The WCAS Analytical Digest

WEST COAST ANALYTICAL SERVICE INC

THE QUARTERLY NEWSLETTER ON PROFESSIONAL ANALYTICAL CHEMISTRY

Aristolochic Acid by LC-MS/MS

Contents

Calendar

Nov 7-11, 2004
American Association of Pharmaceutical Scientists (AAPS) Show
Baltimore, Maryland

Quick Quotes

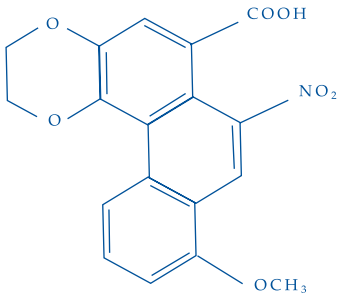
"The world cares very little about what a man or woman knows; it is what the man or woman is able to do that counts."

- Booker T. Washington

"He who comes up to his own idea of greatness must always have had a very low standard of it in his mind."

- William Hazlitt

Aristolochic acid (AA) is a known nephrotoxin (kidney) and potential carcinogen. It occurs in several different plant species, especially Aristolochia, which are sometimes found in traditional Chinese medicines. Due to the similarity of Chinese names for different herbs, it is possible for Aristolochia to be present in traditional medicines or in botanical-containing supplements.



Aristolochic Acid I

FDA requires some imported products to be tested before they will be released from customs and has warned consumers to immediately discontinue use of any product containing AA. FDA warns that products containing "Aristolochia," "Bragantia" or "Asarum" should be avoided. FDA has developed a method for determination of aristolochic acid in botanical products. Samples are powdered, then extracted using 80:18:2 methanol:water:formic acid. This extract is filtered, then analyzed for the primary isomer, aristolochic acid I, using HPLC with UV detection. Extracts are then analyzed by LC-MS/MS for confirmation of both positive and negative results. Since LC-MS/MS is used in all cases, WCAS uses this technique exclusively for measurement of aristolochic acid. In the MS/MS analysis, the ammoniated molecular ion is selected in the first quadrupole, and three different product ions are monitored. All three must be present to generate a positive result. This essentially eliminates false positive results. We have applied this technique to several products with good results. Detection limits of approximately 0.5 µg/g (ppm) can be achieved. For more information or for a quote, please call Eric Lindsay at 562.948.2225 x300. ■

Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) is a powerful tool for identifying types of chemical bonds in a molecule by producing an infrared absorption spectrum that is like a molecular "fingerprint". FTIR is most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantitate some components of an unknown mixture and it can be applied to the analysis of solids, liquids, and gasses. The term Fourier Transform Infrared Spectroscopy refers to a fairly recent development in the manner in which the data is collected and converted from an interference pattern to a spectrum. Today's FTIR instruments are computerized which makes them faster and much more sensitive than the older dispersive instruments.

Qualitative Analysis

FTIR can be used to identify chemicals from spills, paints, polymers, coatings, drugs, and contaminants. FTIR is perhaps the most powerful tool for identifying types of chemical bonds (functional groups). The wavelength of light absorbed is characteristic of the chemical bond as can be seen in the annotated spectrum on the next page.

By interpreting the infrared absorption spectrum, the chemical bonds in a molecule can be determined. FTIR spectra of pure compounds are generally so unique that they are like a molecular "fingerprint". While organic compounds have very rich, detailed spectra, inorganic compounds are usually much simpler. For most common materials, the spectrum of an unknown can be identified by comparison to a

(see FTIR ►)

Lab Notebook

WCAS just purchased two new pieces of test equipment, an **elemental (CHN) analyzer** from Exeter Analytical and a new **ICP-MS system** from Agilent. The new Agilent 7500ce ICPMS includes an octapole reaction cell as well as interfaces with GC and HPLC. We plan to expand on our element speciation capabilities. The elemental analyzer will be performing carbon, hydrogen, and nitrogen analysis in organic materials (0.01-100% range). These are due to be installed in July-August 2004. You'll be hearing a lot more in the future.

Mike Shelton presented a poster at the annual meeting of the American Society for Mass Spectrometry (ASMS) in Nashville. The poster summarized our development of an **LC-MS/MS method for determining very low levels of perchlorate** in water and produce. Data was presented for perchlorate levels down to 0.02 ppb in water and 1 ppb in produce (lettuce, zucchini, celery, green beans and dates). We can now also analyze for perchlorate in milk and other dairy products. The poster has been added to our technical articles section of our web site at **www.wcas.com**.

As always feel free to call our Client Service department at **562.948.2225** for more information or to schedule testing.

RoHS and the EU

Europe has issued a directive on Waste Electrical and Electronic Equipment (WEEE) followed by another directive on Restriction of Hazardous Substances (RoHS). Essentially, certain electrical and electronic equipment must be free of various hazardous materials so that it does not produce an environmental problem upon disposal.

To be marketable in the EU, certain non-exempt products must contain less than 0.1% lead (Pb), mercury (Hg), hexavalent chromium (Cr^{+6}), polybrominated biphenyls (PBB), and polybrominated diphenylethers (PBDE), and less than 0.01% cadmium (Cd). Target dates are Summer 2004 for product registrations and July 1, 2006 for the RoHS ban.

RoHS Testing

Industry representatives the world over have been discussing various test methods to comply with RoHS. So far the most economical method appears to be screening samples for Cd, Cr, Pb, Hg, and Br by x-ray fluorescence spectrometry (XRF) followed by confirmatory tests if necessary. Confirmatory tests would include Cr^{+6} by a diphenyl carbazide color test (such as EPA 7196 or 7199) and PBB and PBDE by GC or GCMS analysis.

While most of these tests are fairly routine, obtaining a representative sample from electrical and electronic equipment may be complicated. Analytical tests are performed on gram sized samples while the electronic components can be large and non-homogenous. Samples should be submitted which have been shredded, ground, or powdered so that gram sized sub-samples are representative of the component. ■

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FTIR ▸*continued*

library of known compounds. WCAS has several infrared spectral libraries including on-line computer libraries. To identify less common materials, IR can be combined with nuclear magnetic resonance, mass spectrometry, emission spectroscopy, X-ray diffraction, and/or other techniques.

Quantitative Analysis

Because the strength of the absorption is proportional to the concentration, FTIR can be used for some quantitative analyses. Usually these are rather simple types of tests in the concentration range of a few ppm up to the percent level. For example, EPA test methods 418.1 and 413.2 measure the C-H absorption for either petroleum or total hydrocarbons. The amount of silica trapped on an industrial hygiene filter is determined by FTIR using NIOSH method 7602.

For sample preparation and physical principles of FTIR please visit our technical articles section of our web site at **www.wcas.com.** If you have any other questions please give our Client Services group a call at 562.948.2225. ■

Infrared Spectrum of Silicone (polydimethylsiloxane)