Experimental programme

Practical tools · SL

When does a graph count as evidence?

A straight line can still be biased

An investigation needs a measurable question, appropriate methods, honest raw data and an uncertainty-aware conclusion.

This unit connects laboratory techniques, conservative uncertainty propagation, gradients and model testing. Numerical examples are invented.

Use these methods throughout supervised practical work and justify each model you use.

From a broad topic to a measurable question

Choose variables with an operational definition

Question: how does initial temperature affect the time for a specified magnesium strip to produce 20 cm^3 hydrogen in HCl of fixed concentration?

Independent variable: initial temperature. Dependent variable: elapsed time to the specified gas volume.

For a fixed collected volume, $1/t$ can compare average rates. It is not automatically an initial rate.

Explain controls and repeat choices

A list of constants needs reasons

Control acid concentration/volume and magnesium mass/exposed area: they can alter the reacting amount or rate.

Choose a measurable safe range; use a pilot to detect a reaction too fast to time or a gas leak.

Repeat each condition to judge scatter. Randomize condition order where useful to reduce drift; one condition with many repeats does not establish a trend.

Risk assessment follows the actual method

Hazard → exposure → control

Acid splash → eye/skin exposure → suitable eye protection and controlled handling.

Flammable solvent → ignition → approved heating and removal of flames. Follow apparatus limits and disposal instructions.

Plan waste minimization and environmental/ethical issues before collecting data.

Choose volume equipment by purpose

Instrument	What it measures or delivers
Volumetric pipette	One specified delivered volume
Burette	Variable delivered volume: final minus initial
Volumetric flask	Specified total contained volume at its mark
Beaker	Approximate volume, usually unsuitable for precise concentration

Other quantities need their own measurement model

Instrument choice is more than resolution

Tare a stable balance, keep vessels dry and record the displayed precision. Define timing start/finish events and avoid scale parallax.

Use temperature probes consistently and allow stabilization; a fast sensor can still lag or have a calibration offset.

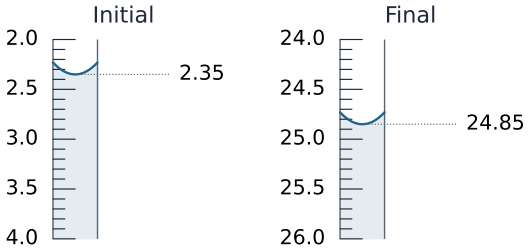
Calibrate a pH probe with suitable buffers. Measure current with an ammeter in series and potential difference with a voltmeter in parallel.

Precision does not establish accuracy

Idea	Interpretation
Random error	Unpredictable scatter; repeats help estimate it
Systematic error	Directional bias, including a calibration offset
Precision	Spread or resolution of measurements
Accuracy	Agreement with a suitable reference under matching conditions

Read both menisci at eye level

Magnified burette scales / cm³



Delivered volume = 24.85 − 2.35 = 22.50 cm³

Burette values increase downwards; delivered volume is a difference, not the final reading alone.

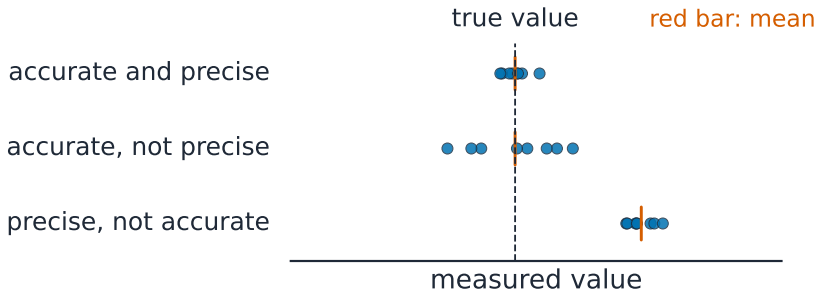
One titre, two reading uncertainties

1. **Invented readings.** Initial 2.35 $\pm$ 0.05 cm^3; final 24.85 $\pm$ 0.05 cm^3.
2. **Difference.** $V = 24.85 - 2.35 = 22.50$ cm^3.
3. **Conservative uncertainty.** $u_V = 0.05 + 0.05 = 0.10$ cm^3; report 22.50 $\pm$ 0.10 cm^3.

