

RESEARCH NOTEBOOK

NAME ?

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TITLE The Gravimetric Determination of Chloride in a Soluble Unknown
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Procedure

1. Wash crucibles with distilled water.
2. Heat up crucible in dryer until mass is constant.
3. When crucible mass is constant mass, record as dry mass of crucible.
4. Add filter mats into every crucible.
5. Put crucibles in watchglasses-covered beakers.
6. Heat crucibles in oven at 110°C for at least one hour.
7. Let crucible cool in a slightly open desiccator, and then measure its mass.
8. If its mass has not changed, record masses of crucibles.
9. Get a sample of the unknown from the oven, and let cool in a desiccator.
10. Obtain Using the method of weighing by difference, measure out approximately 0.1 g of the unknown into three clean beakers.
11. Prepare approximately 30mL of solution by adding 2-3mL of 6M HNO_3 was been added to 30mL of water.
12. Pour approximately 10mL of the acidic solution into each beaker with the unknown.

Week 2

13. Add 0.2M AgNO_3 slowly to sample solutions until AgCl is observed to coagulate.
14. Add 3-5 mL of AgNO_3 to each solution; and add separate stirring rods.
15. Heat almost to boiling, then allow digestion for 10 minutes.
16. Add a few drops of AgNO_3 and test for completeness of precipitation.
17. Let beakers cool. Cover with parafilm, and then store in dark place.

Week 3

18. Prepare 250mL of HNO_3 wash solution and add 1mL 6M HNO_3 .
19. Decant each sample's extra (supernatant) liquid through a crucible.
20. Wash precipitate (in beaker) several times with HNO_3 wash solution.
21. Decant washings into crucible. Transfer AgCl to crucible using deionized water and a rubber policeman.
22. Keep washing until no turbidity (cloudiness) is observed when a few drops of KCl are added.
23. Let each precipitate be dried in the oven at 110°C for at least 1 hr.
24. Repeat cooling-heating cycles for drying like cooling the initial crucibles.

Chemical SDS Information HNO_3 , 6M

- ✓ May cause headache, shortness of breath, irritation for any exposure. Exposure to eyes may cause blindness. May cause ulcerations of skin, redness, pain, gastrointestinal pain, nausea, and vomiting (if ingested).

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(HNO₃ Safety Information, continued)

- wear protective equipment and ensure adequate ventilation
- For safe handling, prevent formation of aerosols, follow good hygiene practice
- ✓ Keep away from flame or sources of ignition.

AgNO₃ : 0.2M

Health: Very dangerous after skin contact or ingestion. Contact with eye may result in damage to corneas or blindness. Ingestion will cause gastritis, irritation or respiratory irritation, resulting in burning feeling, coughing or sneezing. Can cause permanent damage to eyes, lungs, skin, and/or stomach on contact.

Safety: Non-flammable. Oxidizing and corrosive.

Handling: Keep container dry, and away from sources of ignition. Do not add to water. Store in light-resistant containers. Keep in cool, well-ventilated storage.

Concentrated HCl : HCl

Health: HCl is very hazardous on contact with skin (corrosive, irritant), severe contact with eyes, or ingestion. HCl is slightly hazardous when inhaled. Vapor may damage membranous tissues. Exposure can cause redness, itching of eyes and breathing when inhaled. Eye exposure may cause death.

Safety: Non-flammable. Non-combustible. Corrosive and poisonous, with respiratory irritation.

Handling: Keep locked up in dry container. Never add water to HCl. Keep away from oxidizing agents and metals.

NaCl (white part of unknown chloride solution)

Health: Fatal if ingested when in contact with skin, eyes, lungs (inhalation) or ingestion. No acute health effects.

Safety: Non-flammable. Non-combustible. Non-corrosive. Emits toxic fumes when heated - decomposition.

Handling: Keep locked up in a cool, dry place. Do not ingest or breathe dust.

