ITFI Laboratory Work References

- Welcome to the laboratory session of the ITFI project! In this phase of the program, your team will analyze and test the samples collected during field investigations to acquire critical data for your final report. **Please assign two team members to lead this analytical work**. These members should have normal color vision (no color blindness or color weakness), and no serious allergies at present. Only these members will be allowed to enter the laboratory. This laboratory session is shared between Project I and Project II, and the same guidelines apply to both.

  The emphasis is on exploration and hands-on experience—we hope you enjoy your time in the lab!
- Physical and chemical analyses cannot replace your careful observation and description of the sample. *Sometimes, your eyes are the most powerful instrument.*
- Before using the laboratory, prepare your experimental plan using this appendix and any additional information you have gathered in advance.

  This guide outlines a total of 11 experiments. Each experiment is labeled with a **difficulty level**, using atmospheric layers (e.g., Troposphere, Stratosphere) to represent increasing complexity. **The higher the layer, the more challenging the task.** Please select experiments that align with your team's skills and objectives. Rest assured, **your choice of experiments will not impact the final evaluation.** Note that these outlines are not full protocols — some minor details may be omitted — but they include the necessary guidance and formulas.

  Alternatively, you may design your own procedures from scratch, without relying on the examples in this appendix, provided that safety requirements are fully met.

  You are encouraged to think creatively and design some basic procedures yourself, based on the instruments and reagents listed in this appendix, if they serve your investigation goals.

  The laboratory session is scheduled for **4 hours**. Please note that it will **not be possible** to complete all experiments listed in this appendix. You should select a subset of experiments that are most relevant to your project objectives. When planning your time, please allocate at least 1 hour for waiting periods when using shared instruments and common reagents.
- Please arrive at the laboratory **at least 15 minutes**, but no more than 30 minutes, before the scheduled start time. Upon arrival, sign in with the *Laboratory Record Sheet* (hereafter referred to as the *Sheet*) by the staff at the laboratory entrance. Collect personal protective equipment (PPE) and put it on properly. Entry into the laboratory is only allowed after passing the staff's inspection. **Remember to bring your ID badges and the samples your team collected to the laboratory.**

  Late arrivals will be recorded. Teams arriving more than 45 minutes after the scheduled start time will be disqualified from the laboratory session.

  Please wear long pants that cover the ankles, and closed-toe, flat, non-slip shoes. Inappropriate clothing or footwear includes, but is not limited to: shorts, skirts, dresses, sandals, slippers, high heels, roller shoes—and of course, the emperor's new clothes. Participants with long hair must tie it back securely.
- Upon entering the laboratory, locate your pre-assigned work bench and check that **all your exclusive instruments and non-controlled reagents are complete and undamaged**. If anything is missing or damaged, report it to a staff immediately (within 10 minutes of entry).

  Refer to the ***Instruments*** **and** ***Reagents*** provided at the end of this appendix. Note that certain instruments and reagents are not routinely supplied. To request these materials, fill out the *Sheet* and submit it to a staff.

  Remember to designate a container (e.g., the largest beaker) as **waste container**.
- During the experiment, carefully record raw data in the designated area on the reverse side of the *Sheet*. Data falsification or plagiarism is strictly prohibited.

  You are expected to strictly follow **laboratory safety rules** and **academic integrity policies**, use reagents responsibly and efficiently, and avoid interfering with the experiments of other teams. Any violations—whether safety-related or disciplinary—will be recorded by the lab staff and be reported, which **may affect your final score**. However, unless a violation occurs, this lab session (including but not limited to the accuracy or precision of your experimental results) **will not be graded.**

  We encourage (but do not require) teams working on the same project topic to **share data fairly and mutually**, and cite them properly. For example, exchanging results with teammates from your national team might be a good strategy. However, one-sided data sharing is discouraged.
- After completing the experiment, properly **dispose of waste**, check that **all non-consumable instruments and reagents are complete and intact**, return any borrowed instruments and reagents, return all instruments and reagents to their designated locations, clean the workspace, push in the chair; inform a staff when you have finished, wait for them to verify your station before leaving, and leave the laboratory only after the staff has verified everything and signed the *Sheet*.

  After leaving the laboratory, return your personal protective equipment to the staff.
- In case of any special circumstances, be sure to follow the instructions of the exam affairs staff.

Table of Contents

Laboratory Seating Arrangement

Room 105, the Examination Room

	Blackboard	Circuit Breaker
	Staff's Workstation (Public Instruments & Reagents)	Door #105

Window

WS Cambrian (Ꞓ)	**WS Ordovician (O)**	**WS Neogene (N)**	**WS Quaternary (Q)**
Instruments and Reagents for Ꞓ, O, S, and D		Instruments and Reagents for K, E, N, and Q	
WS Silurian (S)	**WS Devonian (D)**	**WS Cretaceous (K)**	**WS Paleogene (E)**
WS Carboniferous (C)	**WS Permian (P)**	**WS Triassic (T)**	**WS Jurassic (J)**
Instruments and Reagents for C, P, T, and J		Staff's Workstation	

Fire Extinguisher | Door #107

WS = Workstation

Workstation		Ꞓ	O	S	D	C	P	T	J	K	E	N	Q
PM, Aug. 13	Team	F	O	L	J	—	—	—	—	I	A	M	—
AM, Aug. 14		H	B	S	D	—	—	—	—	K	G	Q	—

Room 109, the Preparation Room

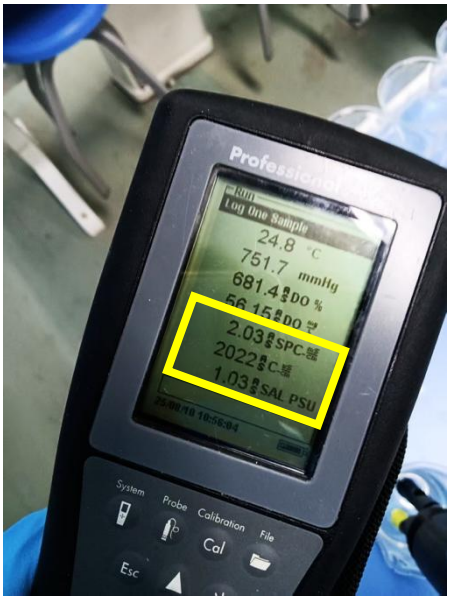
	Blackboard	Circuit Breaker
	Unused	Door #109

Window

Team A	**Team F**	**Team K**	Unused
Team B	**Team G**	**Team L**	**Team Q**
Unused	**Team H**	**Team M**	Unused
Team D	**Team I**	Unused	**Team S**
Unused	**Team J**	**Team O**	

Fire Extinguisher | Door #111

Experiment 1. Water Conductivity and Salinity Measurement



I. Principle

The laboratory will provide a YSI Pro Plus multiparameter meter for this experiment. It operates on electrochemical principles to measure key water quality parameters:

- Temperature (°C)
- Pressure (mmHg)
- Dissolved oxygen (DO, % or mg/L)
- Conductivity (SPC: µS/cm; C: mS/cm)
- Salinity (PSU, Practical Salinity Unit)

As samples are not measured in situ, primary attention should be given to conductivity and salinity data, which reflect ionic content and dissolved solids in water.

II. Main Reagents

Water samples your team have collected (max. **3 samples** per group).

III. Experimental Procedure

1. ***Pretreatment***

   Transfer water samples into clean beakers. Ensure samples are free of debris to avoid electrode contamination.

2. ***Measurement (Conducted by Staff)***

   The staff will test samples in seating order (front-to-back, left-to-right). Wait for readings to stabilize before recording data.

