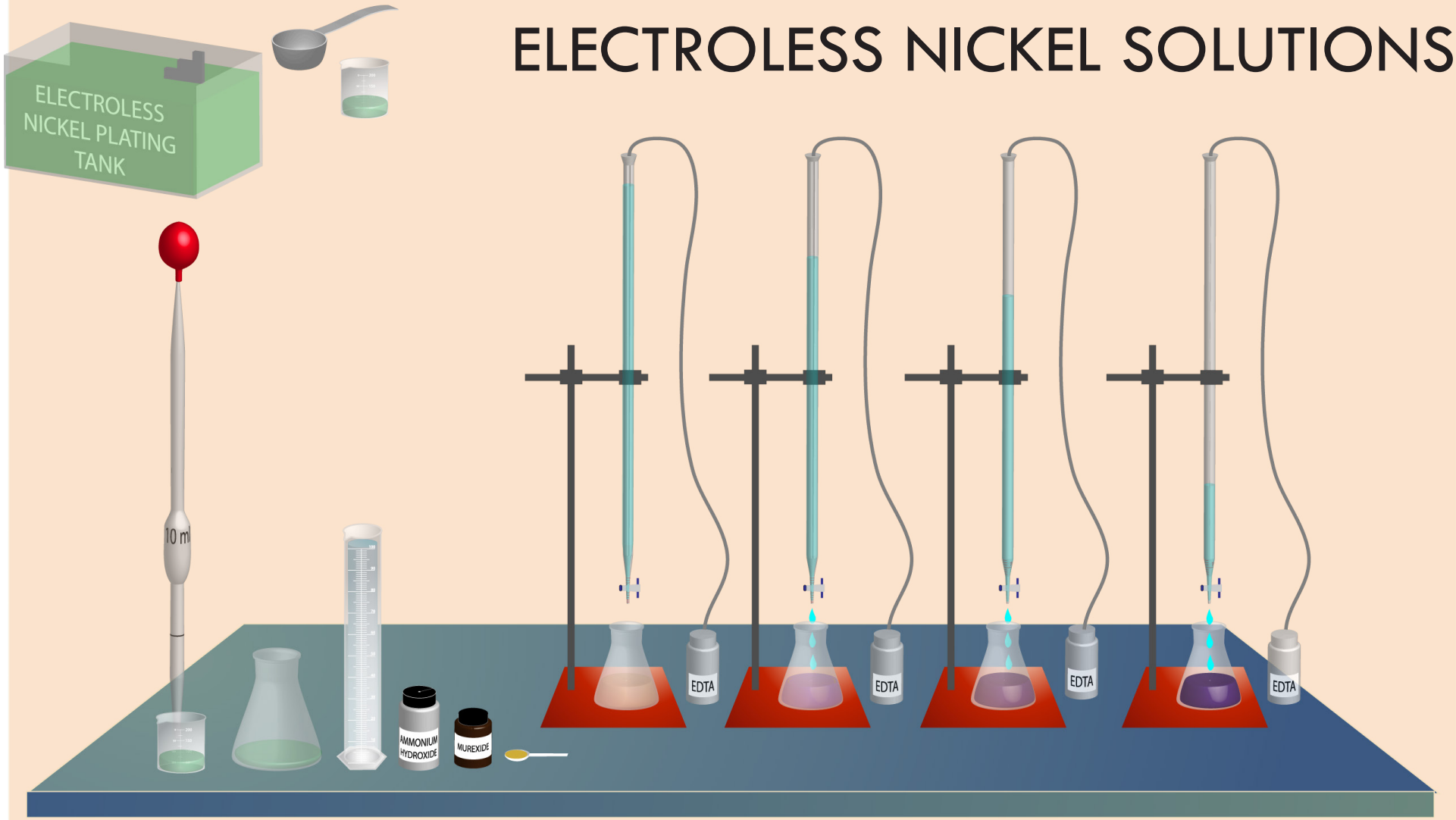
HOW TO DETERMINE NICKEL CONCENTRATION IN ELECTROLESS NICKEL SOLUTIONS



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A STEP-BY-STEP GUIDE TO PERFORMING NICKEL TITRATIONS

HOW TO DETERMINE NICKEL CONCENTRATION IN ELECTROLESS NICKEL SOLUTIONS

Table of Contents

INTRODUCTION:

- **Course description & intended audience:**
  - A titration a standardized procedure to determine the nickel concentration in electroless nickel solutions.
  - This lesson is designed to enable skilled workers who don't necessarily have formal technical training to perform a nickel titration.
- **How to use this instruction:**
  - **Individual or classroom learning guide:**
    - ☐ This manual can be used by independent learners or in a classroom setting to become familiar with the basics of nickel titration.
    - ☐ However, hands-on learning using lab equipment and solutions are necessary for actual proficiency.
  - **Laboratory guide:** This manual provides the key points of laboratory procedures needed to ensure valid and consistent results.
  - **Review & Reference:** For those who have learned how to perform nickel titrations, but don't perform them regularly, this manual can serve as a refresher or as a reference.

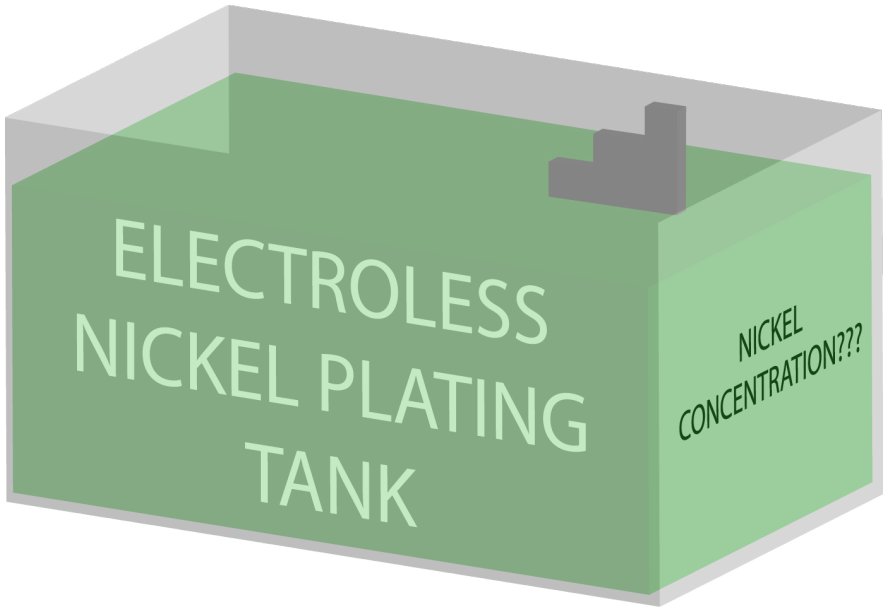
LEARNING OBJECTIVES:

