Volumetric Flasks

Measuring Liquids

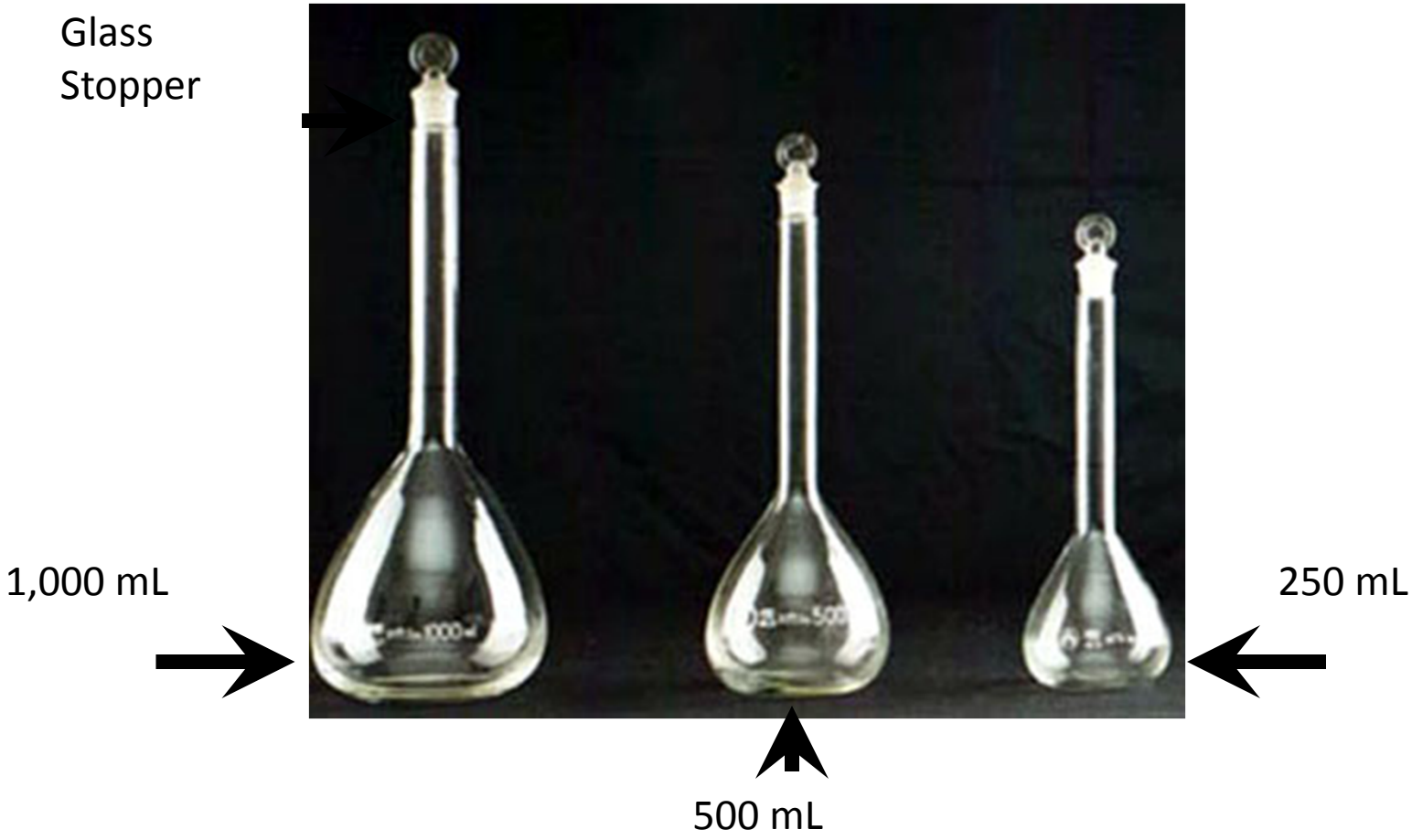
Preparing Dilutions

DEQ

Volumetric Flasks

- Typically are "TC"
- Come in a variety of sizes
  - 25, 50, 100, 250, 500, 1,000 mL
- Only have one graduation
- Can not be used to measure any other volume
- May use plastic caps or ground glass stoppers

Examples



Using the Volumetric Flask to Prepare Dilutions or Standards

DEQ

Preparing Standards and Dilutions

- True (primary) purpose of volumetric flasks
- Allows a volume of one solution (chemical or sample) to be precisely diluted to produce:
  - A sample concentration that is within the limits of the test procedure
  - A chemical solution with a known lower concentration.