IV. Difficulty Level Ground

Experiment 2. XRF Elemental Testing

I. Principle

The laboratory will provide an OLYMPUS VANTA C series handheld XRF fluorescence spectrometer for this experiment. XRF spectroscopy works by irradiating a sample with high-energy X-rays, causing the sample's atoms to emit secondary (fluorescent) X-rays. Each element produces a unique spectral signature, allowing non-destructive, qualitative and quantitative analysis. For accurate measurements, the sample must have an approximately flat surface area of 3 cm × 3 cm.

II. Main Reagents

Soil or rock samples your team have collected (max. **3 samples** per group).

III. Experimental Procedure

1. ***Pretreatment***

   Adjust the sample shape to ensure a flat surface suitable for measurement.

2. ***Measurement (Conducted by Staff)***

   The staff will test samples in reverse order (back-to-front, right-to-left). The XRF instrument's display will show detected elements (above the detection limit) along with their concentrations.

   Note: *Maintain a safe distance from the X-ray gun aperture during testing to avoid direct exposure to X-rays.*

IV. Difficulty Level Ground

Experiment 3. Determination of Soil Dry Matter and Moisture Content

I. Principle

The soil sample is dried to constant weight at (105 ± 5) °C. The mass difference before and after drying is used to calculate the contents of dry matter and moisture, expressed as mass fractions.

II. Main Reagents

Soil samples your team have collected.

III. Experimental Procedure

1. ***Pretreatment***

   Take an appropriate amount of fresh soil sample and mix thoroughly. Remove visible debris such as stones and twigs larger than 2 mm in diameter. Set aside for use.

2. ***Gravimetric Determination***

   Note: *When removing items from the oven, you must* ***WEAR COTTON GLOVES*** *to avoid burns.*

   Weigh a beaker.

   Add 30–40 g of the pretreated fresh soil sample into the beaker. Weigh the beaker (with the soil). Place the beaker into the oven and dry the sample to constant weight.

   Seal the beaker with plastic wrap and allow it to cool to room temperature. Then remove the plastic wrap and weigh the beaker (with the dried soil) again.

IV. Difficulty Level Troposphere

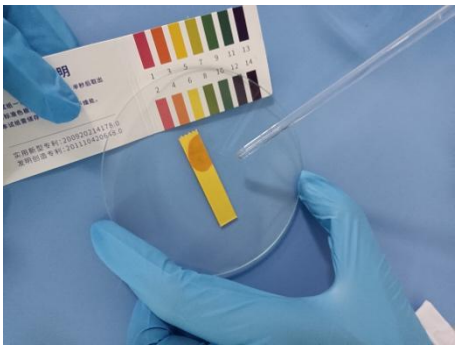
Experiment 4. Use of pH and Nitrate/Nitrite Test Strips

I. Principle

1. ***pH Test Strips***

   pH test strips are categorized into universal pH test strips and precision pH test strips.

   - **Universal pH test strips** cover a broad pH range (typically 1–14) and are suitable for rough estimation of a solution's acidity or alkalinity.
   - **Precision pH test strips** are designed for a narrower pH range (e.g., pH 0.5–5.0), allowing for more accurate pH determination, especially when higher precision is required.



2. ***Nitrate / Nitrite Test Strips***

   These test strips include separate detection zones for multiple parameters.

II. Main Reagents

pH test strips, nitrate/nitrite test strips, samples to be tested.

III. Experimental Procedure

1. ***Usage of pH Test Strips***

   (This is a translation of the instructions shown on the packaging of the pH test strips)

   Take the paper strip and immerse it in the solution to be tested, then take it out after half a second and compare it with the standard color chart to obtain the pH value.

   Note: *This test strip is not suitable for weak buffer solutions and acid-base solutions with concentrations lower than 0.01%, as well as solutions that interfere with coloration.*

2. ***Usage of Nitrate/Nitrite Test Strips***

   (This is a translation of the instructions shown on the packaging of the nitrate/nitrite test strips)

   Dip the test strip detection block completely into the liquid to be tested, then wait for 2 seconds before taking it out. Do not shake off excess moisture from the detection block.

   After standing for 60 seconds, compare the filter paper strip with the color card on the bottle to obtain the final test result.

IV. Difficulty Level Troposphere

Experiment 5. Determination of Soil pH

I. Principle

Soil colloids and minerals can adsorb or release H^+ and OH^- ions. After air-drying and grinding, soil is mixed with distilled water to form a suspension. The ions released into the water phase determine the pH of the suspension, which reflects the soil's natural acid-base characteristics.

II. Main Reagents

Distilled water, soil samples your team have collected.

III. Experimental Procedure

1. Pretreatment

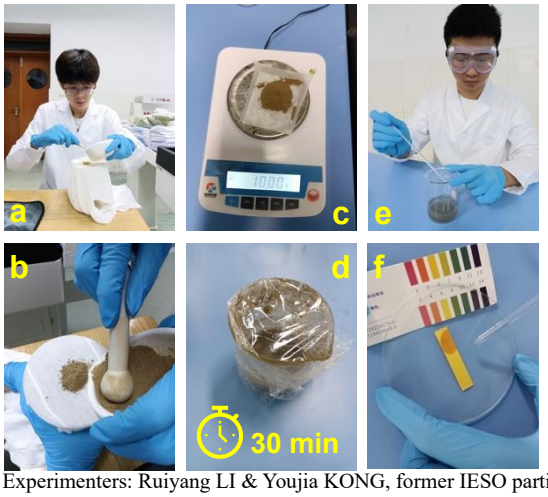
Air-dry the soil sample in a well-ventilated area. Remove visible debris such as gravel, roots, and plant material. Transfer the cleaned sample to a mortar and grind until no particles larger than 2 mm remain. Thoroughly mix to homogenize.

Note: *Air-drying typically takes 2–4 hours. If you plan to conduct this experiment, consider air-drying at least 20 g of soil sample* ***before arriving at the laboratory****, as only 4 hours will be available during the lab session.*

2. Preparation of Soil Suspension and pH Measurement

Weigh 10.0 g of pretreated soil into a beaker. Add 25 mL of distilled water. Cover the beaker with plastic wrap and shake vigorously for 2 minutes. Allow the suspension to stand undisturbed for 30 minutes.

Use suitable pH test paper to measure the pH of the supernatant (the upper layer of the suspension). Compare the test paper to the standard color chart to determine the pH value.



Experimenters: Ruiyang LI & Youjia KONG, former IESO participants

IV. Difficulty Level Troposphere

Experiment 6. Preparation of Soil Leachate

I. Principle

Extracting reagents dissolve soluble substances in soil to produce a leachate for further analysis. Different extracting reagents serve different purposes. You are encouraged to consult reliable sources to choose an appropriate reagent according to your objectives. For example, when an electrolyte solution such as $NaHCO_3$ is used to treat soil, the ions adsorbed on soil colloids can be replaced by ions in the extracting solution (e.g., Na^+). The replaced ions, along with soluble nutrients originally present in the soil solution, enter the leachate.

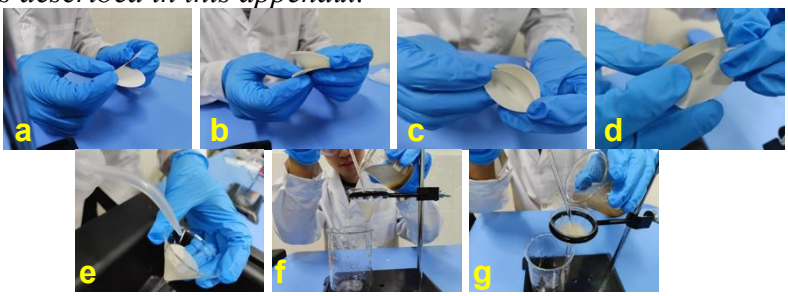
II. Main Reagents

Your chosen extracting reagent, soil samples your team have collected.

