Method for Analysis of Mercury in Compact Fluorescent Lamps

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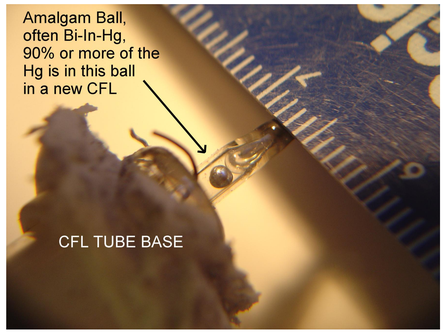
Abstract

Compact Fluorescent Lamps (CFLs) have increasingly been used to replace relatively inefficient incandescent light bulbs since their commercial release in 1985. After lamp failure, used CFLs are typically disposed of in public landfills through normal household disposal. Each CFL contains between 1 and 5 mg of mercury, which is required for its proper operation. These concentrations raise concerns over the total cumulative mercury in landfills contributed from CFL disposal, and the possible mercury ground water contamination which may occur over time. Typical mercury analysis procedures employing nitric and hydrochloric acids and permanganate salts are ineffective in releasing all of the mercury from the CFLs, making calculations of mercury content per CFL, or collectively per landfill, difficult. Shaw's Quality Assurance Technical Support (QATS) program was tasked by the U.S. Environmental Protection Agency's (USEPA) Analytical Services Branch (ASB) in 2009 to develop a method more effective in releasing bound mercury in the CFLs. This poster describes several CFL sample preparation and analysis techniques that were investigated, including the typical acid-permanganate digestion, as well as a new CFL preparation method using a hydrofluoric acid/microwave digestion technique, and three (3) new quantitative thermal desorption techniques. The thermal desorption techniques were developed with the intent of releasing mercury bound to the CFL components for trapping by absorbing media followed by analysis using mercury cold vapor atomic absorption (CVAA) analysis. One thermal desorption technique using hydrochloric acid-impregnated activated carbon is described, which has not been previously employed for quantitative mercury analysis. Recoveries of CFL mercury using these new techniques show significant improvement over the typical mercury analysis methods using acid-permanganate digestion procedures coupled with CVAA.

Method Challenges

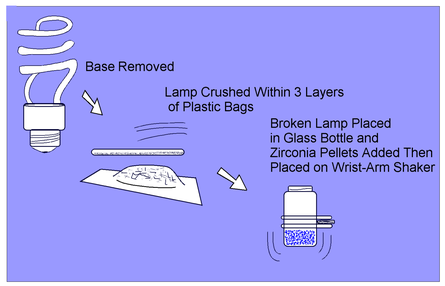
- CFL mercury analysis is difficult due to both homogeneity and dissolution problems
- The EPA acid - permanganate digestion method yields poor mercury recoveries on crushed CFL residue
- Eleven (11) alternative digestion methods were evaluated

The Presence of a 1.5 mm Amalgam Ball in Base of CFL Results in Sample Inhomogeneity



Methods

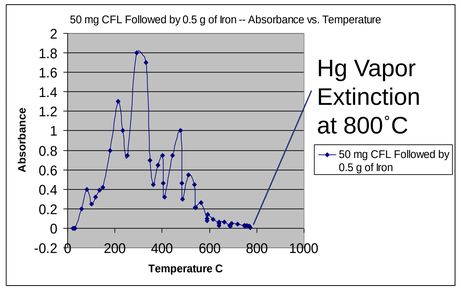
CFL Sample Preparation



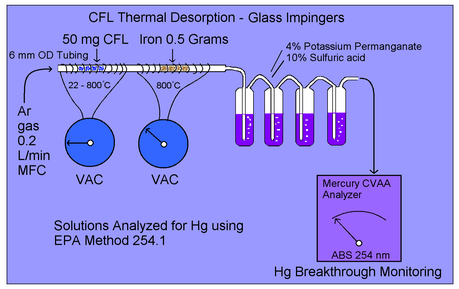
Hydrofluoric Acid Microwave Digestion Procedure

