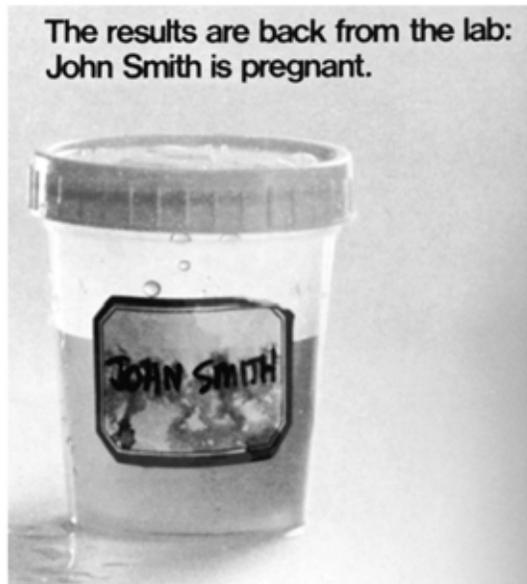


Chapters 3 and 4

Experimental Error



Experimental Error

All measurements have error

precision

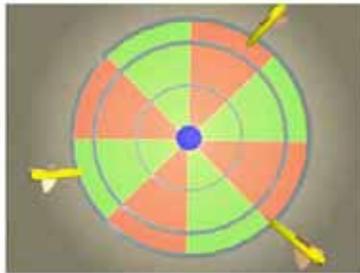
- reproducibility

accuracy

- nearness to the “true value”
- Data with unknown quality is **useless!**

Accuracy

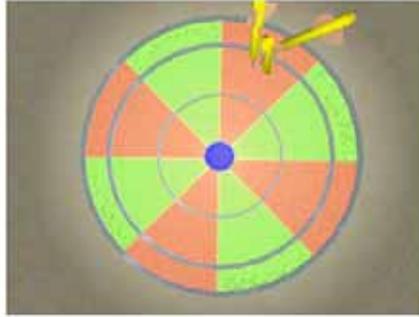
How close our
'average value'
is to the correct
one.



4

Precision

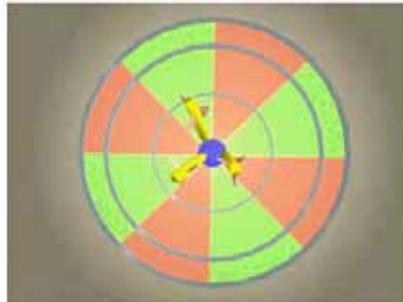
How close our values agree to each other.



5

Accuracy and Precision

Our goal. Accurate values that agree well with each other.



6

3.1 Significant Figures

"The number of **significant figures** is the minimum number of digits needed to write a given value in scientific notation without loss of accuracy."

Counting Significant Figures

Rules for determining which digits are significant

1. All non-zero numbers are significant.
2. Zeros between non-zero numbers are significant.
3. Zeros to the right of the non-zero number **and** to the right of the decimal point are significant.
4. Zeros before non-zero numbers are **not** significant.

Significant Figures

When reading the scale of any apparatus, you should interpolate between the markings. It is usually possible to estimate to the nearest tenth of the distance between two marks.

Significant Figures in Arithmetic

Exact numbers

conversion factors,
significant figure rules do
not apply

Significant Figures in Arithmetic

Addition and Subtraction

For addition and subtraction, the number of significant figures is determined by the piece of data with the fewest number of decimal places.

$$\begin{array}{r} 4.371 \\ 302.5 \\ \hline 306.8 \end{array}$$

11

Significant Figures in Arithmetic

Multiplication and Division

For multiplication and division, the number of significant figures used in the answer is the number in the value with the fewest significant figures.

$$\begin{array}{r} (2075) * (14) \\ \hline 10^2 \quad \quad \quad = 2.0 \times \\ \quad \quad \quad (144) \end{array}$$

12

Significant Figures in Arithmetic

Logarithms and Antilogarithms

logarithm of n :

$$n = 10^a \quad \Leftrightarrow \log n = a$$

n is the antilogarithm of a

$$\log 339 = 2.530$$

2 => character

.530 => mantissa

13

Significant Figures in Arithmetic

Logarithms and Antilogarithms

The number of significant figures in the **mantissa** of the logarithm of the number should equal the number of significant figures in the number.

14

Significant Figures in Arithmetic

Logarithms and Antilogarithms

The **character** in the logarithm corresponds to the exponent of the number written in scientific notation.

15

Significant Figures in Arithmetic

Logarithms and Antilogarithms

The number of significant figures in the antilogarithm should equal the number of digits in the **mantissa**.

$$\begin{array}{ccccccc} \text{antilog}(-3.42) & = & 10^{-3.42} & = & 3.8 \times 10^{-4} \\ | & & | & & | \\ 2 \text{ s.f.} & & 2 \text{ s.f.} & & 2 \text{ s.f.} \end{array}$$

16

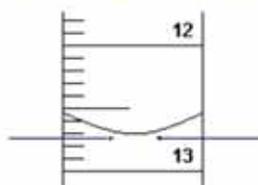
Significant Figures and Graphs

The rulings on a sheet of graph paper should be compatible with the number of significant figures of the coordinates.

In general, a graph must be at least as accurate as the data being plotted. For this to happen, it must be properly scaled. Contrary to "popular belief", a zero-zero origin of a graph is very **rare**.

