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Pre-IB Chemical Research Skills Programme

Chemical Research Skills

A Skills-Building Programme for Independent Chemical Investigation



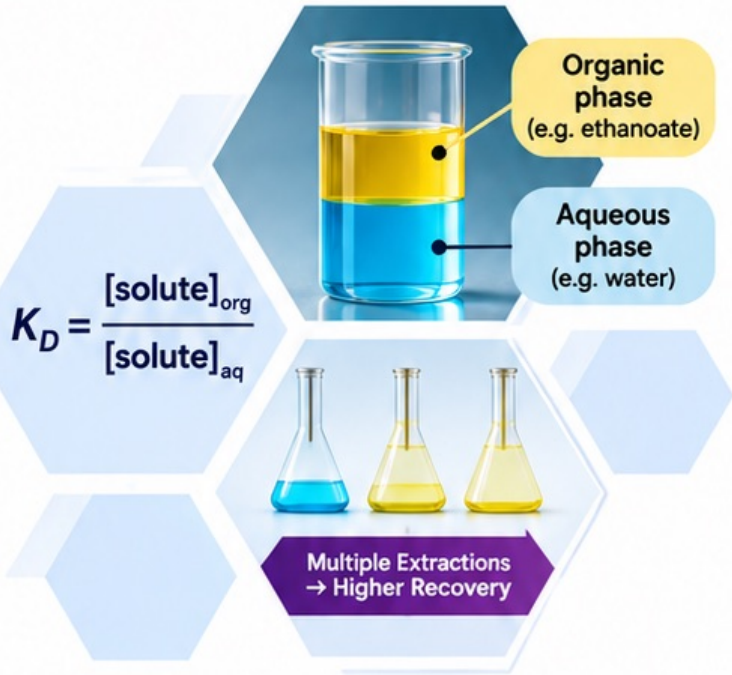
Module 6

Liquid–Liquid Extraction

Separation, Distribution and Recovery

In this module, you will:

- Understand the principle of liquid–liquid extraction
- Use the distribution coefficient (K_D) to rationalise extraction efficiency
- Design and perform an extraction procedure
- Calculate percentage recovery and evaluate results
- Identify limitations and suggest improvements



Sunny Chiam

IB Chemistry Specialist & Examiner-Level Educator
Founder, LearningSparks Education Services
Creator of the CHIAM Framework™

PREFACE

Learning to Think Like a Chemical Researcher

Pre-IB Chemical Research Skills Programme | Modules 1–10

Chemistry is more than the study of substances, reactions, and scientific principles. It is also a discipline built upon observation, questioning, experimentation, measurement, and evidence-based reasoning. For many students, learning chemistry begins with understanding concepts, applying equations, and performing laboratory experiments according to established instructions. These are essential foundations. However, there is an important difference between following an experimental procedure and understanding how to design one.

This distinction becomes particularly significant when students progress into the International Baccalaureate Diploma Programme (IBDP), where independent scientific investigation forms an important part of their learning experience. Students are increasingly expected to formulate meaningful research questions, select appropriate analytical techniques, justify experimental decisions, evaluate the quality of their measurements, and draw conclusions supported by scientific evidence. Yet these research skills do not necessarily develop simply through learning more chemistry content or completing more laboratory experiments. They require deliberate preparation.

The **Pre-IB Chemical Research Skills Programme** was developed to address this need.
Drawing upon my experience as an IB Chemistry educator and examiner, I have observed that students may possess a reasonable understanding of chemical concepts yet encounter considerable difficulty when asked to translate those concepts into an independent investigation.

A student may understand concentration but struggle to prepare a suitable series of standard solutions. Another may know the Beer–Lambert law but remain uncertain about constructing and interpreting a calibration curve. Others may be familiar with experimental procedures yet find it challenging to explain why a particular analytical technique is appropriate, how many measurements are necessary, or whether their results are sufficiently reliable to support a conclusion.

These difficulties are not always the result of insufficient chemical knowledge. Often, they reflect a missing connection between conceptual understanding and experimental research design. This programme seeks to build that connection.

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Organised into ten progressive modules, the programme introduces students to the essential thinking processes and practical skills involved in chemical research. Beginning with the formulation of research questions, students gradually develop their understanding of quantitative solution preparation, dilution, calibration, analytical techniques, standardisation, measurement reliability, and experimental planning.

Each module builds upon the skills established in the preceding modules, encouraging students to recognise that scientific research is not a collection of isolated laboratory techniques. Rather, it is a connected process in which every experimental decision must serve a clear scientific purpose.

Throughout the programme, particular emphasis is placed on a fundamental principle:
A sophisticated-looking experiment is not necessarily a good experiment. A simple experiment that is thoughtfully designed, carefully measured, and critically evaluated can be an excellent scientific investigation.