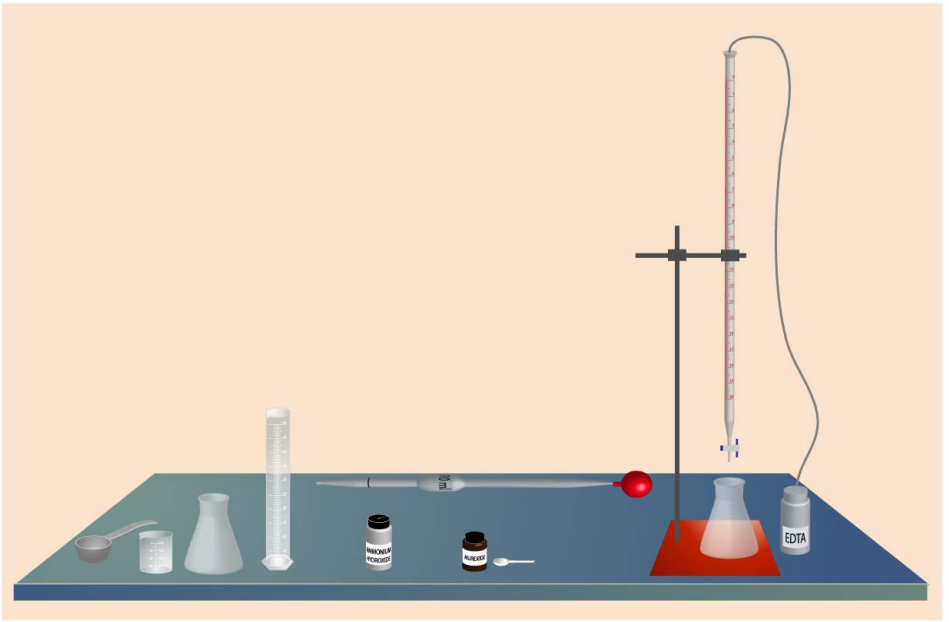
By the end of the written part of this instruction, you should be able to …

- Recognize and name laboratory equipment used;
- Define "titration" & other terms used and describe basic titration process;
- Discuss importance of determining nickel concentration in solution;
- List steps in titration process;
- Describe how to perform each step & sub-step;
- State reasons for performing each step correctly.

By the end of the hands-on part of this instruction, you should be able to…

- Clean each piece of equipment used;
- Perform each step in titration process correctly;
- Maintain equipment correctly;
- Gather and handle solutions & equipment safely;
- Replace all equipment in usable state for next titration.





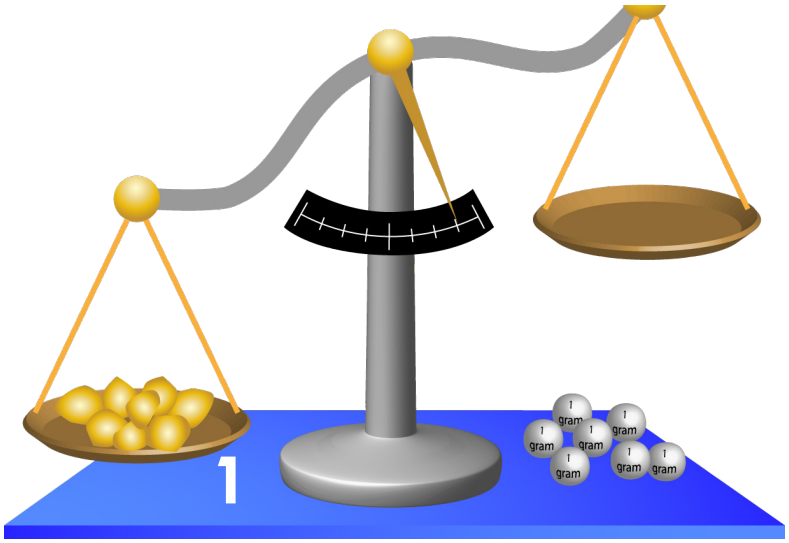
DEFINITION & IMPORTANCE OF TITRATIONS

- A titration is a procedure to analyze a solution & determine the concentration of a substance (in this case, nickel).
- Electroless nickel platers should know how to check the nickel concentration at regular intervals and whenever they suspect a problem.
- **Why is it important to know how much nickel is in an electroless nickel solution?**
- Nickel in EN solutions usually gets used up at a steady rate, but...
- Nickel concentrations can change suddenly with expensive consequences.
- Maintaining nickel concentration at a consistent level is one of the most important variables in successful electroless nickel plating.
- If nickel concentration is not optimal, plating quality is affected.
- If we don't know the nickel concentration in the solution, we won't know how much to add.
- Too big an add made to a solution low in nickel may "shock" the solution, which could mean replacing the contents of the entire tank.

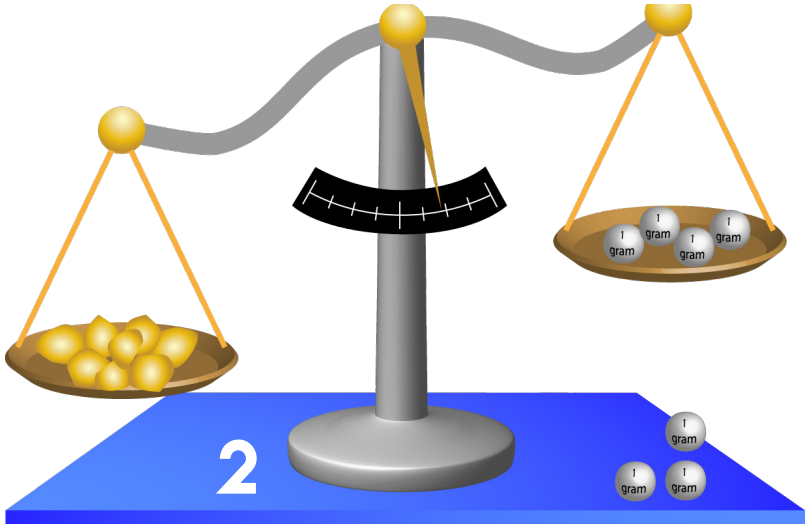
EXPLANATION OF TITRATION PROCESS

Performing a titration is like balancing a scale…

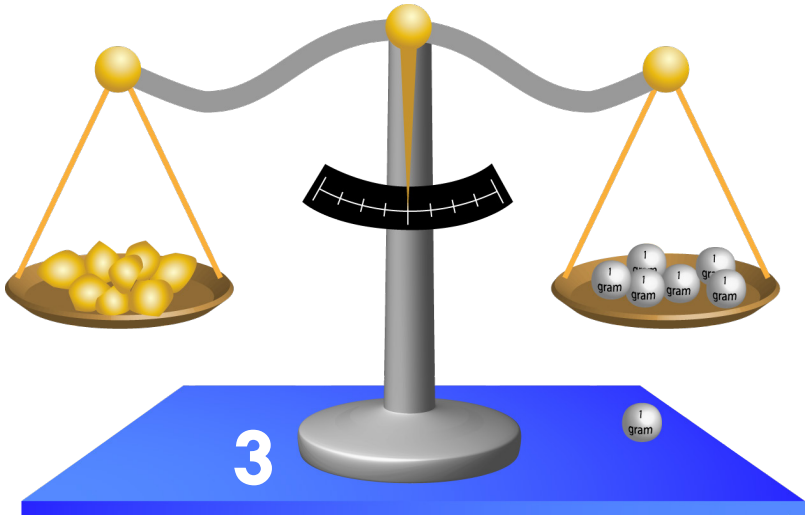
Suppose we need to know how much a certain amount of gold weighs...