Calculations

How much of 0.2M AgNO₃ needed to dissolve 0.0g NaCl.

$$\frac{0.0\text{ g NaCl}}{58.44 \text{ g NaCl}} \times \frac{\text{mol NaCl}}{\text{mol NaCl}} \times \frac{\text{mol AgNO}_3}{\text{mol NaCl}} \times \frac{L \text{ 0.2M AgNO}_3}{0.2\text{ mol AgNO}_3} \times \frac{1000\text{ mL 0.2M AgNO}_3}{L \text{ 0.2M AgNO}_3}$$

$$= \frac{0.0\text{ g}}{58.44 \text{ g}} \times \frac{1}{1} \times \frac{1}{1} \times \frac{0.2\text{ mol}}{0.2\text{ mol}} \times \frac{1000\text{ mL}}{1} = 1.71\text{ mL 0.2M AgNO}_3$$

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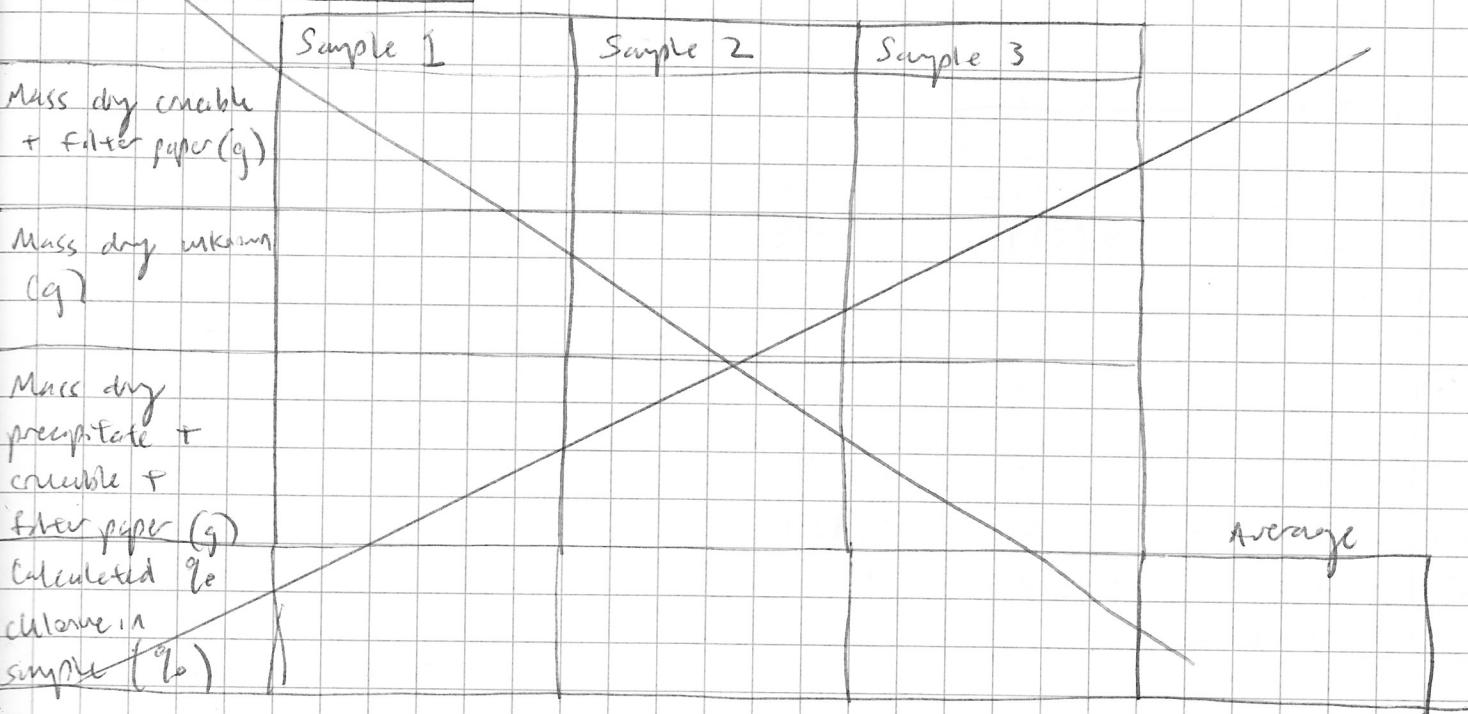
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TITLE The Gravimetric Determination of Chloride in a Soluble Chloride Book No.