III. Experimental Procedure

Weigh an appropriate amount of soil (you may need to pretreat the sample, e.g., air-dry, grind, remove debris, and mix thoroughly), place it in a beaker, and add an appropriate volume of your selected extracting reagent. Stir thoroughly, then filter using filter paper. The filtrate is the soil leachate. To ensure complete extraction, you may rinse the residue with a small amount of extracting reagent and collect the rinsing as part of the leachate. Repeat the same for other leachates as needed.

Note: *You may treat the soil leachate as a water sample and conduct other experiments about water analysis described in this appendix.*

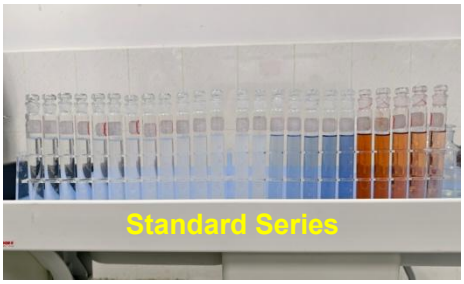


Experimenter: Tongyan ZHANG, former IESO participant

IV. Difficulty Level Stratosphere

Introduction to Visual Colorimetry

Visual colorimetry (including **visual turbidimetry**) is a simple and effective analytical method used to estimate the concentration of a substance in solution based on the intensity of its color. This method relies on the principle that the deeper the color of a solution, the higher the concentration of the colored substance—provided that all other conditions remain the same.



In colorimetric analysis, the substance being tested either has a natural color or reacts with specific reagents to form a colored compound. The sample's color is then visually compared to a series of **standard solutions** with known concentrations that have been prepared and treated in the same way. By matching the color intensity of the sample to the closest standard—or estimating between two standards—the concentration of the target substance can be determined.

This method follows the principle of **Beer's Law**, which states that the absorbance (or perceived color intensity) is proportional to the concentration of the colored species, as long as the path length and conditions are constant.

Experiment 7. Determination of Sulfate, Phosphorus, and Total Iron by Visual Colorimetry

I. Principle

1. *Determination of Sulfate*

Sulfate ions (SO_4^{2-}) react with barium chloride ($BaCl_2$) to form a white precipitate of barium sulfate ($BaSO_4$):

$$SO_4^{2-} + Ba^{2+} \equiv\equiv BaSO_4\downarrow \text{ (white)}$$

The precipitate causes turbidity in the solution. The degree of turbidity is proportional to the sulfate concentration. By comparing the turbidity with a standard series, the sulfate content in the sample can be estimated.

Carbonate ions (CO_3^{2-}) can also react with barium ions to form a white precipitate of barium carbonate ($BaCO_3$), which interferes with sulfate detection.

Barium carbonate dissolves in hydrochloric acid, so adding hydrochloric acid can eliminate this interference.

$$CO_3^{2-} + Ba^{2+} \equiv\equiv BaCO_3\downarrow \text{ (white)}, \qquad BaCO_3 + 2H^+ \equiv\equiv Ba^{2+} + CO_2\uparrow + H_2O$$

2. *Determination of Phosphorus*

Inorganic phosphorus in the test sample is oxidized to phosphate (PO_4^{3-}) by nitric acid. Phosphate ions (PO_4^{3-}) react with ammonium molybdate $\{(NH_4)_2MoO_4\}$ under acidic conditions to form a yellow heteropoly acid complex. This complex is then reduced by sodium sulfite (Na_2SO_3) to produce a blue-colored compound (molybdenum blue):

$$PO_4^{3-} + 12MoO_4^{2-} + 2NH_4^+ + 25H^+ \equiv\equiv (NH_4)_2H[PMo_{12}O_{40}]\cdot H_2O\downarrow + 11H_2O$$

$$(NH_4)_2H[PMo_{12}O_{40}]\cdot H_2O + \text{reducing agent} \longrightarrow \text{molybdenum blue}$$

The color intensity correlates with phosphate concentration. By comparing with a standard series, the phosphate content in the sample can be estimated.

3. *Determination of Total Iron*

Ferrous ions (Fe^{2+}) are oxidized to ferric ions (Fe^{3+}) by nitric acid:

$$2Fe^{2+} + NO_3^- + 2H^+ \equiv\equiv 2Fe^{3+} + NO_2^- + H_2O$$

In HNO_3 solution, Fe^{3+} reacts with potassium thiocyanate (KSCN) to form a complex:

$$Fe^{3+} + 3SCN^- \rightleftharpoons Fe(SCN)_3 \text{ (blood-red)}$$

The color intensity is proportional to the total iron concentration. By comparing with a standard series, the concentration of total iron can be estimated.

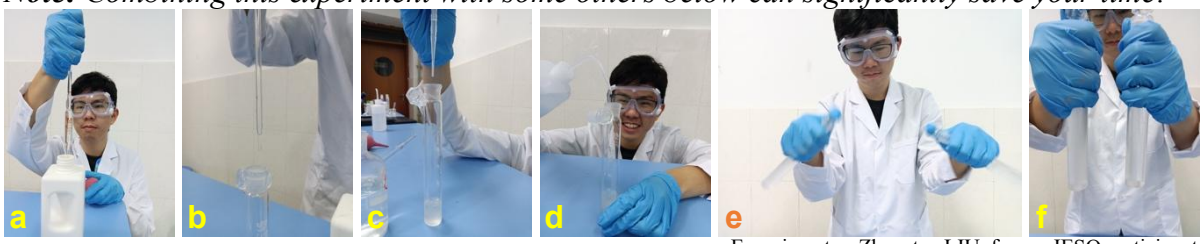
II. Main Reagents

$BaCl_2$ solution, $(NH_4)_2MoO_4$ solution, Na_2SO_3 solution, HNO_3 solution (2 mol/L), KSCN solution, sample to be tested.

III. Experimental Procedure

1. *Determination of Sulfate*

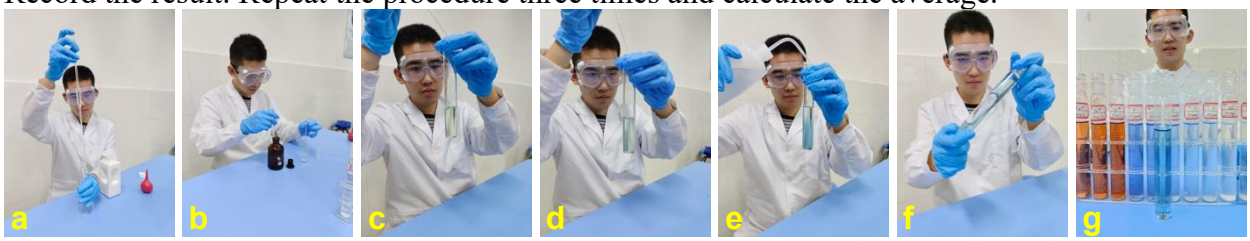
Add, in sequence, to a colorimetric tube: 25.00 mL of the sample, 10.00 mL of $BaCl_2$ solution, 5 mL of HCl solution.
Dilute to exactly 50.0 mL with distilled water and mix well, then let stand for 1 minute.
Compare the turbidity visually with the sulfate standard series.
Record the result. Repeat the procedure three times and calculate the average.
Note: *Combining this experiment with some others below can significantly save your time!*



Experimenter: Zhengtao LIU, former IESO participant

2. *Determination of Phosphorus*

Add, in sequence, to a colorimetric tube: 10.00 mL of the sample, 5.0 mL of HNO_3, 5.0 mL of $(NH_4)_2MoO_4$ solution, 10.0 mL of Na_2SO_3 solution.
Dilute to exactly 50.0 mL with distilled water and mix well, then let stand for 5 minutes.
Compare the color with the phosphate standard series.
Record the result. Repeat the procedure three times and calculate the average.