17

MEASUREMENT WITH A BURET



Read the bottom of meniscus at eye level.

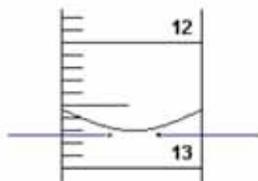
$12.75 \pm 0.01\text{ml}$

Four significant figures

The liquid level is always read at the bottom of the meniscus for transparent liquids

18

MEASUREMENT WITH A BURET



Read the bottom of meniscus at eye level.

$12.75 \pm 0.01\text{ml}$

Four significant figures

The number of significant figures in a measurement is the number of digits that are known with certainty plus the last one that is not absolutely certain

19

1. Using a ruler that has divisions which mark every mm, which of the following would be an appropriately recorded length?

- a) 45.02 mm
- b) 45 mm
- c) 45.02 m
- d) 45.0 mm

20

2. How many significant figures are understood in the following recorded measurement?

0.007805400 g

- a) ten
- b) four
- c) seven
- d) five

21

3. In the following recorded measurement, which digit(s) are estimated?

34.096 g

- a) the 6
- b) the 9 and the 6
- c) the zero
- d) all of them

22

4. When adding 0.67 g to 5.009 g, the proper answer would be

a) 5.679 g

b) 5.68 g

c) 5.6 g

d) 5.7 g

23

5. When dividing 3.09 g by $.85 \text{ cm}^3$, the correct answer would be

a) 3.6352941 g/cm^3

b) 3.64 g/cm^3

c) 4.0 g/cm^3

d) 3.6 g/cm^3

24

Rounding and Truncating Numbers

Round numbers with digits less than '5' down and numbers with digits greater than '5' up (some people round exactly '5' up and some round it down).

25

$$7.799 \text{ g} - 6.25 \text{ g} = 1.549 \text{ g} = 1.55 \text{ g}$$

A student finds the mass of a crucible three times and determines the mass to be 7.34 g, 7.35 g, and 7.38 g. How should the average mass be reported, keeping in mind the rules for operations with significant digits?

- a) 7.35666666 g
- b) 7.36 g
- c) 7 g
- d) 8.45 g

26

3.2 Types of Error

Error - difference between your answer and the 'true' one.

Systematic - problem with a method, all errors are of the same size, magnitude and direction - **Determinate errors**.

Random - based on limits and precision of a measurement - **Indeterminate errors**.
Can be treated statistically.

Blunders - you screw up. Best to just repeat the work.

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3.2 Types of Error

Systematic Error (determinate error) The key feature of systematic error is that, with care and cleverness, you can detect and correct it.

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Systematic or determinate errors

1. **Instrument errors** - failure to calibrate, degradation of parts in the instrument, power fluctuations, variation in temperature, etc.
2. **Method errors** - errors due to no ideal physical or chemical behavior - completeness and speed of reaction, interfering side reactions, sampling problems
3. **Personal errors** - occur where measurements require judgment, result from prejudice, color acuity problems

.

29

Potential Instrument Errors

Variation in temperature

Contamination of the equipment

Power fluctuations

Component failure

All of these can be corrected by calibration or proper instrumentation maintenance.

.

30

Method Errors

Slow or incomplete reactions

Unstable species

Nonspecific reagents

Side reactions

These can be corrected with proper method development.

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Personal Errors

Misreading of data

Improper calibration

Poor technique/sample preparation

Personal bias

Improper calculation of results

These are blunders that can be minimized or eliminated with proper training and experience.

32

The Effect of Systematic Error - normally "biased"

and often very "reproducible".

1. **Constant errors** - E_s is of the same magnitude, regardless of the size of the measurement.

This error can be minimized when larger samples are used. In other words, the relative error decreases with increasing amount of analyte.

$$E_r = (E_s/X_t) \times 100\%$$

Constant

eg. Solubility loss in gravimetric analysis

eg. Reading a buret

2. **Proportional errors** - E_s increases or decreases with

increasing or decreasing sample size, respectively. In other words, the relative error remains constant.

Proportional

Typically a contaminant or interference in the sample

33

Detection of Systematic Method Errors

1. Analysis of standard samples

2. Independent Analysis: Analysis using a "Reference Method" or "Reference Lab"

3. Blank determinations

4. Variation in sample size: detects constant error only

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Random Errors

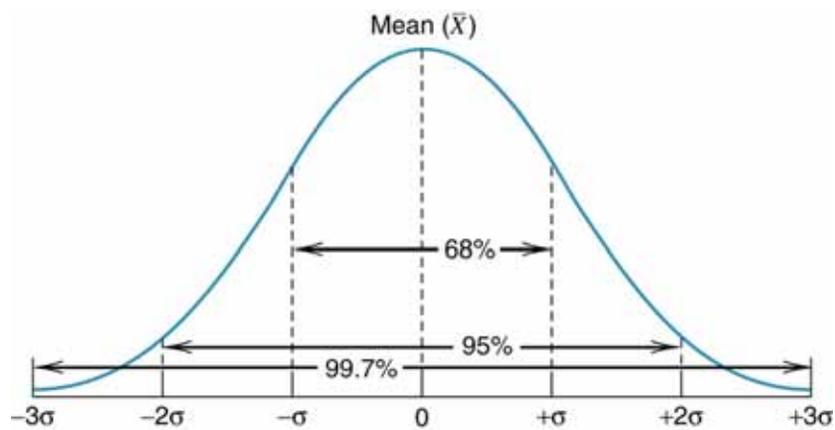
- caused by uncontrollable variables which normally **cannot** be defined
- The accumulated effect causes replicate measurements to fluctuate randomly around the mean.
- Random errors give rise to a normal or gaussian curve.
- Results can be evaluated using statistics
- Usually statistical analysis assumes a normal distribution

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Random errors follow a Gaussian or normal distribution.

