

# The **WCAS** Analytical Digest

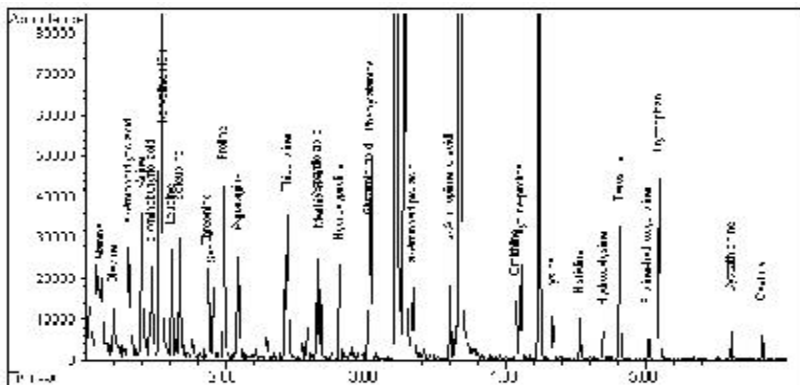
WEST COAST ANALYTICAL SERVICE, INC.

## Amino Acid Analysis by GCMS

w c a s . c o m

WCAS now offers Amino Acid Analysis (AAA) using the Phenomenex kit. This test uses a proprietary reagent to form derivatives of both the carboxylic acid and free amine groups. We perform the test using gas chromatography-mass spectrometry (GCMS).

The test is not applicable to arginine. However the sensitivity is excellent; the chromatogram below is a 20  $\mu$ molar standard. The test is applicable to over 38 amino acids, dipeptides, and other amines.



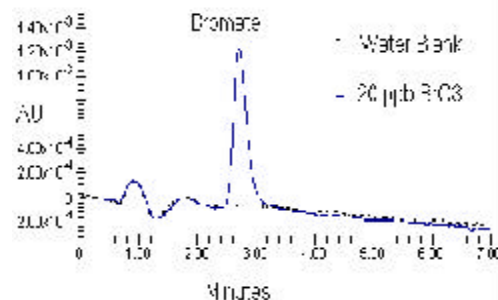
## Bromate by EPA Method 317 - IC

Bromate and other oxyhalides are often unwanted by-products of water disinfection. We have been analyzing samples for bromate by EPA 300.0 using suppressed conductivity detection. This method is not useful for low ppb levels (< 15 ppb). EPA Method 317.0 uses post-column derivatization to increase the specificity of the test and achieve detection limits below 1 ppb (see chromatogram below).

In this method, bromate is specifically reacted with a post-column reagent and detected by visible absorbance detection (450 nm). Separation is basically the same as in EPA 300.0. Neither bromide nor chlorate interfere with this analysis. Potential interference from high levels of chlorite can be eliminated by treatment of the sample with ferrous iron, followed by cleanup with an H + SPE cartridge. This should be done for water samples from treatment plants using chlorine dioxide as a disinfectant. The chromatogram in the EPA method, demonstrates the specificity gained from the post-column chemistry versus conductivity detection.

Water samples may be preserved in the field with ethylenediamine (EDA, 50 ppm) and refrigeration. The holding time after this preservation is 28 days according to the EPA method. Let us know if you need ferrous iron pretreatment or if EDA has been added to your samples, as this information is necessary for us to properly analyze your samples.

Our current MDL is 0.4  $\mu$ g/L with a Minimum Reporting Level (MRL, lowest calibration standard) of 1  $\mu$ g/L. Please give us a call if you have any questions.



## Aldehydes by EPA Method 556 - GCMS

EPA Method 556 offers yet another alternative to measuring formaldehyde and other aldehydes. This method uses pentafluorobenzyl hydroxylamine (PFBHA) to form oxime derivatives of the aldehydes in an aqueous solution at pH 4. The derivatives are extracted into hexane and analyzed by GC-ECD or GCMS. Of course the ECD offers the best detection limits (<1  $\mu$ g/L for most compounds). GCMS has better specificity but only has detection limits in the range of ~10  $\mu$ g/L.

The chromatogram as seen at <http://www.wcas.com/tech/tech2.htm> is taken from EPA Method 556. Note that most aldehydes which are asymmetrical around the carbonyl group produce both E and Z isomers, i.e. two peaks per compound.

### Quick Quotes

... What is a committee? A group of the unwilling, picked from the unfit, to do the unnecessary.

*Richard Harkness*

No government can be maintained without the principle of fear as well as of duty. Good men will obey the last, but bad ones the former only.

*Thomas Jefferson*

# WCAS Laboratory Tidbits



For more information on any of the following call 562-948-2225 and ask for the person mentioned or Eric Lindsay.

**We will be closed July 4<sup>th</sup> and 5<sup>th</sup>.** If you need to have a sample delivered on the 4<sup>th</sup> or 5<sup>th</sup> please give one of our Project Managers a call and we will accommodate you.