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80/80

Data CollectionUnknown:Discussion Questions

- A colloidal suspension is a form of heterogeneous mixture with very small particles, such that it does not separate when left alone.
- Coagulation is necessary to get maximize the size of the AgCl precipitate particles so that the majority are caught by the filter paper.
- Coagulation is accomplished by adding Ag⁺ ions (as AgNO₃) and heating the colloidal suspension almost to boiling (digestion).
- Carbonate ions may be introduced if any CO₂ from the atmosphere enters the solution.
- If carbonate ions form Ag₂CO₃ precipitate, the mass of the ~~chloride~~^{total} precipitate would increase and the % Cl⁻ would also increase. This would be a systematic overestimation of the % Cl⁻.
- ~~Excess~~ HNO₃ acid is added to react with any excess CO₃²⁻ ions. to prevent
- Pepitration is the reverting of a coagulated colloid to a dispersed state.
- It would cause some of the AgCl to disperse lowering the calculated % Cl⁻.
- Heating the solution with Nitric acid prevents precipitation.
- The major source of the silver ion is the silver nitrate.
- a) % Cl⁻ would be too low, as some would form HClO₄⁻ and evaporate.
- b) % Cl⁻ would be too low, as some would form Cl₂(g) and not be included in the mass.

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Data Collection

Unknown #: 1801

		Sample 1 18.8425	Sample 2. 18.9989	Sample 3 18.9049
Mass dry	Trial 1	12.578791325g	18.9989	18.9049
concrete +	Trial 2	18.8430	18.9971	18.9026
filter paper (g)	Trial 3	18.8442	18.9969	18.9048
	Trial 4	18.8399	18.9965	18.9009
Mass dry		2.1325g	0.1011g	0.1161g
unknown (g)				
Mass dry	Trial 1	19.1288	19.2104	18.2536
precipitate +	Trial 2	19.1970	19.2104	18.2532
concrete +	Trial 3	19.1068		18.2544
filter paper (g)	Trial 4			

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Book No.

TITLE The Determination of Percent Chloride by Ion Exchange and

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Procedure week 1

- Measure the mass of one NaOH pellet in a tared watchglass
- Calculate the # of pellets necessary to get correct mass for approximately 0.1 NaOH
- Put pellets in large clean beaker using NaOH spatula
- Add 500mL deionized water
- Store solution in 500mL plastic bottle
- Obtain KHP from over, and place in clean dry beaker
- Measure ~~to~~ the mass of three samples of KHP on the analytical balances by weighing by difference
- Dissolve the KHP in approximately 50mL of ~~water~~ deionized water
- Add some drops of phenolphthalein
- Put a ~~plastic~~ white sheet of paper under the beaker, put a stirring bar in the beaker.
- Rinse barrel with detergent solution followed by tap water, and drain through the valve.
- Rinse with a few mL of deionized water.
- Rinse with a few mL of NaOH stock solution.
- Fill the barrel with NaOH solution above the level of the zero calibration mark.
- Drain some solution so no air is trapped in the barrel.
- Titrate KHP until pink color for half a minute. Repeat for other KHP solutions.

Week 2:

- Obtain a 50mL buret and ~~not~~ clean very well. Make it ^{sure} is free draining at water level
- Put a small glass wool plug in with clean tongs. A long glass rod may be used to gently nudge it to the bottom.
- Test buret by setting it up and making sure water can flow.
- Obtain about 75mL of ion exchange ~~resin~~ resin and carefully pour into a ~~100mL~~ beaker ~~or~~ or a 125mL Erlenmeyer flask using a funnel. Make sure it stays wet at all times.
- Use a plastic funnel to pour the slurry of resin and water into the buret. Top off to avoid uneven packing.
- Add 35mL of NaOH solution at ~~3.5~~ mL/min, 5-10mL at a time.
- Wash in at least 52.5 mL of deionized water. Test pH to make sure
- Measure the mass of a sample by difference by pouring into a clean ~~100mL~~ volumetric flask. It should be between 0.20g and 0.50g. Use deionized water to wash down any additional solid.
- Prepare 100mL of solution by rinsing the tube until it becomes homogeneous.

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TITLE The Determination of Percent Chloride by Ion Exchange and Back Titration

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Procedure (contd.)

