technical articles & abstracts

Analysis of Low Levels of PolyVinylPyrrolidone in PolySulfone by FTIR and Pyrolysis GC/MS

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Dr. Wayne Way will present this paper at ANTEC 2002, the Annual Technical Conference & Exhibition of the Society of Plastics Engineers May 5-9, San Francisco, CA. The abstract is reprinted below.

FTIR and pyrolysis GC/MS were used to investigate the detection of polyvinylpyrrolidone (PVP) in polysulfone (PSO) at low levels. By monitoring the absorption stretch of PVP at 1680 cm-1 and PSO at 1586 cm-1, it was possible to detect and quantitate PVP in PSO to a level of 0.5%. This method allows for rapid monitoring of the two blended polymers. Furthermore, pyrolysis GC/MS was used to investigate low levels of PVP in PSO. Pyrolysis at 700C proved advantageous in allowing for detection of PVP by monitoring the major product, 2-pyrrolidinone.

DEFORMULATION: The Art and Science of Separation, Identification and Quantification of Ingredients in a Complex Formulation.

Over the years, Chemir/Polytech Laboratories, Inc. has developed expertise in the process of deformulating or reverse engineering a product. Our experienced chemists, including several Ph.D. chemists, have performed many deformulations and have the problem-solving skills and the tools to successfully deformulate almost any type of product, simple or complex.

This analytical service uses state-of-the-art spectroscopic, thermal, chromatographic and microscopic techniques and conventional extraction methods to identify and semi-quantify the components of a complex mixture. The components may include polymers, plasticizers, fillers and other minor components such as stabilizers, lubricants, antioxidants, flame retardants, etc. Although we do not guarantee a "cookbook recipe", our analysis, in most instances, identifies the chemical class of major and minor components. A computer library search is also performed when needed to locate the trade names of similar, commercially-available materials.

Types of Products Deformulated
Chemir/Polytech has deformulated a wide variety of products which include: plastics and rubber from the automotive, medical device and injection molding industries; consumer and industrial detergents and cleaners; textiles used in garments and upholstery applications; pharmaceutical products; inks for laser printers, finger printing and packaging; cosmetics and toiletries like shampoos, face creams and sunscreen lotions. Our customers also use the deformulation service for different reasons. It is used to identify batch-to-batch raw material variations, ingredients in a competitor's product, product contamination, comparative analysis of a "good vs. bad" product or for products involved in patent infringement litigation.

Preliminary Information
The deformulation process can be quite complex depending on the product or material being analyzed. It benefits the customer to provide us with as much information as possible about the sample to be deformulated. This includes Material Safety Data Sheets, technical data sheets, a description of how the product is used and any theories as to what the ingredients might be. This is valuable information that the chemist will use to customize the method of analysis.

Initial Studies
When a sample is received at Chemir/Polytech Laboratories, any product information from the customer is reviewed, and an initial assessment of the material's bulk properties is made. This evaluation may include color, form, appearance, odor and other properties. In the hands of an experienced chemist, this information may give some valuable clues which will lead to a more productive analysis.

Chromatography & Solvent Extractions
Sample preparation is the key to the success of any analysis. Chromatography and solvent extractions are used as an effective tool for preparing materials for deformulation. Chromatography, including Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC), serves to separate complex materials into their components. Solvent extraction methods generally utilize larger samples in an effort to separate components for identification. These techniques give specific information as to the identity or chemical class of a product or material when utilized in conjunction with spectroscopic techniques.

Spectroscopy

Spectroscopic techniques are used to provide specific information that helps identify the chemical class of the various components. Chemir/Polytech Laboratories' chemists typically use one or more of the following during a deformulation analysis:

Fourier Transform Infrared Spectroscopy (FT-IR) - The material is analyzed with infrared light which is absorbed as specific parts of the molecules are caused to bend and stretch. Different parts of a molecule absorb at specific frequencies and the spectrum gives information about what functional groups are present in the material. This can also be used as a fingerprint of the material.

Mass Spectroscopy - The chemical component is bombarded with electrons producing a charged material which is then subjected to an electric field. It is possible to separate and count the charged particles according to their molecular weight which helps to positively identify the components.

Nuclear Magnetic Resonance Spectroscopy (NMR) - This technique can be used to determine the structure of many organic compounds. It gives information regarding how the atoms in a molecule are connected and how they are positioned relative to other functional groups.