Dilution

- Procedure to accurately prepare a new solution with a reduced concentration from an existing solution.
- Uses
  - Reducing the concentration of a sample
  - Preparing a series of standard solutions for use in a laboratory test.

Terminology

- Stock solution
  - Solution used to prepare the dilution
- Standard solution
  - The solution resulting from the dilution of the stock solution.
- Serial Dilution
  - Series of dilutions in which each succeeding dilution has a concentration that is a factor of ten less than the previous dilution.

Note

- Preparing diluted samples or chemicals will require the use of measurement devices (i.e. graduated cylinders, pipets, etc.).
- For accuracy all required measurements must be done using procedures discussed in earlier sections.

Sample Dilution

Purpose

- Reduce sample concentration to levels that can be determined using the selected testing method.
- Examples:
  - Total residual chlorine sample contains 3.5 mg/L of chlorine. Test method is only valid for 0.1 – 2.0 mg/L
  - Bacteria sample contains 500,000 organisms per 100 mL. The method only works for 20 – 60 organisms per 100 mL.

Dilution Methods

- Serial Dilution
- Volumetric Dilution

Equipment Used

- The type of equipment used for dilution depends on the level of accuracy required.
- Minimal accuracy
  - Graduated cylinders
- High Accuracy
  - Volumetric flasks & pipets are required

Serial Dilution

- Serial dilution is:
  - A series of dilutions designed to very accurately measure small volumes.
  - Based on factors of 10 (10, 1, 0.1, 0.01, etc)
  - Uses the previous dilution to prepare the next lower dilution.

Preparing a 0.1 mL Dilution

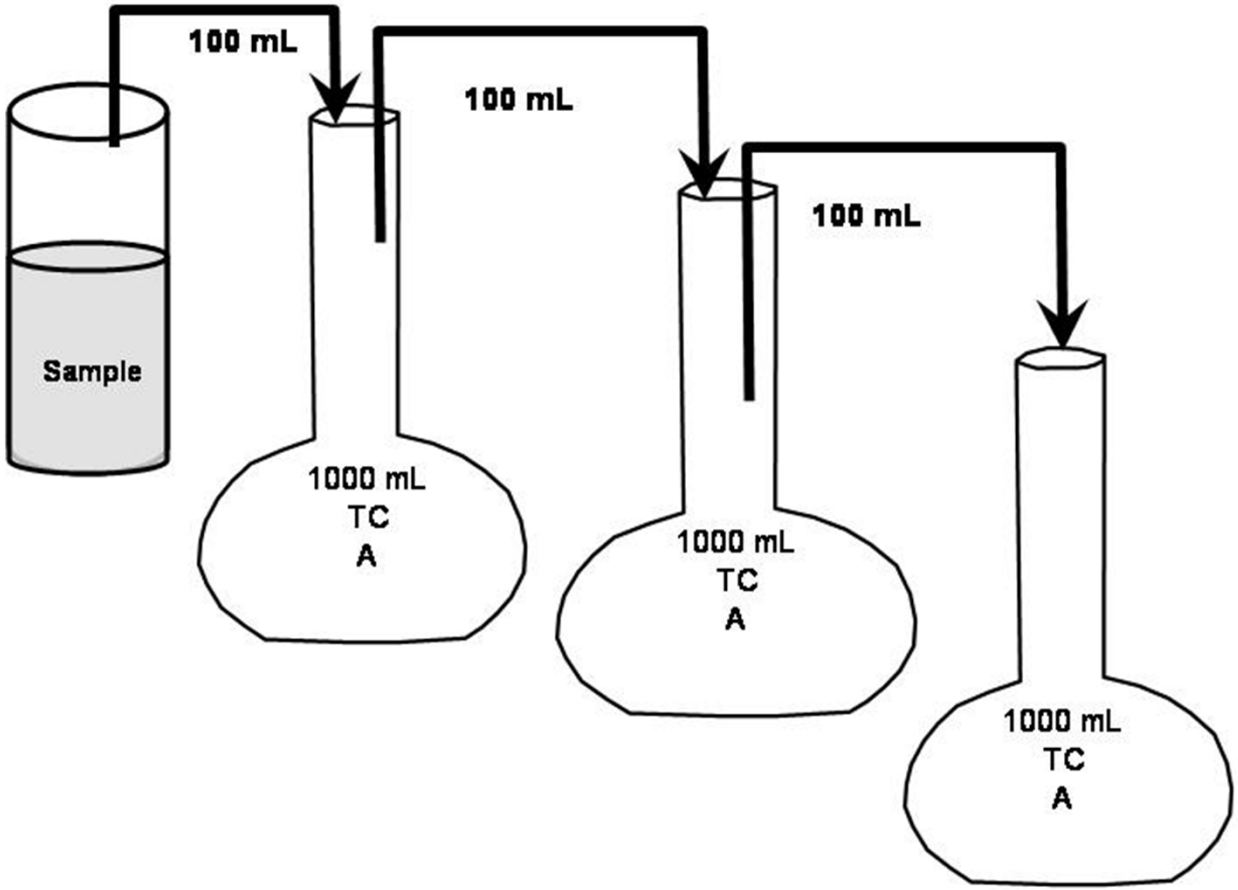
- Starting with the sample
  - Carefully pipette 100 mL of sample into a 1,000 mL volumetric flask
  - Fill the volumetric flask to the line with lab grade water
  - Mix thoroughly
- Each mL of the dilution contains 0.1 mL of the original sample.