Decision

Use apparatus specifications and the stated method; do not assume this uncertainty for every burette.

Accuracy and precision



A **systematic error** spoils accuracy, not precision

Prepare a standard solution and a dilution

Conserve the solute amount

Use $n = cV$ and $m = nM$. Dissolve, transfer quantitatively, rinse into the volumetric flask, make up to its mark and mix.

Purity, hydration and water absorption matter: a precise mass alone does not guarantee an accurate concentration.

Dilution uses $c_1 V_1 = c_2 V_2$. Dilute an aliquot to the final flask volume, not by adding that entire volume of solvent.

Titration: the endpoint is an observation

The equivalence point is stoichiometric

Rinse burette with titrant and pipette with its solution. Use a clean conical flask, normally rinsed with deionized water.

Swirl and approach the endpoint dropwise. Choose an indicator changing in the appropriate steep region; some redox titrations are self-indicating.

Use a balanced equation. Keep a rough titre separate and state the rule for choosing concordant careful trials.

Separate an insoluble solid or purify a soluble product

Technique	Purpose and critical detail
Filtration	Retain insoluble solid; dissolved salt passes through
Recrystallization	Use suitable hot/cold solubility difference; wash with little cold solvent
Constant mass	Repeat suitable heat–cool–weigh cycle; cool before weighing
Separating funnel	Separate suitable immiscible liquid layers

Distillation, reflux and chemical evidence

Technique	Purpose and limitation
Distillation	Collect a condensed volatile component using volatility differences
Reflux	Return condensate during prolonged reaction; do not seal ordinary atmospheric apparatus
Chromatography	Compare migration under specified conditions; matching Rf alone does not prove identity
Melting range	Support purity assessment; not a complete structural identification

Measure chemical behaviour with a stated model

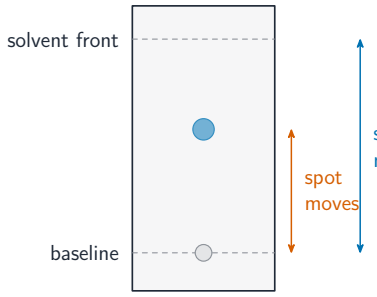
Connect the observable to the chemistry

Calorimetry uses heated mass and temperature change; account for heat exchange and apparatus heat capacity where relevant.

For cells, control electrolyte, electrode surface and temperature. A high-resistance voltmeter reduces current drawn in a potential measurement.

Colorimetry needs suitable wavelength, blank, consistent cuvettes and a justified calibration range. Physical/digital models represent connectivity and geometry under assumptions.

Thin-layer chromatography

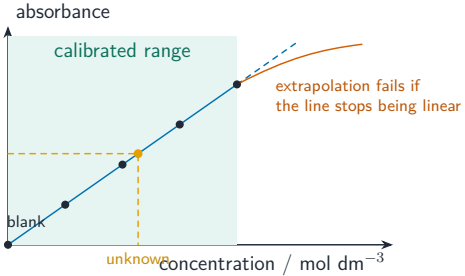


$$R_f = \frac{\text{distance moved by the spot}}{\text{distance moved by the solvent}}$$

always between 0 and 1

Compare R_f with a known in the same solvent

Stay inside the calibration range



Beyond it the true response curves away

Preserve the raw record before processing

A spreadsheet is not the original observation

Record original readings, units, apparatus uncertainties and qualitative changes. Keep consistent decimal places for the same instrument.

For databases retain source, conditions and filtering choices; for sensors record calibration and sampling settings.

Check spreadsheet formulae and units, preserve provenance, and distinguish simulated data from measurements.

Absolute, fractional and percentage uncertainty

1. **Absolute.** For $x \pm u_x$, absolute uncertainty has the same unit as x.
2. **Fractional.** Divide uncertainty by the magnitude of the value: $u_x/|x|$.
3. **Percentage.** Multiply the fraction by 100. A 22.50 ± 0.10 cm^3 titre has 0.44% uncertainty.

