Name_____Lab Day_____Lab Time_____

Experiment 2 · Analysis of magnesium

Pre-lab questions

Answer these questions and hand them to the TF before beginning work.

(1) What is the purpose of this experiment?

_____

_____

(2) You will take 25 mL of a solution that has 0.01 mol Mg^{2+}/L and dilute it to 100 mL. What is the concentration of Mg^{2+} in the new solution in units of moles per liter? Show the calculation.

(3) You will "prepare" several glassware items before use; what does it mean to "prepare" glassware?

_____

_____

(4) You will titrate $Mg(NO_3)_2$(aq) against $EDTA^{4-}$(aq) to the endpoint of the indicator Eriochrome Black T: which solution will be placed in the burette?

_____

_____

(5) You will titrate $Mg(NO_3)_2$(aq) against $EDTA^{4-}$(aq) to the endpoint of the indicator Eriochrome Black T: how will you know when the endpoint is reached?

_____

_____

Analysis of magnesium

Magnesium (Mg) ranks as the fourth most abundant terrestrial element – only iron, oxygen, and silicon are more plentiful. Because it is highly reactive, free magnesium metal, that is, Mg in its zero oxidation state, does not occur naturally on Earth: all the magnesium in minerals, seawater, and in living organisms is present as the divalent cation Mg^{2+}.

An adult human has about 24 g of magnesium, 80% of which is in the skeleton and in muscle; magnesium concentrations in bodily fluids typically range from 0.7–1.0 mmol/L. Mg^{2+} plays an essential role in the biochemistry of all living cells, where its interaction with DNA, RNA, ATP, chlorophyll, and hundreds of enzymes is required for normal function.

Spices, nuts, cereals, coffee, cocoa, tea, and leafy green vegetables are good sources of magnesium. Human magnesium deficiency is relatively rare although only about 30% of adults in the United States consume the recommended daily allowance (RDA) of magnesium, which is 300–400 mg/day. Low levels of magnesium in the body have been associated with asthma, diabetes, and osteoporosis. Taken in proper amount magnesium prevents stroke, heart attack, and reduces symptoms due to fibromyalgia, migraines, and PMS. Magnesium supplements are available for those individuals who are diagnosed with magnesium deficiency. You will be analyzing one such supplement in pill form.

Procedure

Preliminaries

Do not contaminate the stock solutions! Pour the approximate volumes of $Mg^{2+}(aq)$ and $EDTA^{4-}(aq)$ stock solutions that you need into the 250-mL beakers, take the beakers back to your lab bench and continue working with the solutions there. If you take too much solution, do not pour the excess back into the reagent bottle: dispose of the excess in a hazardous-waste receptacle.

Clean, rinse with deionized water and dry two 250-mL beakers. Place about 100 mL of $EDTA^{4-}(aq)$ stock solution in one beaker and label it. The concentration of the $EDTA^{4-}(aq)$ stock solution is 0.010 *M*, that is, there are 0.010 moles of $EDTA^{4-}$ per liter of solution. Place about 100 mL of magnesium nitrate ($Mg(NO_3)_2(aq)$) stock solution in the second beaker and label it. The concentration of the $Mg(NO_3)_2(aq)$ stock solution is 0.010 *M*, that is, there are 0.010 moles of $Mg(NO_3)_2$ per liter of solution.

Obtain a burette and fasten it in a burette holder (see Figures 2-1 and 2-2). Lower the burette or the burette holder to a comfortable working height. Before using the burette it must be prepared by coating its interior surfaces with $EDTA^{4-}(aq)$ stock solution. The purpose of this operation is to dissolve away any contaminants that may be present in the burette. Close the stopcock, position a funnel in the barrel of the burette and pour through enough $EDTA^{4-}(aq)$ so that the burette is about 1/5 full. Remove the funnel from the burette and the burette from the holder; now tip and rotate the burette, coating all inside surfaces. Return the burette to the upright position, open the stopcock and allow the $EDTA^{4-}(aq)$ to drain into a hazardous-waste receptacle. Repeat this procedure again with fresh $EDTA^{4-}(aq)$. After you have prepared the burette, fill it to below the highest graduation mark with $EDTA^{4-}(aq)$.