Thermogravimetric Analysis & Differential Scanning Calorimetry

Thermogravimetric Analysis (TGA) is used to analyze a material with respect to changes due to heating of the sample. Common information that is gained from TGA includes percentage of moisture or fragrance, percentage of organic compounds and the amount of inorganic material in a sample. Differential Scanning Calorimetry (DSC) gives information about transformations which materials undergo as they are heated. The technique detects minute changes in the properties and can offer valuable information regarding material identities, and melting and softening properties.

Conclusion

Chemir/Polytech Laboratories' deformulation service combines the problem-solving skills of our experienced chemists, many of whom hold a Ph.D. in their area of expertise, and powerful analytical techniques to make possible the determination of the composition of complex formulations. We have the experience and the capabilities to work with you and improve your business with valuable information about your product or your competitor's product.

THE USE OF A COLUMN KIT FOR RAPID HPLC METHOD DEVELOPMENT AND OPTIMIZATION

Dr. Wayne Way, Group Leader - Separations Science Section, presented a paper titled The Use of a Column Kit for Rapid HPLC Method Development and Optimization at HPLC ’98. This conference was held in May 1998, in St. Louis, MO. The abstract is reprinted below, and the full article is available upon request.

High Performance Liquid Chromatography (HPLC) coupled with a column kit has been employed in the rapid method development and optimization of hindered phenol antioxidants. Hindered phenol antioxidants are widely used in a variety of products ranging from polymers to foods. The combinations of these materials and others in additive packages can change frequently, thus analysis methods must be continually optimized. While structurally similar, the hindered phenols show a range of retention characteristics, making the development of a single isocratic or simple gradient method by traditional means a time-consuming, trial-and-error effort.

The column kit includes a series of columns with the same ligand group (e.g., C8, C18 or phenyl) bonded to the support with varying hydrophobicity. The unique advantage of the new technology behind this column kit is that it can reliably produce an infinite number of selectivities. The utility of the column kit is that it offers rapid and linear progress towards determining an optimized method for a given set of analytes.

CHARACTERIZATION OF AN UNKNOWN POLYOLEFIN

Dr. Fred Willard, Group Leader - Materials Science Section, presented a paper titled Characterization of an Unknown Polyolefin at a Polyolefins and Elastomers Conference in Houston, TX, in February 1998. The abstract is reprinted below, and the full article is available upon request.

There are many occasions that might warrant the analysis of an unknown polyolefin. It may be that a simple material identification is required in order to classify the material for recycling purposes. Perhaps there is a quality issue regarding one lot of materials so a comparative analysis of a known good lot and a suspect lot might be needed. The material may be a competitive product so a more detailed analysis might be required.

This paper explores the techniques that can give information regarding an unknown polyolefin sample. The techniques include Fourier Transform Infrared Spectrometry (FT-IR), Differential Scanning Calorimetry (DSC), Nuclear Magnetic Resonance Spectrometry (NMR), Thermogravimetric Analysis (TGA) and Gas Chromatography with a Mass Spectrometer detector (GC/MS). Each technique is used to provide a specific piece of information that helps to characterize a polyolefin.

QUALITY PHILOSOPHY

Our mission is to provide our clients with customized problem solving analysis in a timely and accurate manner...
Simply put, our goal at Chemir/Polytech Laboratories' is to provide a quality service that totally satisfies the customer. One way that we achieve quality service is by focusing on a Quality Assurance/Quality Control program. This program ensures the integrity and reliability of our laboratory practices which ensures accurate analyses for our customers. Secondly, we track customer satisfaction. Surveying customers gives us feedback on areas that need improvement.

The details of our QA/QC program are documented in a Quality Assurance/Quality Control Manual covering everything from sample log-in and chain of custody procedures to guidelines for laboratory notebooks. All employees are required to follow the policies in the manual. Furthermore, our QA/QC Director, reporting directly to the Technical Director, oversees company compliance as well as training.

In addition, more than fifty detailed standard operating and calibration procedures have been developed for the instruments in our laboratory. Instruments are calibrated and routine maintenance is performed at regular intervals.

Chemir/Polytech Laboratories is FDA registered and inspected and meets federal regulations and guidelines required for analyses involving pharmaceuticals and pharmaceutical raw material testing. Our QA/QC policies also meet current cGMP/ GLP regulations. If your analysis does require cGMP/ GLP compliance, please notify us in writing at the time of sample submission.

We encourage visits to Chemir/Polytech Laboratories by our customers. You can examine our laboratory practices and review our comprehensive Quality Assurance/Quality program. For additional information about our QA/QC policies or to arrange a visit, please contact us.