

Experimental programme

International Baccalaureate · IB Diploma · Chemistry · HL

Scope and prerequisites

This handout teaches experimental planning, chemical measurement, uncertainty, graph interpretation and evaluation for the IB Chemistry experimental programme. Use these methods throughout supervised practical work. Sections marked **AHL** are for Higher Level only.

Prerequisites: mole and concentration calculations, balanced equations and straight-line graphs. All numerical datasets and investigations below are original, invented teaching examples. They illustrate methods rather than reporting real measurements.

Design a question that measurements can answer

A useful research question specifies the independent variable, dependent variable and chemical system. “How does temperature affect chemistry?” is too broad. “How does initial temperature affect the time for a specified magnesium strip to produce 20 cm³ of hydrogen in hydrochloric acid of fixed concentration?” identifies quantities that can be measured. Temperature is the independent variable; elapsed time is the dependent variable. For the same collected volume, $1/t$ can compare average rates, but it is not automatically an initial rate.

Identify control variables and explain their effects: acid concentration and volume, magnesium mass and exposed area, apparatus geometry and mixing. “Keep everything the same” gives neither a method nor a reason. Select a range wide enough to reveal a trend while keeping the process measurable and the apparatus within approved limits. A pilot trial can reveal a reaction too fast to time or gas leakage before the full investigation.

Use repeated trials at each condition to estimate scatter and judge reliability. Randomizing the order of conditions can reduce the influence of drift, such as gradual room-temperature change. Repeats at one concentration do not replace a range of concentrations. Continuous variables can take intermediate values; a discrete variable such as the number of identical pieces takes separate countable values.

A risk assessment connects a hazard, exposure route and control: acid splashes → eye/skin exposure → appropriate eye protection and controlled handling; flammable solvent → ignition → suitable heating and removal of flames. Follow the teacher’s apparatus limits and disposal instructions. Plan waste minimization and any environmental or ethical issues before collecting data.

Choose and use measuring equipment

Choose the instrument for the quantity and required precision, not merely the nearest container.

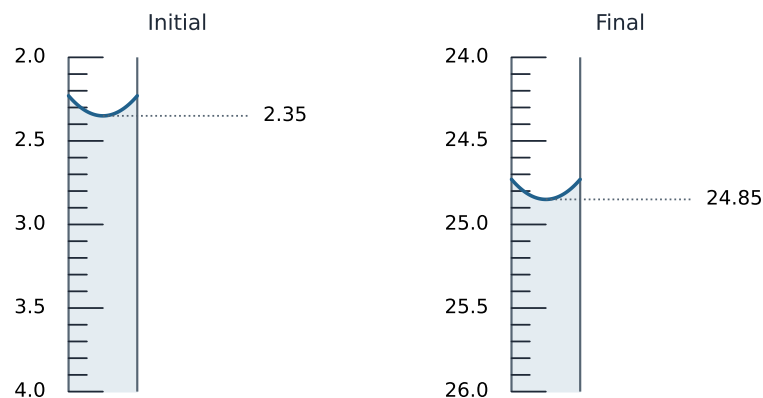
- **Mass:** tare a stable balance; keep the vessel dry and record its displayed precision. Weighing by difference subtracts the final container mass from its initial mass.
- **Volume:** a volumetric pipette delivers one specified volume; a burette delivers a variable volume by subtracting readings; a volumetric flask contains a specified total volume at its mark. A beaker is normally unsuitable for precise concentration preparation.
- **Time, temperature and length:** define the start/finish event, immerse a temperature probe consistently without touching the vessel, and avoid parallax when reading a scale. A fast sensor can still have a calibration offset or response lag.
- **pH:** calibrate an appropriate probe using suitable buffers, rinse between samples and allow stabilization. A pH reading is not a direct measurement of acid concentration for every chemical system.
- **Electrical quantities:** an ammeter measures current in series; a voltmeter measures potential difference in parallel. Choose a suitable range and account for electrode/contact conditions when comparing cells.