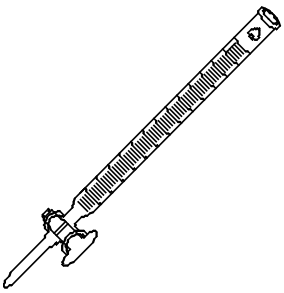


Figure 2-1 A burette.

Obtain a 25-mL volumetric pipette and a 25-mL pipette pump. You will now prepare the volumetric pipette for use. After making sure that the pump's plunger is down, insert the pipette into the pump (see Figure 2-3) and, by rotating the wheel that lifts the plunger, draw in enough $Mg(NO_3)_2(aq)$ stock solution so that the pipette is about 1/5 full. Remove the pump and quickly stopper the end of the pipette with finger pressure so that the liquid does not run out. Tip the pipette at an angle, release finger pressure slightly and twirl the pipette, allowing the solution to coat the inside surface of the pipette. Return the pipette to the upright position and let the solution

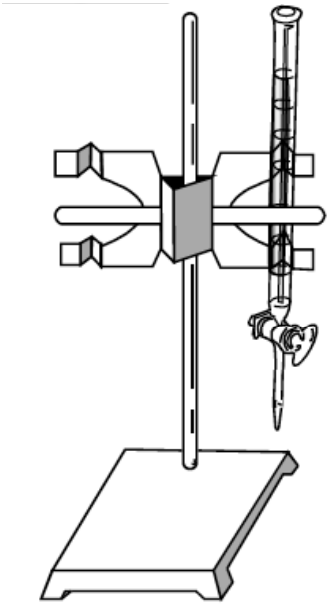


Figure 2-2 A burette fastened in a burette holder.

Figure 2-3 A volumetric pipette fitted with a pipette pump.

drain into a hazardous-waste receptacle. Repeat this procedure again with fresh $Mg(NO_3)_2(aq)$.

Obtain a 100-mL volumetric flask (see Figure 2-4). You will now prepare the volumetric flask for use. Position a funnel in the neck of the flask and pour through enough deionized water so that the flask is about 1/5 full. Swirl and rotate the flask, coating all inside surfaces; dispose of the water in a hazardous-waste receptacle.

Using the prepared 25-mL volumetric pipette – **do not use a graduated cylinder! it is not accurate enough!** – and a pipette pump, transfer exactly 25 mL of $Mg(NO_3)_2(aq)$ stock solution to the prepared 100-mL volumetric flask, then dilute to the mark with deionized water. The concentration of $Mg^{2+}(aq)$ in units of mole of $Mg^{2+}(aq)$ per liter in this new $Mg(NO_3)_2(aq)$ solution is the answer to pre-lab question (2). Place about 25 mL of ammonia (NH_3/NH_4OH) buffer solution in a beaker and cover the beaker with a watchglass (looks like a huge contact lens): ammonia fumes irritate your eyes.

Titrating solutions containing a known amount of Mg^{2+}

You are now almost ready to begin your first titration. You will add $EDTA^{4-}(aq)$ to a solution containing a known amount of $Mg(NO_3)_2(aq)$ and the dye Eriochrome Black T (EBT) until you observe a red-to-blue color change. The chemistry of the titration reaction is discussed in Box 2-1. Your first titration will be the most difficult to execute because you initially have no idea how much $EDTA^{4-}(aq)$ to add in order to realize the color change.

Pipette exactly 25 mL of $Mg(NO_3)_2(aq)$ stock solution into a clean and dry 250-mL Erlenmeyer flask (see Figure 2-5). Add ammonia buffer so that the pH of the solution is about 10: a dropperful should be sufficient, but check using pH paper. Ask the TF to add Eriochrome Black T (EBT) indicator; the solution must turn a deep wine-red color. Place a piece of white paper under the Erlenmeyer flask so that you can more easily perceive the color changes that are about to take place.

