call, it was decided the three labs would codevelop and validate a QuEChERS-based (Quick, Easy, Cheap, Effective, Rugged, Safe) extraction method for seafood followed by an HPLC with FLD (Fluorescence Detection) screening procedure to estimate fifteen PAHs of concern and total oil contamination. The advantages of this approach were that 1) materials required for the QuEChERS extraction were readily available in most labs, 2) the equipment required for the analysis was widely available, and 3) the method would have a throughput of 100 samples per week per lab.

By July 19th, the FCC lab in Ohio and its Connecticut and Minnesota partners had 1) finalized the extraction protocol to be employed and 2) developed suitable HPLC-FLD conditions to resolve the fifteen PAHs in question. On that same day, four days after the well head had initially been capped; method validation at the three labs began. The fifteen PAHs were spiked into four separate matrices (crab, finfish, oyster and shrimp) at three separate levels (0.025, 1 and 10 ppm). The percent recoveries, method detection level (MDL), limit of quantitation (LOQ), and other quality assurance and quality control parameters were separately calculated for each of the fifteen analytes in each matrix.

All validation work in the three labs was completed on Wednesday July 21, 2010. The FDA FCC was to compile the data into a working method and present it to the FDA commissioner on Monday, July 26th. On Thursday July 22nd, the meeting was changed to Saturday July 24th. Members of the Connecticut and Minnesota laboratories flew to Ohio for the meeting which started at 9:00 am CST. At the close of that meeting, the Connecticut and Minnesota labs were told to expect up to twenty samples per day, six days a week for up to six months.

On Monday July 26th, the interlaboratory validated method (1) was posted on the FDA website. On Wednesday, July 28th, CAES received its first shipment of seafood from the Gulf of Mexico waters for testing. We continued to receive samples until October 28, 2010. The anticipated numbers of samples, however, never materialized. The total number of seafood samples tested nationwide in response to the Deepwater Horizon incident was 2,400.

CAES Seafood Testing Program

Samples were shipped to the CAES following organoleptic/olfactory testing in Pascagoula Mississippi, usually with twentyfour hours' advanced notice. The results of these analyses were reported to the FDA via a secure website within twenty-four to thirty-six hours of the receipt of the samples.

During the first month of performing seafood analysis at the CAES, FDA CFSAN (Center for Food Safety and Applied Nutrition), FDA DFS (Division of Field Science) and the Whitehouse were reviewing the data. Reopening decisions were made by the Administration within two to three hours of receiving the data. The data provided to the FDA did not just consist of part per million values for each of the fifteen PAHs. It consisted of full data packages of forty to sixty pages including the chromatograms and the excel spreadsheets showing all calculations from which the final numbers were derived. Following each re-opening decision, the results of the analysis and the decision itself was posted on the FDA website.

Seafood Testing Wrap Up and Results

By mid-November of 2010, Connecticut, Minnesota and FCC were still the only labs running the new screening method. Eighteen laboratories, both FDA and cCAP, were in various stages of ramping up to run the validated method. A total of eight laboratories, four cCAP and four FDA, were testing samples from Federal waters by the NOAA method. Of the 2400 seafood samples tested by this response, none failed the screening analysis. Failure was defined as either 1) one of the 15 PAHs was present at fifty percent of its LOC (level of concern) OR 2) if the estimated PAH content in an HPLC trace between 2.5 and 20 minutes was fifty percent of the FDA LOC for naphthalene in that matrix (16 - 62 mg/kg). Samples that did not pass the screening method would have been sent to a laboratory capable of running the NOAA method for re-analysis.

There was tremendous interest in the state regarding the CAES role in the oil spill response. The CAES issued two press releases and staff performed fourteen interviews for local radio, television and the newspapers in a 10-day timeframe.

Status as of November 2011

Effective November 30, 2010, the FERN cCAP labs were deactivated for this exercise. The new HPLC-FLD screening method had been accepted in the *JAOAC International* and was published in early 2011 (2). A letter of appreciation was sent from the FDA commissioner to the Connecticut Governor in January of 2011.

