you-try-it-01.xlsx
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For use with:
Brian M. Tissue, *Basics of Analytical Chemistry and Chemical Equilibria,* (John Wiley: New York, 2013).
http://www.achem.org

Worksheets in this file

notes	This page with background information.
1.A conversions	Converting between various measurement units.
1.B precision	Data to find mean, standard deviation, etc.
1.C outlier	Test to determine if an apparent outlier may be discarded.
1.D LOD-LOQ	Determining limit of detection and limit of quantitation.
1.E calibration	Calculations based on one-point calibration.
1.F standard-addition	Calibration using standard addition.

Background
Refer to Chapter 1 in the text for equations and explanations.
Each worksheet has instructions in the blue shaded box.
For step-by-step help see you-try-it-01guide.pdf.
For help getting started with Excel see spreadsheet-help.pdf.

You-Try-It 1.A **Unit Conversions**

1. Table 1.A.1 provides several examples of physical measurements in common units.
   Express each case in Table 1.A.1 in the other units that are listed.
   Some conversion factors are below the table.

2. Table 1.A.2 provides a list of analytical results with common units.
   Express each case in Table 1.A.2 in the other units that are listed.
   The density of each solution is given in column E.

Notes: mph is 'miles per hour'
‰ is 'parts per thousand' (by weight if not specified otherwise)

Table 1.A.1

sample	measurement	value	units	Convert to:	
vehicle	velocity	25.00	mph	km/h	m/s
10 % sucrose solution	density	1.034	g/cm^3	g/mL	kg/m^3
7Ag85Cu8Sn brazing alloy	density	4.80	oz/cu in	g/mL	kg/m^3

value	units	value	units	value	units	value	units
1.6093	km/mi	1000	g/kg	1	mL/cm^3	10000	ppm/%
28.3495	g/oz	100	cm/m	1000	mL/L	1000	ppm/‰
16.3871	mL/cu in	1000	mm/m	1000	L/m^3		

Table 1.A.2

analysis	density (g/mL)	value	units	Convert to:	
nitrate in water	1.000	1.9	ppm	wt %	M
NaCl in seawater	1.025	30.0	‰	ppm	M
acetic acid in vinegar	1.010	5.5	% by wt	ppm	M
ethanol in beverage	1.020	4.2	% (v/v)	wt %	M

species	formula wt		density	
NO_3^-	62.005	g/mol		
NaCl	58.443	g/mol		
CH_3COOH	60.052	g/mol		
C_2H_5OH	46.068	g/mol	0.789	g/mL

You-Try-It 1.B Precision

1. Calculate the wt-% for each measurement in Table 1.B.1.
   Find the mean, standard deviation, and %-RSD.

2. Column E contains formulas for statistical results for up to 20 data points in Table 1.B.2.
   Write formulas to calculate the variance and standard error.
   Use the table of *t* values to the right to find the 90 and 99 % confidence levels.
   Verify your results using Data -> Data Analysis -> Descriptive Statistics.

Table 1.B.1 Protein analysis of extra crunchy peanut butter.

Trial	Sample wt (g)	Protein wt (g)	wt-%
1	9.91	1.92	19.37
2	10.17	2.04	20.06
3	10.54	2.09	19.83
4	10.01	2.05	20.48

mean:	%
std dev:	%
relative standard deviation:	%-RSD

Table 1.B.2 Descriptive Statistics

Trial	Data
1	3.44
2	3.11
3	2.98
4	3.27
5	3.03
6	
7	
8	
9	
10	
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	

	Statistics
N =	5
sum =	15.830
mean =	3.166
std dev =	0.188
%-RSD =	
variance =	
std error =	
90-% C.L. =	
95-% C.L. =	0.234
99-% C.L. =	

t values
2.776

	Table of *t* values for given alpha levels					
	0.2	0.1	0.05	0.025	0.01	0.001
N-1	(80%)	(90%)	(95%)	(97.5%)	(99%)	(99.9%)
1	3.078	6.314	12.706	31.821	63.657	636.600
2	1.886	2.92	4.303	6.965	9.925	31.590
3	1.638	2.353	3.182	4.541	5.841	12.920
4	1.533	2.132	2.776	3.747	4.604	8.610
5	1.476	2.015	2.571	3.365	4.032	6.869
6	1.44	1.943	2.447	3.143	3.707	5.959
7	1.415	1.895	2.365	2.998	3.5	5.408
8	1.397	1.86	2.306	2.896	3.355	5.041
9	1.383	1.833	2.262	2.821	3.25	4.781
10	1.372	1.812	2.228	2.764	3.169	4.587
15	1.341	1.753	2.131	2.602	2.947	4.073
20	1.325	1.725	2.086	2.528	2.845	3.850
25	1.316	1.708	2.068	2.485	2.787	3.725
30	1.31	1.697	2.068	2.457	2.75	3.646
50	1.299	1.676	2.068	2.403	2.678	3.496
100	1.29	1.66	2.068	2.364	2.626	3.391
infinity	1.31	1.645	2.068	2.326	2.576	3.300

You-Try-It 1.C Discarding an Outlier

1. Determine which data value in Table 1.C.1 is a potential outlier and calculate Q. Determine if the outlier may be discarded at the 95-% or 99-% confidence levels.