We are 95% certain that the true value falls within 2σ (infinite population),
there is no systematic error.

IF



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Standard deviations from the mean

Fig. 3.2 Normal error curve.

Types of Error

Random Error (indeterminate error)

It is always present, cannot be corrected, and is the ultimate limitation on the determination of a quantity.

Types of Random Errors

- reading a scale on an instrument caused by the finite thickness of the lines on the scale
- electrical noise

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Systematic or determinate errors

affect accuracy!

Random or indeterminate errors

affect precision!

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1. Systematic errors affect _____

(a) accuracy (b) precision (c) none of these

2. Random errors affect _____

(a) accuracy (b) precision (c) none of these

39

3. A systematic error:

(a) can be discovered and corrected.

(b) arises from the limitations on the ability to make a physical measurement.

(c) is also known as an indeterminate error.

(d) All of above.

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Mean

Median

Precision: S, RSD, CV

Accuracy: Error, Relative Error

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Terms & Definitions

Replicates - two or more determinations on the same sample

Example: One student measures Fe (III) concentrations six times
The results are listed below:

19.4, 19.5, 19.6, 19.8, 20.1, 20.3 ppm (parts per million)

6 replicates = 6 measurements

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The "middle" or "central" value for a group of results:

➤ **Mean:** average or arithmetic mean

$$\bar{x} = \frac{\sum_{i=1}^N x_i}{N}$$

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The "middle" or "central" value for a group of results:

➤ **Mean:** average or arithmetic mean

$$\bar{x} = \frac{\sum_{i=1}^N x_i}{N}$$

➤ **Median:** the middle value of replicate data

44

The "middle" or "central" value for a group of results:

➤ **Mean:** average or arithmetic mean

$$\bar{x} = \frac{\sum_{i=1}^N x_i}{N}$$

➤ **Median:** the middle value of replicate data

- If an odd number of replicates, the middle value of replicate data
- If an even number of replicates, the middle two values are averaged to obtain the median

45

Calculation: Mean and Median

Example I: measurements of Fe (III) concentrations:
19.4, 19.5, 19.6, 19.8, 20.1, 20.3 ppm (parts per million)
What are the **mean** and **median** of these measurements

46

Calculation: Mean and Median

Example II: measurements of Fe (III) concentrations:

19.4, 19.5, 19.6, 19.8, 20.1 ppm (parts per million)

What are the mean and median of these measurements

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Calculation: Mean and Median

Example III: measurements of Fe (III) concentrations:

19.6, 19.4, 20.1, 19.5, 19.8 ppm (parts per million)

What are the mean and median of these measurements

48

Standard Deviation

standard deviation, small data set

$$s = \sqrt{\frac{\sum(x_i - x_{av})^2}{(n-1)}}$$

where $x_i - x_{av} \Rightarrow$ deviation from the mean
 $n \Rightarrow$ number of measurements
 $n - 1 \Rightarrow$ degrees of freedom

Standard Deviation

standard deviation, large data set

$$\sigma = \sqrt{\frac{\sum(x_i - \mu)^2}{(n)}}$$

where $\mu \Rightarrow$ population mean, most popular value

4-3 Standard Deviation of Calculated Results

Sample Standard Deviation

$$RSD = \frac{S}{X}, \text{ Relative Standard Deviation}$$

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4-3 Standard Deviation of Calculated Results

$$CV = \frac{S}{X} \times 100\% , \text{ Coefficient of Variation}$$

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4-3 Standard Deviation of Calculated Results

Standard Deviation of a Sum or Difference

$$y = a(\pm s_a) + b(\pm s_b) - c(\pm s_c)$$

$$s_y = \sqrt{s_a^2 + s_b^2 + s_c^2}$$

53

Estimate the absolute standard deviation and RSD.

$$y = 5.75(\pm 0.03) + 0.833(\pm 0.001) - 8.02(\pm 0.001)$$

54

1. Estimate the absolute standard deviation and RSD.

A measurement of unknown sample:

Original: 25.3322, 25.3325, 25.3325 g

Taking out some sample: 24.6228, 24.6230, 24.6231

Sample Weight=?

55

4-3 Standard Deviation of Calculated Results

Standard Deviation of a Product or Quotient

$$y = \frac{a \times b}{c}$$

$$\frac{s_y}{y} = \sqrt{\left(\frac{s_a}{a}\right)^2 + \left(\frac{s_b}{b}\right)^2 + \left(\frac{s_c}{c}\right)^2}$$

56

4-3 Standard Deviation of Calculated Results

Standard Deviation of a Product or Quotient

$$y = 251(\pm 1) \times \frac{860(\pm 2)}{1.673(\pm 0.006)}$$

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4-3 Standard Deviation of Calculated Results

Standard Deviation of a Product or Quotient

A Na_2CO_3 was used to standardize HCl solution, a soda sample was determined by the HCl solution. Calculate the content of Na_2CO_3 in sample and the RSD value upon following result.