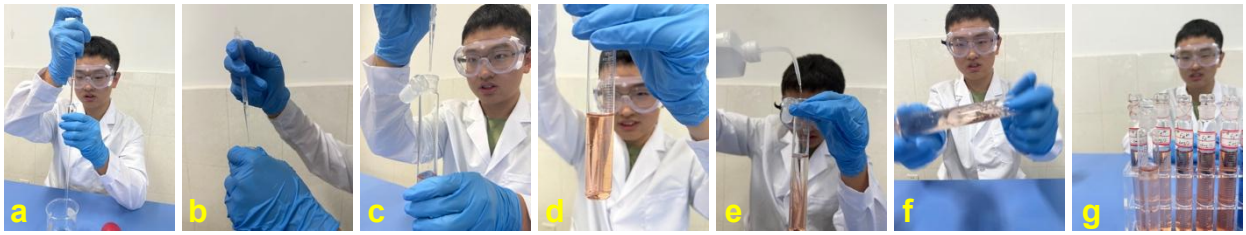


Experimenter: Yanlin WU, former IESO participant

3. *Determination of Total Iron*

Add, in sequence, to a colorimetric tube: 25.0 mL of the sample, 5.0 mL of HNO_3 solution, 5.0 mL of KSCN solution.
Dilute to exactly 50.0 mL with distilled water and mix well, then let stand for 1 minute.
Compare the color with the iron standard series.
Record the result. Repeat the procedure three times and calculate the average.

Note: *The determination of total iron applies only to soil leachates or coal gangue leachates, not to water samples collected in the field, as ferric ion concentrations in water samples from both Taiping and Yangshan are below the detection limit.*



Experimenter: Zhihang ZHONG, former IESO participant

IV. Difficulty Level Stratosphere

Experiment 8. Determination of Dissolved Inorganic Carbon (DIC) in Water Samples

I. Principle

1. *Conversion of Inorganic Carbon Species*

Dissolved inorganic carbon (DIC) in water mainly exists in the forms of carbonate ions (CO_3^{2-}), bicarbonate ions (HCO_3^-), and carbonic acid (H_2CO_3). By adding sodium hydroxide (NaOH) solution to the water sample and adjusting the pH to 12, bicarbonate ions and carbonic acid are converted into carbonate ions:

$$HCO_3^- + OH^- = CO_3^{2-} + H_2O, \qquad H_2CO_3 + 2OH^- = CO_3^{2-} + 2H_2O$$

Since calcium carbonate ($CaCO_3$) is less soluble than calcium bicarbonate $\{Ca(HCO_3)_2\}$, $CaCO_3$ may form as a white precipitate:

$$Ca^{2+} + HCO_3^- + OH^- = CaCO_3\downarrow \text{ (white)} + H_2O$$

2. *Complexation of Interfering Metal Cations*

Certain metal cations (e.g., Mg^{2+}, Fe^{3+}, Al^{3+}) can hydrolyze to form hydroxide precipitates, which dissolve in dilute hydrochloric acid (HCl):

$$M^{n+} + nOH^- = M(OH)_n\downarrow, \qquad M(OH)_n + nH^+ = M^{n+} + nH_2O$$

Disodium ethylenediaminetetraacetate (Na_2H_2Y) can form stable complexes with these cations, preventing their hydrolysis:

$$M^{n+} + Na_2H_2Y = MY^{n-4} + 2Na^+ + 2H^+$$

3. *Precipitation and Conversion*

Upon adding an excess of barium chloride ($BaCl_2$) solution, carbonate ions react with barium ions to form barium carbonate ($BaCO_3$) precipitate:

$$Ba^{2+} + CO_3^{2-} = BaCO_3\downarrow \text{ (white)}$$

Since $BaCO_3$ is less soluble than $CaCO_3$, $CaCO_3$ precipitate will be converted to $BaCO_3$:

$$CaCO_3 + Ba^{2+} \rightleftharpoons BaCO_3\downarrow \text{ (white)} + Ca^{2+}$$

4. *Removal of Sulfate Interference*

Sulfate ions (SO_4^{2-}) can also react with barium ions to form barium sulfate:

$$Ba^{2+} + SO_4^{2-} = BaSO_4\downarrow \text{ (white)}$$

$BaSO_4$ is insoluble in dilute HCl, whereas $BaCO_3$ dissolves readily:

$$BaCO_3 + 2H^+ = Ba^{2+} + H_2O + CO_2\uparrow$$

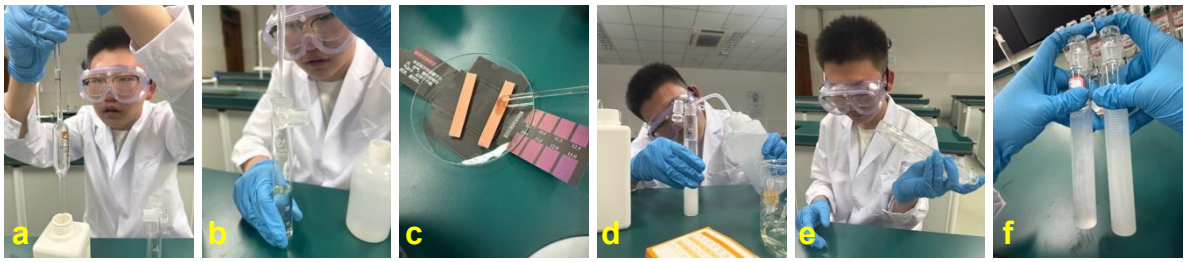
Therefore, the mass difference of the precipitate before and after HCl treatment can be used to calculate the DIC content.

5. *Turbidity Equivalence*

Experimental results show that the turbidity produced by carbonate ions (CO_3^{2-}) with an excess of barium ions is approximately equal to that produced by **an equal mass** of sulfate ions (SO_4^{2-}) with the same and sufficient amount of barium ions.

II. Main Reagents

EDTA solution (0. 1 mol/L), NaOH solution, $BaCl_2$ solution, HCl solution, water sample collected by your team.



Experimenter: Yifan WANG, former IESO participant

III. Experimental Procedure

1. ***Sample Preparation***
   Pipette 100 mL of the water sample into a beaker. Add 3 mL of EDTA solution.
2. ***Alkalinization***
   Adjust the pH of the solution to 12 using NaOH solution. Dilute the solution to 50.0 mL.
3. ***First turbidimetry***
   Add an excess amount of $BaCl_2$ solution to the alkaline sample. Filter the resulting precipitate. Dry the precipitate together with the filter paper, and record the combined mass. Add 25.00 mL of the solution in step 2 and 10.0 mL of $BaCl_2$ to a colorimetric tube. Dilute it to exactly 50.0 mL.
   Compare the turbidity visually with the sulfate standard series. Record the result.
4. ***Second turbidimetry***
   Add, in sequence, to a colorimetric tube: 25.00 mL of the solution in step 2, 10.0 mL of $BaCl_2$, and a 5 mL of HCl. Dilute it to exactly 50.0 mL.
   Compare the turbidity visually with the sulfate standard series. Record the result again.
   Calculate the difference.