Students are therefore encouraged not merely to ask *what should I do?* but also *why should I do it this way?*

Why is a volumetric flask preferable to a measuring cylinder when preparing a standard solution? Why might several smaller extractions be more effective than a single extraction? Why is a calibration curve necessary? Why can repeated measurements still produce inaccurate results? How can we determine whether an experimental method is capable of answering a research question?

These questions represent the foundations of scientific reasoning.
Importantly, this programme is not intended to replace the IB Chemistry syllabus, nor is it a shortcut to completing an Internal Assessment (IA) or Extended Essay (EE). Instead, it provides an early introduction to the research skills that students will eventually need when undertaking more demanding independent investigations.

The intention is to help students enter the IB Diploma Programme with greater confidence, stronger experimental awareness, and a more structured approach to scientific problem-solving. By the end of these ten modules, students should begin to appreciate that successful chemical research does not start with selecting an impressive experiment or sophisticated instrument.

It begins with a meaningful question, followed by careful consideration of the evidence required to answer it.

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Ultimately, the purpose of this programme is not simply to teach students how to conduct experiments. It is to develop their ability to think independently, make scientifically justified decisions, and approach chemical investigations with clarity, confidence, and intellectual curiosity.That is where meaningful scientific research begins.

Sunny Chiam
IB Chemistry Specialist & Examiner-Level Educator
Founder, LearningSparks Education Services
Creator of the CHIAM Framework™
Think Chemistry. Think Structurally.

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Module 6 — Liquid–Liquid Extraction
Separating Chemicals Using Solubility & Partitioning
Learning objectives
By the end of this module, students should be able to:

- explain what liquid–liquid extraction (LLE) is
- distinguish between miscible and immiscible liquids
- explain why two liquid layers form
- relate molecular polarity and intermolecular forces to solubility
- explain why a solute distributes between two immiscible phases
- identify aqueous and organic layers
- explain why the organic layer is **not always the upper layer**
- use density information to identify layer position
- describe correct use of a separating funnel
- explain why a separating funnel must be vented
- explain the purpose of mixing and allowing layers to separate
- distinguish extraction from washing
- understand the partition/distribution coefficient concept
- explain why several smaller extractions can be more effective than one large extraction
- recognise common experimental problems such as emulsions
- design an extraction strategy appropriate to a research problem
- connect extraction to later chemical analysis

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1. Why do we need separation?
Real chemical samples are rarely pure.
A sample may contain:

Analyte + solvent + impurities + other dissolved substances

Suppose the compound we want to analyse is mixed with several unwanted substances.
Measuring the original mixture directly may give unreliable results.
One strategy is:

Separate first → Measure later

Liquid–liquid extraction is one method for doing this.

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2. What is liquid–liquid extraction?
Liquid–liquid extraction uses **two immiscible liquid phases** to separate substances according to their different affinities for the two phases.
A simple system may contain:

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- an **aqueous phase**
- an **organic phase**

and a solute that can distribute between them.

Aqueous phase ⇌ Solute ⇌ Organic phase

The solute does not necessarily move completely into one layer.
Instead, it usually **partitions** between the two.

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3. Miscible vs immiscible

Miscible liquids

Two liquids are miscible if they mix completely to form one homogeneous phase.
Example:

water + ethanol

Immiscible liquids

Two liquids are immiscible if they do not mix completely and form separate phases.
For example, under ordinary conditions:

water + hexane

forms two layers.
For LLE, the two chosen solvents must have sufficiently low mutual miscibility to form distinct phases.

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4. Why do the layers separate?

This is where chemistry matters.
Water molecules are highly polar and interact strongly through hydrogen bonding.
A non-polar organic solvent such as hexane interacts mainly through London dispersion forces.
Mixing the two would require disrupting favourable water–water interactions without replacing them with equally favourable water–hexane interactions.
As a result, the liquids have limited mutual solubility and separate into phases.
At Pre-IB level, a useful principle is:

"Like dissolves like"

but students should understand that this is a **guideline**, not a complete law.
More scientifically:
A substance tends to dissolve better in a solvent when the intermolecular interactions between solute and solvent are sufficiently favourable.

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5. Polarity and solubility

Consider two solutes.

Solute A

Highly polar and capable of strong interactions with water.

It is likely to have greater affinity for:

$$\boxed{\text{aqueous phase}}$$

Solute B

Mostly non-polar.

It may have greater affinity for:

$$\boxed{\text{organic phase}}$$

Therefore, differences in molecular properties can be used to separate substances.

This is the chemical foundation of LLE.

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6. A solute does not necessarily choose only one layer

A common misconception is:

"If the solute prefers the organic solvent, all of it moves into the organic layer."

Usually this is not true.

Instead, at equilibrium:

$$\boxed{X_{\text{aq}} \rightleftharpoons X_{\text{org}}}$$

Some X may remain in the aqueous phase while some is present in the organic phase.

The relative amounts depend on how strongly X favours each phase.

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7. Partitioning

Imagine a solute X is shaken with water and an immiscible organic solvent.