Weight of Na_2CO_3 : 0.2000g, 0.2002g, 0.2005g

Volume of HCl used to standardization: 15.25, 15.26, 15.28 ml

Volume of HCl for the titration of 1.000g sample is 20.05ml, 1.208 g sample is 24.06 ml, 1.122 g sample is 22.09 ml, respectively.

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Pooled Data

To achieve a value of s which is a good approximation to σ , i.e. $N \geq 20$, it is sometimes necessary to *pool* data from a number of sets of measurements (all taken in the same way).

Suppose that there are t small sets of data, comprising N_1, N_2, \dots, N_t measurements. The equation for the resultant sample standard deviation is:

$$S_{pooled} = \sqrt{\frac{\sum_{i=1}^{N_1} (X_i - \bar{X}_1)^2 + \sum_{i=1}^{N_2} (X_i - \bar{X}_2)^2 + \sum_{i=1}^{N_3} (X_i - \bar{X}_3)^2 + \dots}{N_1 + N_2 + N_3 + \dots - t}}$$

(Note: one degree of freedom is lost for each set of data)

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Standard Error of a Mean

The standard deviation relates to the probable error in a *single* measurement. If we take a series of N measurements, the **probable error of the mean is less than the probable error of any one measurement.**

The **standard error of the mean**, is defined as follows:

$$s_m = \frac{s}{\sqrt{N}}$$

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Pooled Standard Deviation

Analysis of 6 bottles of wine for residual sugar.

Bottle	Sugar % (w/v)	No. of obs.	Deviations from mean
1	0.94	3	0.05, 0.10, 0.08
2	1.08	4	0.06, 0.05, 0.09, 0.06
3	1.20	5	0.05, 0.12, 0.07, 0.00, 0.08
4	0.67	4	0.05, 0.10, 0.06, 0.09
5	0.83	3	0.07, 0.09, 0.10
6	0.76	4	0.06, 0.12, 0.04, 0.03

$$s_1 = \sqrt{\frac{(0.05)^2 + (0.10)^2 + (0.08)^2}{2}} = \sqrt{\frac{0.0189}{2}} = 0.0972 = 0.097$$

and similarly for all s_n .

Set n	$\sum (x - \bar{x})^2$	s_n
1	0.0189	0.097
2	0.0178	0.077
3	0.0282	0.084
4	0.0242	0.090
5	0.0230	0.107
6	0.0205	0.083
Total	0.1326	

$$s_{pooled} = \sqrt{\frac{0.1326}{23-6}} = 0.088\%$$

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4-3 Standard Deviation of Calculated Results

Standard Deviation of Exponential calculations

$$y = a^x$$

$$\frac{s_y}{y} = x \left(\frac{s_a}{a} \right)$$

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4-3 Standard Deviation of Calculated Results

Standard Deviation of logarithms and antilogarithms

$$y = \log a$$

$$s_y = 0.434 \frac{s_a}{a}$$

$$y = \text{antilog } a$$

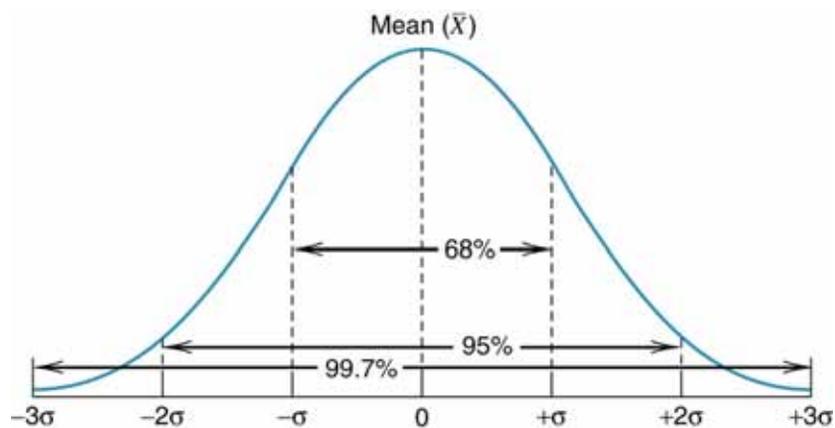
$$\frac{s_y}{y} = 2.303 s_a$$

63

Random errors follow a Gaussian or normal distribution.

We are 95% certain that the true value falls within 2σ (infinite population),
there is no systematic error.

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Standard deviations from the mean

Fig. 3.2 Normal error curve.

Define some terms:

CONFIDENCE LIMITS

interval around the mean that probably contains μ

CONFIDENCE INTERVAL

the magnitude of the confidence limits

CONFIDENCE LEVEL

fixes the level of probability that the mean *is* within the confidence limits

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$$\text{CL for } \mu = \bar{x} \pm \frac{z\sigma}{\sqrt{N}}$$

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Values of z for determining Confidence Limits

Confidence level, %	z
50	0.67
68	1.0
80	1.29
90	1.64
95	1.96
96	2.00
99	2.58
99.7	3.00
99.9	3.29

Note: these figures assume that an excellent approximation to the real standard deviation is known.

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Confidence Limits when σ is known

Atomic absorption analysis for copper concentration in aircraft engine oil gave a value of $8.53 \mu\text{g Cu/ml}$. Pooled results of many analyses showed $s \rightarrow \sigma = 0.32 \mu\text{g Cu/ml}$. Calculate 90% and 99% confidence limits if the above result were based on (a) 1, (b) 4, (c) 16 measurements.

