BUCK Scientific

# Application Note #AA3012

# Determination of Trace Elements in Lead for Battery Applications using Atomic Absorption Analysis

**Sample and Standard Preparation procedures for Trace Elements in Lead**

*[1] Preparation of Lead (Pb) Samples:*
Drill Pb metal to make small pieces. Weigh 2.0 grams of these pieces on a balance. Place the sample in a 400ml Pyrex breaker and add 10ml $H_2O$, 2.5 grams of tartaric acid and 7ml $HNO_3$. Warm on a hot-plate until the sample dissolves. Dilute to 100ml in a volumetric flask with water ($H_2O$). This prepares a 2% sample solution, with a dilution factor of 50.

*[2] Preparation of Sulfuric Acid ($H_2SO_4$) Samples:*
Using a graduated cylinder, place 75ml of water ($H_2O$) into a 100ml volumetric flask. Slowly add sulfuric acid ($H_2SO_4$) to the water, swirling to mix. **Solution will bubble up and get hot, do NOT spill it on yourself – wear gloves!** Add $H2SO_4$ to bring the final volume to 100ml. This prepares a 25% sample solution, with a dilution factor of 4.

*[3] Preparation of Pure / Waste Water ($H_20$) Samples:*
Using a digital pipettor or glass pipet, add 1ml nitric acid ($HNO_3$) to 100ml volumetric flask. Add water sample to 100ml mark. There is basically no dilution and the energy relates directly to the sample.

*[4] Preparation of Multi-element Standards for Calibration:*
Using a pipet or pipettor, add 100ml of each of the 1000ppm stock concentrate standards (Buck standards) to a 1 Liter volumetric flask containing 250ml water ($H_2O$) and 50ml nitric acid ($HNO_3$). The following groupings will prepare stable 100ppm Stock Standards . (Add water to bring final volume to 1 Liter):

A) Bi, Ni, Ag, Zn, Cu, Cd
B) Fe, Sb, Sn, As, Al, Ca

Dilute the 100ppm Stock Standards into the following Working Standards:

| | | | |
|---|---|---|---|
| 25ml | Stock + 1ml $HNO_3$ to 100ml in Volumetric Flask = | | 25ppm |
| 10ml | " | " | 10ppm |
| 5ml | " | " | 5ppm |
| 2ml | " | " | 2ppm |
| 1ml | " | " | 1ppm |
| 0.5ml | " | " | 0.5ppm |

Use these standards for Cu, Ag, Fe, Ca:
0, 0.5, 2, 5ppm.

Use these standards for Ni, Zn, Cd:
0, 0.5, 1, 2ppm.

Use these standards for Bi, Sb, Sn, As, Al, Pb:
0, 5, 10, 25ppm.

SIC: 173, 351, 353, 361, 362, 366, 3691, 3692, 3714, 3724, 5013

## Determination of Trace Element in Lead

**Samples:** Lead Sample #19, Lead Sample #21, Sulfuric Acid (~98%)

**Sample Prep:** 2% solutions of lead in 5% $HNO_3$ / 2.5% tartric acid; 10% solution of sulfuric acid (1:10 dilution)

**Calibration:** 0.5 and 2.5 µg / ml (ppm) analyte metal standard in 2% high-purity lead matrix, 2% high-purity lead matrix blank; for lead samples.
1.0 µg / ml (ppm) analyte metal standard in pure (distilled / deionized) water, pure water blank; for sulfuric acid sample.

**Instrument:** Buck 210VGP Atomic Absorption Spectrophotometer, Giant Pulse and In-Line $D_2$ Correction, and Model 420 Hydride Generation system.

**Conditions:** Standard operating conditions for 210 unit; analytical parameters and correction modes as listed per element; air / acetylene flame for Ni, Ag, Zn, Cu, Fe, Cd; nitrous oxide / acetylene for Al, Ca; argon hydrogen for As, Sb, Sn, Bi.

**Results:** Values are weight percent (% w/w) in original sample: Data based on 1:50 Pb dilution and 1:10 $H_2SO_4$ dilution: D.L. [detectability] based on 2-sigma statistics for Pb samples.

| Element | Wavelength | D.L. | Lead # 19 | Lead # 21 | $H_2SO_4$ |
|---|---|---|---|---|---|
| Ni | 232nm | 0.0015% | <0.0015% | 0.0018% | <0.0015% |
| Ag | 328 | 0.0003 | 0.0017 | 0.0018 | 0.0005 |
| Zn | 213 | 0.0003 | 0.0009 | 0.0004 | 0.0027 |
| Cu | 324 | 0.0005 | 0.0150 | 0.0093 | 0.0021 |
| Bi | 223 | 0.0008 | 0.0167 | 0.0184 | <0.0008 |
| Fe | 248 | 0.001 | <0.001 | 0.002 | 0.005 |
| Sb | 217 | 0.0005 | <0.0005 | 0.0012 | <0.0005 |
| Sn | 224 | 0.0007 | <0.0007 | 0.0009 | <0.0007 |
| As | 193 | 0.0001 | <0.0001 | 0.0011 | <0.0001 |
| Cd | 228 | 0.0005 | 0.0006 | 0.0009 | 0.0008 |
| Al | 309 | 0.005 | <0.005 | <0.005 | 0.006 |
| Ca | 422 | 0.0005 | 0.0006 | 0.0022 | 0.0472 |

These data show the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the manufacturing industry. The overall high sensitivity of the various trace metals supports the interference-free quality of the data. The combination of unique components provides an un-matched system in performance and economy.

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