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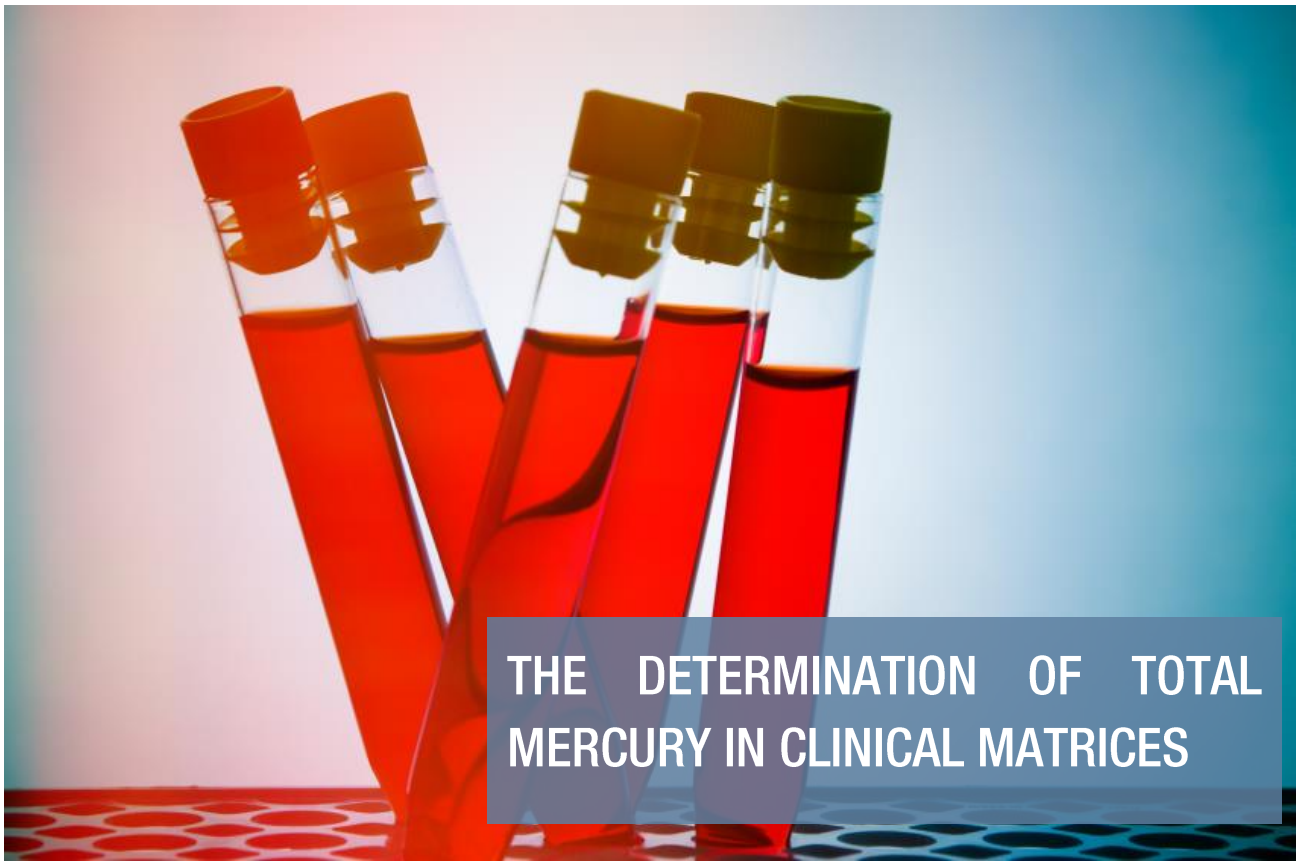

THE DETERMINATION OF TOTAL MERCURY IN CLINICAL MATRICES

The Determination of Total Mercury in Clinical Matrices Utilizing Direct Analysis for Mercury Detection in Blood, Hair and Urine Samples

| SUMMARY

Excessive exposure to mercury has been linked to, among others, neurological and developmental disorders in children, as well as, cardiovascular disease, neurological and other problems in adults. Individuals at a high risk of mercury exposure are typically monitored through the analysis of blood, urine and, occasionally, hair samples.

Many laboratories have testing procedures in place to analyze for mercury in clinical samples.

These procedures have typically included the use of Cold Vapor Atomic Absorption (CVAA) or ICP-MS.

Both of these techniques, although effective, require costly and time-consuming sample digestion prior to analysis. Direct mercury analysis, an alternative to these methods, has been used successfully to determine total mercury in clinical matrices. This technique requires no sample preparation and delivers results in as little as six (6) minutes per sample.

| INTRODUCTION

Methyl mercury is a well-known neurotoxin that has been shown to effect brain development.

Studies have shown that, when ingested by pregnant women, the methyl mercury can cross the placenta and effect development of the central nervous system. Even small

amount of methyl mercury can impact the time it takes a child to walk, talk, hear and write. The most common mechanism for humans to be exposed to this neurotoxin is through the consumption of fish and seafood. However, mercury can also enter the body via inhalation and absorption through skin. The most effective way to monitor people who are suspected of mercury intoxication is through the analysis of blood, urine and hair samples. Because methyl mercury is not excreted from the body, blood analysis is the preferred method to determine total mercury in the human body.

Several methods exist for the determination of mercury in clinical matrices. Traditional analytical methods such as Cold Vapor Atomic Absorption (CVAA) and ICP-MS both require sample preparation prior to analysis. This results in both techniques being costly, labor-intensive and subsequently, having a long turnaround time. Direct mercury analysis, as described in EPA Method 7473 and ASTM Method 6722-01, is a cost-effective, proven alternative to these labor-intensive, wet chemistry techniques.