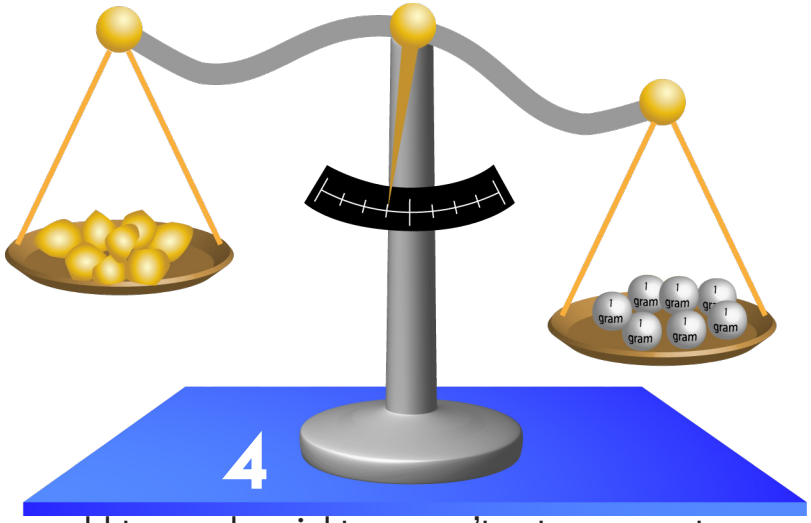
We place the gold on the left side of the scale.



We start to add 1 gram weights to the other side of the scale...



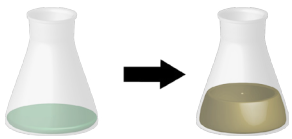
When the scales are balanced, we record the number of 1 gram weights so that we know how much gold we have.

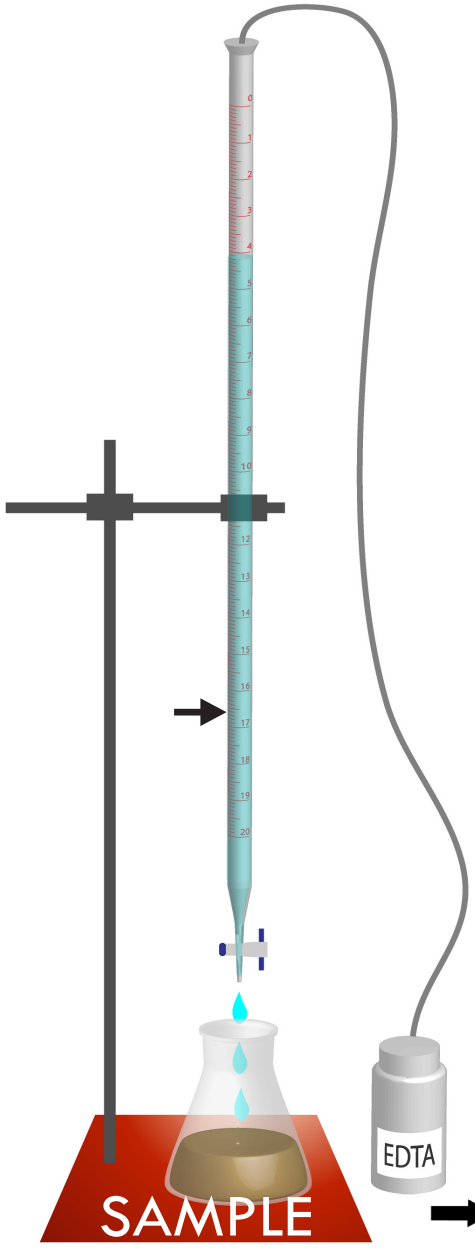


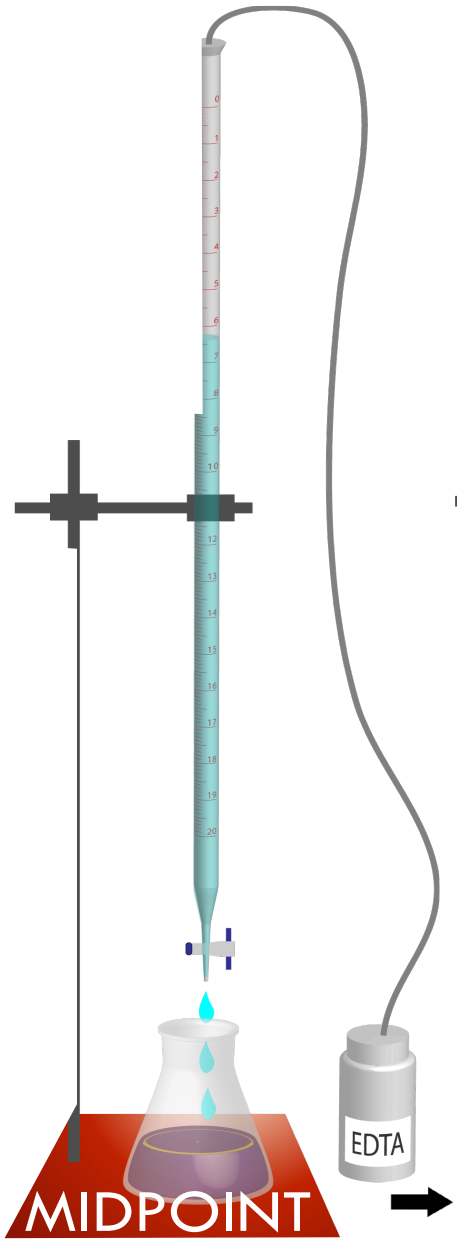
If we add too much weight, we won't get an accurate measurement. The point is, we have to add just enough in order to get an accurate measurement without going over.

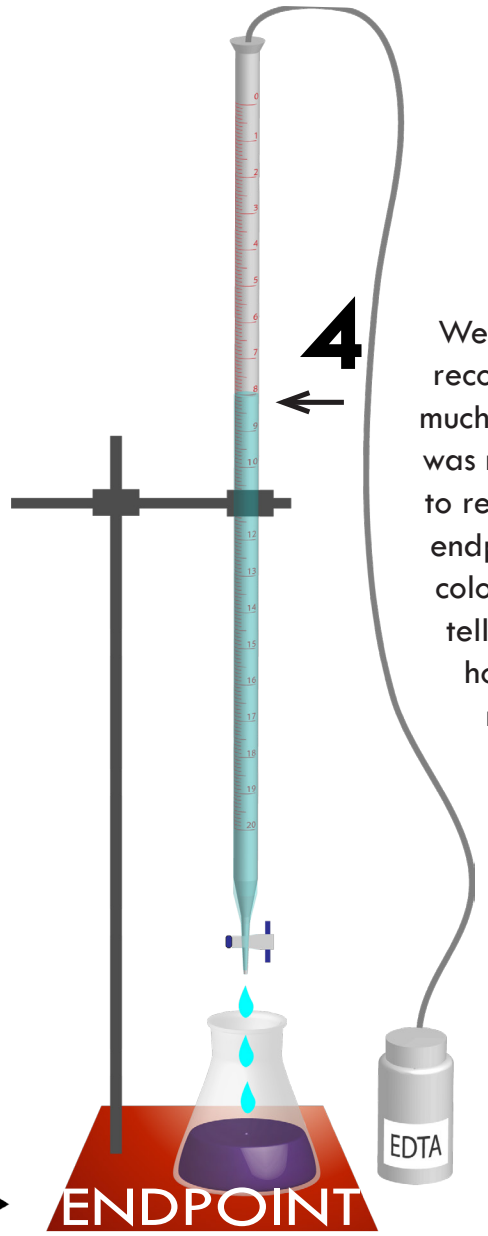
TITRATION PROCESS OVERVIEW

- In a titration, we can find out how much nickel is in the sample by adding a chemical (a titrant called EDTA) that reacts with the nickel sample.
- When the color of the sample stops changing, we have balanced the titrant with the nickel.
- By measuring how much titrant has been used up, we will know how much nickel is in solution.