Preparing a 0.01 Dilution

- Starting with the 0.1 dilution
  - Pipet 100 mL of the 0.1 dilution into a 1,000 mL volumetric flask
  - Fill the volumetric flask to the line with lab grade water
  - Mix thoroughly
- Each mL of the dilution contains 0.01 mL of the original sample.

Preparing a 0.001 Dilution

- Starting with the 0.01 dilution
  - Pipet 100 mL of the 0.01 dilution into a 1,000 mL volumetric flask
  - Fill the volumetric flask to the line with lab grade water
  - Mix thoroughly
- Each mL of the dilution contains 0.001 mL of the original sample



Continue until the required sample concentration is achieved

Target Concentrations

- Some test procedures cover a limited range of concentrations
- Samples with higher levels must be diluted before testing
- Requires some knowledge of the sample
  - Results of past analyses
  - Results for similar samples
- Initial testing always requires multiple dilutions to cover range of results

Procedure

- Determine concentration range of test procedure
- Based on available information determine expected concentration range of sample
- Using calculation determine volume of sample to be diluted to produce a concentration within the range of the test.

Calculation

Highest Sample Volume

$$Volume_{High} = \frac{Volume_{Test} \times Concentration_{Test\ High}}{Concentration_{Sample\ High}}$$

Lowest Sample Volume

$$Volume_{Low} = \frac{Volume_{Test} \times Concentration_{Test\ Low}}{Concentration_{Sample\ Low}}$$

Note

- Since concentrations can vary, it is best to select several dilution concentrations to ensure one or more will fall within the range of the test.

- Example:
- The phosphorus test requires:
  - A 50 mL sample volume
  - A concentration in the range 2 – 5 mg/L
  - Previous tests indicate the sample has a concentration of 12.5 – 18 mg/L

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  - A 50 mL sample volume
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Step 1

- Calculate the sample volume required to fall within the upper concentration limit.

$$Volume_{High} = \frac{Volume_{Test} \times Concentration_{Test\ High}}{Concentration_{Sample\ High}}$$

$$= \frac{50\ mL \times 5\ mg/L}{18\ mg/L}$$

$$= 13.9\ mL$$

- Based on this 13.9 mL of sample would be diluted to 50 mL

Step 2

- Calculate the sample volume required to fall within the lower concentration limit.

$$Volume_{Low} = \frac{Volume_{Test} \times Concentration_{Test\ Low}}{Concentration_{Sample\ Low}}$$

$$= \frac{50\ mL \times 2\ mg/L}{12.5\ mg/L}$$

$$= 8\ mL$$

- Based on this 8 mL of sample would be diluted to 50 mL

Note

- In most cases, it would be more appropriate to prepare larger volumes of the diluted samples. This would:
  - Provide extra diluted sample for spikes, duplicates, and other QA related requirements.
  - Reduce potential preparation errors

Chemical Dilutions

- Not all labs do this
- Useful when:
  - Solutions are not available at the required concentration
  - Shelf life is extremely short at the required concentration