Decision

Percentage disagreement with a reference is a different calculation and can reflect systematic bias.

Three percentages, three denominators

1. **Given.** Measured mass 10.40 ± 0.10 g; accepted reference 10.00 g.
2. **Uncertainty.** $100(0.10/10.40) = 0.96\%$: divide by the measured value.
3. **Error magnitude.** $100|10.40 - 10.00|/10.00 = 4.0\%$: divide by the reference.
4. **Symmetric difference.** For two comparable measurements, divide their difference by their mean: $100(0.40/10.20) = 3.92\%$.

Decision

State the comparison convention; a small uncertainty does not prove accuracy.

Choose one propagation convention

Operation	**Conservative small-uncertainty estimate**
Addition/subtraction	Add absolute uncertainties
Product/quotient	Add fractional or percentage uncertainties
Exact coefficient	Scales absolute uncertainty, not fractional uncertainty
Shared errors	Consider correlations; a constant zero offset can cancel in a difference

Dilution: concentration and uncertainty

1. **Invented data.** Stock 0.2000 mol dm^{-3}, uncertainty 0.50%; pipette 25.00 $\pm$ 0.03 cm^3 to flask 250.0 $\pm$ 0.15 cm^3.
2. **Concentration.** $c_2 = c_1 V_1 / V_2 = 0.2000(25.00/250.0) = 0.02000$ mol dm^{-3}.
3. **Maximum percentage.** $0.50 + 100(0.03/25.00) + 100(0.15/250.0) = 0.68\%$.

Decision

Absolute uncertainty $\approx$ 0.00014 mol dm^{-3}; report 0.02000 $\pm$ 0.00014 mol dm^{-3} with two uncertainty digits.

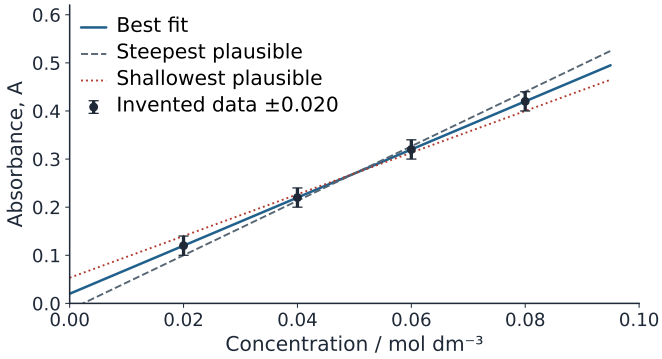
Titration concentration: keep the mole ratio visible

1. **Invented 1:1 reaction.** 25.00 $\pm$ 0.03 cm^3 NaOH; HCl titre 22.50 $\pm$ 0.10 cm^3 at 0.1000 mol dm^{-3} (0.20% uncertainty).
2. **Concentration.** $c_{\mathrm{NaOH}} = c_{\mathrm{HCl}} V_{\mathrm{HCl}} / V_{\mathrm{NaOH}} = 0.09000$ mol dm^{-3}.
3. **Maximum percentage.** $0.20 + 100(0.10/22.50) + 100(0.03/25.00) \approx 0.76\%$.

Decision

Absolute uncertainty $\approx$ 0.0007 mol dm^{-3}; final result 0.0900 $\pm$ 0.0007 mol dm^{-3}.

Constrain the line with all the bars



Invented calibration: the best, steepest and shallowest lines must be consistent with the full dataset.

Gradient units come from the axes

1. **Best-fit model.** $A = 5.00c + 0.020$, with concentration c in mol dm^{-3}; absorbance is dimensionless.
2. **Steepest.** $m_{\max} = (0.440 - 0.100)/(0.080 - 0.020) = 5.67$ dm^3 mol^{-1}.
3. **Shallowest.** $m_{\min} = (0.400 - 0.140)/(0.080 - 0.020) = 4.33$ dm^3 mol^{-1}.

Decision

Check intermediate bars too; two extreme endpoint choices alone are insufficient.