<p>(a)</p> $90\% \text{ CL} = 8.53 \pm \frac{(1.64)(0.32)}{\sqrt{1}} = 8.53 \pm 0.52 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.5 \mu\text{g/ml}$</p> $99\% \text{ CL} = 8.53 \pm \frac{(2.58)(0.32)}{\sqrt{1}} = 8.53 \pm 0.83 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.8 \mu\text{g/ml}$</p>	<p>(b)</p> $90\% \text{ CL} = 8.53 \pm \frac{(1.64)(0.32)}{\sqrt{4}} = 8.53 \pm 0.26 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.3 \mu\text{g/ml}$</p> $99\% \text{ CL} = 8.53 \pm \frac{(2.58)(0.32)}{\sqrt{4}} = 8.53 \pm 0.41 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.4 \mu\text{g/ml}$</p>
<p>(c)</p> $90\% \text{ CL} = 8.53 \pm \frac{(1.64)(0.32)}{\sqrt{16}} = 8.53 \pm 0.13 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.1 \mu\text{g/ml}$</p> $99\% \text{ CL} = 8.53 \pm \frac{(2.58)(0.32)}{\sqrt{16}} = 8.53 \pm 0.21 \mu\text{g/ml}$ <p>i.e. $8.5 \pm 0.2 \mu\text{g/ml}$</p>	

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Values of t for various levels of probability

Degrees of freedom (N-1)	80%	90%	95%	99%
1		3.08	6.31	12.7
2		1.89	2.92	4.30
3		1.64	2.35	3.18
4		1.53	2.13	2.78
5		1.48	2.02	2.57
6		1.44	1.94	2.45
7		1.42	1.90	2.36
8		1.40	1.86	2.31
9		1.38	1.83	2.26
19		1.33	1.73	2.10
59		1.30	1.67	2.00
∞		1.29	1.64	1.96

- Note:**
- (1) As $(N-1) \rightarrow \infty$, so $t \rightarrow z$
 - (2) For all values of $(N-1) < \infty$, $t > z$, I.e. greater uncertainty

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$$\text{CL for } \mu = \bar{x} \pm \frac{ts}{\sqrt{N}}$$

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Confidence Limits where σ is not known

Analysis of an insecticide gave the following values for % of the chemical lindane: 7.47, 6.98, 7.27. Calculate the CL for the mean value at the 90% confidence level.

$x_i, \%$	x_i^2
7.47	55.8009
6.98	48.7204
7.27	52.8529

$$\Sigma x_i = 21.72$$

$$\Sigma x_i^2 = 157.3742$$

$$\bar{x} = \frac{\Sigma x_i}{N} = \frac{21.72}{3} = 7.24$$

$$s = \sqrt{\frac{\Sigma x_i^2 - \frac{(\Sigma x_i)^2}{N}}{N-1}} = \sqrt{\frac{157.3742 - \frac{(21.72)^2}{3}}{2}} = 0.246 = 0.25\%$$

$$90\% \text{ CL} = \bar{x} \pm \frac{t_s}{\sqrt{N}} = 7.24 \pm \frac{(2.92)(0.25)}{\sqrt{3}} = 7.24 \pm 0.42\%$$

If repeated analyses showed that $s \rightarrow \sigma = 0.28\%$:

$$90\% \text{ CL} = \bar{x} \pm \frac{z\sigma}{\sqrt{N}} = 7.24 \pm \frac{(1.64)(0.28)}{\sqrt{3}} = 7.24 \pm 0.27\%$$

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Terms & Definitions

➤ **Accuracy** - the closeness of the measurement to the true or accepted value.

This "closeness" called as the **error**:

absolute or **relative error** of a result to its **true value**.

➤ **absolute error**

➤ **relative error**

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Absolute Uncertainty

- same units as measurement

absolute uncertainty = your value
- true value

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Absolute and Relative Errors

➤ **Absolute Error** (E) - the difference between the **experimental value** and **the true value**. Has **a sign** and **experimental units**:

$$E = x_i - x_t$$

Experimental value - true (acceptable) value

➤ **Relative Error** (E_r) - the absolute error corrected for the size of the measurement or expressed as the fraction, %, or parts-per-thousand (ppt) of the true value. E_r has a sign, but no units.

$$E_r = \frac{x_i - x_t}{x_t} \times 100\%$$

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Enter text, numbers, or formulas in specific cells.

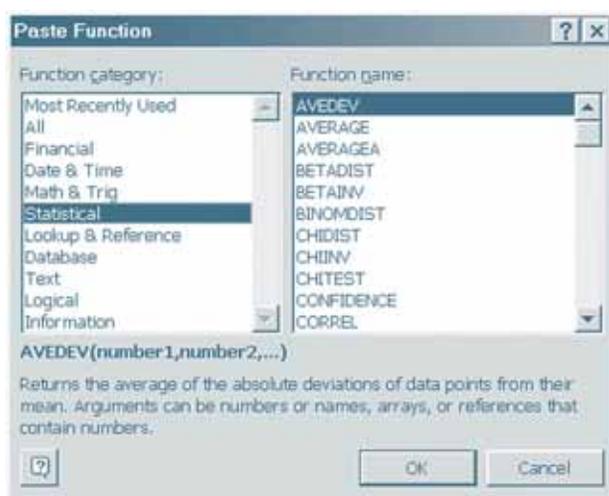
	A	B	C	D	E
1	A1	B1	C1	etc.	
2	A2	B2	C2	etc.	
3	A3	B3	C3	etc.	
4					
5					

Fig. 3.3. Spreadsheet cells.