Dilution Formula

- Works with any concentrations if:
  - Stock and diluted concentrations are expressed in the same units (i.e. %, mg/L, Normality, Molarity, etc.)
  - Stock and diluted volumes are expressed in the same units (mL, L, gallons, etc.)
- Formula can also be used when preparing dilutions of process chemicals

Dilution Formula

$$Volume_1 = \frac{Volume_2 \times Concentration_2}{Concentration_1}$$

1 = Stock Solution (solution used to prepare dilution)

2 = Standard Solution (solution being prepared)

Dilution Procedure

- Determine the volume of stock solution needed to prepare the desired volume of the standard (dilute) solution
- Using a volumetric pipet, transfer the required volume to a volumetric flask
- Fill the volumetric flask to about 1 inch below the graduation

Dilution Procedure

- Insert the stopper and mix
- If required, let the contents of the volumetric flask return to room temperature
- Finish filling the volumetric flask so the meniscus is just sitting on the line

Example

- Prepare 1,000 mL of 0.025N sodium thiosulfate solution. The concentration of the stock sodium thiosulfate solution is 1.0N

Step 1

- Calculate the required volume of stock solution.

$$Volume_{1.0N} = \frac{1{,}000\ mL_{0.025N} \times 0.025N}{1.0\ N}$$

$$= 25\ mL$$

Step 2

- Pour a small amount of the stock solution into a clean beaker and swirl to wet the inside surfaces

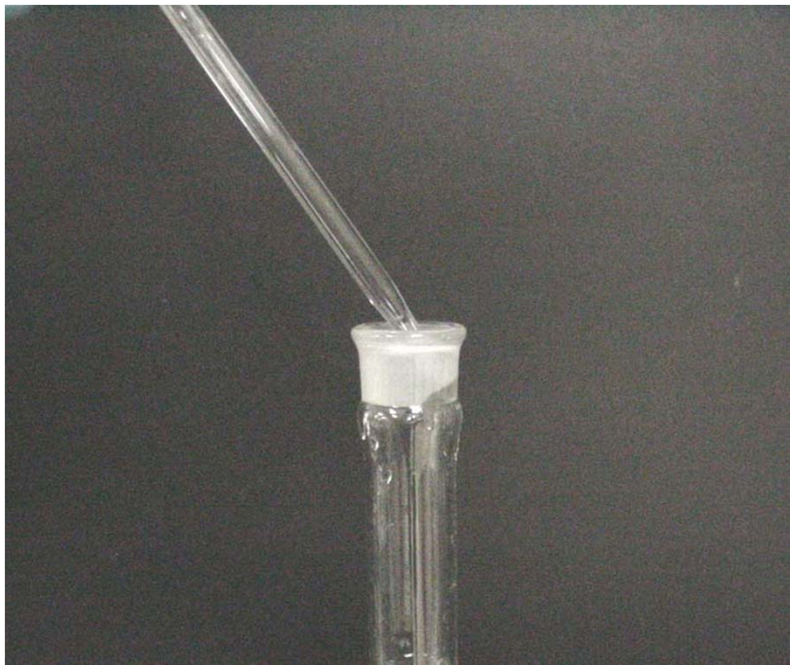
Step 3

- Discard the thiosulfate used to rinse the beaker

Step 4

- Pour enough 1.0 N Thiosulfate into the beaker to:
  - Rinse the pipet
  - Provide the required 25 mL