- ~~Fill~~ a 10mL pipette with the unknown, and use it to add exactly 10.00mL of the unknown solution to the top of the column. The tip of the pipette should be dried using a Kim wipe.
- Pipette 10.00mL of unknown onto the top of the column.
- Keep adding deionized water at the rate of 3mL/min for 70mL of deionized water.
- Repeat for other two samples of unknown solution.
- Add 2-3 drops of phenolphthalein to a solution.
- Fill a buret with 0.410M KHP. ~~Addition~~
- Add enough KHP to make the solution colorless, and then add 3 additional mL. The total volume of KHP added is recorded.
- Add 2-3 drops of CCl₄ and ~~seal~~ with Parafilm before storing.
- Do the same extraction with the other two samples.

Week 3

- Repeat week 1 titration procedure. Measure the exact amount of standardized NaOH used. Each sample of overtitrated unknown KHP will be titrated.

SDS InformationAmbertite IRA 910

~~Health~~ May cause eye and skin irritation. Wash with soap and water. Use dry chemicals in case of fire. Do not pour down the drain. Keep away from dust. Keep in a well-ventilated place free of dust.

Sodium Hydroxide (NaOH)

Flush with water if it comes in contact with skin or eyes. Strong corrosive agent and irritant. May cause lung damage if inhaled. Neutralize before cleaning up. Do not add water. Keep in a cool place with adequate ventilation.

Carbon Tetrafluoride (CCl₄)

Flush with lots of water if it comes in contact with skin or eyes. Irritant of skin, eyes. Carcinogenic, ~~teratogenic~~, and mutagenic. Toxic to vital organs. Store with proper ventilation.

Potassium hydrogen phthalate (KHC₈H₄O₄)

May cause eye, skin, digestive tract, respiratory tract irritation. Flush with water for at least 15 minutes if in contact with skin or eyes. Weak acid. Avoid strong oxidizing agents and excess heat. Use adequate ventilation. Store in a tightly closed container.

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Phenolphthalein

May cause skin, eyes, digestive system, respiratory tract irritation. May cause fever, hypertension if ingested. May be carcinogenic. Flush with water for at least 15 minutes if on skin or clothes. Avoid dusty conditions. Use only in a chemical fume hood. Do not let this chemical enter the environment. Keep away from strong oxidizing agents and heat. Store in a tightly-closed container in a cool, dry place.

Glass Wool

Irritant for skin and eye contact. Carcinogen. Toxic to lungs and mucous membranes. Wash with water on skin or eye contact. Non-flammable, keep locked tightly in a container.

Calculations

1. # grams of NaOH to prepare 500mL 0.1M NaOH solution:

$$500\text{mL } 0.1\text{M NaOH} \times \frac{1\text{L}}{1000\text{ mL}} \times \frac{0.1\text{ mol}}{1\text{ L}} = 0.05\text{ mol NaOH}$$

$$0.05\text{ mol NaOH} \times \frac{39.997\text{ g NaOH}}{\text{mol NaOH}} = 2\text{ g NaOH}$$

2. # grams KHP necessary to titrate 25mL 0.1M NaOH

$$25\text{mL } 0.1\text{M NaOH} \times \frac{L}{1000\text{mL}} \times \frac{0.1\text{ mol NaOH}}{L} \times \frac{\text{mol KHP}}{\text{mol NaOH}} \times \frac{204.22\text{ g KHP}}{\text{mol KHP}}$$

$$= 0.5\text{ g KHP}$$

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TITLE The Determination of Product Chloride by Ion Exchange and Backtitration
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Data CollectionStandardization of NaOH solution:

mass of NaOH pellet (g): 2.2115 g

actual # pellets necessary: 10

Total mass pellets: 2.2091 g

	Sample 1	Sample 2	Sample 3	Sample 4
Mass of dry KHP (g)	0.5022 g	0.4793	0.5009 g	0.4997
Volume NaOH added to titrate KHP (mL)	6.20 mL	3.60 mL	1.80 mL	Initial Volume 11.50
-Final volume (mL)	23.75 mL	25.50 mL	25.00 mL	Final Volume 37.00

Overtitration and Backtitration of Unknown:

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Mass of unknown (g)	0.5022 g	0.4743 g	0.5009 g	0.3957 g	Initial Volume
Total volume KHP to overtitrated OH- ions	8.05	0.998 g	0.3957	8.92 mL	Final Volume
Volume NaOH to back-titrate overtitrated KHP	16.16	22.10 mL	22.45 mL	28.00 mL	Initial Volume
-Final volume (mL)	6.00 mL	4.30 mL	4.20 mL	9.20 mL	Final Volume
	34.10 mL	39.20 mL	39.91 mL	40.25 mL	