IV. Difficulty Level Stratosphere

Introduction to Titration

Titration is a widely used analytical technique for determining the concentration of a dissolved substance (called the *analyte*) by reacting it with a solution of known concentration (called the *titrant*). The titrant is added slowly and precisely using a device called a *burette* (or *titration burette*) until the chemical reaction between the two substances is complete.

The point at which the reaction is exactly complete is called the **equivalence point**. It is often detected by a sharp change in a measurable signal—commonly a color change from a chemical *indicator*, a sudden pH shift, or a change in electrical potential. The volume of titrant used at the equivalence point allows us to calculate the exact concentration of the analyte based on the stoichiometry of the reaction.

Types of Titration

There are four major types of titration, each based on the kind of chemical reaction involved.

Type	Main Reaction	Typical Use	Example Reagents / Indicators
Acid–Base	Neutralization	Measuring acidity or basicity	Phenolphthalein, Methyl orange
Redox	Electron transfer	Dissolved oxygen (DO), chemical oxygen demand (COD), iron ions	$KMnO_4$, I_2, $Na_2S_2O_3$, Starch
Precipitation	Formation of a precipitate	Cl^- determination	$AgNO_3$, K_2CrO_4, Fluorescein
Complexometric	Formation of stable complexes	Water hardness, metal ions	EDTA, Eriochrome Black T

Standard Procedure for Titration

Performing a titration requires care, consistency, and attention to detail. Below is a general procedure:

1. ***Rinsing Equipment***
   Rinse the burette with tap water, then with distilled water 3 times and with a small amount of the titrant 3 times. This ensures that no residual water dilutes the titrant.
   Rinse the pipette with tap water, then with distilled water 3 times and with a small amount of the analyte solution 3 times before transferring it to the titration flask.
   Rinse the Erlenmeyer flask with distilled water only, never with analyte, to ensure precise volume measurements.
2. ***Filling the Burette***
   Close the stopcock and fill the burette with the titrant using a funnel.
   Remove the funnel after filling to prevent additional drops from entering.
   Record the initial reading of the burette (to 0.01 mL precision).
   Ensure there are no air bubbles in the burette tip. If bubbles exist, gently tap the burette or quickly open the stopcock to expel them.
3. ***Adding the Analyte***
   Use a pipette to transfer a fixed volume of the analyte into a clean Erlenmeyer flask.
   Add a few drops of a suitable indicator if necessary.

4. ***Performing the Titration***
   Slowly add the titrant from the burette while gently swirling the flask.
   As you approach the expected endpoint, add the titrant dropwise and observe carefully for a color change or other indicator.
   Record the final volume when the endpoint is reached.
5. ***Post-Titration Handling***
   After finishing the titration, wash all glassware thoroughly with tap water and then distilled water.
   Open the burette stopcock and invert the burette (tip pointing upward) to empty remaining liquid. This prevents long-term damage from residues.

Note*: If you wish to perform standard titration procedures, you may request a burette from the staff.*

Simplified Titration Procedure without Burettes

Although the standard titration procedure described above is recommended, this simplified method is also allowed. This approach aligns with the goals of the ITFI project, which emphasize contestants' quantitative awareness and analytical thinking over strict volumetric accuracy. The simplified method remains sufficient for interpreting results and solving problems. If used, the final measurement should be reported with **no more than three significant figures**.

1. ***Adding the Analyte***
   Use a pipette to transfer a fixed volume of the analyte into a clean Erlenmeyer flask.
2. ***Preparing the Titrant***
   Rinse a 10 mL measuring cylinder sequentially with tap water, distilled water, and a small amount of the titrant. Then fill the measuring cylinder with approximately 10 mL of titrant.
   Rinse the dropping pipette with distilled water (inside and outside).
   Expel any remaining distilled water from inside the dropping pipette. Wipe the outer surface dry with a clean paper towel (or filter paper.
   While holding the dropping pipette in the air, fully squeeze the bulb, then insert the dropping pipette into the titrant in the measuring cylinder and release the bulb to draw in the solution.
   Withdraw the dropping pipette from the liquid and dispense the titrant only by squeezing the bulb **once**. Do **not blow out** any remaining drops at the tip.
3. ***Performing the Titration***
   Record the initial volume of the titrant in the measuring cylinder (at eye level).
   Slowly add the titrant using the rinsed dropping pipette while gently swirling the flask.
   As you approach the expected endpoint, add the titrant dropwise and observe carefully for a color change or other indicator.
   When the endpoint is reached, return the remaining titrant in the dropping pipette to the measuring cylinder by squeezing the bulb **once** only. Do **not blow out** any remaining drops at the tip. Record the final volume in the measuring cylinder.
   Calculate the total volume of titrant used.
   Note: *If the titrant in the measuring cylinder is nearly depleted during titration, record the remaining volume, refill the cylinder with more titrant (without exceeding the full scale), and record the new total volume.*
4. ***Post-Titration Handling***

After finishing the titration, wash all glassware thoroughly with tap water and then distilled water.

Experiment 9. Determination of Water Hardness by Complexometric Titration

I. Principle

1. *Total Hardness Determination*

Under pH = 10 (adjusted by the ammonia buffer), disodium EDTA (Na_2H_2Y) reacts with Ca^{2+} and Mg^{2+} to form colorless stable complexes:

$$Ca^{2+} + Y^{4-} \rightleftharpoons CaY^{2-}\ \text{(colorless)}, \qquad Mg^{2+} + Y^{4-} \rightleftharpoons MgY^{2-}\ \text{(colorless)}$$

Eriochrome Black T (EBT) is used as an indicator. It first forms a wine-red complex with Mg^{2+}. As EDTA is added, it preferentially binds Mg^{2+}, displacing it from the Mg–EBT complex, freeing the EBT, which appears pure blue:

$$Mg^{2+} + EBT^{3-} \rightleftharpoons Mg\text{-}EBT^{-}\ \text{(wine red)}, \qquad Mg\text{-}EBT^{-} + Y^{4-} \rightleftharpoons MgY^{2-} + EBT^{3-}\ \text{(pure blue)}$$

2. *Calcium Hardness Determination*

NaOH is added to raise the pH to 12–13, causing Mg^{2+} to precipitate as $Mg(OH)_2$, thus eliminating interference. Ammonium purpurate (AP) is used as an indicator, forming a purple-red complex with Ca^{2+}. EDTA displaces Ca^{2+} from this complex, and the free indicator gives a violet-blue color.

$$Ca^{2+} + AP^{2-} \rightleftharpoons Ca\text{-}AP\ \text{(purple-red)}, \qquad Ca\text{-}AP + Y^{4-} \rightleftharpoons CaY^{2-} + AP^{2-}\ \text{(violet-blue)}$$

3. *Elimination of Interference*

Fe^{3+} and Al^{3+} may interfere with EDTA titration. These ions are masked using triethanolamine (TEA), which forms stable complexes:

$$Fe^{3+} + TEA^{3-} \rightleftharpoons Fe\text{-}TEA, \qquad Al^{3+} + TEA^{3-} \rightleftharpoons Al\text{-}TEA$$

4. *Calculation of Magnesium Hardness*

$$\text{Magnesium hardness} = \text{Total hardness} - \text{Calcium hardness}$$

II. Main Reagents

Standard EDTA solution, EBT solution, AP solution, TEA solution, ammonia buffer (pH 10.0), NaOH solution, water samples your team have collected.