Record the initial volume reading on the burette to two decimal places, open the stopcock of the burette, add a few milliliters of the $EDTA^{4-}(aq)$ to the $Mg(NO_3)_2(aq)$ in the Erlenmeyer flask, close the stopcock and swirl the flask. If a blue

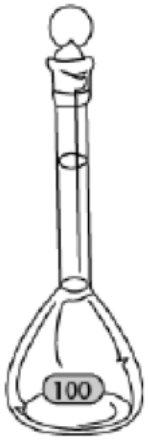


Figure 2-4 A 100-mL volumetric flask.

color doesn't appear, you are far from the red-to-blue color change (i.e., the endpoint of the titration). Continue adding a few milliliters of $EDTA^{4-}(aq)$ at a time and note the color as before. When a blue or purple color begins to make its first appearance, the endpoint is close and you must begin adding $EDTA^{4-}(aq)$ dropwise and with constant swirling. The endpoint of the titration is reached when just one drop of the $EDTA^{4-}(aq)$ changes the color of the $Mg(NO_3)_2(aq)$ to a permanent pure blue color with no trace of purple remaining. **Do not overshoot the endpoint by adding too much $EDTA^{4-}(aq)$!** Record the final volume reading on the burette to two decimal places and dispose of the solution that you just titrated in a hazardous-waste receptacle.

Repeat the titration procedure on a fresh 25-mL sample of the $Mg(NO_3)_2(aq)$ stock solution. You can complete your second titration more quickly than the first because you now know approximately how much $EDTA^{4-}(aq)$ is needed to reach the endpoint. Once again, do not overshoot the endpoint by adding too much $EDTA^{4-}(aq)$. Record the initial and final volume readings on the burette to two decimal places and dispose of the solution that you just titrated in a hazardous-waste receptacle.

Next, perform the titration procedure on a fresh 25-mL sample of the $Mg(NO_3)_2(aq)$ solution you prepared. Finally, repeat the titration procedure on a second fresh 25-mL sample of the $Mg(NO_3)_2(aq)$ solution you prepared. At this point you should have performed a total of four titrations: two on the $Mg(NO_3)_2(aq)$ stock solution and two on the $Mg(NO_3)_2(aq)$ solution you prepared.

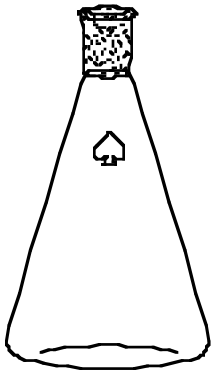


Figure 2-5 An Erlenmeyer flask.

Determination of reaction stoichiometry

The titration data you have just collected must now be worked up to establish the coefficients *x* and *y* and the charge *n* on the product of the reaction between $Mg^{2+}(aq)$ and $EDTA^{4-}(aq)$:

$$x\,Mg^{2+}(aq) + y\,EDTA^{4-}(aq) \rightarrow Mg_x(EDTA)_y{}^n(aq) \quad \text{(Eqn. 2-1)}$$

The ratio of moles of Mg^{2+} (x) to moles of $EDTA^{4-}$ (y) is given by

Box 2-1 At the start of the titration, magnesium ion (Mg^{2+}) is associated with Eriochrome Black T (EBT): the resulting Mg^{2+}·EBT complex has a characteristic wine-red color. As $EDTA^{4-}$ is added, $EDTA^{4-}$ begins to replace EBT in the complex: the products of this reaction are $Mg_x(EDTA)_y^n$ and free EBT, which is blue:

$$\underset{\textit{wine-red}}{x\,Mg^{2+}\cdot EBT} + y\,EDTA^{4-} \rightarrow Mg_x(EDTA)_y^n + \underset{\textit{blue}}{x\,EBT}$$

At the endpoint of the titration, all of the EBT has been displaced and the solution assumes the blue color characteristic of free, unassociated EBT.

$$\frac{x\text{ mol } Mg^{2+}}{y\text{ mol EDTA}} = \frac{C_{Mg}V_{Mg}}{C_{EDTA}V_{EDTA}} \qquad \text{(Eqn. 2-2)}$$

where C_{Mg} is the concentration of Mg^{2+}(aq) in units of mole of Mg^{2+} per liter, V_{Mg} is the volume of Mg^{2+}(aq) in units of liter titrated, C_{EDTA} is the concentration of $EDTA^{4-}$(aq) in units of mole of $EDTA^{4-}$ per liter, and V_{EDTA} is the volume of $EDTA^{4-}$(aq) in units of liter needed to reach the endpoint. If you followed instructions, V_{Mg} = 0.025 L and C_{EDTA} = 0.010 mol/L. **Important!** Keep in mind that C_{Mg} is not the same for all four titrations: C_{Mg} = 0.010 mol/L when the stock solution is titrated, but C_{Mg} has a different value (the answer to pre-lab question (2)) when the magnesium solution you prepared is titrated.