X discarded

Calculations

$$\text{Concentration NaOH} = \frac{\text{mass KHP/g}}{204.22 \text{ g KHP}} \times \frac{\text{mol KHP}}{\text{mol NaOH}} \times \frac{1}{(\text{final - initial volume NaOH}) \times \frac{L}{1000 \text{ mL}}}$$

	Sample 1	Sample 2	Sample 3	Sample 4	Average
Concentration NaOH (mol)	0.1064	0.1061	0.1057	0.1083	0.1066

Noteswell 30 mL mol delivered after- discarded sample 2 b/c stopped at well for a week- not clean stirring rod- spilled a little NaOH- mag of expand not entirely stable

- didn't shake mag
 - brief stopcock let sit and stirring
 - too much in overtitration
 - too much in backtitration in sample 3
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Calculations

$$\text{mL NaOH for back titration} \times \frac{0.1066 \text{ mol NaOH}}{\cancel{0.1066 \text{ mL NaOH}}} \times \frac{\text{mol KHP}}{\text{mol NaOH}} \times \frac{\cancel{0.400 \text{ mol KHP}}}{\cancel{0.400 \text{ mol NaOH}}} = \text{excess mol KHP}$$

$$(\text{total mol KHP for neutralization} - \text{excess mol KHP}) \times \frac{\text{mL KHP for titration}}{\text{mL KHP}} = \text{mL KHP for titration}$$

$$\text{KHP for titration} \times \frac{0.400 \text{ mol KHP}}{\cancel{0.400 \text{ mL}}} \times \frac{\text{mol } \text{O}\text{H}^-}{\text{mol KHP}} \times \frac{\text{mol Cl}^-}{\text{mol } \text{O}\text{H}^-} = \text{mol Cl}^-$$

$$\% \text{ chloride} = \text{mol Cl}^- \times \frac{35.45 \text{ g Cl}^-}{\text{mol Cl}^-} \times \frac{1}{\text{mass unknown}} \times 100$$

Overall calculation:

$$\% \text{ chloride} = \left(\frac{\text{total mL KHP} - \left(\text{mL NaOH} \times \frac{0.1066 \text{ mol NaOH}}{1000 \text{ mL NaOH}} \times \frac{\text{mol KHP}}{\text{mol NaOH}} \times \frac{1000 \text{ mL KHP}}{0.400 \text{ mol KHP}} \right)}{\text{total mL KHP}} \times \frac{0.400 \text{ mol KHP}}{1000 \text{ mL}} \times \frac{\text{mol } \text{O}\text{H}^-}{\text{mol KHP}} \times \frac{\text{mol Cl}^-}{\text{mol } \text{O}\text{H}^-} \times \frac{35.45 \text{ g Cl}^-}{\text{mol Cl}^-} \times \frac{100}{\text{mass unknown}} \right)$$

$$\text{For soln 1: } \left((8.1 \text{ mL} - (28.1 \text{ mL} \times \frac{0.1066 \text{ mol}}{1000 \text{ mL}} \times \frac{\text{mol KHP}}{\text{mol NaOH}} \times \frac{1000 \text{ mL KHP}}{0.400 \text{ mol KHP}})) \times \frac{0.400 \text{ mol KHP}}{1000 \text{ mL}} \times \frac{\text{mol } \text{O}\text{H}^-}{\text{mol KHP}} \times \frac{\text{mol Cl}^-}{\text{mol } \text{O}\text{H}^-} \times \frac{35.45 \text{ g Cl}^-}{\text{mol Cl}^-} \times \frac{100}{\text{mass unknown}} \right)$$

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TITLE Spectrophotometric Determination of the Identity of an Unknown

