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Utilizing Direct Mercury Analysis for Mercury Detection in Botanical Extracts, Vitamins, Minerals and Dietary Supplements

Mercury in Nutraceutical Samples Utilizing Direct Mercury Analysis for Mercury Detection in Botanical Extracts, Vitamins, Minerals and Dietary Supplements

This report was extracted from the Milestone Industry Report DMA–80 *evo* / Nutraceutical

With the expansion of the global nutraceutical market, the spotlight on the analysis of its raw materials is ever increasing. Testing of nutraceutical products for heavy metals like lead, arsenic, cadmium and mercury has gained tremendous attention, and the low level limits in these products makes the analysis particularly challenging. Analytical chemists have to rely on techniques like CVAA and ICP-MS, which involve a time consuming and a labor-intensive sample preparation step. Direct mercury analysis on the other hand, as described by EPA 7473, is an alternative method to traditional techniques that requires no sample prep and delivers results in as little as 6 min per sample. This makes it significantly faster with comparable or better recoveries than CVAA and ICP-MS.

INTRODUCTION

The nutraceutical industry covers a broad spectrum of products including botanical extracts, vitamins, minerals and dietary supplements. A rise in the level of health consciousness among consumers has led to an exponential growth of the nutraceutical market. However, the industry has come under scrutiny to ensure control over toxic heavy metals such as mercury. Given the volatile nature of mercury and the industry's need to test it regularly at low concentrations, sample preparation can often become complicated and confusing. Traditional techniques used to analyze mercury in nutraceuticals involve a sample digestion step followed by CVAA or ICP-MS. Although effective, sample preparation requires manpower, equipment, handling and disposing large amounts of acid resulting in hours for completion.

Alternatively, the U.S. EPA developed a method 7473 for rapid determination of mercury in solids and aqueous samples without sample preparation. This method, known as direct mercury analysis, uses an integrated sequence of thermal decomposition followed by catalytic conversion, amalgamation and atomic absorption spectrophotometry.

The main benefits of direct mercury analysis include:

- Reduced Sample Turnaround (6 Minutes)
- No Sample Preparation
- Reduced Hazardous Waste Generation
- Reduction of Analytical Errors
- General Cost Savings (70% versus CVAA)

EXPERIMENTAL

Instrument

The DMA-80evoDirect Mercury Analyzer from Milestone, as referenced in EPA Method 7473, was used in this study (Figure 1).

Figure 1. Milestone's DMA-80 evo

The DMA-80 *evo* features a circular, stainless steel, interchangeable 40 position autosampler for virtually limitless throughput and can accommodate both nickel (500 mg) and quartz boats (1500 uL) depending on the requirements of the application. It operates from a single phase 110/220V, 50/60 Hz power supply and requires regular grade oxygen as a carrier gas.

As the process does not require the conversion of mercury to mercuric ions, both solid and liquid matrices can be analysed without the need for acid digestion or other sample preparation. The fact that zero sample preparation is required also eliminates all hazardous waste generation.

All results, instrument parameters including furnace temperatures, are controlled and saved with easy export capabilities to Excel or LIMS.

Principles of operation

Direct mercury analysis incorporates the following sequence: Thermal Decomposition, Catalytic Conversion, Amalgamation, and Atomic Absorption Spectrophotometry. Controlled heating stages are implemented to first dry and then thermally decompose a sample introduced into a quartz tube. A continuous flow of oxygen carries the decomposition products through a hot catalyst bed where halogens, nitrogen, and sulphur oxides are trapped.

