

Question 12

Sample calc for dilution of Lake Nakuru samples

typical sample concentration in solid (ug/g)

147

Step 1: Solid sampled and dried

Step 2: 1.0 g of dried solid removed

1.0

Step 3: the 1.0 g of dried solid was digested and diluted to 100.0 mL

expected concentration 1470 ug/L (ppb)
(ug/g * g) / L

Step 4: This solution was diluted 1:20 before electrochemical analysis

Final Concentration 73.5 ug/L (ppb)
above conc /20

Note: this question and answer take into consideration the sediment and suspended solid samples described (which analyses samples by XRF and AAS) and the sample preparation described in Locatelli 1999, which pre

in Nelson 1998
epares sediment samples for ASV analysis.

Determination of Pb in Sediment Samples

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Pb ug/g
4.980	(10 into 500)	0.1011	0.3045	0.0244	24.4	0.4877	0.04877	48.8
		0.1008	0.3026	0.0245	24.5	0.4901	0.04901	49.0
		0.1205	0.3225	0.0292	29.2	0.5848	0.05848	58.5
mass Pb(NO3)2								
0.1990	target = 0.1998							
249.0	ppm (stock std)							
				avg	26.0	0.5	0.1	52.1
				stdev	2.8	0.1	0.0	5.5
				RSD	0.106	0.106	0.106	0.106

Determination of Zn in Sediment Samples

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Zn ug/g
5.006	(5 into 1000)	0.2134	0.3152	0.1013	101.3	2.0251	0.20251	202.5
		0.2159	0.3165	0.1036	103.6	2.0723	0.20723	207.2
		0.2200	0.3251	0.1011	101.1	2.0222	0.20222	202.2
mass Zn								
1.0011	target = 1.0000							
1001.1	ppm (stock std)							
				avg	102.4	2.0	0.2	204.0
				stdev	1.4	0.0	0.0	2.8
				RSD	0.014	0.014	0.014	0.014

Determination of Cu in Sediment Samples

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Cu ug/g
5.001	(10 into 500)	0.0985	0.2000	0.0474	47.4	0.9478	0.09478	94.8
		0.0996	0.2015	0.0477	47.7	0.9546	0.09546	95.5
		0.1000	0.2099	0.0445	44.5	0.8893	0.08893	88.9
mass CuCl2 . 2H2O								
0.3354	target = 0.3354							
250.0	ppm (stock std)							
				avg	46.5	0.9	0.1	93.1
				stdev	1.8	0.0	0.0	3.6
				RSD	0.039	0.039	0.039	0.039

In the paper Locatelli 1999, they used 1000 ppm standard solutions (purchased) and diluted for working standard. Here I have the students prepare the standards from scratch and calculate concentrations. To simplify or shorten the problem, I could have students start with the 1000 ppm standards rather than solid materials.

Stock standard: Weigh out lead nitrate to prepare 250.0 ppm Pb (weigh 0.1998 g and dilute to 500.0 mL).

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Stock standard: Prepare 1000 ppm Zn - weigh out 1.0000 g Zn(s); dissolve in conc HCl and dilute to 1.000 L.

Working standard: dilute to 5.00 ppm (5.00 mL of stock into 1000.0 mL) with appropriate electrolyte

Stock standard: Weigh out cupric chloride to prepare 250.0 ppm Cu (weigh 0.3354 g and dilute to 500.0 mL).

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Sample preparation is described in the document: briefly, 1 g sediment samples were acid digested and diluted to 100 mL. This solution was diluted by a factor of 20 with appropriate electrolyte. The analysis was performed on this second solution.

Determination of Cr in Sediment Samples

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Cr ug/g
5.000	(10 into 500)	0.1111	0.2522	0.0387	38.7	0.7736	0.07736	77.4
		0.1212	0.2855	0.0363	36.3	0.7251	0.07251	72.5
		0.1359	0.2989	0.0409	40.9	0.8187	0.08187	81.9
Stock Standard conc (ppm)								
	1000.0							
250.0	ppm (stock std)			avg	38.6	0.8	0.1	77.2
				stdev	2.3	0.0	0.0	4.7
				RSD	0.061	0.061	0.061	0.061

Modified from Wang 1997 and Locatelli 1999

Stock standard: 1000 mg/L standard solution is diluted appropriately

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Sample preparation is described in the document: briefly, 1 g sediment samples were acid digested and diluted to 100 mL. This solution was diluted by a factor of 20 with the following solution:

0.01 M DTPA (complexing agent), 0.04 M sodium acetate (buffer), and 0.5 M sodium nitrate (pH 5.6)

The analysis was performed on 10.00 mL this second solution.

