

Quantitative Determination of Citric and Ascorbic Acid in Powdered Drink Mixes

Notes to the Instructor

The following instructions are for a class of 24 students working in pairs. The solutions must be prepared before class time.

Solution Preparation Directions

- 1) 400 mL of 1 M HCl [7647-01-0] - Dilute **35 mL of concentrated (12 M) HCl** to a total volume of 400 mL with distilled water.
- 2) 250 mL of 0.5% soluble starch solution – Make a paste by rubbing **1.25 g of soluble starch** [9005-84-9] in about 2 mL of water. Pour ~248 mL of boiling water into this solution and if necessary, heat with stirring until solution is clear. Cool to room temperature and store in a closed container. This solution must be freshly prepared as the shelf life is only a few days.
- 3) A total of 2 liters of 0.00100 M KIO₃ is required – Dissolve **exactly 0.214 g of KIO₃** [7758-05-6] in 1.00 L of distilled water. Repeat.
- 4) 2 liters of ~0.1 M NaOH [1310-73-2] – Dissolve **~4 g of NaOH** in 1.00 L of distilled water. Repeat and combine the two solutions. This solution must be standardized. Use a solid acid (e.g. potassium hydrogen phthalate, KHP [877-24-7]) for the standardization.

Standardization procedure. Weigh at least **3 samples of KHP (0.500 g – 0.600 g)** and dissolve each sample in 10 mL of distilled water. Add **3 drops of phenolphthalein** [77-09-8] to each solution. Titrate each acidic solution with the NaOH solution. KHP (molecular weight = 204.23 g/mol) reacts with NaOH in a 1:1 molar ratio. Therefore, moles of KHP = moles of NaOH at the endpoint. Moles of NaOH / volume of NaOH (in L) = molarity (around 0.10 M). Average the values obtained and provide this information to the students with 3 significant figures.

- 5) Thymol blue indicator [76-61-9] – 0.04% (w/w) aqueous.

Solids Required

- 1) **50 grams of KI** [7681-11-0] are required for this experiment.
- 2) **12 packages of various flavors of powdered drink mixes.**
Avoid blue and purple colored mixes.

A **pair of goggles** is required for each student. Provide each student a set of instructions and data sheets. A set of these sheets follows these directions.

Student Materials (per pair of students)

Glassware

2 burets
250 mL volumetric flask*
50 mL volumetric pipet*
10 mL volumetric pipet*
pipet bulb
6 Erlenmeyer flasks

Reagents

150 mL 0.00100 M KIO_3
3 g KI (solid)
15 mL 1 M HCl
10 mL 0.5% starch solution
150 mL 0.1 M NaOH (standardized to 3 sf)
thymol blue indicator
1 package or approximately 5 g of powdered drink mix sample

*Graduated glassware may be substituted for volumetric glassware.

Additional Comments

As with any new experiment, test the procedure and solutions before the students. While this procedure works well, it is advisable to observe the endpoint color changes beforehand.

All solutions may be safely be disposed of down the drain with water.

Calculations & Results

Name _____

Date _____

Data Table 1 - Total Acid**Partner's Name**

Flavor of Powdered Drink Mix	Orange Flavored Kool-Aid®		
Mass of sample, g	4.6641 g		
	Trial 1	Trial 2	Trial 3
Sample volume, mL	10.00 mL	10.00 mL	10.00 mL
Molarity NaOH, <i>M</i>	0.104 M		
Volume NaOH used in titration, mL			
Final buret reading, mL	19.62 mL	19.61 mL	19.64 mL
Initial buret reading, mL	0.00 mL	0.00 mL	0.00 mL
Volume NaOH used, mL	19.62 mL	19.61 mL	19.64 mL
Average volume NaOH used in titration, mL	19.62 mL		

