



Determination of Mercury in Whole Blood Using the CETAC M-6000A Automated Mercury Analyzer

Problem: The complex matrix of whole blood makes mercury analysis challenging. Foaming of samples, inadequate detection limits and slow sample throughput make mercury analysis problematic and difficult.

Solution: The CETAC M-6000A Automated Mercury Analyzer

Advantages:

- Non-foaming gas liquid separator
- Maximum required sample 4 mL
- Fast rinsing gas liquid separator
- Software over-range protection
- Auto dilutor capability
- Simplified maintenance

Analytes:	Hg
Matrix:	Digested Whole Blood (3% HNO ₃)

Performance: The whole blood samples and standards were prepared by a five-stage microwave digestion procedure recommended by CEM (Table 1). 0.5 mL of whole blood was placed in the digestion vessel and 2.5 mL HNO₃ was added to assist in the dissolution of the sample. The samples were diluted to 50 mL and analyzed in the ‘high sensitivity’ mode of the M-6000A. The high sensitivity mode consists of low gas flow (30-40 mL/min) and longer sample uptake and rinse times (60 second uptake, 120 second rinse). Figure 1 is the calibration curve obtained with digested standards.

Table 1. Microwave digestion parameters

Stage	1	2	3	4	5
% Power	100	100	100	100	100
Pressure psi	20	45	85	150	200
Ramp (min)	10	10	10	10	10
Hold (min)	3	3	3	3	5

Whole blood control samples were obtained from The Center of Toxicology in Quebec, Canada. Controls contained both organic and inorganic forms of Hg prior to digestion. Table 2 shows the excellent recovery achieved by using microwave digestion for sample preparation and the M-6000A for determination of mercury levels.

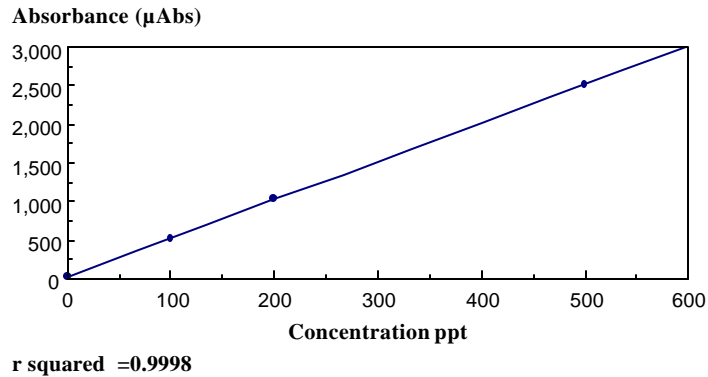


Figure 1: Hg calibration curve.

Sample	Measured Value (µg/L)	Reference Value (µg/L)	% Recovery	%RSD
M9605	146	145	100	10
M9602	230	239	96	2.4
M9511	202	210	96	5

Table 2: Recoveries in reference materials (n=12).

Appreciation is expressed to The Center of Toxicology in Quebec, Canada for supplying the reference materials and helpful consultation with sample handling and preparation of this application.

Instrumentation: CETAC M-6000A Automated Mercury Analyzer
CEM MDS-2000 Microwave Digestion System

Operating Parameters: Instrumental Parameters

Gas flow: 40 mL/min
Sip time: 60 sec
Rinse time: 120 sec
Detection limit: 1.8 ppt

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} in solution is reduced by Sn^{+2} to form Hg° while the mixture is enroute to the gas-liquid separator. The resulting finely dispersed $\text{Hg}^{\circ}/\text{SnCl}_2$ 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 $\text{Hg}^{\circ}/\text{SnCl}_2$ film and toward the gas exhaust port. Hg° 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° vapor, passes through a drying tube where water vapor is removed and then into the sample cell for measurement of transmitted radiant power. Finally, the carrier/ Hg° gas stream is exhausted to a vapor trap where Hg° is absorbed and clean carrier gas passes to the atmosphere.

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