Initially:

$$X_{\text{aq}}$$

may dominate.

After mixing, some molecules move into the organic phase.

Eventually a dynamic equilibrium is established:

$$\boxed{X_{\text{aq}} \rightleftharpoons X_{\text{org}}}$$

Molecules continue moving between phases, but the concentration ratio becomes approximately constant under defined conditions.

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8. Partition coefficient

A simplified partition coefficient may be written:

$$\boxed{K = \frac{[X]_{\text{org}}}{[X]_{\text{aq}}}}$$

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for the same molecular form of X, under specified conditions.
If:

$$K > 1$$

the solute favours the organic phase.
If:

$$K < 1$$

the solute favours the aqueous phase.
If:

$$K \gg 1$$

extraction into the organic phase may be particularly effective.

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9. Example of partitioning

Suppose:

$$K = \frac{[X]_{\text{org}}}{[X]_{\text{aq}}} = 4.0$$

This means that at equilibrium:

$$[X]_{\text{org}} = 4[X]_{\text{aq}}$$

under the stated conditions.
It does **not** mean:

- exactly four times as many total moles must be in the organic layer regardless of phase volumes
- 100% of $X$is extracted
- four extraction steps are required

The **phase volumes also matter** when determining the amount of solute in each layer.

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10. Concentration is not the same as amount

Remember:

$$n = cV$$

Suppose:

$$[X]_{\text{org}} = 4[X]_{\text{aq}}$$

but the two phases have different volumes.
The number of moles in each phase depends on both:

concentration AND volume

Therefore, partition coefficient alone does not directly tell us the percentage extracted unless the phase volumes are known.

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11. Which layer is on top?

Students often learn:

"The organic layer is on top."

This is **not always true**.

Layer position depends primarily on **density**.

The less dense phase is normally above the denser phase.

For example:

Hexane

Density approximately:

$$0.66\ \text{g cm}^{-3}$$

Water:

$$\approx 1.00\ \text{g cm}^{-3}$$

Therefore:

hexane layer on top

But an organic solvent denser than water would normally form the **bottom layer**.

So:

Organic ≠ automatically top

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12. How can we identify the aqueous layer?

If uncertain, a simple experimental strategy can be used.

Add a small drop of water to the separating funnel.

Observe which layer the drop joins.

That layer is the:

aqueous layer

This is safer scientifically than guessing based on "organic = top".

Density data and solvent identity can also be used.

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13. The separating funnel

A separating funnel allows two immiscible liquid phases to be:

1. mixed
2. allowed to separate
3. physically separated

Important components include:

- stopper
- funnel body
- stopcock/tap

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- outlet

The basic process is:

Add phases → Mix → Vent → Allow separation → Separate layers

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14. Why do we mix the phases?

If the two layers simply sit separately, only a relatively small interfacial area exists between them.

Mixing temporarily creates much greater contact between the phases.

This allows solute molecules to transfer more effectively between them.

Therefore:

Mixing increases contact between phases

which helps the solute approach its partition equilibrium.

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15. Why must we vent?

Pressure can build inside a closed separating funnel.

Possible causes include:

- volatile solvents
- warming during mixing
- gas formation in some chemical systems

Therefore, the funnel should be vented appropriately during mixing.

Conceptually:

Mix → Vent → Mix → Vent

Students should never treat venting as an optional decorative step.

It is a **safety requirement**.

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16. Basic separating-funnel procedure

A simplified procedure:

1. Ensure the stopcock is closed.
2. Add the solution containing the solute.
3. Add the extraction solvent.
4. Insert the stopper.
5. Invert/mix appropriately.
6. Vent regularly and safely.
7. Continue controlled mixing.
8. Return the funnel to the stand.
9. Remove the stopper as appropriate before draining.
10. Allow the layers to separate clearly.
11. Identify the layers.

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12. Drain the lower layer through the stopcock.
13. Collect the upper layer separately.

The exact procedure depends on the chemicals and safety requirements involved.

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17. Never identify layers by colour alone

Suppose the desired compound is coloured.

Students may assume:

"The coloured layer must be the organic layer."

Not necessarily.

Colour tells us where the coloured species is concentrated.

It does **not** by itself identify whether that phase is aqueous or organic.

Layer identity should be based on:

- solvent identity
- density
- water-drop test where appropriate

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18. Extraction vs washing

These terms describe similar physical operations but different purposes.

Extraction

The desired compound is transferred into another phase.

Desired solute → new phase

Washing

Unwanted impurities are transferred away while the desired compound remains mainly in its current phase.

Impurities → wash phase

Therefore:

The apparatus may look similar, but the **purpose** is different.

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19. Why one extraction does not remove everything

Suppose a solute strongly favours the organic phase.

After extraction, some solute usually still remains in the aqueous phase because partition equilibrium is established.

Therefore:

One extraction ≠ complete extraction

This leads to an important research strategy:

Use multiple extractions.

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