Data Table 2 - Ascorbic Acid

Flavor of Powdered Drink Mix	Orange Flavored Kool-Aid®		
Mass of sample, g	4.6641 g		
	Trial 1	Trial 2	Trial 3
Sample volume, mL	50.00 mL	50.00 mL	50.00 mL
Molarity KIO ₃ , <i>M</i>	0.00100 M		
Volume KIO ₃ used in titration, mL			
Final buret reading, mL	21.21 mL	21.36 mL	21.21 mL
Initial buret reading, mL	0.00 mL	0.00 mL	0.00 mL
Volume KIO ₃ used, mL	21.21 mL	21.36 mL	21.21 mL
Average volume KIO ₃ used in Titration, mL	21.26 mL		

Calculations & Results**Data Table 3**

1. Moles ascorbic acid in 50.00 mL

$$= \left[\frac{0.00100 \text{ mols KIO}_3}{1 \text{ L KIO}_3} \right] \left[\frac{1 \text{ Liter}}{1000 \text{ mL}} \right] \left[\frac{21.26 \text{ mL}}{1} \right] \left[\frac{3 \text{ mols I}_2}{1 \text{ mol KIO}_3} \right] \left[\frac{1 \text{ mol C}_6\text{H}_8\text{O}_6}{1 \text{ mol I}_2} \right]$$

$$= 6.38 \times 10^{-5} \text{ mols}$$

2. Moles ascorbic acid in 10.00 mL =
- $(6.38 \times 10^{-5} \text{ mols}) / 5 = 1.28 \times 10^{-5} \text{ mols}$

3. Moles ascorbic acid in 250 mL =
- $(1.28 \times 10^{-5} \text{ mols}) \times 25 = 3.20 \times 10^{-4} \text{ mols}$

4. Moles NaOH used in titration =
- $(0.104 \text{ M})(0.01962 \text{ L}) = 2.04 \times 10^{-3} \text{ mols}$

5. Moles NaOH neutralized due to citric acid in 10.00 mL =
-
- $(2.04 \times 10^{-3} \text{ mols}) - (1.28 \times 10^{-5} \text{ mols}) = 2.03 \times 10^{-3} \text{ mols}$

6. Moles citric acid in 10.00 mL =
-
- $(2.03 \times 10^{-3} \text{ mol NaOH}) \times (1 \text{ mol citric acid} / 3 \text{ mols NaOH}) = 6.77 \times 10^{-4} \text{ mols}$

7. Moles citric acid in 250 mL =
- $(6.77 \times 10^{-4}) \times 25 = 1.69 \times 10^{-2} \text{ mols}$

Table 3 - Determination of mols of citric and ascorbic acid

		AVERAGE
1	Moles ascorbic acid in 50.00 mL, mols	$6.38 \times 10^{-5} \text{ mols}$
2	Moles ascorbic acid in 10.00 mL, mols	$1.28 \times 10^{-5} \text{ mols}$
3	Moles ascorbic acid in 250 mL, mols	$3.20 \times 10^{-4} \text{ mols}$
4	Moles NaOH used in titration, mols	$2.04 \times 10^{-3} \text{ mols}$
5	Moles NaOH neutralized due to citric acid in 10.00 mL, mols	$2.03 \times 10^{-3} \text{ mols}$
6	Moles citric acid in 10.00 mL, mols	$6.77 \times 10^{-4} \text{ mols}$
7	Moles citric acid in 250 mL, mols	$1.69 \times 10^{-2} \text{ mols}$

Calculations & Results**Data Table 4**

8. Grams of citric acid in the sample = $(1.69 \times 10^{-2} \text{ mols})(210.14 \text{ g/mols}) = \mathbf{3.55 \text{ g}}$
9. Percent by mass of citric acid in sample = $(3.55 \text{ g} / 4.6641\text{g}) \times 100 = \mathbf{76.1 \%}$
10. Average grams of citric acid per serving = $3.55 \text{ g} / 8 \text{ servings} = \mathbf{0.444 \text{ g}}$
11. Grams of ascorbic acid in sample = $(3.20 \times 10^{-4} \text{ mols})(176.14 \text{ g/mols}) = \mathbf{0.0564 \text{ g}}$
12. Percent by mass of ascorbic acid in sample = $(0.0564 \text{ g} / 4.6641\text{g}) \times 100 = \mathbf{1.21 \%}$
13. Milligrams of ascorbic acid in sample = $0.0564 \text{ g} \times 1000 \text{ mg/g} = \mathbf{56.4 \text{ mg}}$
14. Milligrams of ascorbic acid per serving = $56.4 \text{ mg} / 8 = \mathbf{7.05 \text{ mg}}$
15. Percent of RDA = $(7.05 \text{ mg} / 70 \text{ mg}) \times 100 = \mathbf{10 \%}$