I (1) corresponds to the peak current from a scan of this solution

I (2) corresponds to the peak current after addition of a 100 uL spike of chromium standard

Determination of Pb in Suspended Solids

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Pb ug/g
4.980	(10 into 500)	0.1511	0.9513	0.0093	9.3	0.1859	0.01859	18.6
		0.1623	0.9253	0.0105	10.5	0.2093	0.02093	20.9
		0.1506	0.9222	0.0096	9.6	0.1921	0.01921	19.2
mass Pb(NO3)2								
0.1990	target = 0.1998							
249.0	ppm (stock std)							
				avg	9.8	0.2	0.0	19.6
				stdev	0.6	0.0	0.0	1.2
				RSD	0.062	0.062	0.062	0.062

Determination of Zn in Suspended Solids

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Zn ug/g
5.006	(5 into 1000)	0.1985	0.4545	0.0379	37.9	0.7588	0.07588	75.9
		0.1752	0.4326	0.0333	33.3	0.6667	0.06667	66.7
		0.1863	0.4863	0.0304	30.4	0.6086	0.06086	60.9
mass Zn								
1.0011	target = 1.0000							
1001.1	ppm (stock std)							
				avg	35.6	0.7	0.1	67.8
				stdev	3.8	0.1	0.0	7.6
				RSD	0.106	0.112	0.112	0.112

Determination of Cu in Suspended Solids

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Cu ug/g
5.001	(10 into 500)	0.0999	0.4697	0.0133	13.3	0.2657	0.02657	26.6
		0.1001	0.4898	0.0126	12.6	0.2527	0.02527	25.3
		0.0959	0.4800	0.0123	12.3	0.2456	0.02456	24.6
mass CuCl2 . 2H2O								
0.3354	target = 0.3354							
250.0	ppm (stock std)							
				avg	12.7	0.3	0.0	25.5
				stdev	0.5	0.0	0.0	1.0
				RSD	0.040	0.040	0.040	0.040

In the paper Locatelli 1999, they used 1000 ppm standard solutions (purchased) and diluted for working standard. Here I have the students prepare the standards from scratch and calculate concentrations. To simplify or shorten the problem, I could have students start with the 1000 ppm standards rather than solid materials.

Stock standard: Weigh out lead nitrate to prepare 250.0 ppm Pb (weigh 0.1998 g and dilute to 500.0 mL).

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Stock standard: Prepare 1000 ppm Zn - weigh out 1.0000 g Zn(s); dissolve in conc HCl and dilute to 1.000 L.

Working standard: dilute to 5.00 ppm (5.00 mL of stock into 1000.0 mL) with appropriate electrolyte

Stock standard: Weigh out cupric chloride to prepare 250.0 ppm Cu (weigh 0.3354 g and dilute to 500.0 mL).

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Sample preparation: Water samples were collected and filtered to obtain 1 g (dry weight) of suspended solids. The solids were acid digested and diluted to 100 mL. This solution was diluted by a factor of 20 with appropriate electrolyte. The analysis was performed on this second solution.

Determination of Cr in Sediment Samples

Working Std conc		I (1) / uA	I (2) / uA	ppm	ppb	x 20 (ppm)	x 0.100 L (mg)	Cr ug/g
5.000	(10 into 500)	0.1015	0.8986	0.0063	6.3	0.1259	0.01259	12.6
		0.1985	0.9563	0.0129	12.9	0.2587	0.02587	25.9
		0.1574	0.9254	0.0101	10.1	0.2025	0.02025	20.3
Stock Standard conc (ppm)								
1000.0								
250.0 ppm (stock std)								
		avg		9.8		0.2	0.0	19.6
		stdev		3.3		0.1	0.0	6.7
		RSD		0.341		0.341	0.341	0.341

Modified from Wang 1997 and Locatelli 1999

Stock standard: 1000 mg/L standard solution is diluted appropriately

Working standard: Dilute 250.0 ppm standard to 5.000 ppm (10.00 mL into 500.0 mL) with appropriate electrolyte

Sample preparation: Water samples were collected and filtered to obtain 1 g (dry weight) of suspended solids.

The solids were acid digested and diluted to 100 mL. This solution was diluted by a factor of 20 with appropriate electrolyte:

0.01 M DTPA (complexing agent), 0.04 M sodium acetate (buffer), and 0.5 M sodium nitrate (pH 5.6)

The analysis was performed on 10.00 mL this second solution.

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