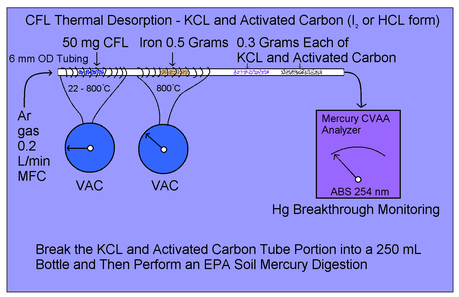
- Place 100 mg CFL sample in a microwave vessel
- Add 0.5 mL HF + 5 mL sulfuric acid
- Microwave for 15 minutes at 180 W and 5 minutes at 240 W
- Add boric acid to digest, and bring to 100 mL volume
- Digest using EPA Method 245.1
- CVAA analysis using a Leeman Hydra-II™ mercury analyzer

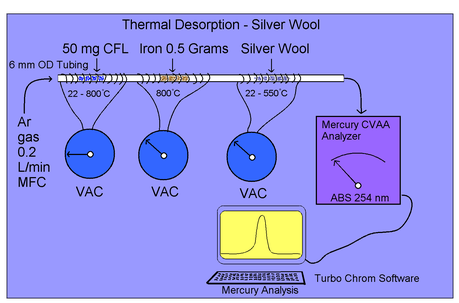
Thermal Desorption Techniques Are Used to Verify Digestion Effectiveness



Thermal Desorption Mercury Trapping Techniques (3 Evaluated)







Results

Initial Digestion Results (Philips Marathon 27 W)

Digestion Technique	Mercury mg/Bulb N = 3	RSD N = 3	Matrix Spike Recovery (%)
EPA Soil Method	0.1	28.8	78.2
Aqua Regia + Acid-Permanganate	0.1	NA	NA
Aqua Regia	0.3	56.6	88.0
HF-Microwave + EPA Aqueous Digestion	1.2 *	3.7	105.9

CFL Mercury Results by Manufacturer HF- Microwave Digestion

CFL NAME Wattage	Mercury mg/Bulb N = 3	RSD N = 3	Matrix Spike Recovery (%)	Amalgam Ball Observed
Philips 27 W	1.22*	3.7	105.9	No
Philips 13 W	2.83	31.9	303.1	Yes
Philips 13 W	1.71	46	119.4	Yes
Philips 13 W Without Ball	0.04	51.9	104.9	NA
Philips Ball Alone	3.55	NA	110.6	NA
ECO Smart 27 W	2.60	11.5	106.4	Yes
ECO Smart 14 W	1.16	21.9	106.4	Yes
Fiet 23 W	4.28	9.3	107.0	Yes
Fiet 15 W	5.26	1.5	106.8	Yes
Fiet 15 W ~ 2 yrs old	4.45	4.4	52.4	No

*Red Font is for Philips Marathon CFL 27 W by Various Techniques

CFL Results Thermal Desorption (Philips Marathon 27 W)

Digestion Technique	Mercury mg/Bulb N = 3	RSD N = 3	Matrix Spike Recovery (%)
Glass Impingers	1.1*	10.2	NA
KCL - Iodine Activated Carbon	1.4*	NA (N =1)	147
KCL – HCL Activated Carbon	1.4*	4.2	109.7
Silver Wool	1.3*	6.0	81.3

Conclusion

Of the eleven (11) digestion methods evaluated, the hydrofluoric acid microwave digestion provided the best results. This method allows for the complete, or nearly complete, dissolution of mercury in the CFL, which has been verified by several thermal desorption techniques.

Commercial soil/solid mercury analyzers are available that may be able to analyze CFL residue. Verification of this technology may be the subject of future studies.

References

Larrea, M., Gomez-pinilla, I., Farinnas, J. (1997): Microwave-assisted Acid Dissolution of Sintered Advanced Ceramics for ICP-AES. J. Anal. At. Spectrom., 1997, 12, 1323 – 1332. (procedure 5, p. 1330)

Acknowledgement

This study was performed by Shaw Environmental, Inc. under USEPA's QATS contract EP-W-06-005, under the direction of the USEPA Analytical Services Branch (ASB).

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