Table 4 - Determination of Percent by Mass and % RDA

		AVERAGE
8	Grams of citric acid in sample, g	3.55 g
9	Percent by mass of citric acid in sample, %	76.1 %
10	Grams of citric acid per serving, g	0.444 g
11	Grams ascorbic acid in sample, grams	0.0564 g
12	Percent by mass of ascorbic acid in sample, %	1.21 %
13	Milligrams of ascorbic acid in sample, mg	56.4 mg
14	Milligrams of ascorbic acid per serving, mg	7.05 mg
15	Percent of Recommended Dietary Allowance (RDA)*, %	10 %

* RDA is in the process of being revised and will be called the Dietary Reference Intake (DRI). For purposes of calculation 70 mg of ascorbic acid was used as the RDA.

End Instructor Materials

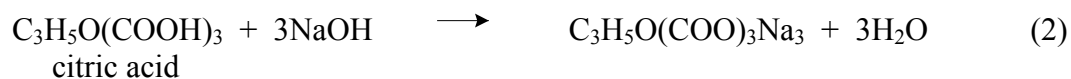
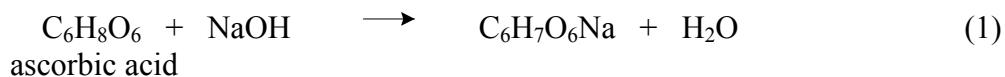
Quantitative Determination of Citric and Ascorbic Acid in Powdered Drink Mixes

Purpose

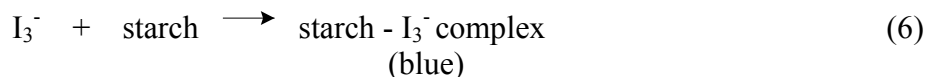
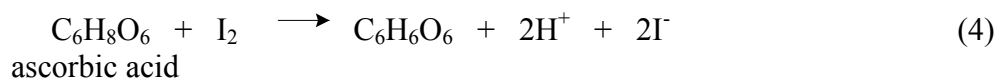
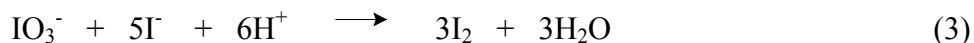
To quantitatively determine the amount of citric acid and ascorbic acid (Vitamin C) in a sample of powdered drink mix.

Introduction

Commercially available powdered drink mixes contain both citric acid and ascorbic acid. In any given sample of powdered drink mix, both acids react with the NaOH according to equations 1 and 2.



Titration with NaOH therefore determines the amount of *total acid* in the sample. The amount of ascorbic acid is determined by titration with KIO_3 . The reaction equations are shown as equations 3 - 6.



The amount of citric acid determined by difference as shown in equation 7.

$$\begin{aligned} & \text{moles NaOH neutralized (total acid)} - \text{moles NaOH neutralized (ascorbic acid)} \\ & = \text{moles NaOH neutralized (citric acid)} \end{aligned} \quad (7)$$

Calculations for the grams and percent by mass of each acid in the sample is determined and reported in Data Table 3. The percent % RDA per serving of ascorbic acid is also determined.

Procedure

Safety Precautions

1 M HCl is corrosive and 0.1 M NaOH is caustic. Handle both with care. In case of contact with skin, rinse the area with large amounts of water. Solid KI is a possible skin and lung irritant. Wear eye protection at all times in the chemistry laboratory. Wash your hands thoroughly before leaving the lab.

1. Weigh the sample (package) of powdered drink mix. Record the weight in the appropriate space in Data Table 1.
2. Dilute the sample to 250 mL with distilled water.
3. **Determine the Moles of Total Acid (Citric & Ascorbic)**

Obtain approximately 150 mL of the 0.1 M standardized sodium hydroxide (NaOH) solution in a clean, dry, labeled beaker. Be sure to record the *exact molarity* on your data sheet. Prepare and fill a buret according to your instructor's directions with the standardized NaOH solution.