We then record how much EDTA was needed to reach the endpoint color. This tells us how much nickel is in the solution.

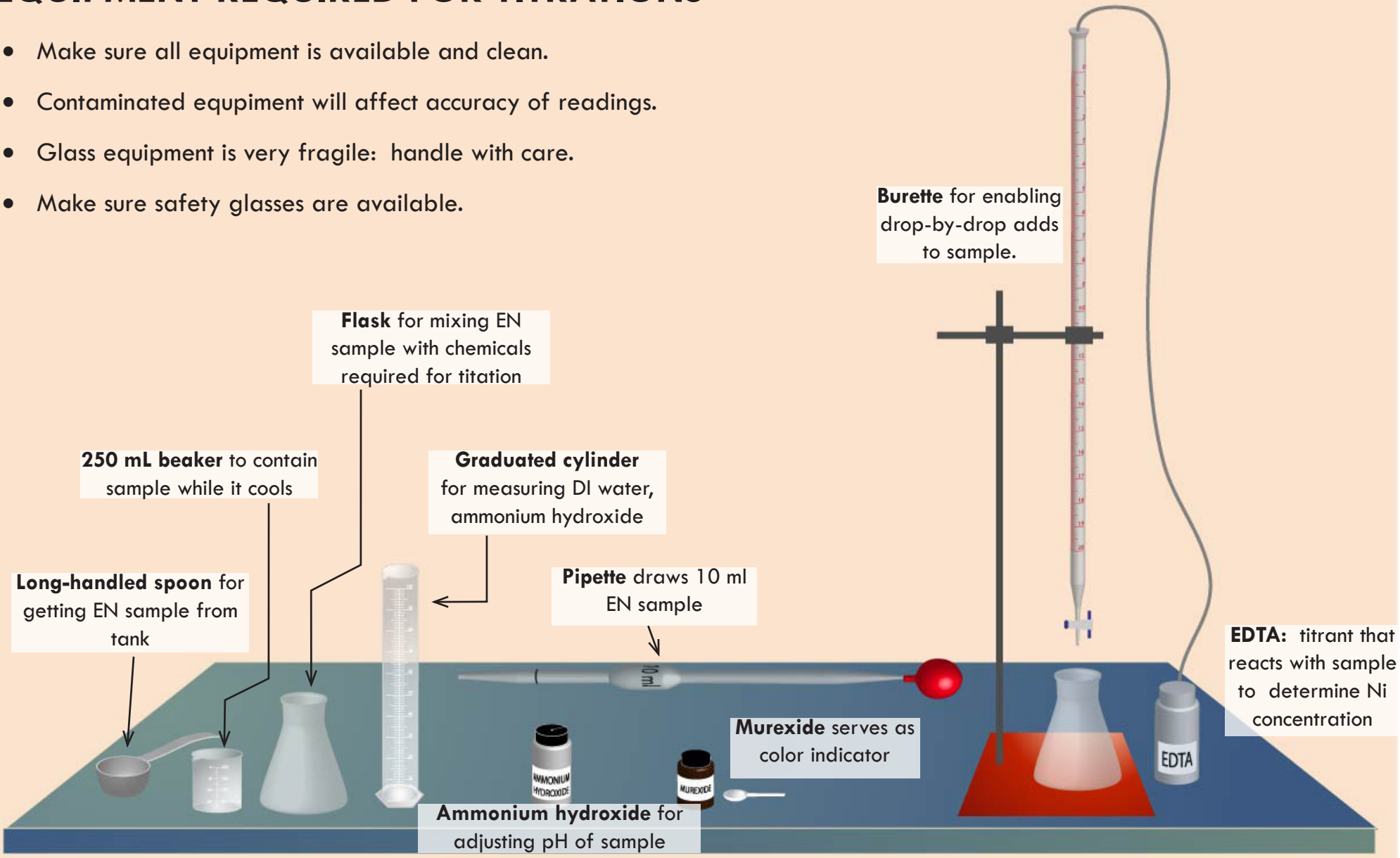
1 We gather a precise amount of the sample & prepare it by adding water & other chemicals.

2 We then add the titrant drop by drop to the sample until the reactions are balanced. (The drops of titrant are comparable to the 1 gram weights in the comparison.)

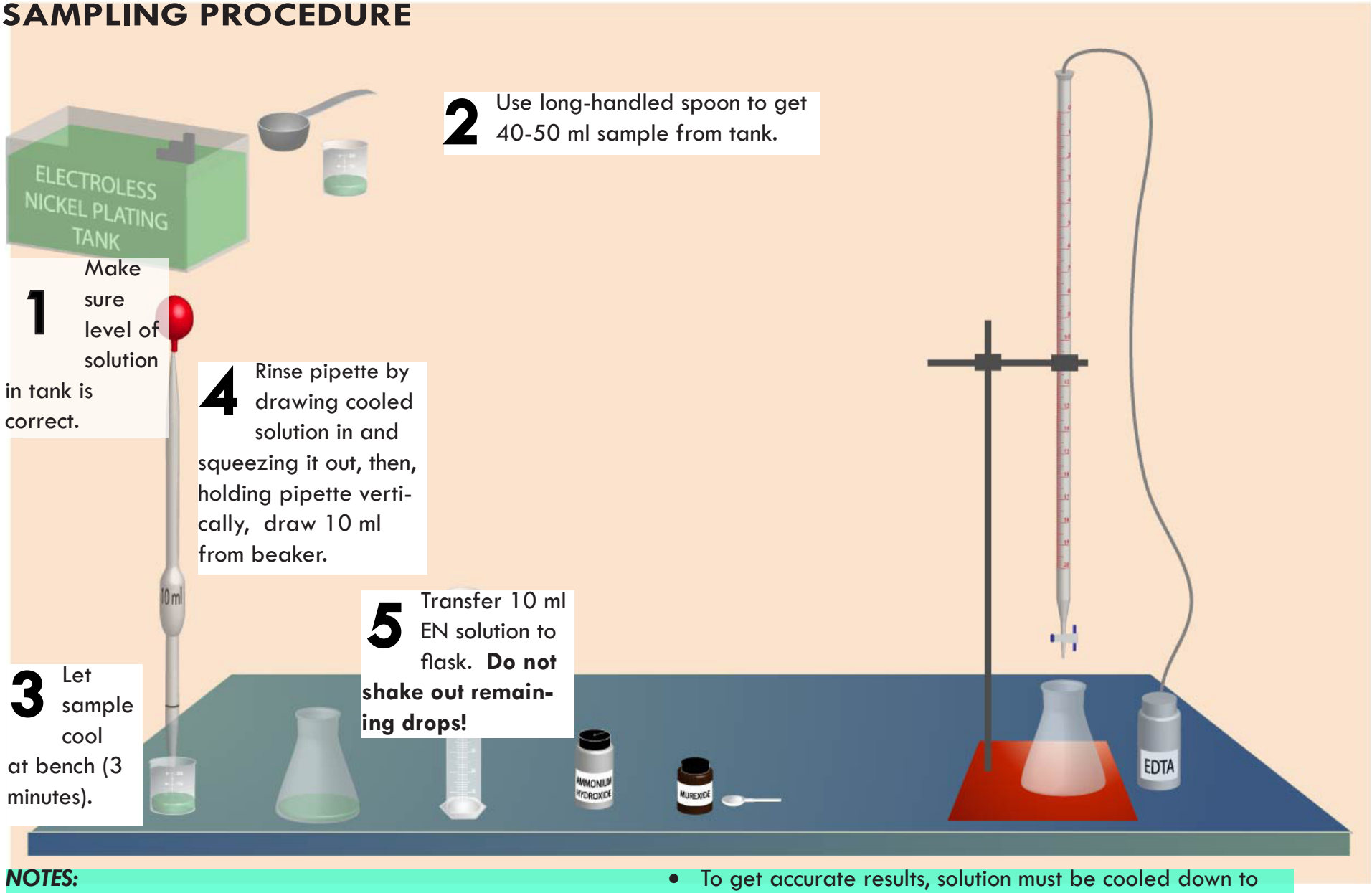
3 We can tell when the reactions are balanced because the sample solution reaches the endpoint color: that is, it stops changing color.

