TELEDYNE LEEMAN LABS
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Technical Note: Hg/FD-1902

Tuna Fish (BCR-463)

Category: Food **Technique: CVAA**

Summary

This technical note describes the analysis of Tuna Fish (BCR-463) by FDA EAM 4.5 on the QuickTrace® M-7600 mercury analyzer. The analysis utilized a reduced internal diameter (ID) tubing for the stannous chloride ($SnCl_2$), sample and waste lines. This configuration reduces reagent and waste and is validated in Application Note: AN1905 – Green Chemistry: Decreased Reagent Consumption and Waste Using Reduced ID Tubing on the QuickTrace® M-7600 CVAA Mercury Analyzer (Viewable Here).

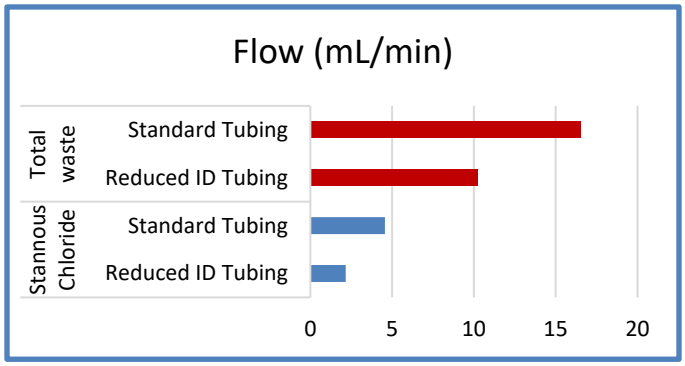
Instrumentation

Analyzer and ASX-560 autosampler for automated analysis. $SnCl_2$ pump tubing was orange/yellow 0.51 mm (TLL PN 15-4309-102). Sample and waste tubing were white/white 1.02 mm (TLL PN 15-4308-102).

Method Parameters

Parameter	Value
Sample Uptake (sec)	30
Rinse Time (sec)	65
Gas Flow (mL/min)	125
Pump Speed (%)	80
Replicate Read Time (sec)	1
Replicates	3

Sample flows were optimized by adjusting clamp pressure according to the *QuickTrace® M-7600 User Manual*. The reduced ID tubing (0.51 mm) used 53% less $SnCl_2$ than the standard tubing (0.76 mm). Total waste was reduced by ~40%.



Calibration

Six Standards (0, 50, 100, 250, 500 and 1000 ng/L) were prepared in 10% nitric acid (HNO_3), 7% hydrochloric acid (HCl) and 1% sodium chloride (NaCl). The calibration curve must be linear with a correlation of 0.998 or better. The calibration was verified with a 500 ng/L second-source standard with a ± 10% acceptance limit.

Sample Preparation

The sample weight average was ~0.1 g. Samples were digested in a CEM MARS® 6 microwave. 5 mL of HNO_3 and 1 mL of 1% NaCl were added to each sample. Samples were capped and microwaved according to EAM 4.5 method parameters. After digestion, samples were transferred to 50 mL vials containing 10 mL of DI water and 3.5 mL of HCl. Samples were then filled to a final volume of 50 mL and mixed. Samples were diluted 10X with a digested reagent blank prior to analysis.

Procedure

Samples and standards were loaded onto the ASX 560. Inorganic mercury was reduced to elemental mercury by excess online addition of 10% $SnCl_2$ in 7% HCl.

Results

ICV (second source) - ng/L	489.90	98.0 % Recovery
	µg/g	
Hg in Tuna Fish (BCR-463) 1	2.99	
Hg in Tuna Fish (BCR-463) 2	2.91	
Hg in Tuna Fish (BCR-463) 3	2.90	
Hg in Tuna Fish (BCR-463) 4	2.86	
Hg in Tuna Fish (BCR-463) 5	2.86	
Hg in Tuna Fish (BCR-463) 6	2.87	
Hg in Tuna Fish (BCR-463) 7	2.81	
Avg	2.89 ± 0.042 @ 95 %	
STDEV	0.06	
MDL	0.14 @ 95 %	
Min	2.81	
Max	2.99	
CCV (second source) - ng/L	491.04	98.2 % Recovery

Conclusion

The QC recoveries of 98.0 to 98.2% demonstrate that the system is in control and stable for analysis of trace Hg in tuna fish. The trace Hg certified value for BCR-463 is 2.85 ± 0.16 µg/g. The calculated MDL for this analytical system under the method conditions presented in this technical note is ≤ 0.14 µg/g with a confidence level of 95%.

Using the reduced ID pump tubing saved reagent consumption, decreased waste and was an ideal configuration for the determination of mercury in Tuna Fish (BCR-463).