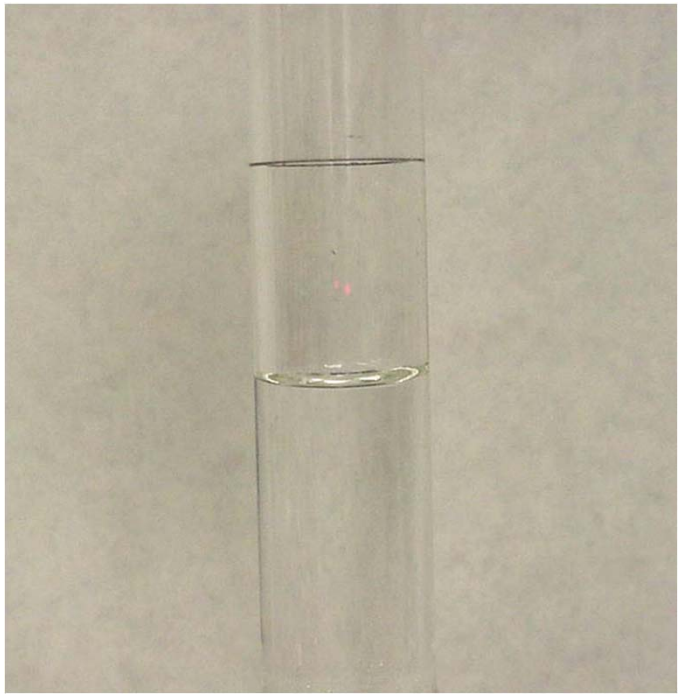
Step 5



- Pipet 25 mL of 1.0N sodium thiosulfate solution into a 1,000 mL volumetric flask

Pipetting must be done using the procedure discussed earlier

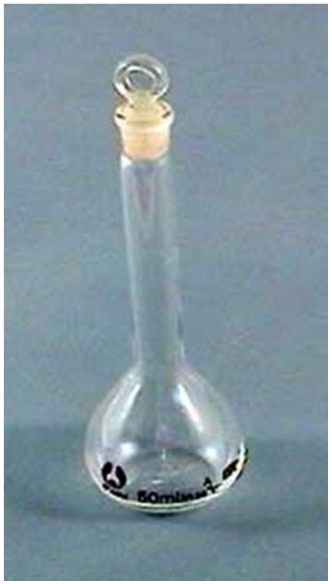
Step 6



- Fill the volumetric flask with lab grade water until the meniscus is approximately 1 inch below the graduation.
- Type of water to be used for dilution may vary with the procedure the chemical is being prepared for (i.e. ammonia free water for nitrogen)

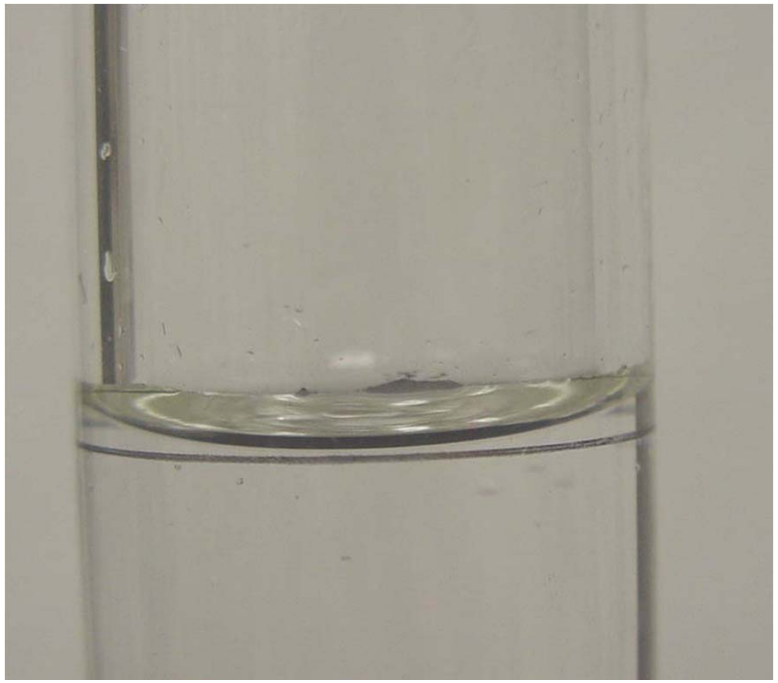
Step 7

- Replace the stopper and mix thoroughly



- If needed allow the contents of the flask to return to room temperature.

Step 8



- Carefully remove the stopper
- Using a dropper add lab grade water until the meniscus is just sitting on the line.