III. Experimental Procedure

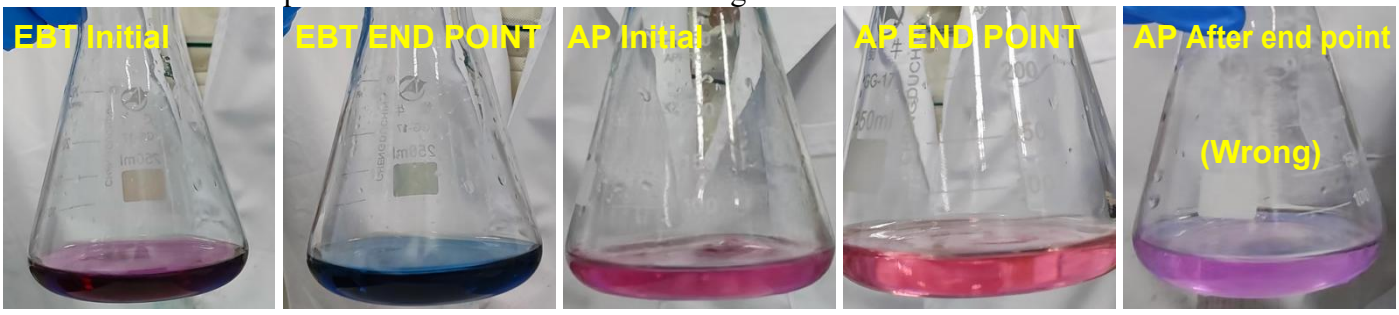
Experimenter: Shunning ZHANG, former IESO participant

1. *Total Hardness Determination*

Use a pipette to transfer 25.00 mL of the water sample into an Erlenmeyer flask.
Add, in sequence: 5 mL TEA solution, 5 mL ammonia buffer (pH 10.0), and 3–5 drops of EBT indicator. Swirl to mix.
Titrate with standard EDTA solution. **Endpoint:** solution changes from **wine red** to **pure blue**.
Record the volume of EDTA used.
Perform three parallel titrations and use the average volume to calculate total hardness.

2. *Calcium Hardness Determination*

Use a pipette to transfer 25.00 mL of the water sample into another Erlenmeyer flask.
Add, in sequence: 5 mL TEA solution, NaOH solution (to adjust pH to 12–13), and a few drops of AP indicator. Swirl to mix.
Titrate with standard EDTA solution. **Endpoint:** solution changes from **purple-red** to **violet-blue**.
Record the volume of EDTA used.
Perform three parallel titrations and use the average volume to calculate calcium hardness.



IV. Difficulty Level

Mesosphere

Experiment 10. Determination of Chemical Oxygen Demand (COD) by Acidic Permanganate Titration

I. Principle

1. *Oxidation of Reducing Substances*

Under acidic conditions, potassium permanganate ($KMnO_4$) acts as a strong oxidizing agent, oxidizing the reducing substances in the water sample. $KMnO_4$ is reduced to colorless Mn^{2+}:

$$4MnO_4^- + 5[C]\ (\text{reductants}) + 12H^+ \longrightarrow 4Mn^{2+} + 5CO_2\uparrow + 6H_2O$$

2. *Reduction by Sodium Oxalate*

Excess $KMnO_4$ is then reduced by a known volume of standard sodium oxalate ($Na_2C_2O_4$) solution:

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \longrightarrow 2Mn^{2+} + 10CO_2\uparrow + 8H_2O$$

3. *Back Titration*

The remaining sodium oxalate is back-titrated with standard $KMnO_4$ solution. The volume difference between the initially added and the back-titrated $KMnO_4$ corresponds to the amount that reacted with reducing substances in the sample. Based on electron balance, 1 mol $KMnO_4$ is equivalent to 40 g of oxygen in terms of oxygen demand, which is used to calculate the COD value.

4. *Color Indicator*

$KMnO_4$ has a characteristic purple-red color and serves as its own indicator.

II. Main Reagents

Standard $KMnO_4$ solution, standard $Na_2C_2O_4$ solution, H_2SO_4 solution (2 mol/L), water samples your team have collected.

III. Experimental Procedure

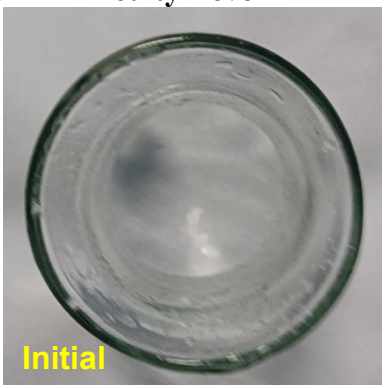
1. *Oxidation Stage*

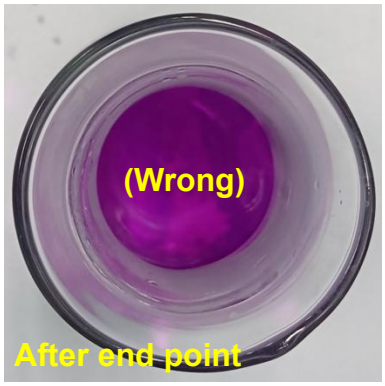
Use a pipette to transfer 10.00 mL of the water sample into a 250 mL Erlenmeyer flask.
Add 10.00 mL of sulfuric acid, followed by 10.00 mL of standard $KMnO_4$ solution (using a pipette).
Swirl to mix, then heat the mixture in a boiling water bath for about 30 minutes.
Note: *If the purple-red color fades significantly during heating, the sample must be diluted and the procedure repeated.*

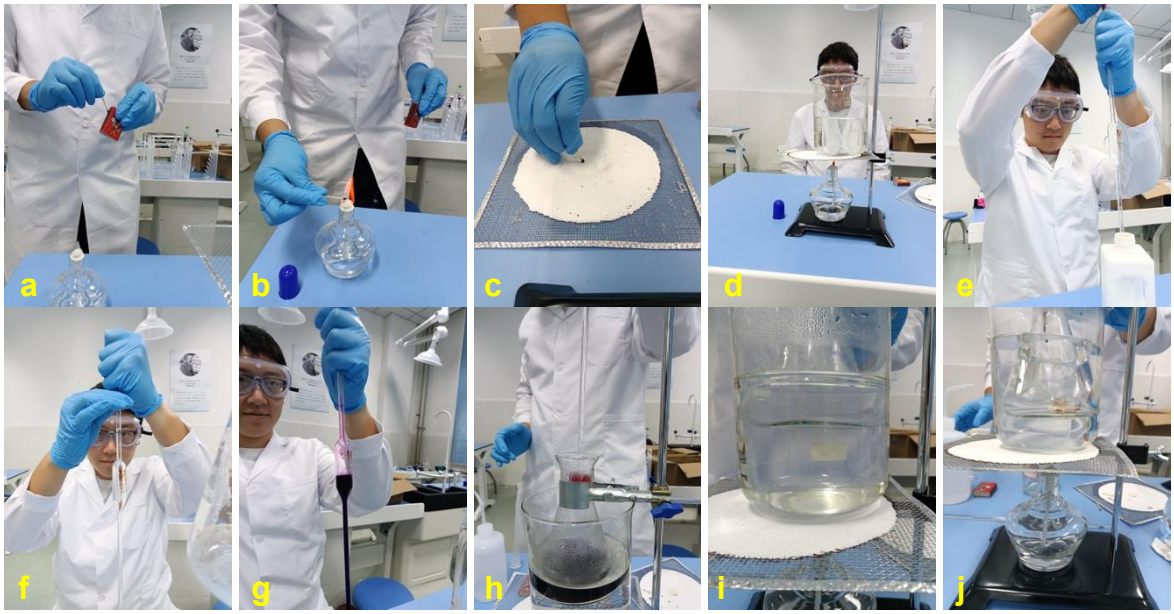
2. *Back Titration*

Cool the flask to approximately 70–80°C. Add 10.00 mL of standard sodium oxalate solution using a pipette. Immediately titrate the excess oxalate with standard $KMnO_4$ solution.
Endpoint: solution changes from **colorless** to **faint pink**.
Record the volume of $KMnO_4$ used in the back titration.
Perform three parallel titrations and use the average volume to calculate chemical oxygen demand (COD).
Note: *Consider setting up a blank using distilled water to correct for background oxygen demand from reagents.*