Calculate the value of x/y of each of your four runs (two using the data obtained when you titrated the $Mg(NO_3)_2$(aq) stock solution and two using the data obtained when you titrated the $Mg(NO_3)_2$(aq) solution you prepared). Using the formulas given in Appendix A "Statistical Treatment of Data" of this lab manual, calculate the mean value of x/y, the standard deviation of the mean, and the 95% confidence interval about the mean. **Do not wait to do these calculations at home after lab!** If your data are no good, you must know so immediately so that you can take remedial action.

Table 2-1 Possible values of x/y in Eqn. 2-1.

decimal	*x*	*y*
0.25	1	4
0.33	1	3
0.50	1	2
0.67	2	3
0.75	3	4
1.00	1	1
1.33	4	3
1.50	3	2
2.00	2	1
3.00	3	1
4.00	4	1

The value of x/y should work out to a value approximating the ratio of two small whole numbers (see Table 2-1 for some possibilities). If the confidence interval you calculate is so large that it contains more than one of the possibilities listed in Table 2-1, your data are not sufficiently precise and you must perform more titrations to narrow down the possibilities.

Preparing the magnesium pill solution for analysis

Obtain a magnesium pill and weigh it to two decimal places; record the weight in your notebook. Crush the pill to a fine powder in a mortar and pestle. Tare a 250-mL beaker and transfer about 0.1 g of the powder to the beaker; be sure to record the mass of powder transferred to the beaker to two decimal places. Do not discard the powder remaining in the mortar: you may need it later if something goes wrong with your experiment.

Add 40 mL of 1.5 *M* hydrochloric acid (HCl) solution to the beaker to dissolve as much of the powder as possible; stirring with a glass rod and swirling will be necessary. The pill contains insoluble starch and binders so don't be concerned if the solution is not crystal clear at this point: you will remove undissolved solids in the next step.

Obtain a ring stand, a Büchner funnel, a filter flask equipped with a side-arm, heavy-walled tubing, a filtervac and filter paper. Make sure that the filter flask is clean; otherwise, wash it with detergent. The filter paper must lie perfectly flat on the floor of the funnel, yet cover all the holes.

Assemble the apparatus sketched in Figure 2-6, ensuring that the filter flask is securely clamped to a ring stand. Turn on the vacuum located on your bench top. Swirl the beaker containing the pill solution to mobilize the contents and then slowly pour a portion **(not all of it!)** of the solution down a stirring rod into the funnel. Swirl the beaker once again to mobilize the contents and pour another portion of the solution down a stirring rod into the funnel. Continue in this manner until all the solution has been filtered. Rinse the beaker with a 10-mL portion of deionized water and pour the rinse through the residue on the filter paper. Turn off the vacuum.

The liquid that collects in the filter flask (called the "filtrate") should be clear and all the solid residue should have

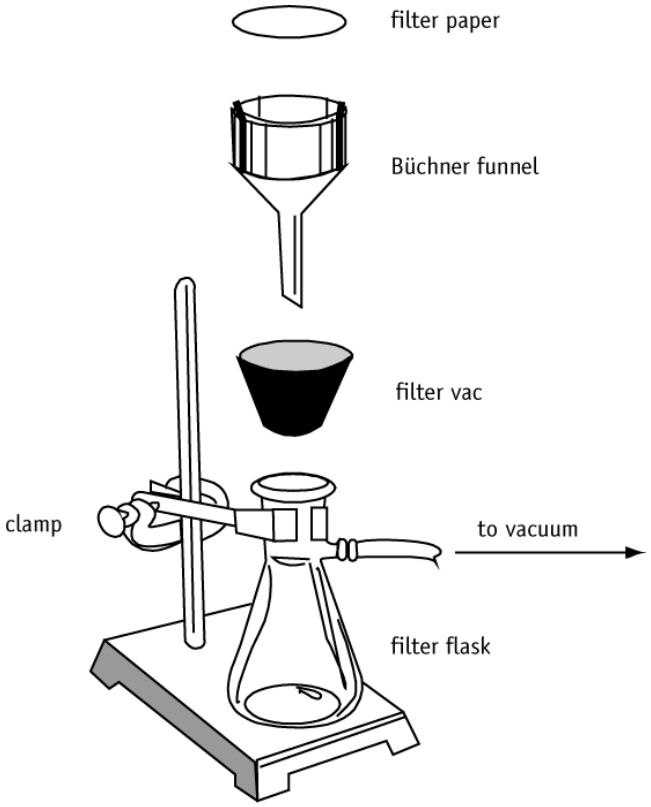
been trapped on the filter paper. Yes, if the filtrate is not free of suspended solids, the solution should be filtered again.