**A NEW report format** has been developed which we believe makes it easier for you to read and understand. A large percentage of our reports are now being produced using this new format. We would like to hear from you on what you think of this new format. Please give Eric Lindsay a call at extension 300 with any comments you may have.

**We have settled into our new lab!** If you are in the area please come by and take a tour. Call Eric Lindsay or Louis Albanese at extensions 300 and 303 respectively and they will be happy to set it up.

**Virtual Lab Tours on the Web** are now available at our web site! Just go to our site at [www.wcas.com](http://www.wcas.com) and click on **About WCAS** and then **Lab Tour**. Click on one of the pictures and there will be a clickable line stating [Click Here for a QuickTime VR Panoramic](#). Click on it and have a great tour!

**Craig Hechanova has retired!** After working with us for about 13 ½ years Craig has taken early, and I mean early, retirement. A lot of you have talked to him over the years as he had been one of our Project Managers. He had also worked in our organics group. He has decided to stay home full time with his two beautiful daughters. He had been working part time for the last five years and helping out at their school. He is going to keep doing this as well as his bike riding which is a big part of his families life. We wish him luck and hope he enjoys his time with his kids!

## Arsenic Speciation Update

WCAS currently offers two arsenic speciation techniques: (1) Hydride Generation - ICPMS and (2) Ion Chromatography - ICPMS.

### IC-ICPMS

Ion chromatography can be used to separate the common arsenic (As) species. The chromatogram to the right shows the determination of As(III), dimethylarsenate (DMA), monomethylarsenate (MMA), and As(V). Coupling the IC column directly to the ICPMS results in detection limits near 0.5 µg/L in clean water. This method should be used whenever the organic species are expected, i.e. organic vegetation. Under these chromatographic conditions, arsenobetaine (AsB) is not separated from As(III), so this method should not be used for shellfish or urine. However the AsB interference can be removed by using IC-HG-ICPMS or under different chromatographic conditions.

### Hydride Generation - ICPMS

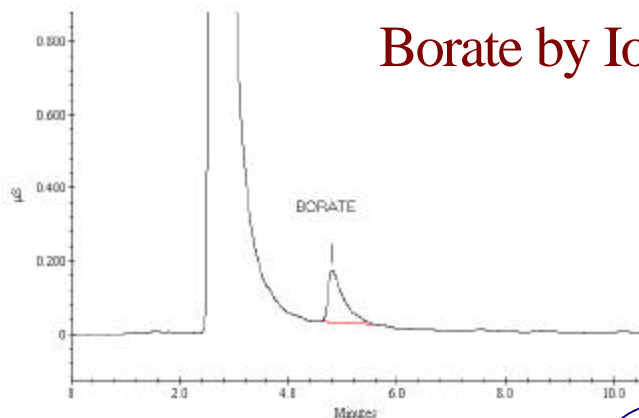
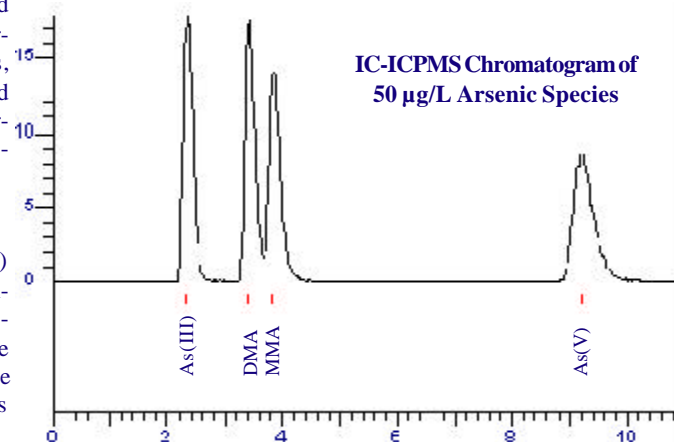
Within a certain pH range, arsine is produced only from the As(III) species, not from As(V). First As(III) is determined under the controlled pH conditions. Then total As is determined. As(V) is then determined by difference. Because DMA and MMA also form volatile hydrides, this speciation technique should not be used when these species may be present. The advantage of the hydride technique is sensitivity. The detection limit is less than 0.02 µg/L.

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## Borate by Ion Exclusion Chromatography

At WCAS we have, in addition to the more common Ion Exchange Chromatography (IC), the capacity to run Ion Exclusion chromatography using the Dionex ICE-AS6 column. Using suppressed conductivity, we can detect borate down to 1 ppm in solutions. In ion exclusion chromatography, the anions of weak acids are retained while anions of strong acids (like Cl<sup>-</sup>) are unretained due to Donnan exclusion. The chromatogram shown here is of an actual sample containing 1 M hydrochloric acid and 6 µg/mL borate at a 1:10 dilution in water. The eluent is dilute heptafluorobutyric acid. This column can separate other anions of weak acids such as carbonate. Give us a call for more information.