Measure a 10.0 mL aliquot of the powdered drink mix solution and quantitatively transfer it to your Erlenmeyer flask. Add 5 drops of thymol blue indicator to the flask. Titrate the contents of the flask until a permanent color change (the color persists for 30 seconds or longer) in the solution is observed. **NOTE:** The endpoint color varies according to the initial color of the drink solution being tested. Save the first titrated sample in a beaker to use as a color reference for the next titrations.

Repeat with two additional 10.0 mL aliquots. The titrated solutions may be flushed down the drain with water.

Clean up according to the instructor's directions. Wash your hands.

4. **Determine the Moles of Ascorbic Acid**

Prepare and fill a buret according to your instructor's directions with 0.00100 M KIO₃ solution.

Measure a 50.0 mL aliquot of the powdered drink mix solution and quantitatively transfer it to your Erlenmeyer flask. Add 1 gram of KI, 5 mL of 1 M HCl and 3 mL of 0.5 % starch solution to the flask. Titrate with 0.00100 M KIO₃ immediately to the first

permanent color change (the color persists for 1 minute or longer). The endpoint color varies according to the initial color of the drink solution being tested. Save the first titrated sample in a beaker to use as a color reference for the next titrations.

Repeat with two additional 50.0 mL aliquots. The titrated solutions may be flushed down the drain with water.

5. Clean up according to the instructor's directions. Wash your hands.

Name _____

Date _____

Data Table 1 - Total Acid

Partner's Name

Flavor of Powdered Drink Mix			
Mass of sample, g			
	Trial 1	Trial 2	Trial 3
Sample volume, mL	10.00 mL	10.00 mL	10.00 mL
Molarity NaOH, M			
Volume NaOH used in titration, mL			
Final buret reading, mL			
Initial buret reading, mL			
Volume NaOH used, mL			
Average volume NaOH used in titration, mL			

Data Table 2 - Ascorbic Acid

Flavor of Powdered Drink Mix			
Mass of sample, g			
	Trial 1	Trial 2	Trial 3
Sample volume, mL	50.00 mL	50.00 mL	50.00 mL
Molarity KIO_3 , M	0.00100 M		
Volume KIO_3 used in titration, mL			
Final buret reading, mL			
Initial buret reading, mL			
Volume KIO_3 used, mL			
Average volume KIO_3 used in titration, mL			

Calculations & Results

Use the following calculations to complete Tables 3 and 4

1. Determine the number of moles of ascorbic acid in a 50.0 mL aliquot where the average volume of KIO_3 is the volume, **in mL**, from Data Table 2.

Moles of Ascorbic Acid in 50.00 mL

$$= \left[\frac{0.00100 \text{ mols } \text{KIO}_3}{1 \text{ L } \text{KIO}_3} \right] \left[\frac{1 \text{ Liter}}{1000 \text{ mL}} \right] \left[\frac{\text{Average Volume (mL) } \text{KIO}_3}{1 \text{ mol } \text{KIO}_3} \right] \left[\frac{3 \text{ mols } \text{I}_2}{1 \text{ mol } \text{KIO}_3} \right] \left[\frac{1 \text{ mol } \text{C}_6\text{H}_8\text{O}_6}{1 \text{ mol } \text{I}_2} \right]$$

2. Determine the moles of ascorbic acid in 10.00 mL where the moles of ascorbic acid in 50 mL are the amount calculated in #1.

$$\text{Moles of Ascorbic Acid in 10.00 mL} = (\text{Moles of Ascorbic Acid in 50 mL}) / 5$$

3. Determine the moles of ascorbic acid in 250 mL.

$$\text{Moles of Ascorbic Acid in 250 mL} = (\text{Moles of Ascorbic Acid in 10 mL}) \times 25$$

4. Determine the moles of NaOH used in the Total Acid titration, where the average volume of NaOH is the volume, **in mL**, from Data Table 1.