By November 30, 2010, ninety-seven percent of state fisheries had been reopened, with a small section of the Louisiana fisheries closed until the spring of 2011. Of the 88,522 sq. mi. originally closed, roughly 1,100 sq. mi. remained closed as of November 24, 2010. On November 24, 2010an additional 4,200 sq. mi. of Federal waters were closed to fishing after a shrimper recovered tar-balls with his catch of royal red shrimp (depth of 600 feet).

At that same time, British Petroleum (BP) had pledged \$450 million in competitive oil spill research funding. The request for proposals (RFP) for consortia was released on April 25th, 2011. Funding of \$37.5 million per year was slated to establish a total of four to eight research consortia. The Environmental Protection Agency (EPA) has released a RFP for grant funding worth \$2.5 million, with a focus on funding Gulf Coast states. By mid-April of 2011, the remaining closed state and federal waters were reopened with the exception of a 10 sq. mi. zone around the well-head. The FDA had also established a two-year surveillance program for Gulf seafood. The sampling will occur at processing plants, and the FDA labs will perform the analyses. FERN cCAP labs were told to be available for surge capacity in the event that the sample load becomes too great for FDA to process efficiently. The Gulf Coast states have also instituted surveillance programs, with some of them receiving funding directly from BP.

Although the FERN was initially established to act in rapid response to chemical terrorism against the nation's food supply, it has gained considerable attention at the Congressional level for its flexibility in dealing with other national food crises. The FERN has repeatedly demonstrated its capabilities in its response to melamine in pet food in 2007 and in the Chinese melamine outbreak in 2008. Moreover FERN and its partners developed and validated new analytical methods to analyze seafood from the Gulf of in response to the Deepwater Horizon Oil spill. All actions were performed in very short timeframes. Clearly, the FERN is an invaluable resource to all the citizens of the US.

 "Screen for the Presence of Polycyclic Aromatic Hydrocarbons in Select Seafoods Using LC-Fluorescence", Gratz, S., Mohrhaus, A., Gamble, B., Gracie, J., Jackson, D., Roetting, J., Ciolino, L., McCauley, H., Schneider, G., Crockett, D., Krol, W., Arsenault, T., White, J., Flottmeyer, M., Johnson, Y., Heitkemper, D., Fricke, F. Laboratory Information Bulletin (LIB) FDA/ORA/DFS No. 4475, **2010**. Available at: <u>http://www.fda.gov/downloads/ScienceResearch/UC</u> M220209.pdf (**2010**).

(2) "Screening and Determination of Polycyclic Aromatic Hydrocarbons in Seafoods Using QuEChERS Based Extraction and High Performance Liquid Chromatography with Fluorescence Detection", Gratz, S., Ciolino, L.A., Mohrhaus, A.S., Gamble, B.M., Gracie, J.M., Jackson, D.S., Roetting II, J.P, McCauley, H.A., Schneider, G.T., Crockett, D.F., Heitkemper, D.T., Fricke, F.L., Krol, W.J., Arsenault, T.L., White, J.C., Flottmeyer, M.M., Johnson Y.S. J AOAC Int., 2011, 94, (5) 1601 – 1616.

2011 New Haven Science Fair Program David Smudin

The 17th Annual New Haven Science Fair was held at Commons Hall, Yale University on May 10 to 12, 2011. This annual program is committed to making learning both meaningful and enjoyable by using "hands-on" science as a vehicle for developing and answering questions that a curious person might have about the world around him or her. This program functions as a partnership involving businesses, universities, associations, foundations, and private citizens, working with the public schools' teachers, students and administrators. This year there were 269 projects entered in the Science Fair by students in grades PreK through 12, from schools in New Haven.

The New Haven Section is proud to participate in this program. Several years ago we made a commitment to support it both financially and professionally. In addition to providing monetary awards for *Excellence in a Chemistry Related Science Fair Project* in the pre-K to 8, and 9 to 12 grade categories, the Section also provides judges for this event. Judges from the Section for the 2011 Science Fair were Prof. Maria Parr, Trinity College, and David Smudin. The award for *Excellence in a Chemistry* Related Science Fair Project from grades Pre-K through grade 8, was given to a team of 23 students from Larissa Giordano's 4th grade class at the Nathan Hale School in New Haven. The title of the project was, The Solution to Pollution. These students spent a large part of the school year studying pollution. They learned what constitutes pollution, where it can be found, how it is recognized, and how it can be prevented. They learned that certain kinds of pollution can be harmful to our health, but materials called antioxidants can help protect us from the effects of pollution. They then set out to learn something about antioxidants; what they are, how they behave, and where they can be found. They selected a group of compounds with antioxidant properties and evaluated them with respect to how they would protect potato and apple slices from air oxidation. They hypothesized that Vitamin C would be the best antioxidant of the group, and the study that they chose to do showed this to be true. Afterward they evaluated different citrus fruits and juices to determine which had the highest Vitamin C content.

