

Experiment 1

Things that go wrong

1. Dilution error

- bad calibration curve
- weird ICP-MS & AA data


2. Not letting the product dry all the way

3. Not pre-drying your filters

how to avoid things going wrong

- Practice, Experience, Mistakes
- Always use the best instrumentation
ie balance, not volumetric flasks
- always look @ your data right away.
do you need to redo it?
- Start w/ more reps - allow for mistakes
- Do a trial run

Example Calculation, Experiment 1

1.  1.273g Metal
in 15 mL HNO_3
diluted to 100.3762 ^{ml or g} H_2O

Initial dilution factor =

$$\frac{100.3762 \text{ g H}_2\text{O}}{1.273 \text{ g metal}} = 78.85 \frac{\text{g H}_2\text{O}}{\text{g metal}}$$

for each replicate

2. Dilutions - see attached sheet

3. Measure solutions on the instrument

4. Your results are either in ppb (ICP-MS) or ppm (flame AA). For your diluted solutions

4a. Subtract $\frac{\mu\text{g Fe}}{\text{g solvent}}$ your blank

4b. apply dilution factors (From Both steps 1 & 2)

$$\frac{\mu\text{g Fe}}{\text{g solvent}} \times \underbrace{\frac{\text{g solvent}}{\text{g solvent}}}_{\text{step 2}} \times \frac{\text{g solvent}}{\text{g metal}} = \frac{\mu\text{g Fe}}{\text{g metal}}$$

for each replicate

4c. Calculate average \bar{x} St. dev.

5. Tabulate the concentration \bar{x} error for each element in ppm.

Report any below detection Limit as BDL

6. Sum to determine total ppm measured \bar{x} the error* associated with that.

*error is calculated by formula in attached table (or appendix 1)

Essentially the question is how many of the million bits you've divided your sample into have you accounted for?

7. Calculate % recovery \bar{x} error.

$$\frac{\text{weight recovered}}{\text{original sample mass}} \times 100\%$$

Dilutions

10x			
sample	aliquot	+ diluent	Dilution Factor
ideal	1.5 ml	13.5 ml	10
1	1.5298	14.9725	9.787227
2	1.4871	14.9965	10.08439

50x			
sample	aliquot	+ diluent	Dilution Factor
ideal	0.3 ml	14.7 ml	50
1	0.2974	15.0515	50.61029
2	0.3007	15.0731	50.1267

		1000x	* use 50x as new sample	
sample	aliquot*	+ diluent	Dilution Factor	
ideal	0.75	14.25	1000	
1	0.7447	15.5012	1053.471	
2	0.7419	15.5479	1050.499	

TABLE 3-1 Summary of rules for propagation of uncertainty

Function	Uncertainty	Function ^a	Uncertainty ^b
$y = x_1 + x_2$	$e_y = \sqrt{e_{x_1}^2 + e_{x_2}^2}$	$y = x^a$	$\%e_y = a\%e_x$
$y = x_1 - x_2$	$e_y = \sqrt{e_{x_1}^2 + e_{x_2}^2}$	$y = \log x$	$e_y = \frac{1}{\ln 10} \frac{e_x}{x} \approx 0.434\,29 \frac{e_x}{x}$
$y = x_1 \cdot x_2$	$\%e_y = \sqrt{\%e_{x_1}^2 + \%e_{x_2}^2}$	$y = \ln x$	$e_y = \frac{e_x}{x}$
$y = \frac{x_1}{x_2}$	$\%e_y = \sqrt{\%e_{x_1}^2 + \%e_{x_2}^2}$	$y = 10^x$	$\frac{e_y}{y} = (\ln 10)e_x \approx 2.302\,6 e_x$
		$y = e^x$	$\frac{e_y}{y} = e_x$

a. x represents a variable and a represents a constant that has no uncertainty.

b. e_x/x is the relative error in x and $\%e_x$ is $100 \times e_x/x$.

Harris, *Quantitative Chemical Analysis*, 8e

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