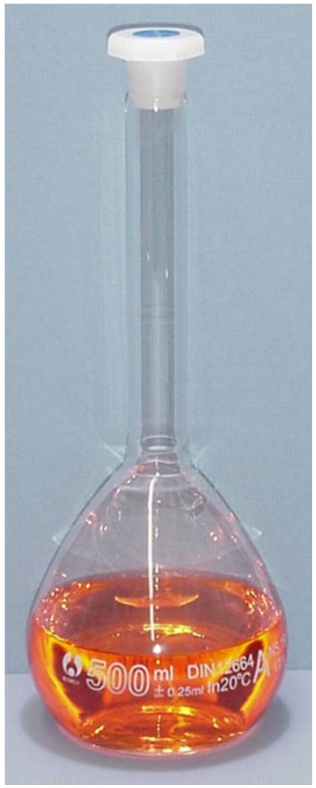
Step 9

- Replace the stopper and mix again

Step 10

- Using appropriate technique, transfer the standard solution to a properly labeled storage bottle
- Remember
  - Transfer a small volume
  - Rinse the inside of the bottle completely
  - Discard
  - Fill the bottle

Using Volumetric Flasks For Measuring Volumes



- Secondary use
- Not really designed for measurement
- Can be useful when precise measurement of larger volumes (i.e. 50 – 1,000 mL) is required.
- Normally are ‘TC’ devices

Note

- Using a volumetric flask to measure volumes is a much different procedure than that used when preparing standards or dilutions.

Use the following procedure only when using the volumetric flask as a measurement device.

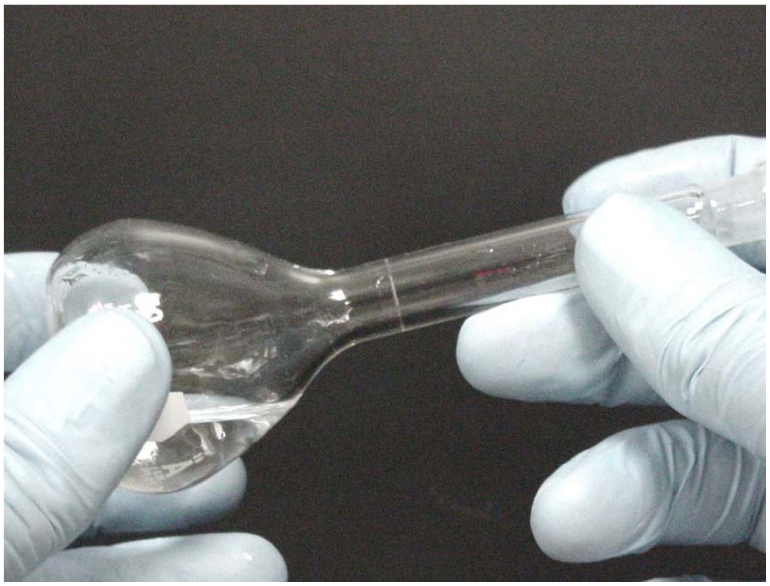
Preparation

- Mix the solution (chemical or sample) to be measured thoroughly
- Pour small amount into a clean beaker (capacity selected based on volume of solution to be measured).
- Rotate the beaker until all the interior surfaces have been rinsed
- Discard the solution
- Refill the beaker with the solution to be measured

Flask Step 1

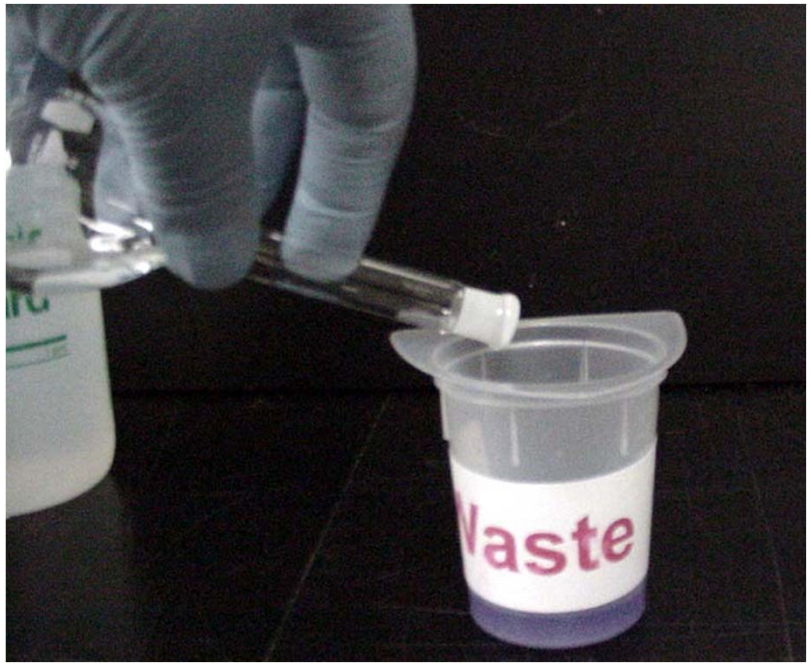
- Using a funnel, pour a small amount of the solution from the beaker into the volumetric flask to be used as a measurement device.