A **systematic error** shifts measurements consistently, for example a calibration offset. Random error produces unpredictable scatter. Repetition can reduce uncertainty in a mean caused by random variation, but it does not remove a shared offset. A gas leak or heat loss may create a directional bias without being a simple constant zero error.

Accuracy concerns agreement with the appropriate reference; precision concerns the spread or resolution of measurements. Closely agreeing repeats can all be inaccurate. A reference comparison must use matching conditions and a trustworthy reference value.

■ Read a burette as a difference

For a clear aqueous solution with a concave meniscus, read the bottom at eye level. Burette values increase downwards. Record initial and final readings to the precision supported by the scale and stated method; do not assume the initial reading is zero. Check that the tip is filled and free of air bubbles before starting.

Magnified burette scales / cm³

$$\text{Delivered volume} = 24.85 - 2.35 = 22.50 \text{ cm}^3$$

Delivered volume = final reading minus initial reading; read the meniscus consistently at eye level.

For the invented readings 2.35 ± 0.05 and $24.85 \pm 0.05 \text{ cm}^3$,

$$V = 24.85 - 2.35 = 22.50 \text{ cm}^3,$$

$$u_V = 0.05 + 0.05 = 0.10 \text{ cm}^3.$$

The conservative result is $22.50 \pm 0.10 \text{ cm}^3$. The uncertainty values are given for this example; consult the apparatus specification and method rather than assuming every burette has this uncertainty.

Chemical techniques: purpose and critical detail

■ Make a known concentration

A **standard solution** has a reliably known concentration. Calculate the solute amount and mass using $n = cV$ and $m = nM$. Dissolve the solid, transfer it quantitatively into a suitable volumetric flask, rinse the original vessel into the flask, then add solvent to the mark and mix thoroughly. Make the final adjustment at the appropriate temperature. Purity, hydration and the suitability of the solute as a standard matter; an accurately weighed impure or water-absorbing sample does not automatically give an accurate concentration.

For dilution, use $c_1V_1 = c_2V_2$ because solute amount is conserved, provided there is no reaction or loss. Transfer the aliquot with a pipette and dilute **to** the final flask volume, not by adding that entire volume of solvent. Never pipette by mouth.

In a titration, rinse a burette with its titrant and a pipette with the solution it will deliver; use a clean conical flask, normally rinsed with deionized water. Swirl, approach the endpoint dropwise and choose an indicator whose change occurs in the appropriate steep region. The observed endpoint is not the definition of the stoichiometric equivalence point. Acid–base and redox titrations both require a balanced equation; some redox systems are self-indicating. Keep a rough titre separate from the carefully obtained concordant trials and state the selection rule.

■ Separate, purify and characterize

- **Filtration** separates an insoluble solid from a liquid; it does not remove dissolved salt. A separating funnel separates suitable immiscible liquid layers.
- **Recrystallization** uses a suitable solvent in which the product is much more soluble hot than cold. Cool to crystallize, collect and wash with a little cold solvent; too much solvent can reduce recovery.
- **Drying to constant mass** means repeating a suitable heat–cool–weigh cycle until successive masses agree within the stated criterion. Cool before weighing; constant mass does not prove chemical purity or justify heating a decomposing compound more strongly.
- **Distillation** vaporizes and condenses a component for collection, using volatility differences. **Reflux** returns condensed vapour to the reaction vessel, allowing prolonged heating with less volatile material lost. Neither setup should be sealed during ordinary atmospheric heating.
- **Paper/TLC chromatography** compares movement of substances under specified conditions. Keep the sample above the initial solvent level; mark the solvent front promptly. R_f is distance moved by the spot from the baseline divided by distance moved by the solvent front from the same baseline. Matching R_f alone does not prove identity.
- **Melting-point determination** can support a purity assessment: impurities often depress and broaden a melting range, but comparison requires suitable heating and a relevant reference. A single melting point is not a complete structural identification.

■ Measure chemical behaviour

- **Calorimetry:** measure temperature change and the mass being heated; use insulation and account for apparatus heat capacity and heat exchange where relevant. The reaction and surroundings have opposite heat signs.
- **Electrochemical cells:** keep electrode surfaces, electrolyte conditions and temperature consistent; a high-resistance voltmeter reduces current drawn during a potential-difference measurement.