Obtain a clean 100-mL volumetric flask and rinse it with deionized water; dispose of the water in a hazardous-waste receptacle. Transfer the filtrate to the 100-mL volumetric flask and dilute to the mark with deionized water.

Titrating the magnesium pill solution

You are now ready to analyze the pill solution to determine the amount of magnesium it contains. Titrate at least two fresh 25-mL samples of the magnesium pill solution against the $EDTA^{4-}(aq)$ stock solution following the same procedure that you used earlier in the "Titrating solutions containing a known amount of Mg^{2+}" section of this lab. **Important!** You may have to add two or three dropperfuls of ammonia buffer to obtain a pH of about 10: check using pH paper.

Figure 2-6 Filtration apparatus.



Data analysis

The goal here is to determine the mass of magnesium $m_{\text{Mg,pill}}$ in the original pill.

Because by this point you know how many moles of Mg^{2+} react per mole of $EDTA^{4-}$, that is, you know the quantity *x/y* in

$$x\ Mg^{2+}(aq)\ +\ y\ EDTA^{4-}(aq)\ \rightarrow\ Mg_x(EDTA)_y^{n}(aq) \qquad \text{(Eqn. 2-1)}$$

the number of moles of Mg^{2+} in each of the 25.00-mL pill solution samples you titrated is equal to

$$\text{mole } Mg^{2+} \text{ in each 25.00-mL sample} = (x/y)(C_{\text{EDTA}}V_{\text{EDTA}})$$

where C_{EDTA} is the concentration of $EDTA^{4-}(aq)$ (i.e., 0.01 mol/L) and V_{EDTA} is the volume of $EDTA^{4-}(aq)$ in units of liter needed to reach the endpoint. Each 25.00-mL sample of pill solution you titrated contains one-fourth of the amount of Mg^{2+} in the 100-mL of pill solution you prepared, so the total number of moles of Mg^{2+} in the 100-mL of pill solution is equal to

$$\text{mole } Mg^{2+} \text{ in pill solution} = (x/y)(4C_{\text{EDTA}}V_{\text{EDTA}})$$

Recall that you did not analyze the entire pill. You only analyzed about 0.1 g of the powdered pill; call this amount m_{powder} and call the mass of the original pill m_{pill}. Thus, the total number of moles of Mg^{2+} in the pill is equal to

$$\text{mole } Mg^{2+} \text{ in pill} = (x/y)(4C_{\text{EDTA}}V_{\text{EDTA}})(m_{\text{pill}}/m_{\text{powder}})$$

Finally, the mass of Mg^{2+} in the pill $m_{\text{Mg,pill}}$ is given by

$$m_{\text{Mg,pill}} = (x/y)(4C_{\text{EDTA}}V_{\text{EDTA}})(m_{\text{pill}}/m_{\text{powder}})(24.30\text{ g Mg}^{2+}/\text{mol Mg}^{2+}) \qquad \text{(Eqn. 2-3)}$$

Calculate $m_{\text{Mg,pill}}$ for each of your two runs and report the average.