What the graphical uncertainty means

1. **Midpoint.** $m \approx (m_{\max} + m_{\min})/2 = 5.00\ \text{dm}^3\ \text{mol}^{-1}$.
2. **Half-range.** $u_m \approx (m_{\max} - m_{\min})/2 = 0.67\ \text{dm}^3\ \text{mol}^{-1}$.
3. **Boundary.** This range reflects the stated bars and straight-line model; it is not a formal statistical confidence interval.

Interpret a calibration fit without claiming accuracy

1. **Given.** A calibration fit reports $R^2 = 0.81$.
2. **Interpret.** The model accounts for 81% of response variation in this dataset; 19% remains in residual variation.
3. **Compare.** A second model with $R^2 = 0.94$ accounts for more of the same response variation.
4. **Evaluate.** Neither percentage measures accuracy or causation. Check the model, residual pattern and possible calibration bias.

Decision

A higher R^2 alone does not validate the measurement method.

Do not let a fitted line answer the wrong question

Interpret intercepts, residuals and range

Use labelled axes and a suitable scale. A gradient needs widely separated fitted-line points and units; a tangent gives an instantaneous gradient, a chord an average.

A nonzero intercept can indicate a blank signal, offset or incomplete model. Investigate rather than forcing the line through zero.

A high R^2 does not prove accuracy or causation. Inspect residuals and chemical plausibility; interpolation and extrapolation have different evidential support.

Build an evaluation from cause to improvement

The conclusion must answer the research question

Use processed data and uncertainty to state a supported trend. Separate an observation from its chemical explanation.

A small titre gives a large percentage reading uncertainty: a suitable larger aliquot may reduce it within approved limits.

Heat loss biases calorimetry: insulation or an appropriate time-series correction addresses it; repetition alone does not remove the bias.

An anomaly needs a reasoned decision

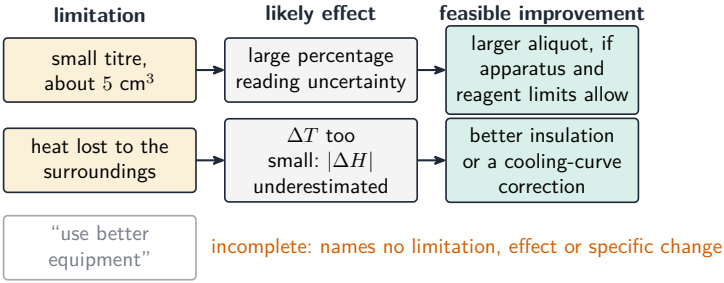
Keep the original data and decision trail

Inspect raw observations and apparatus, repeat if possible and justify inclusion or exclusion. Do not delete a value because it weakens a trend.

Compare with relevant references under matching conditions and distinguish accuracy, precision, reliability and validity.

Limit the conclusion to the supported measurement range and model assumptions.

Weakness, effect, improvement



Use a larger aliquot; insulate better

Try a transferred-mass and calibration problem

Work before checking

A container reads 6.237 $\pm$ 0.001 g before transfer and 2.104 $\pm$ 0.001 g after. Find transferred mass and conservative uncertainty.

Separately, use $A = 5.00c + 0.020$ to find concentration when A $=$ 0.270. What happens if you ignore the intercept?

Then attempt the handout questions on techniques, measurement and model evaluation.

Transferred-mass and calibration reasoning

1. **Difference.** $m = 6.237 - 2.104 = 4.133$ g; $u_m = 0.001 + 0.001 = 0.002$ g.
2. **Calibration.** $c = (A - 0.020)/5.00 = (0.270 - 0.020)/5.00 = 0.0500$ mol dm^{-3}.
3. **Interpret.** Ignoring the intercept assigns background absorbance to analyte and overestimates concentration.

Check yourself

Every number needs a measurement model

Reading or difference? Absolute or percentage? State the uncertainty convention and keep guard digits until final rounding.

Use units in tables, axes and gradients. Close repeats and high R^2 do not prove accuracy.

For evaluation, connect cause, consequence and a specific feasible improvement. Preserve raw data and justify exclusions.