Because of the time devoted to their project, the extent of information that they learned, the amount of data that was collected, the way it was treated, the excellent manner in which the results were presented, and the enthusiasm of the four students selected to present the project at the Science Fair, the judges felt that this project was certainly worthy of selection as the *Best Chemistry Related Science Fair Project* in the Pre-K to 8th grade category.

The award for *Excellence in a Chemistry Related Science Fair Project* submitted by students in grades 8 through 12, went to Lea Winter and Lily Engbith, Wilbur Cross High School, for the project entitled, "*Water Remediation via Double Precipitate Reactions.*" The idea for the project grew out of reports that hydraulic fracturing, the process of forcing water containing salts or other additives into rock formations deep below the earth's surface, to aid in the release and recovery of oil and natural gas contained therein, was resulting in contamination of ground water and potable water wells.

The students explored a way to remediate water contaminated with inorganic salts. They hypothesized that combinations of water soluble salts could be selected, which would combine with water soluble ions in a fracturing solution, resulting ideally in a double precipitate of insoluble salts. Upon removal of the insoluble salts, the aqueous component was determined to be significantly reduced in salt content than it was initially. In planning experiments to test their hypothesis, the students gained an understanding of the periodic table of the elements, the solubility of salts, and methods to determine the purity of water. The idea was unique, a significant amount of data was collected and analyzed, and the project was well presented at the science fair.

In carrying out the project, Lea and Lily received support and direction from Chris Willems and Bernard Hulin, chemistry teachers at Wilbur Cross High School.

2011 Excellence in Chemistry Science Fair Winners



Adeline Ozyck, Nyla Toms, Kayla Dayton, and Michael Tramontano, students in Larissa Giordano's 4th Grade class at Nathan Hale School, receive certificates from Dr. David Smudin for their Science Fair Project entitled, "The Solution to Pollution."



Lea Winter and Lily Engbith, Wilbur Cross High School, receive certificates for their Science Fair Project entitled: "Water Remediation via Double Precipitate Reactions."

An invitation to participate in a Survey from Indiana University of Pennsylvania, Department of Chemistry

You are invited to participate in this research study and at the end of the survey have a chance to win an Apple IPad (Generation 3). You are eligible to participate as you have been identified as a chemist working in industry. Participation in this study will require approximately 15 minutes of your time.

The purpose of this study is to develop the undergraduate organic curriculum to better prepare our undergraduate students for an entry level industrial positions through a more integrated laboratory approach. The goals of this survey are to 1) identify the most commonly used types of chemical techniques and chemical reactions used in industry today and 2) to identify general aspects of the chemical industry, from initial product identification through manufacturing, that students should be aware of prior to entering the job market.

The results of this survey will ensure that future developments in the organic chemistry curriculum satisfy current needs of industry, while at the same time, offer exposure to the core concepts that are fundamental to the chemical industrial processes.

If you are willing to participate in this study, please select "I agree and give my consent for this survey" to continue onto the survey. Names provided for entrance into the Apple IPod drawing will only be used to contact the winner. No names will be used for research purposes.

Survey Link: https://iup.qualtrics.com/SE/?SID=SV_afydYckvntvvWXG

Thank you for your time,

Project Directors:

Dr. Justin D. Fair Dr. Carl LeBlond Assistant Professor Associate Professor Indiana University of Pennsylvania Department of Chemistry 975 Oakland Ave Rm. 143 Weyandt Hall Indiana, PA 15705

A Quarter Century Ago

(Where you there?)



Chairperson Lidia Doweyko presided over the October1986 meeting of the Local Section, which was held at Jimmies of Savin Rock. The featured speaker at that meeting was Dr. Alfred Bader of Sigma-Aldrich, who spoke on the topic of *Chemistry in Art*.