EQUIPMENT REQUIRED FOR TITRATIONS

- Make sure all equipment is available and clean.
- Contaminated equpiment will affect accuracy of readings.
- Glass equipment is very fragile: handle with care.
- Make sure safety glasses are available.



SAMPLING PROCEDURE



1 Make sure level of solution in tank is correct.

2 Use long-handled spoon to get 40-50 ml sample from tank.

3 Let sample cool at bench (3 minutes).

4 Rinse pipette by drawing cooled solution in and squeezing it out, then, holding pipette vertically, draw 10 ml from beaker.

5 Transfer 10 ml EN solution to flask. **Do not shake out remaining drops!**

NOTES:

- If tank is not within 5% of optimal level, sample won't represent solution in tank.
- Getting 40-50 ml of solution will provide enough to check or redo titration, if necessary.
- To get accurate results, solution must be cooled down to near room temperature.
- Rinsing pipette with sample will remove dried solution.
- Reading won't be accurate unless pipette is held vertically.
- 10 ml mark on pipette accounts for remaining drops.

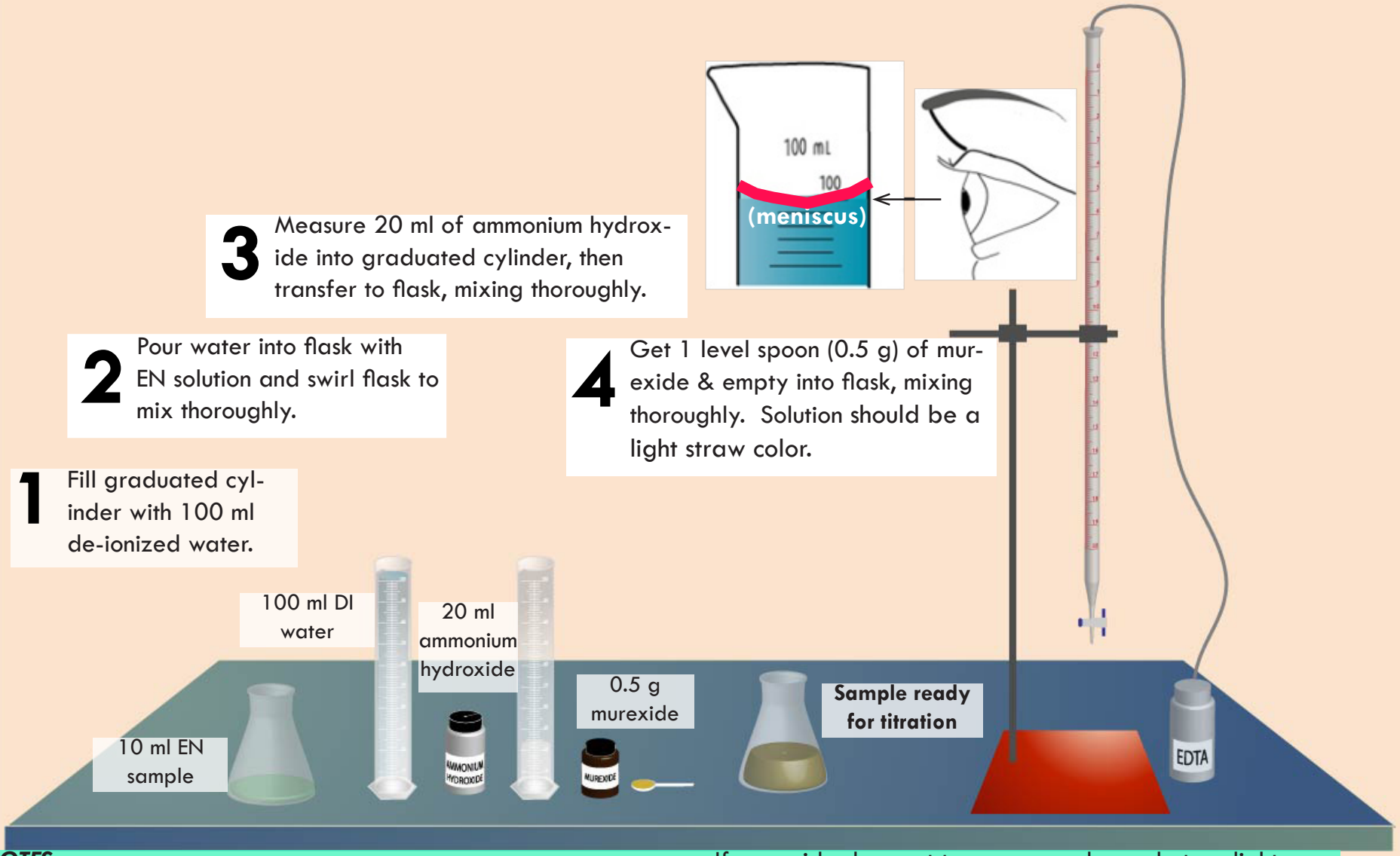
MEASURING & MIXING ADDITIVES