IV. Difficulty Level Thermosphere







Experimenter: Yibo WANG, former IESO participant

Experiment 11. Determination of Inorganic Phosphorus by Back Titration

I. Principle

1. *Formation of Phosphomolybdate Precipitate*

Inorganic phosphorus in the test sample is oxidized to phosphate (PO_4^{3-}) by nitric acid. The phosphate then reacts with ammonium molybdate $\{(NH_4)_2MoO_4\}$ to form a yellow precipitate of ammonium phosphomolybdate $\{(NH_4)_2H[PMo_{12}O_{40}]\cdot H_2O\}$:

$$PO_4^{3-} + 12MoO_4^{2-} + 2NH_4^+ + 25H^+ \equiv (NH_4)_2H[PMo_{12}O_{40}]\cdot H_2O\downarrow + 11H_2O$$

2. *Dissolution of the Precipitate and Reaction with NaOH*

After filtration and washing, the precipitate is dissolved in a known excess of standard sodium hydroxide (NaOH) solution:

$$(NH_4)_2H[PMo_{12}O_{40}]\cdot H_2O + 27NaOH \equiv Na_3PO_4 + 12Na_2MoO_4 + 2NH_3\cdot H_2O + 16H_2O$$

3. *Back Titration of Excess NaOH*

The remaining NaOH is back-titrated with standard nitric acid using phenolphthalein (PP) as the indicator. The endpoint is reached when the pink color disappears (pH ≈ 8), indicating the following reactions:

$$OH^- + H^+ \equiv H_2O, \qquad PO_4^{3-} + H^+ \equiv HPO_4^{2-}, \qquad NH_3\cdot H_2O + H^+ \equiv NH_4^+ + H_2O$$

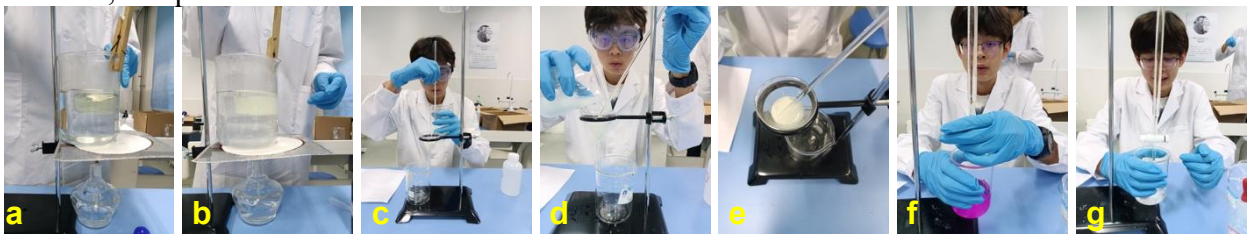
4. *Phosphorus Content Calculation*

The net result is that each mole of phosphorus reacts with 24 moles of NaOH. Therefore,

$$n(\mathrm{P}) = \tfrac{1}{24} \times [\, n(\mathrm{NaOH\ added}) - n(\mathrm{HNO_3\ used\ in\ back\ titration})\,]$$

II. Main Reagents

Standard $(NH_4)_2MoO_4$ solution, standard NaOH solution, standard HNO_3 solution (2 mol/L), PP solution, sample to be tested.



Experimenter: Ruiyang LI, former IESO participant

III. Experimental Procedure

1. Precipitate Formation

Transfer 50.00 mL of the sample into a 250 mL beaker. Add an excess of HNO_3 solution, followed by 20 mL of standard $(NH_4)_2MoO_4$ solution. Stir and heat the mixture in a boiling water bath for 10 minutes. Allow the solution to cool to room temperature.

Filter the yellow precipitate using quantitative filter paper and wash the precipitate with distilled water until the filtrate is free of acidity. Transfer the precipitate along with the filter paper back into the original beaker.

Note: *If the filtration rate of the filter paper is too slow, you can be creative. For example, tissue paper might have unexpected effects.*

2. Titration

Add an excess amount of standard NaOH solution until the precipitate is completely dissolved. Record the volume of NaOH used.

Add 3–5 drops of PP indicator and back-titrate with standard HNO_3 solution.

Endpoint: The solution changes from **pink** to **colorless**.

Record the volume of HNO_3 used.

Perform three parallel titrations and use the average volume to calculate the concentration of inorganic phosphorus in the sample.

IV. Difficulty Level Exosphere

Instruments

In the ITFI program, the laboratory provides the following instruments:

Code	Instrument	Specification	Quantity	Chinese Name
I-01	Beaker	100 mL	1	烧杯
I-02	Beaker	250 mL	2	烧杯
I-03	Beaker	500 mL	1	烧杯
I-04	Glass rod	—	1	玻璃棒
I-05	Dropping pipette	—	5	滴管
I-06	Tweezers	—	1	镊子
I-07	Test tube holder	—	1	试管夹
I-08	Erlenmeyer (conical) flask	250 mL	2	锥形瓶
I-09	Measuring cylinder	10 mL	1	量筒
I-10	Measuring cylinder	50 mL	1	量筒
I-11	Wash bottle	—	1	洗瓶
I-12	Watch glass	—	1	表面皿
I-13	Petri dish	—	1	培养皿
I-14	Sandpaper	—	1	砂纸
I-15	Test tube brush	—	3	试管刷
I-16	Wipe cloth	—	1	抹布
I-20	Spot plate	White	1	点滴板
I-21	Stand	—	1	铁架台
I-22	Volumetric pipette	10 mL	1	移液管
I-23	Volumetric pipette	25 mL	1	移液管
I-24	Pipette rack	—	1	移液管架
I-25	Rubber suction bulb	—	1	洗耳球
I-26	Mortar and pestle	—	1	研钵、研杵
I-30	pH test strip	Universal pH range	0†	pH 试纸
I-31	pH test strip	pH 0.5 – 5.0	0†	pH 试纸
I-32	pH test strip	pH 5.4 – 7.0	0†	pH 试纸
I-33	pH test strip	pH 6.4 – 8.0	0†	pH 试纸
I-34	pH test strip	pH 8.2 – 10.0	0†	pH 试纸
I-35	pH test strip	pH 9.5 – 13.0	0†	pH 试纸
I-36	Nitrate / nitrite test strip	—	0†	硝酸盐-亚硝酸盐试纸
I-40	Burette	50 mL, colorless	0	滴定管(无色)
I-41	Burette	50 mL, amber	0	滴定管(棕色)
I-42	Burette clamp	—	0	蝴蝶夹
I-43	Volumetric flask	50 mL	0	容量瓶
I-44	Volumetric flask	100 mL	0	容量瓶
I-45	Volumetric flask	250 mL	0	容量瓶
I-46	Volumetric flask	500 mL	0	容量瓶
I-48	Round-bottom flask	250 mL	0	圆底烧瓶
I-49	Colorimetric tube	50 mL	0	比色管
I-50	Colorimetric tube rack	50 mL	0	比色管架
I-51	Test tube	—	0	试管
I-52	Filter funnel	—	0	普通漏斗
I-53	Glass tubing	—	0	玻璃导管