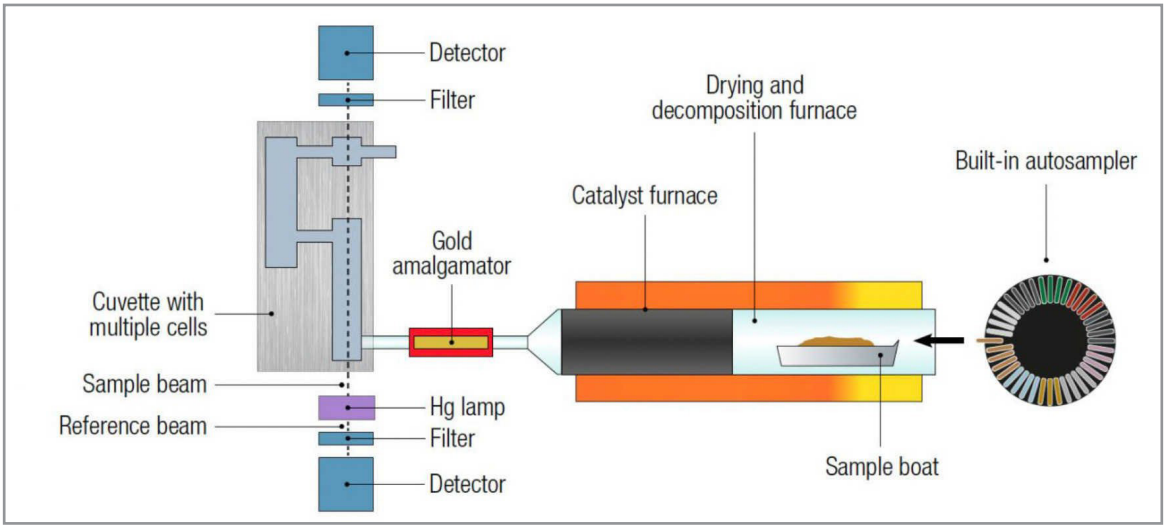

Figure 2. An Internal Schematic of Milestone's DMA-80 evo.

All mercury species are reduced to Hg(0) and are then carried along with reaction gases to a gold amalgamator where the mercury is selectively trapped. All non-mercury vapors and decomposition products are flushed from the system by the continuous flow of gas. The amalgamator is subsequently heated and releases all trapped mercury to the single beam, fixed wavelength atomic absorption spectrophotometer. Absorbance is measured at 253.7 nm as a function of mercury content.

EXPERIMENTAL DISCUSSION

To test the efficiency of the DMA-80 *evo*, three commonly available nutraceutical samples –Valerian Root, Ginkgo Biloba and Glucosamine Chondroitin were spiked by a solution having a mean mercury concentration of ~10.2ppband were run in the instrument to test for spike recoveries. Also, Gingko SRM 3248 was analysed to test if its mercury concentration falls in the NIST certified range.

Calibration

The DMA-80 *evo* can be calibrated using aqueous standards or Standard Reference Materials (SRM). The DMA-80 *evo* used for this experiment had a tricell spectrophotometer and covered adynamic range of 0.0015-1200 ng Hg. Each cell was calibrated using different volumes of 1 ppm and 0.1 ppm stock solutions, prepared from an NIST traceable 1000 ppm stock solution (VHG Labs).

Operating conditions

The DMA-80's operating conditions for all analyses are shown in Table 1.

Table 1. Analysis Operating Parameters

Parameter	Setting
Drying Temp/Time	30 seconds to 200 °C
Decomposition Ramp	90seconds to 660 °C
Decomposition Hold	90 seconds at 660 °C
Catalyst Temp	565 °C
Purge Time	60 seconds
Amalgamation Time	12 seconds at 900 °C
Recording Time	30 seconds
Oxygen Flow	120 mL/min

RESULTS

The concentrations mentioned in Table 2 are mean values obtained after running duplicates for each sample. The 2 concentrations obtained for the Ginkgo SRM –3248 were 0.2544 ppb and 0.2673 ppb respectively. These concentrations were not only in the certify ed range of mercury concentration, but also had an RSD of 0.05%, representing the accuracy and reproducibility of the DMA-80 *evo* at low mercury concentrations. The recovery data mentioned in the Table 2 suggests efficient spike recoveries.

Table 2. Results

Sample	Concentration (ppb)	Expected Conc. (ppb)	Recovery (%)
Valerian Root	4,7447	-	-
Gingko Biloba	2,0537	-	-
Glucosamine Chondroitin	1,5817	-	-
Spike	10,2750	10	102,75
Valerian Root (spiked)	15,5589	15,0197	103,59
Gingjko Biloba (spiked)	12,8810	12,3287	104,48
Glucosamine Chondroitin (spiked)	12,4578	11,8567	105,07
Gingko SRM 3248	0,2609	0,271 +/-0,034	96,27

CONCLUSION

A nutraceutical testing laboratory is required to analysedifferent matrices accurately and quickly while keeping operating costs under control. The DMA-80evois an excellent tool as it yields results in ~6 min/ sample and proves to be proficient, matrix-independent and cost-effective while completely eliminating the challenges of sample preparation posed by conventional mercury analysis techniques.

Further reading

Please visit our Hg info center for complete access to application notes, technical papers, as well as links to valuable resources for mercury testing.

Go to www.milestonesrl.com/dma-80

To learn more about mercury and other related topics, feel free to visit these websites:

- EPA Method 7473: http://www.epa.gov/waste/hazard/testmethods/sw846/pdfs/7473.pdf
- ASTM Method D6722-01: http://www.astm.org/Standards/D6722.htm
- EPA Mercury: http://www.epa.gov/mercury/
- Methyl Mercury: http://en.wikipedia.org/wiki/Methylmercury
- Mercury in Fish: http://www.epa.gov/waterscience/fish/advice/mercupd.pdf
- Mercury in Coal: http://energy.er.usgs.gov/health_environment/mercury/

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