3 Measure 20 ml of ammonium hydroxide into graduated cylinder, then transfer to flask, mixing thoroughly.

2 Pour water into flask with EN solution and swirl flask to mix thoroughly.

4 Get 1 level spoon (0.5 g) of murexide & empty into flask, mixing thoroughly. Solution should be a light straw color.

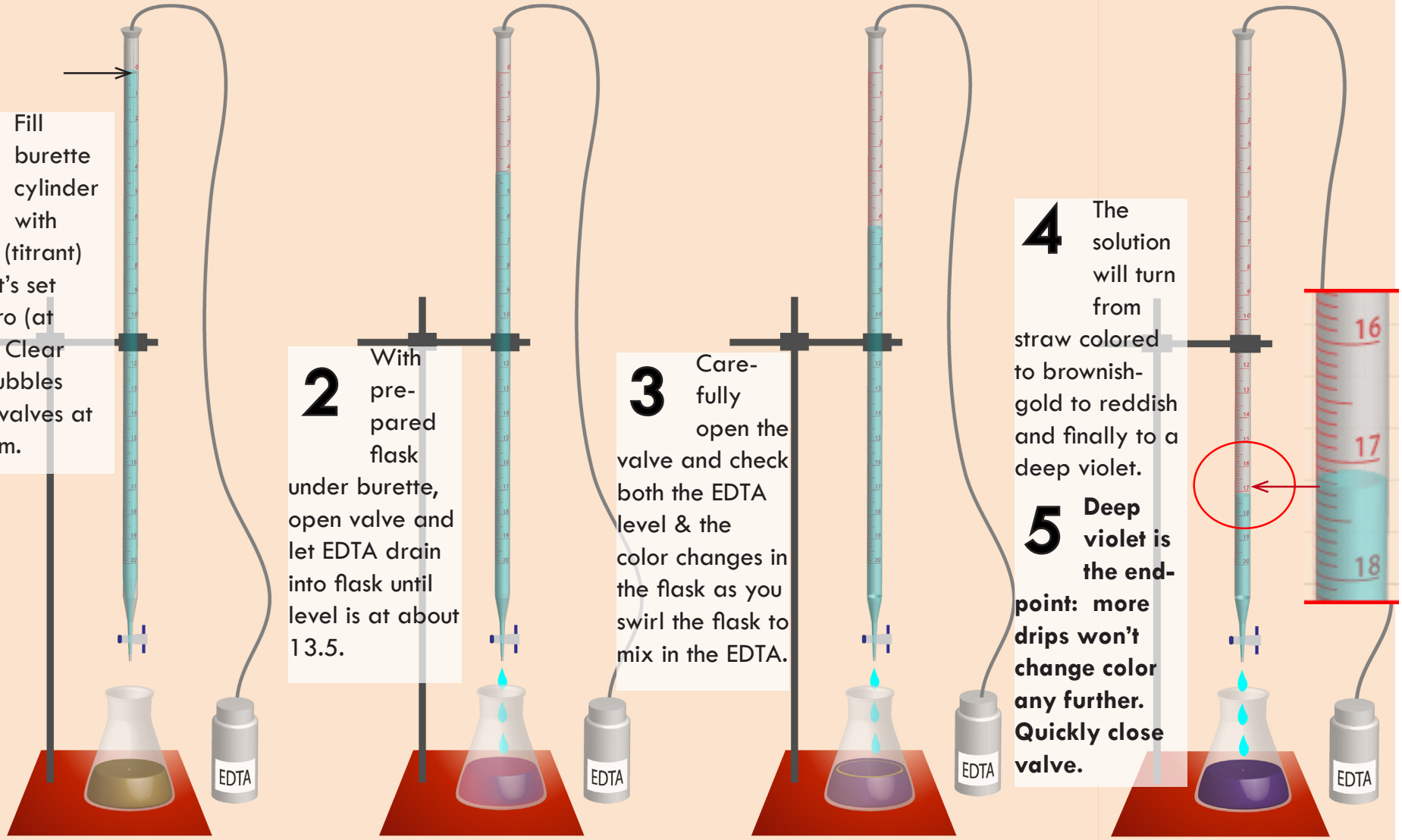
1 Fill graduated cylinder with 100 ml de-ionized water.



NOTES:

- When measuring liquids in a narrow cylinder, the solution will tend to cling to the sides and form a curve called a **meniscus.**
- The measuring devices are meant to be read so that eye level is at bottom of meniscus curve.
- If murexide does not turn prepared sample to a light straw color, discard sample and check quality of murexide.
- ALWAYS WEAR SAFETY GLASSES WHEN USING CHEMICALS.
- AVOID BREATHING IN AMMONIUM HYDORXIDE VAPORS.

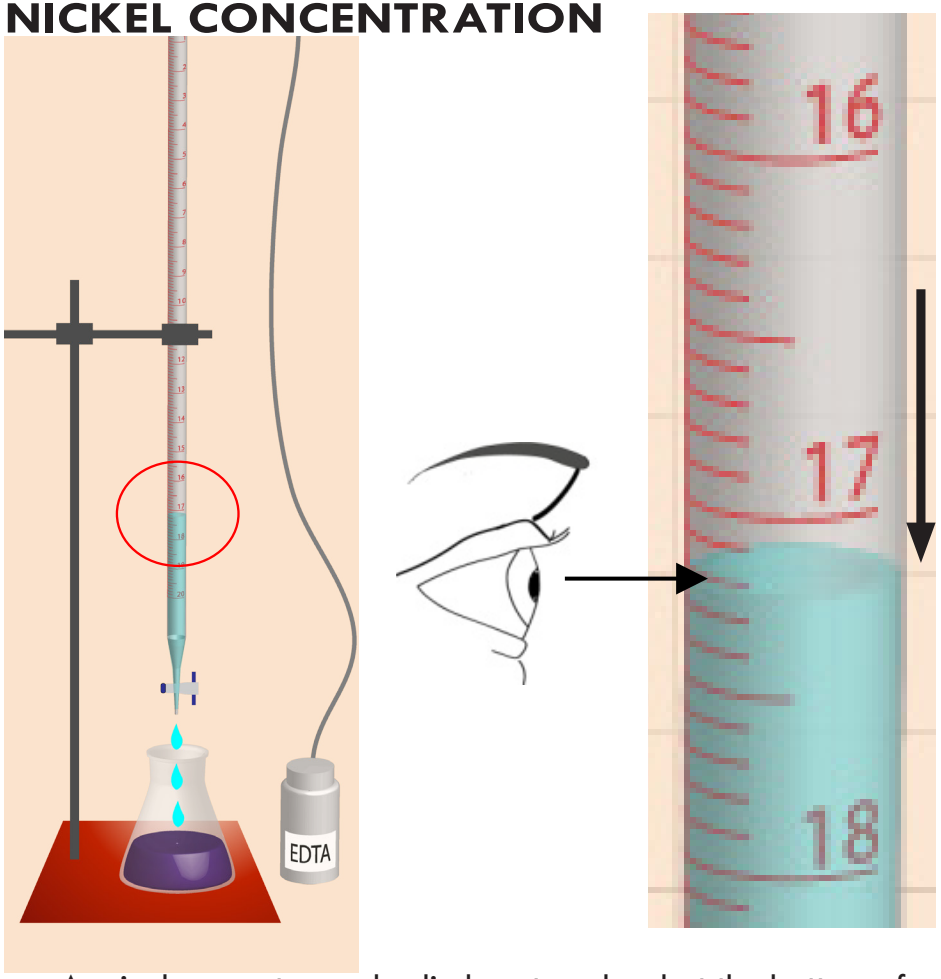
PERFORMING TITRATION



NOTES:

- Controlling the valve carefully requires a little practice. Take it slowly at first until you can control the drips while watching both the level and the color change.
- Slow down as the solution color approaches the endpoint.
- The color change should take around 3 minutes or so.