$$\text{Total Moles of NaOH} = [\text{Molarity of NaOH, } M][\text{Average Volume of NaOH, mL}] \left[\frac{1 \text{ Liter}}{1000 \text{ mL}} \right]$$

5. Determine the moles NaOH neutralized due to citric acid in 10.00 mL

$$\text{Moles of NaOH Neutralized Due to Citric Acid} =$$

$$(\text{Total Moles of NaOH}) - (\text{Moles of Ascorbic Acid in 10 mL})$$

6. Determine the moles of citric acid in 10.00 mL.

$$\text{Moles of Citric Acid in 10.00 mL} =$$

$$(\text{Moles of NaOH Neutralized Due to Citric Acid}) \times (1 \text{ Mole of Citric Acid} / 3 \text{ Moles NaOH})$$

7. Determine the moles of citric acid in 250 mL.

$$\text{Moles of Citric Acid in 250 mL} = (\text{Moles of Citric Acid in 10.00 mL}) \times 25$$

8. Determine the grams of citric acid in the sample. Multiply the **average moles of citric acid in 250 mL** from Data Table 3 by the molecular weight of citric acid (MW = 210.14 g/mol)

$$\text{Grams of Citric Acid in Sample} = [\text{Average Moles Citric Acid}] \left[\frac{210.14 \text{ grams}}{1.00 \text{ mol}} \right]$$

9. Determine the percent by mass of citric acid.

$$\text{Percent by Mass of Citric Acid in Sample} = \left[\frac{\text{Grams Citric Acid in Sample}}{\text{Mass of Sample}} \right] \times 100$$

10. Determine the grams of citric acid per serving. (See package for serving information.)

$$\text{Grams of Citric Acid per Serving} = [\text{Grams of Citric Acid in Sample}] \left[\frac{1}{8 \text{ servings}} \right]$$

11. Determine the grams of ascorbic acid in the sample. Multiply the **average moles of ascorbic acid in 250 mL** from Data Table 3 by the molecular weight of ascorbic acid. (MW = 176.14 g/mol)

$$\text{Grams of Ascorbic Acid in Sample} = [\text{Average Moles Ascorbic Acid}] \left[\frac{176.14 \text{ grams}}{1.00 \text{ mol}} \right]$$

12. Determine the percent by mass of ascorbic acid.

$$\text{Percent by Mass of Ascorbic Acid in Sample} = \left[\frac{\text{Grams Ascorbic Acid in Sample}}{\text{Mass of Sample}} \right] \times 100$$

13. Determine the milligrams of ascorbic acid in the sample.

$$\text{Milligrams of Ascorbic Acid} = (\text{Grams of Ascorbic Acid in Sample}) \times (1000 \text{ mg/g})$$

14. Determine the milligrams of ascorbic acid per serving. (See package for serving information.)

$$\text{Grams of Ascorbic Acid per Serving} = [\text{Grams of Citric Acid in Sample}] \left[\frac{1}{8 \text{ servings}} \right]$$

15. Determine the percent RDA per serving.

$$\% \text{ RDA} = \left[\frac{\text{mg Ascorbic Acid}}{70 \text{ mg}} \right] \times 100$$

Name _____

Date _____

Table 3 - Determination of mols of citric and ascorbic acid

		AVERAGE
1	Moles ascorbic acid in 50.00 mL, mols	
2	Moles ascorbic acid in 10.00 mL, mols	
3	Moles ascorbic acid in 250 mL, mols	
4	Moles NaOH used in titration, mols	
5	Moles NaOH neutralized due to citric acid in 10.00 mL, mols	
6	Moles citric acid in 10.00 mL, mols	
7	Moles citric acid in 250 mL, mols	

Table 4 - Determination of Percent by Mass and % RDA

		AVERAGE
8	Grams of citric acid in sample, g	
9	Percent by mass of citric acid in sample, %	
10	Grams of citric acid per serving, g	
11	Grams ascorbic acid in sample, grams	
12	Percent by mass of ascorbic acid in sample, %	
13	Milligrams of ascorbic acid in sample, mg	
14	Milligrams of ascorbic acid per serving, mg	
15	Percent of Recommended Dietary Allowance (RDA), %	