- **Colorimetry/spectrophotometry:** choose a suitable wavelength, use a blank and consistent clean cuvettes, and build a calibration using known concentrations. A calibration range must be justified; extrapolation outside it can fail if the relationship ceases to be linear.
- **Physical/digital molecular models:** use them to investigate connectivity and geometry, stating model assumptions and limitations. A ball-and-stick bond is a representation, not a rigid stick physically joining atoms.

Record raw data and propagate uncertainty

Keep original readings, units, apparatus uncertainties and qualitative observations. Put units and common uncertainties in column headings; use consistent decimal places for an instrument. Record unexpected changes and failed trials honestly. Preserve raw data before calculations or spreadsheet transformations. For database work, retain the source, conditions, units and any filtering choices; for sensors, record calibration and sampling settings.

An **uncertainty** quantifies a limitation on the result. Absolute uncertainty has the measured quantity's unit; fractional uncertainty is $u_x/|x|$, and percentage uncertainty is that fraction multiplied by 100. These are not the same as percentage disagreement with a reference.

For the conservative maximum-uncertainty method used here:

- In addition or subtraction, add absolute uncertainties: $u_{a\pm b} = u_a + u_b$.
- In multiplication or division, add fractional uncertainties: $u_y/|y| \approx u_a/|a| + u_b/|b|$ for $y = ab$ or a/b .
- A defined exact conversion or stoichiometric coefficient adds no measurement uncertainty. Multiplying a measured value by an exact coefficient scales its absolute uncertainty by the same factor, leaving its percentage uncertainty unchanged.

These are conservative first-order estimates for small uncertainties, not a claim that every error reaches its maximum simultaneously. Statistical combination of independent random standard uncertainties can instead use quadrature; do not switch between conventions halfway through an answer. Shared/correlated errors need separate treatment. Subtracting two readings from the same instrument can cancel a truly constant zero offset, even though the conservative reading-uncertainty sum is still used when requested.

■ Worked example: titration concentration

Use $25.00 \pm 0.03 \text{ cm}^3$ of NaOH and a titre of $22.50 \pm 0.10 \text{ cm}^3$ of HCl of concentration $0.1000 \text{ mol dm}^{-3}$ with 0.20% uncertainty. The balanced reaction is 1:1.

$$c_{\text{NaOH}} = \frac{c_{\text{HCl}} V_{\text{HCl}}}{V_{\text{NaOH}}} = \frac{0.1000 \times 22.50}{25.00} = 0.09000 \text{ mol dm}^{-3}.$$

Volumes share the same units, so their ratio is dimensionless. Maximum percentage uncertainty is

$$100 \frac{u_c}{c} = 0.20 + 100 \frac{0.10}{22.50} + 100 \frac{0.03}{25.00} \approx 0.76\%.$$

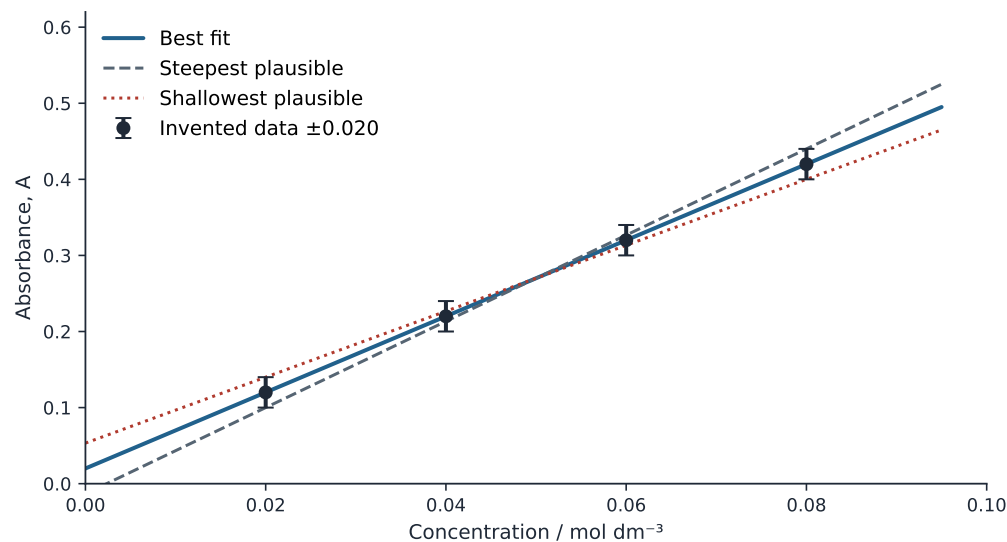
$$u_c = 0.09000 \times 0.00764 \approx 0.00069 \text{ mol dm}^{-3}.$$