READING EDTA & CALCULATING NICKEL CONCENTRATION



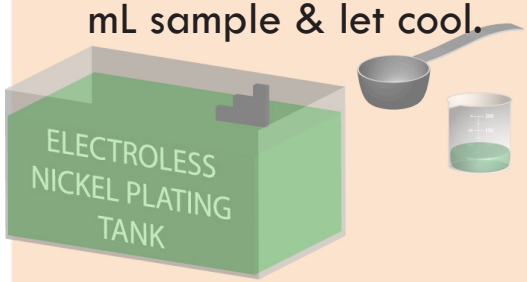
- Again, be sure to read cylinder at eyelevel at the bottom of the meniscus.
- Remember, we're recording **how much EDTA has been used up...**
- **So we read down: here, the level is at 17.2.**
- A common mistake would be to read this as 18.8.
- Using the chart provided, we can see that if we used 17.2 mLs of EDTA to match the nickel in solution, that means that the nickel concentration is 96.8 %.

mLs EDTA	g/L Nickel metal	% Nickel conc.
17.8	6.01	100.2
17.7	5.98	99.6
17.6	5.94	99.0
17.5	5.91	98.4
17.4	5.87	97.8
17.3	5.84	97.3
17.2	5.81	96.8
17.1	5.78	96.3
17.0	5.74	95.7
16.9	5.71	95.1
16.8	5.67	94.5
16.7	5.64	93.9
16.6	5.60	93.3

FINAL STEPS

- Rinse out all equipment and store upside down to drain.
- Replace untested sample in tank.
- Dispose of tested sample properly.

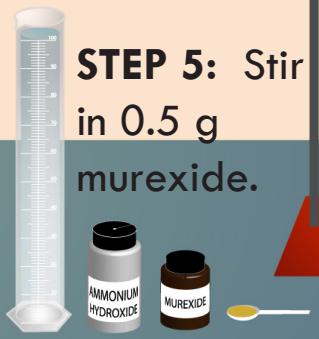
STEP 2: Gather 40-50 mL sample & let cool.



STEP 4: Measure & mix 100 mL DI water & 20 mL ammonium hydroxide into sample flask.

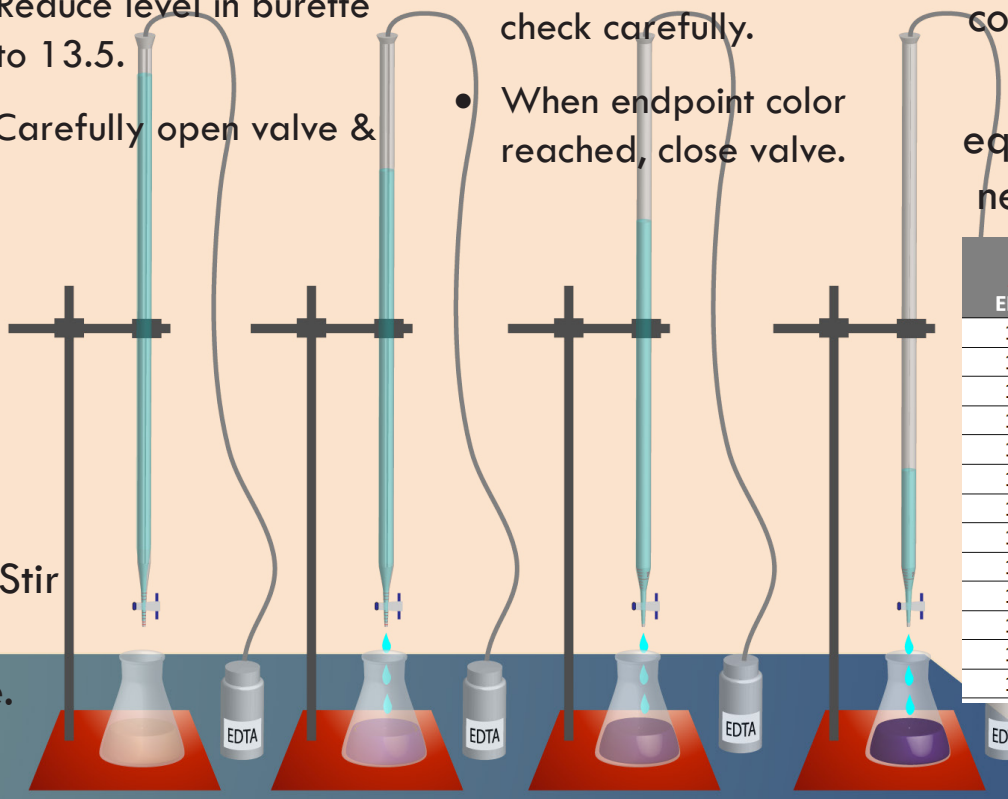
STEP 3: Draw 10 mL & transfer to flask.

STEP 5: Stir in 0.5 g murexide.



STEP 6: TITRATE

- Place prepared sample under tube.
- Set EDTA level to zero.
- Reduce level in burette to 13.5.
- Carefully open valve & let EDTA drip into flask.
- Swirl flask & observe color change.
- When solution nears endpoint color, drip & check carefully.
- When endpoint color reached, close valve.



STEP 7: Record EDTA level & correlate to nickel concentration.

Clean all equipment for next titration.

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17.8	6.01	100.2
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16.7	5.64	93.9
16.6	5.60	93.3

STEP 1: Check all equipment.

OVERVIEW OF STEPS

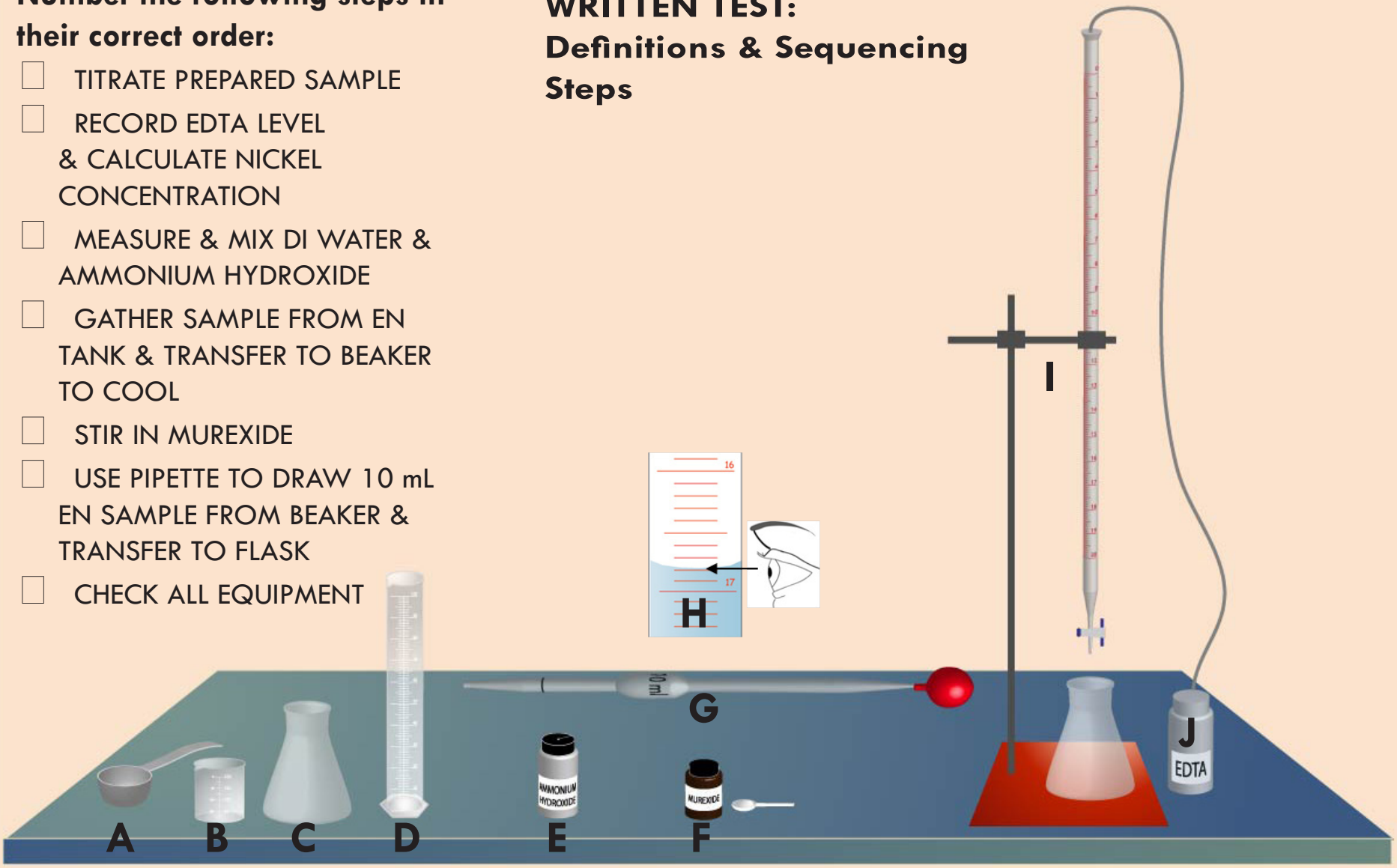
NOTES:

