# Determination of Mercury in Biological Matrices

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|---|---|
| Problem: | Unable to analyze mercury in biological samples without the addition of anti-foaming agents. |
| Solution: | The CETAC M-6000A Automated Mercury Analyzer |
| Advantages: | •Samples do not foam in the gas liquid separator.<br>•High sample throughput.<br>•Exceptional detection limits.<br>•Fully automated, stand alone operation.<br>•Compliant with promulgated EPA methodology.<br>•Windows™ based software. |

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|---|---|
| Analytes: | Mercury |
| Matrix: | Biological Matrices including Urine, Fish Tissue, Hair, Bovine Liver |

Performance: Mercury determination in biological samples has traditionally been difficult due to the foaming properties of the sample matrix. Digestions with minimal amounts of protein will form a non-wetting, soap-like mixture, which creates a foam when gas is bubbled through it. The patented design of the M-6000A gas liquid separator (GLS) eliminates this problem. Figure I illustrates the novel design of the GLS.

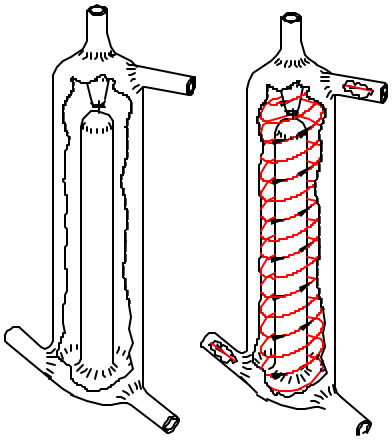


**Figure I:** Novel design Gas Liquid Separator featuring concentric frosted post and efficient swirling of carrier gas.

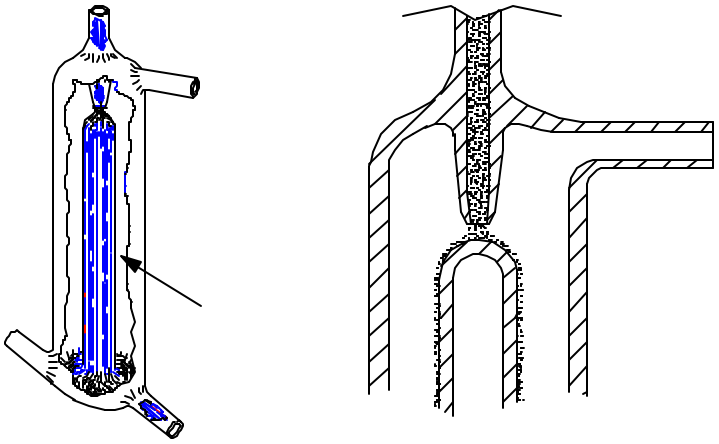


**Figure II:** Gas Liquid Separator illustrating intimate interaction between sample and carrier gas.

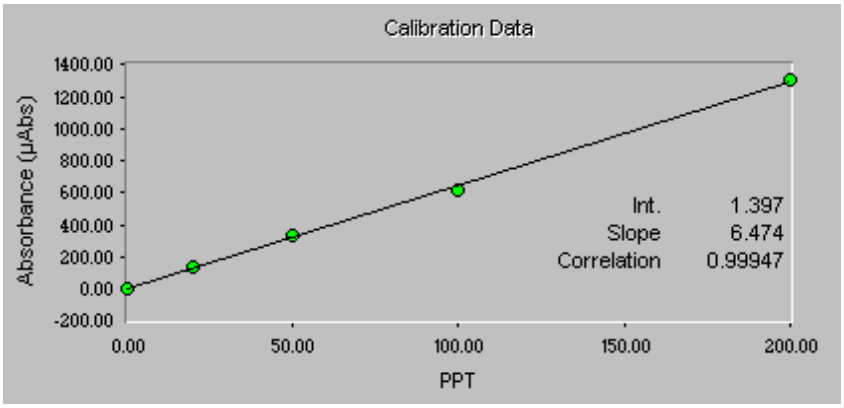
The sample/reductant mix coats the exterior surface of a frosted glass post (Figure I). The carrier gas is introduced tangentially at the bottom of the post, where it swirls upward around the glass post. The elemental mercury vapor ($Hg^0$) evaporates into the gas stream and is swept into the absorption cell for measurement. This design is successful because the carrier gas and sample have enough intimate contact, (Figure II) to evaporate the Hg vapor, but not enough to pass (bubble) through each other. The result is a simple, an effective way to separate the Hg vapor from a complex mixture.

Samples were prepared following EPA water methods, 245.1 ($KMnO_4$), 245.7 ($KBr/KBrO_3$) and 3052 (microwave digestion).

Recoveries for NIST standard reference materials were well within tolerance values (Table 1). Anti-foaming agents were not added to any samples, increasing sample throughput while decreasing laboratory costs and the risk of contamination.

| NIST SRM | Certified Value (mg/g) | Measured Value (mg/g) |
|---|---|---|
| Urine 2760 | 1.05 ± 0.008 | 1.08 ± 0.002 |
| Oyster Tissue 1655a | 0.642 | 0.642 ± 0.003 |
| Bovine Liver 1577a | 0.004 ± 0.002 | 0.0038 ± 0.0003 |

**Table 1:** Recoveries of Hg in NIST tissues.




Instrumentation: CETAC M-6000A Automated Mercury Analyzer

Operating Parameters: Conditions
Gas flow: 40 mL/min
Uptake time: 60 sec
Rinse time: 120 sec

Principal of Operation: An acidified aqueous sample solution containing trace level $Hg^{+2}$ is introduced to the M-6000A by a peristaltic pump. Stannous chloride is used as the reducing agent to generate Hg vapor and joins the sample stream at a mixing tee. The $Hg^{+2}$ is solution is reduced by $Sn^{+2}$ to form $Hg^{o}$ while the mixture is enroute to the gas-liquid separator. The resulting finely dispersed $Hg^{o}/SnCl_2$ emulsion is introduced into the top of the gas-liquid separator, forming a thin film on the entire exterior surface of the frosted glass center post. A dry carrier gas, either nitrogen or argon, first passes through the reference

cell to facilitate measurement of the incident radiant power at 253.6 nm. Next, the carrier gas is introduced tangentially at the bottom of the center post, swirls upward around the post, over the $Hg^0/SnCl_2$ film and toward the gas exhaust port. $Hg^o$ droplets from the emulsion evaporate into the carrier gas and are swept along to the optical section of the M-6000A for analysis. The carrier gas, with $Hg^o$ vapor, passes through a drying tube where water vapor is removed, and then into the sample cell for measurement of transmitted radiant power. The carrier/$Hg^o$ gas stream is ultimately exhausted to a vapor trap where $Hg^o$ is absorbed and clean carrier gas passes to the atmosphere.

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