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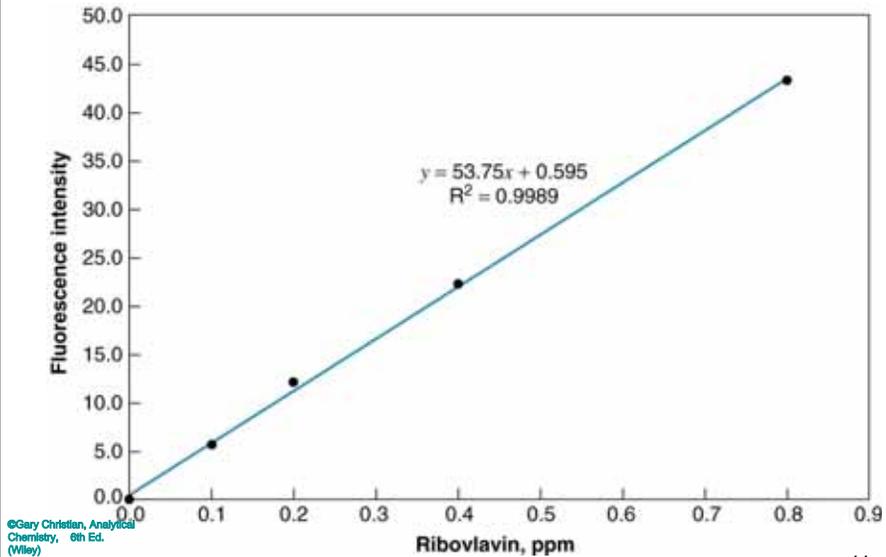
Excel has a number of mathematical and statistical functions.
Click on f_x on the tool bar to open the Paste Function.



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This Excel plot gives the same results for slope and intercept as calculated in the example.



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Fig. 3.8. Least-squares plot of data from Example 3.21.

Chart Wizard is on your tool bar (the icon with vertical bars).
Select XY (Scatter) for making line plots.



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You may insert the graph within the data sheet (Sheet 1), or a new Sheet 2.

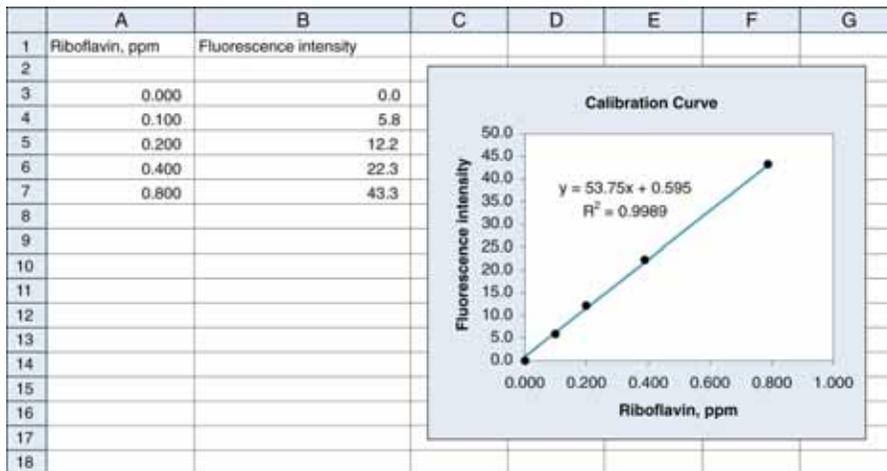


Fig. 3.9. Calibration graph inserted in spreadsheet (Sheet 1).

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Select LINEST from the statistical function list (in the Paste Function window – click on f_x in the tool bar to open).

LINEST calculates key statistical functions for a graph or set of data.

	A	B	C	D	E
1	Riboflavin, ppm	Fluorescence intensity			
2					
3	0.000	0.0			
4	0.100	5.8			
5	0.200	12.2			
6	0.400	22.3			
7	0.800	43.3			
8					
9	slope	53.75	0.595	intercept	
10	std. devn.	1.017759	0.419633	std. devn.	
11	R ²	0.998926	0.643687	std. error of estim.	
12	F	2789.119	3	d.f.	
13	sum sq. regr.	1155.625	1.243	sum sq. resid.	

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Fig. 3.10. Using LINEST for statistics.

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A "detectable" analyte signal would be 12 divisions above a line drawn through the average of the baseline fluctuations.

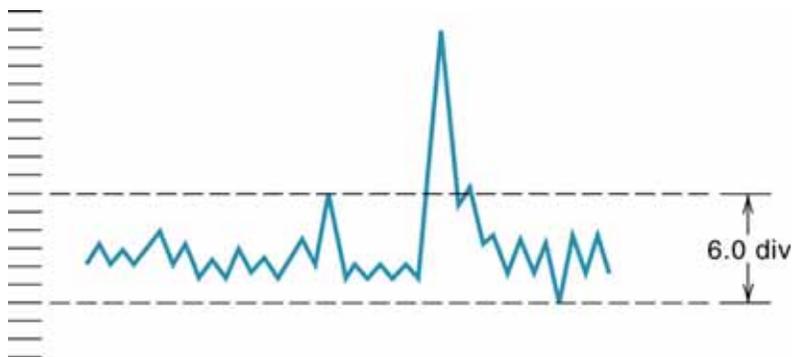


Fig. 3.11. Peak-to-peak noise level as a basis for detection limit.