I-60	Rubber tubing	—	0	橡胶导管
I-61	Scissors	—	0	剪刀
I-62	Spatula	—	0	药匙
I-63	Weighing paper	—	0†	称量纸
I-64	Quantitative filter paper	—	0†	定量滤纸
I-65	Gauze	—	0†	纱布
I-66	Plastic wrap	—	0†	保鲜膜
I-70	Alcohol burner	—	0	酒精灯
I-71	Match	—	0†	安全火柴
I-72	Tripod	—	0	三脚架
I-73	Wire gauze	—	0	石棉网
I-74	Thermometer	0 – 100 ℃	0	温度计
I-75	Boiling chip	—	0†	沸石
I-76	Beaker	1000 mL	0	烧杯
I-80	Oven	105°C	0*	烘箱
I-81	Electronic balance	200g × 0.01g	0*	电子天平
I-82	Multiparameter meter	YSI Pro Plus	0*	多功能水质检测仪
I-83	Handheld XRF analyzer	OLYMPUS VANTA C	0*	手持 XRF 分析仪

Note: *Each team is allocated the quantity shown in the 'Quantity' column. Before starting the experiment, please check whether the quantity of instruments at your team's lab bench matches the list above. If there is any discrepancy, report it to staff immediately. All instruments listed above are available in the laboratory. Instruments with a quantity of 0 are available upon request (see Laboratory Usage Procedure below). Please note that these items are in limited supply and may not be immediately available. Instruments with an asterisk (*) are shared and you cannot take them to your own workstation. Except for items marked with a dagger (†) in the "Quantity" column, which are consumables, all other instruments must not be damaged or lost; otherwise, contestants will be required to compensate at full price.*

Reagents

The laboratory also provides the following reagents:

Code	Reagent	Chemical Formula	Specification	Control	Chinese Name
R-01	Hydrochloric acid	HCl	~ 0.1 mol/L	—	盐酸
R-02	Sulfuric acid	H_2SO_4	~ 0.1 mol/L	—	硫酸
R-03	Oxalic acid	$H_2C_2O_4$	0.1000 mol/L	—	草酸
R-04	Potassium permanganate	$KMnO_4$	~ 0.02 mol/L	—	高锰酸钾
R-05	Potassium hydroxide	KOH	~ 0.1 mol/L	—	氢氧化钾
R-06	Sodium bicarbonate	$NaHCO_3$	~ 0.1 mol/L	—	碳酸氢钠
R-07	Sodium hydroxide	NaOH	~ 0.1 mol/L	—	氢氧化钠
R-08	Sodium carbonate	Na_2CO_3	0.1000 mol/L	—	碳酸钠
R-09	Zinc oxide	ZnO	Suspension	—	氧化锌
R-10	Methyl orange (MO)	$C_{14}H_{14}N_3NaO_3S$	0.1%	—	甲基橙
R-11	Phenolphthalein (PP)	$C_{20}H_{14}O_4$	Ethanol solution, 1%	—	酚酞
R-30	Activated carbon	C	Suspension	General	活性炭
R-31	Ferric chloride	$FeCl_3$	0.1000 mol/L	General	氯化铁
R-32	Hydrochloric acid	HCl	~ 2 mol/L	General	盐酸(2M)
R-33	Nitric acid	HNO_3	~ 0.1 mol/L	General	硝酸
R-34	Sulfuric acid	H_2SO_4	~ 2 mol/L	General	硫酸(2M)
R-35	Potassium phosphate monobasic	KH_2PO_4	0.01000 mol/L	General	磷酸二氢钾

R-36	Potassium hydroxide	KOH	~ 2 mol/L	General	氢氧化钾(2M)
R-37	Ammonium molybdate	$(NH_4)_2MoO_4$	0.5 mol/L	General	钼酸铵
R-38	Sodium hydroxide	NaOH	~ 2 mol/L	General	氢氧化钠(2M)
R-39	Sodium thiosulfate	$Na_2S_2O_3$	~ 0.1 mol/L	General	硫代硫酸钠
R-40	Sodium sulfate	Na_2SO_4	0.1000 mol/L	General	硫酸钠
R-41	Triethanolamine (TEA)	$C_6H_{15}NO_3$	30%	General	三乙醇胺
R-42	Ammonium purpurate (AP)	$C_8H_8N_6O_6$	1%	General	紫脲酸铵
R-43	Disodium ethylenediaminetetraacetate	$C_{10}H_{14}N_2O_8Na_2$	~ 0.01 mol/L	General	EDTA 二钠盐
R-44	Eriochrome Black T (EBT)	$C_{20}H_{12}N_3NaO_7S$	0.5%	General	铬黑 T
R-45	Bromcresol green-methyl red (BCG-MR)	$C_{21}H_{14}Br_4O_5S$-$C_{15}H_{15}N_3O_2$	Ethanol solution	General	溴甲酚绿-甲基红
R-50	Disodium ethylenediaminetetraacetate	$C_{10}H_{14}N_2O_8Na_2$	~ 0.1 mol/L	General	EDTA 二钠盐
R-70	Barium chloride	$BaCl_2$	0.1000 mol/L	**Strict**	氯化钡
R-71	Nitric acid	HNO_3	~ 2 mol/L	**Strict**	硝酸(2M)
R-72	Sulfuric acid	H_2SO_4	6 mol/L	**Strict**	硫酸(6M)
R-73	Potassium thiocyanate	KSCN	0.1 mol/L	**Strict**	硫氰酸钾
R-74	Ammonia solution	$NH_3·H_2O$	2 mol/L	**Strict**	氨水
R-75	Ammonia buffer solution	NH_3–NH_4Cl	pH 10.0	**Strict**	氨缓冲溶液
R-76	Sodium sulfite	Na_2SO_3	1.0 mol/L	**Strict**	亚硫酸钠
R-77	Zinc	Zn	Solid	**Strict**	锌
R-78	Ethanol	C_2H_5OH	Absolute	**Strict**	乙醇

Note: *All controlled reagents (labeled "General" or "Strict") must be requested using the Reagent Request fields of contestants' Laboratory Record Sheet and obtained from staff. Contestants are* ***strictly prohibited*** *from retrieving these reagents without authorization. Reagents under "Strict" control require approval from the exam affairs staff before use.*
Reagents with a tilde (~) in the Specification field are approximately labeled in the table but will be provided in the experiment as precisely standardized solutions. Their exact concentrations are indicated on the reagent labels and should be used as the reference.

Safety Supplies

To ensure laboratory safety, the following items are also provided to each team:

Code	Safety Supply	Specification	Quantity	Comments	Chinese Name
S-01	Laboratory coat	White	2	Collected upon leaving lab	实验服
S-02	Goggles	—	2	Collected upon leaving lab	护目镜
S-03	Nitrile gloves	—	2	—	丁腈手套
S-04	Cotton gloves	—	1	Collected upon leaving lab	线手套

Note: *Additional safety supplies (code S-30 and above) are available in the laboratory for emergency use. These items are managed and used* ***only by staff****. Contestants are* ***not allowed*** *to request or handle these items.*

* * *

ITFI Laboratory Work References