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 beaker 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 H_2SO_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_2O) 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.

Analyst: Gerald J. DeMenna

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% tartaric acid; 10% solution of sulfuric acid (1:10 dilution)

Calibration: 0.5 and 2.5µg/ml (ppm) analyte metal standard in 2% high-purity lead matrix, 2% high-purity lead matrix blank; for lead samples.
1.0µg/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	Wave-Length	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.

Basic System:	**$12,950.00**
Turnkey System:	**$20,593.00** Includes: All recommended lamps, standards, and accessories for normal operation.

For detailed configuration, refer to Quote #AA40012A

Application Note
#AA3012B

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25ml	Stock + 1ml HNO_3 to 100ml in Volumetric Flask =	25ppm
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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.

Analyst: Gerald J. DeMenna

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1.0µg/ml (ppm) analyte metal standard in pure (distilled/deionized) water, pure water blank; for sulfuric acid sample.

Instrument: Buck 200A Atomic Absorption Spectrophotometer, Model 420 Hydride Generation system, and strip chart recorder.

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.

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Basic System: **$8,495.00**

Turnkey System: $16,138.00   Includes: All recommended lamps, standards, and accessories for normal operation.

For detailed configuration, refer to Quote #AA40012B

1-800-562-5566

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