A suitably rounded result is $0.0900 \pm 0.0007 \text{ mol dm}^{-3}$. Keep guard digits until this final rounding; quote the value and absolute uncertainty to the same decimal place.

Graphs, gradients and model testing

Put the independent variable on the horizontal axis and the dependent variable on the vertical axis, with quantities and units. Use a readable scale covering the useful data range; a plotted axis need not always start at zero. A qualitative sketch has labelled but unscaled axes. Scatter plots suit paired continuous measurements; bar charts compare categories, and histograms describe a continuous distribution grouped into intervals.

Plot justified uncertainty bars and a best-fit line or curve. Do not join every noisy point with straight segments and call the changing slopes a physical model. A gradient is $\Delta y/\Delta x$ using two widely separated points on the fitted line, not necessarily two measured points. Its unit is vertical-axis unit divided by horizontal-axis unit. A tangent gives an instantaneous gradient; a chord gives an average gradient.



Treat the full set of uncertainty bars as constraints; endpoint choices alone do not guarantee a plausible line.

In the invented calibration, concentrations 0.020, 0.040, 0.060 and 0.080 mol dm⁻³ give absorbances 0.120, 0.220, 0.320 and 0.420, each with ± 0.020 uncertainty; horizontal uncertainty is neglected. The best line is $A = 5.00c + 0.020$.

The steepest allowed line goes through (0.020, 0.100) and (0.080, 0.440); the shallowest through (0.020, 0.140) and (0.080, 0.400). Both also intersect the intervening bars:

$$m_{\max} = \frac{0.440 - 0.100}{0.080 - 0.020} = 5.67 \text{ dm}^3 \text{ mol}^{-1},$$

$$m_{\min} = \frac{0.400 - 0.140}{0.080 - 0.020} = 4.33 \text{ dm}^3 \text{ mol}^{-1}.$$

A symmetric gradient estimate is $m \approx (m_{\max} + m_{\min})/2 = 5.00$, with $u_m \approx (m_{\max} - m_{\min})/2 = 0.67 \text{ dm}^3 \text{ mol}^{-1}$. This graphical range describes the stated bars and straight-line model, not a formal statistical confidence interval.

A nonzero intercept may suggest a blank offset, a background signal or an incomplete model. Investigate it; do not automatically force the graph through the origin. An excellent R^2 describes how well a fitted model accounts for variation in the chosen response data; it does not prove accuracy, causation or the absence of systematic error. Even an exact line can contain a shared calibration bias. Inspect residuals, the data range and the chemistry too. Interpolation stays within the measured range; extrapolation extends beyond it and needs additional justification.

AHL: Powers and exponential models

For $y = x^n$ with an exact exponent and small uncertainty,

$$\frac{u_y}{|y|} \approx |n| \frac{u_x}{|x|}.$$

The magnitude matters: for $y = 1/x^2$, a 2% uncertainty in x gives about 4% in y , not -4%. Constants known exactly do not contribute. This first-order approximation becomes unreliable for very large fractional uncertainties or values near singularities.

An exponential model can be linearized. If $c = c_0 e^{-kt}$, then $\ln(c/c_0) = -kt$. Plotting the dimensionless logarithm against time gives gradient $-k$. For an invented $k = 0.0200 \text{ s}^{-1}$ and $c_0 = 0.1000 \text{ mol dm}^{-3}$, at 30.0 s:

$$c = 0.1000 e^{-0.0200(30.0)} = 0.0549 \text{ mol dm}^{-3}.$$

The half-time satisfies $1/2 = e^{-kt}$, so $t = \ln 2/k = 34.7 \text{ s}$. An approximately straight transformed plot supports the model over the tested range; it does not independently establish a chemical mechanism.