2. Repeat using Peirce's criterion.

Table 1.C.1 Calcium Potentiometry

Trial	ISE Result (mV)		Results	
1	39.8	N =	5	Q_c (95 %) = 0.710
2	36.5	sum =	195.0	Q_c (99 %) = 0.821
3	39.9	mean =	39.00	
4	39.2	std dev =	1.42	
5	39.6			

Dixon Q-test calculation

	value	deviation	closest value	Dixon's Q	Q_c (95 %)	Q_c (99 %)
min =						
max =						

Peirce's criterion

	value	|deviation|	R	d_{max}	result
min =					
max =					

Critical values of Dixon's *Q* parameter (Q_c)

N	95%	99%
3	0.970	0.994
4	0.829	0.926
5	0.710	0.821
6	0.625	0.740
7	0.568	0.680
8	0.526	0.634
9	0.493	0.598
10	0.466	0.568
15	0.384	0.475
20	0.342	0.425
25	0.317	0.393
30	0.298	0.372

suspect may be rejected if $Q > Q_c$

A more complete list of Q_c values is in:
David B. Rorabacher,
"Statistical treatment for rejection of deviant values: critical values of Dixon's "Q" parameter and related subrange ratios at the 95% confidence level,"
Anal. Chem ., **1991**, *63* (2), 139-146; DOI: 10.1021/ac00002a010.

Values of *R* for Peirce's Criterion

	Number of doubtful observations			
N	1	2	3	4
3	1.196			
4	1.383	1.078		
5	1.509	1.200		
6	1.610	1.299	1.099	
7	1.693	1.382	1.187	1.022
8	1.763	1.453	1.261	1.109
9	1.824	1.515	1.324	1.178
10	1.878	1.570	1.380	1.237
11	1.925	1.619	1.430	1.289
12	1.969	1.663	1.475	1.336
13	2.007	1.704	1.516	1.379
14	2.043	1.741	1.554	1.417
15	2.076	1.775	1.589	1.453
20	2.209	1.914	1.732	1.599
25	2.307	2.019	1.840	1.709

suspect may be rejected if |deviation| > d_{max}

A more complete list of *R* values is in:
Stephen M. Ross,
"Peirce's criterion for the elimination of suspect experimental data,"
Journal of Engineering Technology, Fall 2003.

You-Try-It 1.D Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Tables 1.D.1 and 1.D.2 contain data sets from two different fluorescence experiments.
The fluorescence signal is directly proportional to analyte concentration for both data sets.
The blank measurements are for pure solvent.

1. Determine the S/N, LOD, and LOQ for the measurement in Table 1.D.1.

2. Correct the measurements in Table 1.D.2 for the nonzero background and plot. Determine the LOD and LOQ.

Table 1.D.1. Repetitive fluorescence measurements.1

trial	blank (cps)	standard (cps)
1	753	1667287
2	620	1670694
3	703	1670408
4	620	1668215
5	576	1927720
average		
std dev		

standard concentration:	10.0	ppm
	1000	ppb/ppm
3x noise =		cps
10x noise =		cps
calibration =		cps/ppb
LOD:		ppb
LOQ:		ppb

Table 1.D.2. Fluorescence signal as a function of concentration.2

conc (mM)	corrected signal (mV)	measured signal (mV)	blank signal (mV)
0.300	0.581	0.581	0.006
0.095	0.173	0.173	0.003
0.030	0.059	0.059	0.006
0.009	0.029	0.029	0.005
0.003	0.010	0.010	0.006
0.000	0.007	0.007	0.004
		average	
		std dev	

3x noise =		
10x noise =		
slope =		
LOD:		mM
LOQ:		mM

1. The fluorescence signal is counts per second (cps) from a photomultiplier tube detector and pulse counter.
2. The signal is a voltage from a photon detector and lock-in amplifier.

You-Try-It 1.E Calibration

Acetylsalycilic acid (aspirin or ASA) can be measured using UV light absorption (see procedure).
The measured absorbance is directly proportional to ASA concentration: $A \propto c_{ASA}$.
Table 1.E.1 lists absorbance measurements for a standard solution and two unknowns.

1. Calculate the ASA concentration in the unknown samples using a simple proportionality.

2. Generate a two-point calibration curve using 0,0 and the standard measurement.
   Calculate the ASA calculation in the unknown samples using the calibration curve.

Table 1.E.1

	absorbance	blank	conc (M)
standard	0.363	0.000	5.00E-05
Sample 1	0.222	0.000	
Sample 2	0.311	0.043	

	conc (M)	absorbance
blank	0.00E+00	0.000
Standard	5.00E-05	0.363

Measurement Procedure

Crush an analgesic tablet and weigh ≈ 0.1 g to four places on an analytical balance.
Dissolve in deionized water and allow time for all of the ASA to dissolve (do not heat).
Filter to remove the starch binder and wash with water.
Dilute to a known volume with 0.05 M HCl.
Measure the absorbance of the solution at 227 nm.

A standard ASA solution of 5.0×10^{-5} M in 0.05 M HCl has an absorbance of 0.363 at 227 nm.

Sample 2 exhibited a faint turbidity (cloudiness). It was remeasured at a wavelength where ASA does not absorb to provide an approximate absorbance due to the turbidity
This measurement is listed as the blank measurement for Sample 2.

You-Try-It 1.F **Standard Addition**

Table 1.F.1 gives a set of data for lead analysis using an electrochemical method.
The dependence is linear, i.e., electrochemical current is proportional to analyte concentration.

1. Determine the unknown concentration using a proportionality calculation.

2. Determine the x-intercept for the data set, which equals the unknown concentration.

Table 1.F.1

std addition (µM)	**signal (µA)**	**c_{unk} (µM)**
0.0	2.4	
2.5	5.2	
5.0	8.2	
7.5	11.0	
	average:	µM
	std dev:	

data adapted from:
Andrew J. Saterlay , Shelley J. Wilkins and Richard G. Compton
"Towards greener disposal of waste cathode ray tubes via ultrasonically enhanced lead leaching"
Green Chem., 2001, 3, 149 - 155, DOI: 10.1039/b102671m
http://www.rsc.org/ej/GC/2001/b102671m/