- Step 1: All equipment must be clean or results may be affected.
- Step 2: Make sure tank is within acceptable range.
- Step 3: Rinse pipette with EN solution.
- Step 4: Read measurements of water, ammonium hydroxide from bottom of meniscus.
- Step 5: Use level spoon of murexide.
- Step 6: Don't drip EDTA after endpoint color reached.
- **Step 7:** Record EDTA and nickel concentration immediately to avoid errors.

WRITTEN TEST: Definitions & Sequencing Steps

Number the following steps in their correct order:

- ☐ TITRATE PREPARED SAMPLE
- ☐ RECORD EDTA LEVEL & CALCULATE NICKEL CONCENTRATION
- ☐ MEASURE & MIX DI WATER & AMMONIUM HYDROXIDE
- ☐ GATHER SAMPLE FROM EN TANK & TRANSFER TO BEAKER TO COOL
- ☐ STIR IN MUREXIDE
- ☐ USE PIPETTE TO DRAW 10 mL EN SAMPLE FROM BEAKER & TRANSFER TO FLASK
- ☐ CHECK ALL EQUIPMENT



Match letter on image to name on list:

- ☐ titrant for balancing Ni concentration
- ☐ burette
- ☐ flask
- ☐ beaker
- ☐ pipette w/ bulb
- ☐ solution needed For adjusting pH
- ☐ graduated cylinder
- ☐ powder needed to create color change
- ☐ DI water
- ☐ meniscus

Hands-on test: Instructor's guide

The classroom / individual instruction is designed to provide an overview of the terms, steps, and basic processes of titration.

To enable learners to perform nickel titrations, however, hands-on training is required.

The Job Instruction (JI) Method will enable instructors to ensure that learners can and will perform nickel titrations accurately and consistently.

Use the Job Breakdown on the next page to guide learners through the job.

Step 1: Prepare the learners to perform the task.

- Ask learners what they know about the job already.
- Describe the basic procedure.
- Explain the importance, in terms of quality and productivity, of performing each step correctly.

Step 2: Present the entire job thoroughly, stating each major step to be performed, the key points of each step, and the important reasons underlying those key points.

Step 3: Have each learner perform the titration.

- Ideally, when using the Job Instruction Method, the instructor would require each learner to perform that job four times:
  - once silently,
  - once explaining major steps,
  - once explaining major steps and key points, and
  - once explaining major steps, key points, and important reaons for each key point.
- However, due to the time and resources consumed during each titration, instructors should
  - have each learner perform the task and state the major steps once. While each learner is performing the task,
  - have the other learners to provide the key points about each step, and
  - have the other learners explain the important reasons for each key point.

Step 4: Follow up performance on shop floor.

- When a nickel titration is called for, allow the learner to perform the titration using the job aid provided (see "OVERVIEW OF STEPS", p. 12), & "JOB BREAKDOWN", p. 15).
- Monitor the learner's performance. Encourage questions and be ready to answer any questions the learner might have.
- When the learner has demonstrated proficiency on two separate occasions, certify the learner as having mastered the nickel titration procedure.

NICKEL TITRATION JOB BREAKDOWN

MAJOR STEPS	KEY POINTS	IMPORTANT REASONS
1. CHECK ALL EQUIPMENT & WEAR SAFETY GLASSES.	• Check for contamination. • Rinse carefully.	• Contamination will affect accuracy of results. • Glass lab equipment is easily broken. • Safety glasses will help prevent eye injuries.
2. GATHER 40-50 mL OF ELECTROLESS NICKEL SOLUTION FROM TANK & LET COOL IN BEAKER.	• Make sure solution level in tank is within operating ranges. • Gather more than enough sample than you will need for the titration.	• If solution level is too high or too low, sample will not represent tank solution accurately. • Take enough solution to repeat titration if necessary. • Let the solution cool before testing: hot solution will affect titration results.
3. DRAW 10 mL SOLUTION FROM BEAKER W/ PIPETTE & TRANSFER TO FLASK.	• Draw in & squeeze out enough EN sample to rinse pipette. • Hold pipette vertically & check sample in pipette at bottom of meniscus at eye level.	• Dried solution in pipette from previous titration can affect results. • Use correct reading procedures to get consistent results.
4. MEASURE & MIX IN 100 mL DI WATER & 20 mL AMMONIUM HYDROXIDE.	• Hold graduated cylinders vertically & check amount at bottom of meniscus at eye level.	• Use correct reading procedures to get consistent results.
5. STIR IN 0.5 g MUREXIDE.	• Use level spoon, not heaping.	• Accurate amount of murexide will enable color change to happen consistently.
6. TITRATE	• Place prepared sample under burette tube. • Set EDTA level to zero. • Reduce level in tube to 13.5 & close valve. Swirl flask to mix. • Carefully open valve & let EDTA drip into flask. • Swirl flask & observe color change. • When solution nears endpoint color, slow down drips. • Close valve as soon as endpoint color is reached: do not add EDTA after endpoint reached.	• Reducing the level to a certain point speeds the process up without affecting results. • Swirl flask while mixing to ensure color change reactions are happening as they should. • Adding more EDTA after the endpoint is reached will invalidate the results.
7. RECORD EDTA LEVEL & CORRELATE TO NICKEL CONCENTRATION.	• Record EDTA & correlate to nickel concentration immediately.	• Avoid errors by recording data promptly.