Conclude and evaluate

Answer the research question using processed data and uncertainty. Separate observations from explanations: “the temperature rose” is an observation; “the reaction transferred heat to the solution” is an interpretation supported by a model. Compare relevant references under matching conditions, and distinguish accuracy, precision, reliability and validity.

Do not delete a point because it weakens the trend. Inspect the raw record, repeat where appropriate and give a defensible reason for including or excluding an anomalous trial. Retain the original value and the decision. A spreadsheet trend line or simulation produces an output from inputs and assumptions; check units and formulae, preserve provenance and test whether the model represents the chemical system.

An evaluation should link a limitation to its likely effect and a feasible improvement. “Use better equipment” is incomplete. For a small titre, a suitable larger aliquot can reduce percentage reading uncertainty without changing chemical stoichiometry, provided apparatus and reagent limits allow it. For heat loss, better insulation or time-series correction addresses the bias; repeating the same poorly insulated method does not remove it.

Practice

Use conservative maximum uncertainty unless stated otherwise. All additional data are invented.

1. Turn “Does acid concentration affect reaction speed?” into a measurable research question. Identify independent/dependent variables and two justified controls.
2. A balance reads 6.237 ± 0.001 g before transfer and 2.104 ± 0.001 g after transfer. Find the transferred mass and its uncertainty.
3. A burette gives 2.35 ± 0.05 cm³ initially and 24.85 ± 0.05 cm³ finally. Calculate titre and percentage uncertainty. Explain why using only one reading uncertainty is wrong.
4. A temperature rises from 20.0 ± 0.1 °C to 26.0 ± 0.1 °C. Find the rise and percentage uncertainty. Does adding 273 to both readings change the rise?
5. A 0.2000 mol dm⁻³ stock has 0.50% uncertainty. Dilute 25.00 ± 0.03 cm³ to 250.0 ± 0.15 cm³. Calculate concentration and its maximum percentage uncertainty.
6. Use the titration worked example to calculate the NaOH concentration and uncertainty. What happens to the calculated concentration if an air bubble initially in the burette tip fills with titrant during the titre?
7. Identify a suitable technique to obtain an insoluble solid, purify a soluble crystalline solid, collect a volatile liquid, and heat a reacting mixture while returning vapour. Give one important distinction for each pair.
8. A calibration is $A = 5.00c + 0.020$, with c in mol dm⁻³. Find the concentration for $A = 0.270$. Explain why dividing absorbance by 5.00 without the intercept is wrong.
9. Recalculate the steepest and shallowest gradients in the calibration above and estimate the gradient uncertainty. Why must the lines be checked against intermediate bars too?
10. A chromatography spot moves 3.6 cm and the solvent front 6.0 cm from the baseline. Find R_f . Explain why a matching R_f under different conditions does not prove identity.
11. Three titres are 22.45, 22.50 and 22.55 cm³. Find mean and range. Explain why close agreement and a high calibration R^2 do not prove accuracy, and give a justified approach to an anomalous fourth titre.
12. **AHL.** For a cube edge $x = 2.00 \pm 0.02$ cm, estimate volume and uncertainty using the small-uncertainty power rule. State its approximate nature.
13. **AHL.** For $c = c_0 e^{-kt}$ with $c_0 = 0.1000$ mol dm⁻³ and $k = 0.0200$ s⁻¹, calculate c at 30.0 s and the half-time. State the gradient and unit of a plot of $\ln(c/c_0)$ against time.

■ Worked answers

1. For example: how does HCl concentration over a specified safe range affect the time for a fixed magnesium strip to produce 20 cm³ hydrogen at constant initial temperature? Independent: HCl concentration; dependent: elapsed time to that volume. Control magnesium mass/exposed area because reactive quantity and surface affect the rate; control temperature because it affects reaction rate. State

acid volume and apparatus too.