Direct analysis affords the laboratory many benefits including:

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

I EXPERIMENTAL

INSTRUMENT

The DMA-80 *evo*, Direct Mercury Analyzer, as referenced in EPA Method 7473, from Milestone (www.milestonesrl.com) was used in this study (Figure 1). 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.

Figure 1 Milestone's DMA-80 evo

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 analyzed 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 sulfur oxides are trapped. 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.

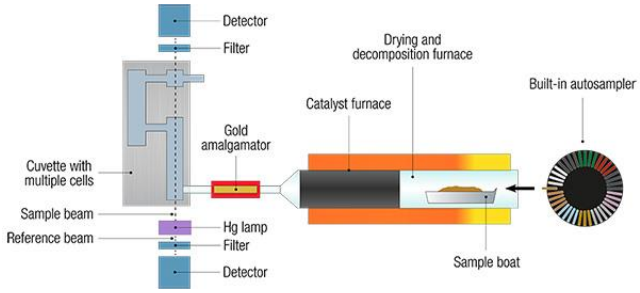

Figure 2 A schematic of Milestone's DMA-80 evo

EXPERIMENTAL DISCUSSION

This study focuses on the effectiveness of the DMA-80 to analyse clinical matrices like blood, hair and urine. Ten blood Proficiency Test (PT) samples were obtained from a state agency. Samples were centrifuged and 100 L aliquots were pipetted into the quartz sample boats (1500 uL) for analysis on the DMA-80. Six urine samples were also analysed. Approximately 200 µL – 300 µL of urine was pipetted directly into the quartz samples boats for analysis.

Finally, hair samples of approximately 5 – 10 mg, in powder form, were weighed out and placed into nickel sample boats for analysis.

CALIBRATION

Calibration standards were prepared using a NIST traceable stock solution of 1000 ppm Hg preserved in 5 % HNO_3.

Working standards of 100 ppb and 1 ppm were prepared and preserved in 37% HCl and stored in amber glass vials. By injecting increasing sample volumes of standard into the quartz sample boats, calibration graphs of 0 – 20 ng (Figure 3) and 20 – 500 ng (Figure 4) of mercury were created using the 100 ppb and 1 ppm standards respectively.

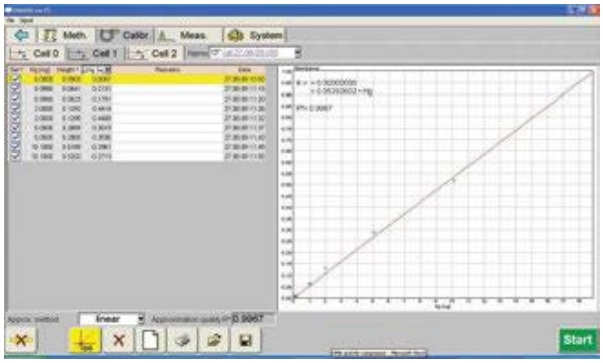

Figure 3 0 ng – 20 ng Calibration Graph for ultra-level

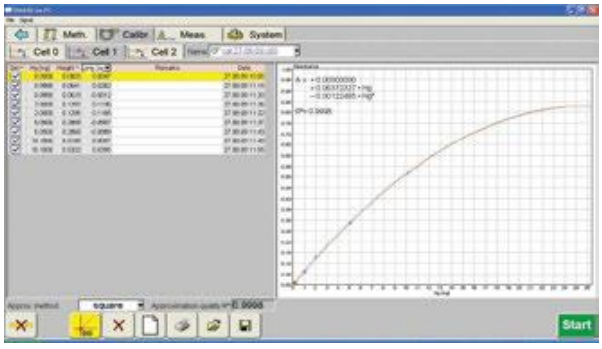

Figure 4 4. 20 ng – 1000 ng Calibration Graph for low to mid-level analysis (ppb, ppm)

INDUSTRY REPORT
DMA-80 *evo* | CLINICAL

| OPERATING CONDITIONS

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

Parameter	Setting
Drying Temp/Time	30 seconds to 200°C
Decomposition Ramp	90 seconds to 650°C
Decomposition Hold	90 seconds at 650°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

Table 1 Analysis Operating Parameters

RESULTS

Results of the blood proficiency samples are shown in Table 3. It is important to point out that all samples were within their certified range. Tables 2 and 4 describe the results of urine and hair analysis respectively. All samples were analysed in triplicate. Good reproducibility was achieved on the hair analysis with most samples having an RSD < 5%.

Sample	Concentration	% RSD
1	5.82±0.06 ppb	1.11
2	21.1±0.4 ppb	1.71
3	47 ± 2 ppb	5.65
4	98 ± 4 ppb	4.18
5	180 ± 4 ppb	2.03
6	200 ± 10 ppb	5.76

Table 2 Urine Analysis

Sample	Weight(g)	Assigned Target(ng/g)	DMA-80 (ng/g)
1	0.1015	32.7	39.3
2	0.1999	1.9	1.3
3	0.1024	5.4	4.5
4	0.1020	17.3	19.8
5	0.1002	12.0	13.1
6	0.1002	25.6	31.0
7	0.1036	2.9	1.2
8	0.1009	1.8	0.0
9	0.1049	10.4	11.3
10	0.1013	6.4	5.8

Table 4 Blood Analysis: DMA-80 vs Known Concentrations

Sample	Concentration	% RSD
1	212 ± 3 ppb	3.5

Table 3 Hair Analysis

| CONCLUSION

The DMA-80 *evo*, direct mercury analyzer, successfully processed all three clinical matrices and provides a fast, accurate and reliable alternative to wet chemistry techniques.

No sample preparation is required meaning results are obtained within six minutes. This is ideal for clinical laboratories looking for quick turnaround of their in-house samples.

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

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