Flask - Step 2



- Rotate the flask so that the solution wets the entire interior surface of the flask.

Flask - Step 3

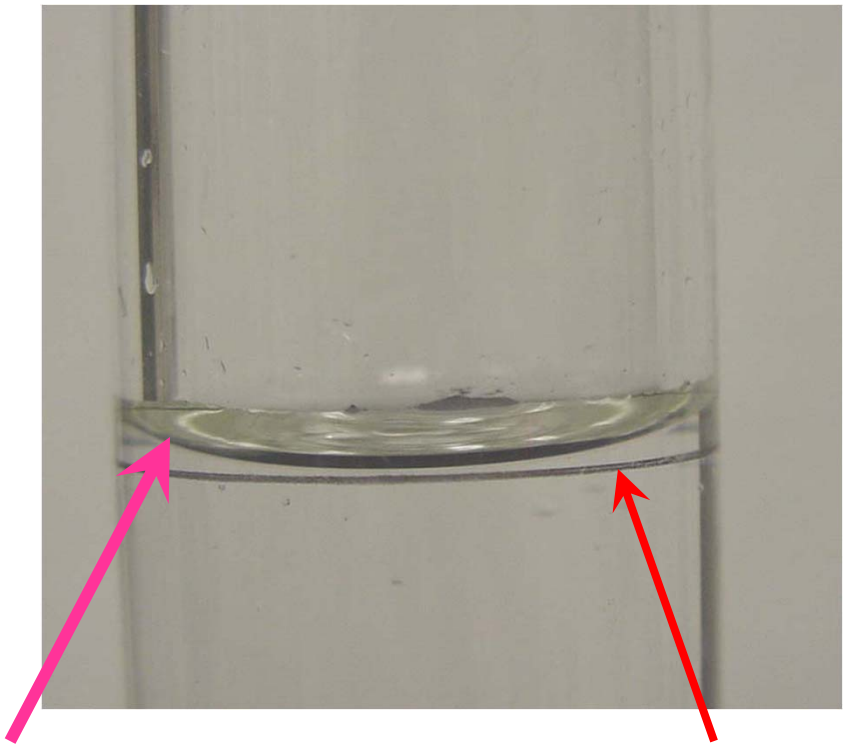


- Drain the volumetric flask into the waste container

Flask - Step 4

- Using the solution in the beaker, refill the volumetric flask to mark.

Flask - Step 5

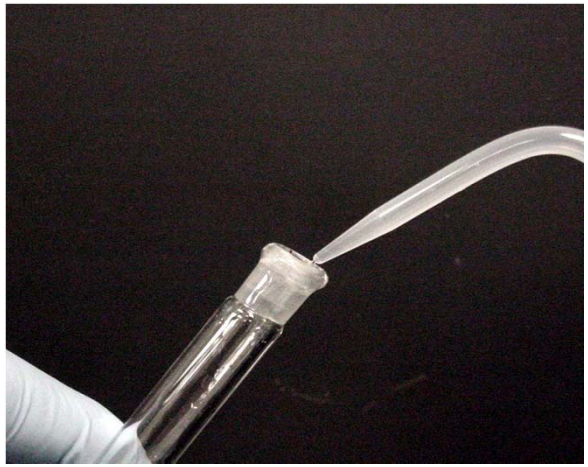


- If necessary use a disposable pipet (dropper) to adjust the volume in the flask until the meniscus is just touching the line.

Flask - Step 6

- Using a funnel, if needed, *Carefully* transfer the contents of the volumetric flask to the designated beaker, flask or other container.

Flask - Step 7



- Rinse the volumetric flask used to measure the volume 3X with lab grade water.

- Add each rinse to the designated (same) beaker, flask or other container

Not!!!

- Extreme care is needed to ensure that all solution and rinse water is transferred to the receiving container.
- The volume of rinse water used depends on:
  - The size volumetric flask being used
  - Final volume requirements of the procedure the solution will be used for.
- This procedure is used for measuring volumes using a volumetric flask.

Department of Environmental Quality