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In-class calculations

In the nuclear industry, detailed records are kept of the quantity of plutonium received, transported, or used. Each shipment of plutonium pellets received is carefully analyzed to check that the purity and hence the total quantity is as the supplier claims. A particular shipment is analyzed with the following results: 99.93, 99.87, 99.91 and 99.86%. Is the shipment acceptable?

82

Various uses of student's t test

1) Comparing a measured result to a "true" value

2) Comparing replicate measurements

$$t = \frac{\bar{x} - \mu}{s_{pooled} \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

3) Comparing individual measurements

$$t = \frac{\bar{d} - \mu_d}{s_d \sqrt{\frac{1}{n} + \frac{\sum (d_i - \bar{d})^2}{n-1}}}$$

83

In-class calculations

An enzymatic method for determining alcohol in wine is evaluated by comparison with a gas chromatographic method. The same sample is analyzed several times by both methods with the following results (%ethanol).

Enzymatic method	GC-method
13.1	13.5
12.7	13.3
12.6	13.0
13.3	12.9

Does the enzymatic method give the same results as the GC method at the 95% confidence level?

84

In-class calculations

Your laboratory is evaluating the precision of a colorimetric method for creatinine in serum in which the sample is reacted with alkaline picrate to produce a color. Rather than perform one set of analyses, several sets with different samples are performed over several days, in order to get a better estimate of the precision of the method. From the following absorbance data, calculate the pooled standard deviation.

Day 1 (sample A)	Day 2 (B)	Day 3 (C)	
0.826		0.682	0.751
0.810		0.655	0.702
0.880		0.661	0.699
0.865			0.724

Averages are
Sample A: 0.845 B: 0.666 C: 0.719

85

4-4 Some Examples

1 The calcium in a 200.0 ml sample of a natural water was determined by precipitating the cation as $\text{Ca}_2\text{C}_2\text{O}_4$. The precipitate was filtered, washed and ignited in a crucible with an empty mass of 26.6002g. The mass of the crucible plus CaO was 26.7134 g. Calculate the concentration of Ca in the water in units of grams per 100ml.

Steps: Mass of CaO
Amount of Ca
Concentration of Ca

86

2 An iron ore was analyzed by dissolving a 1.1324 g sample in concentrated HCl. The resulting solution was diluted with water, and the iron(III) was precipitated as the hydrous oxide $\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}$ by the addition of NH_3 . After filtration and washing, the residue was ignited at a high temperature to give 0.5394 g of pure Fe_2O_3 . Calculate (a) the %Fe and (b) % Fe_3O_4 in the sample.

Procedures:

Amount of Fe_2O_3

Mass of Fe

%Fe

Mass of Fe_3O_4 ,

% Fe_3O_4

87

3. Iron in water was determined by spectroscopic method. The results of five measurements are 0.764 ppm, 0.758 ppm, 0.762 ppm, 0.759 ppm, 0.760 ppm.

Calculate the mean, median, S and RSD.

88

4. What is used to describe precision of measurements?

- (a) relative error (b) standard deviation (c) mean
- (d) medium (e) none of these

89

5. What is used to describe accuracy of measurements?

- (a) relative error (b) standard deviation (c) mean
- (d) medium (e) none of these

90

6. The mercury in samples of three fish taken from Chesapeake Bay was determined by a method based on the adsorption of radiation by gaseous elemental mercury. Calculate a pooled estimate of the standard deviation for the method.

Specimen	Hg, ppm
1	1.80, 1.58, 1.64
2	0.96, 0.98, 1.02, 1.10
3	2.06, 1.93, 2.12, 2.16, 1.89, 1.95

91

Sample weight (g)	Weight of the precipitate (g)		
	1.2206	0.1256	0.1260
1.2216	0.1259	0.1260	0.1258
1.2225	0.1262	0.1268	0.1270

$$S_{pooled} = \sqrt{\frac{\sum_{i=1}^{N_1} (x_i - \bar{x}_1)^2 + \sum_{j=1}^{N_2} (x_j - \bar{x}_2)^2 + \sum_{k=1}^{N_3} (x_k - \bar{x}_3)^2 + L}{N_1 + N_2 + N_3 + L - N_t}}$$



92

7. A soda ash sample is analyzed in the lab by titration with standard HCl solution. The analysis is performed in triplicate with the following results: 93.50, 93.58, 93.43% Na₂CO₃, within what range are you 95% confident that the true value lies?

93

8. A biochemist is beginning to prepare 100.0 mL of a solution to be labeled "0.900% (wt/vol) Sodium Chloride." To prepare this solution, the biochemist would:

- (a) Weigh 0.900 grams of sodium chloride into a beaker and add 100.0 mL of water to dissolve the sodium chloride.
- (b) Weigh 0.900 grams of sodium chloride into a container, add water to dissolve the sodium chloride, and then add water to the full scale mark of a 100 ml volumetric flasks.
- (c) Weigh 9.00 grams of sodium chloride into a container, dissolve the sodium chloride in water, and then add water to produce 100.0 mL of solution.

94

9. A solution is prepared by dissolving 25.8 grams of magnesium chloride (MgCl_2) in water to produce 250.0 mL of solution. Calculate the molarity of the chloride ion in the solution.