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Experiment 2 · Analysis of magnesium

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(I) Report the data relating to the titrations of the $Mg(NO_3)_2(aq)$ stock solution and of the $Mg(NO_3)_2(aq)$ solution you prepared. Using Eqn. 2-2,

$$\frac{x \text{ mol } Mg^{2+}}{y \text{ mol EDTA}} = \frac{C_{Mg}V_{Mg}}{C_{EDTA}V_{EDTA}} \qquad \text{(Eqn. 2-2)}$$

and using the formulas in Appendix A "Statistical Treatment of Data" of this lab manual, calculate the mean value of x/y **as a decimal number**, the standard deviation of the mean, and the 95% confidence interval.

Run	C_{Mg} [mol/L]	V_{Mg} [mL]	C_{EDTA} [mol/L]	Volume readings on the burette: Initial [mL]	Volume readings on the burette: Final [mL]	V_{EDTA} [mL]	x/y
Data relating to the titration of the $Mg(NO_3)_2(aq)$ stock solution							
1							
2							
Data relating to the titrations of the $Mg(NO_3)_2(aq)$ solution you prepared							
1							
2							
						Mean	
						Standard deviation	
						95% confidence interval	

(II) What **whole number** values of x, y and n in the equation

$$x\,Mg^{2+}(aq) + y\,EDTA^{4-}(aq) \rightarrow Mg_x(EDTA)_y^{n}(aq) \qquad \text{(Eqn. 2-1)}$$

are suggested by your data?

x = _____ y = _____ n = _____

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Experiment 2 · Analysis of magnesium

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(III) Report the data relating to the titrations of the magnesium pill solution. Using the **integer values** of x and y reported in (II) and Eqn. 2-3

$$m_{\mathrm{Mg,pill}} = (x/y)(4C_{\mathrm{EDTA}}V_{\mathrm{EDTA}})(m_{\mathrm{pill}}/m_{\mathrm{powder}})(24.30\ \mathrm{g\ Mg^{2+}/mol\ Mg^{2+}}) \qquad \text{(Eqn. 2-3)}$$

report the mass of Mg^{2+} $m_{\mathrm{Mg,pill}}$ obtained in each titration. Using the formulas given in Appendix A "Statistical Treatment of Data" of this lab manual, calculate the mean value of $m_{\mathrm{Mg,pill}}$.

x = _____ y = _____

C_{EDTA} = _____ mol/L

m_{pill} = _____ g m_{powder} = _____ g

Run	C_{EDTA} [mol/L]	Volume readings on the burette		V_{EDTA} [mL]	$m_{\mathrm{Mg,pill}}$ [g]
		Initial [mL]	Final [mL]		
1	0.01				
2	0.01				

Mean = _____ g

Post-lab questions

(1) Suppose that a student reports a value of $m_{\mathrm{Mg,pill}}$ that is higher than what the actual value is supposed to be. What operation did the student most likely execute incorrectly in the lab to cause this error? Assume that no computational blunders were made.

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(2) The concentration of manganese ion ($Mn^{2+}(aq)$) can be determined by titrating against permanganate ion ($MnO_4^-(aq)$) according to the balanced equation

$$3\ Mn^{2+}(aq)\ +\ 2\ MnO_4^-(aq)\ +\ 4\ OH^-(aq)\ \rightarrow\ 5\ MnO_2(aq)\ +\ 2\ H_2O(l)$$

The endpoint is reached when the purple color of MnO_4^- disappears.

Suppose that three 25.00-mL samples containing an unknown amount of $Mn^{2+}(aq)$ are titrated against a solution containing 0.0416 mol MnO_4^-/L. The data in the table below presents the volume of $MnO_4^-(aq)$ required to reach the endpoint.

Run	1	2	3
Volume of $MnO_4^-(aq)$	37.21 mL	38.04 mL	36.12 mL

(2.a) Calculate to four decimal places the concentration of $Mn^{2+}(aq)$ in units of mole per liter in the original samples and the mean concentration of $Mn^{2+}(aq)$. Show all calculations.

Run 1

Run 2

Run 3

Mean

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(2.b) Calculate to four decimal places the standard deviation of the mean concentration of $Mn^{2+}(aq)$ in units of moles per liter in the original samples. Show all calculations.

(2.c) Calculate to four decimal places the 95% confidence interval about the mean concentration of $Mn^{2+}(aq)$ in units of moles per liter in the original samples. Show all calculations.

(2.d) What does the 95% confidence interval imply about the measurement of the concentration of $Mn^{2+}(aq)$?