

# Mercury (Hg) in Soil (NIST 2710a)

**Method ID: USEPA 7473**
**Category: Environmental**
**Technique: CAA**

## Summary

This technical note will describe the analysis of total mercury in soil (NIST 2710a) in the high mg/Kg range, using the Teledyne Leeman Labs Hydra IIc mercury analyzer. This method utilized the moisture control system as described in AN1701 ([viewable here](#)) to enhance the performance of the Hydra IIc in standard, direct combustion mode for total mercury in this matrix.

Direct combustion mercury analysis, as described in USEPA 7473, is a simple method which eliminates lengthy sample preparations and the hazardous wastes generated from wet chemistry techniques. With no sample pretreatment, total mercury results are obtained faster and at less expense using Teledyne Leeman Labs' Hydra IIc mercury analyzer for direct combustion atomic absorption (CAA).

Weighed samples were introduced into the analyzer using an automated sequence. The unattended analysis of samples was completed at a rate of ~ 5 min / sample.

## Instrumentation

Hydra IIc CAA mercury analyzer, Envoy software version 2.2, quartz boats (calibration), nickel boats (samples), analytical balance, hot plate, watch glasses, disposable spatulas, pipettes/tips, and labware/reagents for aqueous calibration standard preparation.

## Method Parameters

	°C	Seconds	Other
Oxygen Flow (mL/min)			500
Drying	300	30	
TempRamp*		60	
Decomposition	800	120	
Catalyst	600	20	
Amalgamator	700	30	
Integration		70	
LowPeakAbsLimit*			1100000
HighSampleAutoCleanAbsLimit*			30000000
NafionFurnaceTemp*			600
EluteWarmTempOverride*			175

\*Adjusted in, or added to, the startup.ini text file located in the Envoy folder.

## Calibration

Aqueous intermediate standards were prepared in 1% HNO<sub>3</sub> acid for mercury stability. Various weights of

intermediate calibration standards were added to quartz boats for total mass in ng of Hg, as listed below. Both Low and High Concentration ranges utilized a quadratic fit.

Low Concentration	Blank, 1, 5, 10, 20, 50, 100 ng
High Concentration	100, 200, 600, 800, 1000, 1500, 2000 ng

## Procedure

1. Homogenized sample in the container
2. Tare boats and add sample to boats
3. Load boats onto the sample boat shuttle
4. Run Hydra IIc using parameters listed with an automated sequence
5. Determine percent moisture in CRM for data analysis

## Sample Weight

Average sample weight range was ~ 0.1 g.

## Results

	mg/Kg	
NIST 2703 (0.474 mg/Kg)	0.459	97 % Recovery
NIST 2704 (1.44 mg/Kg)	1.205	84 % Recovery
NIST 2710a (1)	9.329	
NIST 2710a (2)	8.527	
NIST 2710a (3)	9.253	
NIST 2710a (4)	9.194	
NIST 2710a (5)	9.044	
NIST 2710a (6)	9.021	
NIST 2710a (7)	9.195	
Avg	9.080 ± 0.198 @ 95 % Confidence	
STDEV	0.267	
MDL	0.654 @ 95 % Confidence	
Min	8.527	
Max	9.329	
NIST 2403 (0.474 mg/Kg)	0.442	93 % Recovery
NIST 2704 (1.44 mg/Kg)	1.218	85 % Recovery

## Conclusion

The calibration curve check standard of 97% and 93% recovery for NIST 2703, and 84% and 85% for NIST 2704 demonstrate the system is in control and stable in both the high and low sensitivity calibration ranges. The certified value for NIST 2710a is 9.88 ± 0.21 mg/Kg at 95% confidence. This analysis of NIST 2710a had a recovery of 9.080 ± 0.198 mg/Kg at 95% confidence, which is 92% of the certified value. The Hydra IIc in standard mode is an ideal system for the determination of total mercury in soil, NIST 2710a a high mercury content soil.