2. $m = 6.237 - 2.104 = 4.133$ g; $u_m = 0.001 + 0.001 = 0.002$ g. Report 4.133 ± 0.002 g.
3. $V = 24.85 - 2.35 = 22.50$ cm³; $u_V = 0.10$ cm³. Percentage uncertainty = $100(0.10/22.50) = 0.44\%$. Delivered volume depends on both readings, so both reading uncertainties enter the conservative difference estimate.
4. $\Delta T = 6.0 \pm 0.2$ K; percentage uncertainty = $100(0.2/6.0) = 3.3\%$. Converting both readings by adding the same Celsius-to-kelvin offset leaves their difference unchanged.
5. $c_2 = c_1 V_1/V_2 = 0.2000(25.00/250.0) = 0.02000$ mol dm⁻³. Maximum percentage uncertainty = $0.50 + 100(0.03/25.00) + 100(0.15/250.0) = 0.68\%$. Absolute uncertainty is $0.02000(0.0068) = 0.000136$ mol dm⁻³; with two uncertainty digits, report 0.02000 ± 0.00014 mol dm⁻³.
6. $c = 0.1000(22.50/25.00) = 0.09000$ mol dm⁻³; maximum uncertainty $\approx 0.76\%$, giving 0.0900 ± 0.0007 mol dm⁻³. Filling an initial tip bubble makes the indicated titre exceed liquid delivered into the flask; the calculated amount of acid and hence NaOH concentration is too high in this model.
7. Insoluble solid: filtration. Soluble crystalline product: recrystallization followed by collection/drying; filtration alone cannot remove dissolved impurities. Volatile liquid collection: distillation; prolonged reaction with condensate returned: reflux. Reflux does not collect the distillate as product outside the reaction vessel.
8. $c = (A - 0.020)/5.00 = (0.270 - 0.020)/5.00 = 0.0500$ mol dm⁻³. Ignoring the intercept assigns background absorbance to the analyte and overestimates concentration.
9. $m_{\max} = 0.340/0.060 = 5.67$; $m_{\min} = 0.260/0.060 = 4.33$ dm³ mol⁻¹. Midpoint 5.00 and half-range 0.67 dm³ mol⁻¹. An endpoint-extreme line might miss other bars; it must remain plausible for the whole dataset and model.
10. $R_f = 3.6/6.0 = 0.60$, dimensionless. Solvent, stationary phase and other conditions affect movement; different substances can also share an R_f . Compare under matched conditions with additional evidence.
11. Mean = $(22.45 + 22.50 + 22.55)/3 = 22.50$ cm³; range = $22.55 - 22.45 = 0.10$ cm³. A common calibration bias can preserve both precision and a strong linear fit. Inspect the fourth trial's raw observations and method, repeat if possible and justify any exclusion rather than deleting it because it weakens agreement.
12. **AHL.** $V = x^3 = 8.00$ cm³; fractional uncertainty $u_V/V \approx 3(0.02/2.00) = 0.030$. Thus $u_V \approx 0.24$ cm³, giving 8.00 ± 0.24 cm³ with two uncertainty digits. This is a first-order estimate for small uncertainty, not exact evaluation of both cubed bounds.
13. **AHL.** $c = 0.1000e^{-0.0200(30.0)} = 0.0549$ mol dm⁻³. $t_{1/2} = \ln 2/0.0200 = 34.7$ s. Since $\ln(c/c_0) = -kt$, gradient is -0.0200 s⁻¹.

Exam tips

- Distinguish a reading from a difference and an absolute uncertainty from a percentage. State which uncertainty convention you use.
- Record original values and units, then keep guard digits until final rounding. Precision of repeats is not proof of accuracy.
- A gradient needs units and a model; an intercept, residual pattern or anomaly needs investigation, not automatic deletion.
- Link each evaluation point to a direction or consequence and a specific feasible improvement. Answer the research question within the supported range.
- **AHL.** Multiply fractional uncertainty by the magnitude of an exact exponent; check the axes and sign when linearizing an exponential model.

Reference: IB Chemistry guide: tools and inquiry skills.