- (a) 0.271 Molar
- (b) 1.08 Molar
- (c) 2.17 Molar

95

10. The Great Salt Lake, located in the state of Utah, is approximately eight times saltier than the ocean. The salinity of the lake is said to occasionally be as high as 27,000 ppm sodium chloride. Calculate the molarity of the sodium ion in the Great Salt Lake.

- (a) 4.6×10^{-4} Molar Na^+
- (b) 0.46 Molar Na^+
- (c) 1.2 Molar Na^+

96

11. The ethyl alcohol content of many beers produced in the United States is 4.05% (vol/vol). If the density of ethyl alcohol at room temperature is 0.7893 grams per mL, what is the percent of ethyl alcohol in beer expressed as percent (wt/vol)?

- (a) 3.20% ethyl alcohol (wt/vol)
- (b) 5.13% ethyl alcohol (wt/vol)
- (c) 7.80 ethyl alcohol (wt/vol)

97

Questions

An analytical procedure required the preparation of a solution containing 100.0 ppm chromium. How many grams of potassium dichromate ($K_2Cr_2O_7$) would be required to prepare 1.000 liter of this solution?

- (a) 0.2829 g $K_2Cr_2O_7$
- (b) 0.1000 g $K_2Cr_2O_7$
- (c) 0.5658 g $K_2Cr_2O_7$

98

Questions

Calculate the formal concentration of a sodium acetate solution prepared by diluting 45.0 mL of a solution containing 25.0 ppm sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$) to a total of 0.500 liters of solution.

- (a) 6.10×10^{-4} M $\text{NaC}_2\text{H}_3\text{O}_2$
- (b) 1.37×10^{-6} M $\text{NaC}_2\text{H}_3\text{O}_2$
- (c) 2.75×10^{-5} molar $\text{NaC}_2\text{H}_3\text{O}_2$.

99

Questions

When concentrated sulfuric acid is sold to the chemist, the label contains no mention of the molarity of the acid. Instead, the label normally lists the concentration of sulfuric acid as a wt/wt percent and the density of the sulfuric acid solution. If the solution is 98.0 (wt/wt %) sulfuric acid, and has a density of 1.80 g/mL, calculate the molarity of concentrated sulfuric acid.

- (a) 18.0 Molar
- (b) 18.4 Molar
- (c) 10.0 Molar

100

Questions

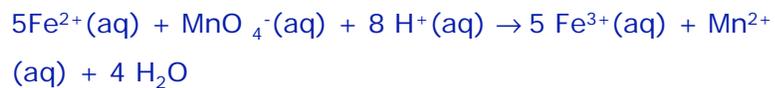
A solution was prepared by diluting 10.0 mL of 1.00×10^{-3} molar solution of potassium dichromate to 100.0 mL. What is the concentration of chromium in the new solution?

- (a) 10.4 ppm Chromium
- (b) 104 ppm Chromium
- (c) 5.20 ppm Chromium

101

Questions

In the analysis of iron in a local water well, 500.0 mL of the water sample was poured through a Jones reductor to convert the iron present to the Fe^{2+} oxidation state. The resulting sample required 1.05 mL of 0.011 molar KMnO_4 to react with the iron present. What is the wt/vol % of iron in the well water? The reaction is



- (a) $1.29 \times 10^{-4} \%$
- (b) 6.45 %
- (c) $6.45 \times 10^{-3} \%$

102

Questions

How many significant figures are in the number 6.230×10^{23} ?

- (a) 4
- (b) 3
- (c) 23

How many significant figures are in the number 0.000120?

- (a) 7
- (b) 3
- (c) 2

103

Questions

Write the answer to the following calculation to the proper number of significant figures.

$$3.86 + 9.1 - 0.231 = 12.7290$$

- (a) 12.7
- (b) 12.73
- (c) 12

Write the answer to the following calculation to the proper number of significant figures.

$$145.68 \times 1.09 \div 87,000 = 11,627.6697$$

- (a) 12,000
- (b) 11,600
- (c) 11,000

104

Questions

Define the term precision.

- (a) Precision refers to how close a measured value is to the "true" value.
- (b) Precision refers to how accurately a given measurement is made.
- (c) Precision is a measure of the reproducibility of a result.

105

Questions

Find the absolute uncertainty of the following calculation, and express the answer to the proper number of significant figures.

$$12.34(0.04) + 178.1(0.2) - 18.493(0.002) = 172.010$$

- (a) 172.01(0.2)
- (b) 172.0(0.2)
- (c) 172.010(0.242)

106

Questions

Find the absolute uncertainty of the following calculation:
 $3.78(.04) \times 6.23(.03) \times 10^{23} = 2.3549 \times 10^{24}$

and round the answer to the proper number of significant figures.

- (a) $2.3(.01) \times 10^{24}$
- (b) $2.35(.01) \times 10^{24}$
- (c) $2.35(.03) \times 10^{24}$

107

Questions

A systematic error:

- (a) can be discovered and corrected.
- (b) arises from the limitations on the ability to make a physical measurement.
- (c) is also known as an indeterminate error.

108

What mass of MgNH_4PO_4 precipitated when 200.0 mL of a 1.000% (w/v) solution of MgCl_2 were treated with 40.0 mL of 0.1753 M Na_3PO_4 and an excess of NH_4^+ ? What was the molarity of the excess reagent (Na_3PO_4 or MgCl_2) after the precipitation was complete?

109