Indicator

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Procedure

- Obtain sample and record sample # of indicator unknown and record sample #1.
- Perform a two-point calibration of pH meter (see laboratory for details).
 - ↳ Clean probe of pH meter, place in one buffer solution, change slope to 100%, and wait for temperature to equalize ($\approx 2\text{ min.}$). Then, change the "standardize" knob until the correct pH value is displayed. Adjust temperature knob.
 - ↳ Clean probe with deionized water, and repeat above procedure in other buffer solution. This time, the slope knob should be adjusted and the "standardize" knob should be adjusted to display the correct pH value.
- Dilute 20 drops (1mL) of sample in approximately 25mL 1M acetic acid in a clean 100mL beaker. Repeat in a second beaker (to be used for color comparison).
- Place white sheet of paper under ~~stirrer~~ beaker on stirrer (for contrast). Place beaker (with stir bar) on stirrer. Stir.
- Add 0.1M NaOH drop-wise. Record color vs. pH for dilutions in pH.
- Add 5mL of KH_2PO_4 solution (roughly) to acetic beaker. Add 1M NaCl , while stirring, to the KH_2PO_4 until the pH reaches roughly 2 units above the high end⁺ of the effective pH range of the indicator. Pour into a volumetric flask.
- Add 300mL of indicator sample by volumetric pipet. Add deionized water to the volumetric flask until it reaches 100mL (the final amount with a small pipet), and mix well.
- Store in a dry or lined 250mL Erlenmeyer flask with a rubber stopper.
- Repeat last three steps to produce two more buffered solutions: one with a pH two units below the effective pH range of the indicator, and the other at the middle of the effective pH range.
- Recalibrate the pH meter to include the range of pH's. (using 2.00, 7.00, and 12.00 pH buffered standard solutions), and measure the pH of each solution.
- Add one drop of CaCl_2 to each flask (preservative).
- Calibrate the spectrometer and obtain "nothing" currents.
- Measure and record the absorbance of each solution as a function of the wavelength from 350 to 650nm at 25nm intervals. Determine the two optimal wavelengths.
 - ↳ Use smaller (5nm) increments near the absorption maxima.
- ~~Perform a serial dilution~~ Prepare solutions with concentrations 0.8 times, 0.6, and 0.4, and 0.2 times that of the low-pH ~~start~~ buffered indicator solution, using a graduated cylinder.
- Measure the absorbance of each of these in a cuvette (use the same one) at the optimal wavelength for the ~~higher~~ low-pH solution.
- Repeat last two steps with high-pH buffered solutions.

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Safety, Health, and Handling1M $\text{HCl}_2\text{H}_3\text{O}_2$ (acetic acid)

Harmful for skin, eyes, and in case of ingestion and inhalation. Corrosive to skin and eyes or other membranes. Toxic to liver and in case of prolonged exposure. Flammable and corrosive. Keep away from heat and source of combustion. Keep in cool, well-ventilated area, in a tightly-sealed grounded container.

1M NaOH (sodium hydroxide)

Extremely hazardous to skin and eyes. Do not inhale or ingest. Rinse immediately with copious amounts of water if contact. Allow victim well-ventilated area if inhaled. Not flammable ~~or~~ corrosive. Corrosive to some metals. Provide ventilation for container. Do not store near extreme heat, food, oxidizing agents. Store cool and well-ventilated.

2dM KH_2PO_4 (potassium dihydrogen phosphate)

Harmful when it contact with eyes, when inhaled, or when ingested. Mild irritant in case of skin contact. Non-flammable and non-corrosive in presence of glass. Keep in a cool, well-ventilated area.

1.0M HCl (hydrochloric acid)

Very hazardous on contact with skin (corrosive, irritant, permeator.), eyes or ingestion. Slightly hazardous when inhaled. Vapor may damage mucous membranes & lungs. Over-exposure may cause death. Non-flammable and non-combustible. Corrosive and very dangerous to personal. Never add water and ^{always} use protective equipment. Keep locked up in dry container. Keep away from oxidizing agent and metals.

 CCl_4 (carbon tetrachloride)

Very hazardous in contact with skin, eyes, ingestion and inhalation. Contact, permeation, carcinogenic. Toxic to kidneys, lungs, nervous system, liver, mucous membranes. Non-flammable. Keep locked up. wear protective clothing, respiratory equipment if needed, gloves.

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TITLE Spectrophotometric Determination of an Unknown Indicator

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DataUnknown Indicator Sample # 1815

Table 1: color vs. pH

pH	color	pH	color	pH	color	pH	color	pH	color
2.3	yellow	3.8	dark red-	5.3		6.8		8.3	
2.4	yellow	3.9	violet	5.4		6.9		8.4	
2.5	yellow	4.0	dark	5.5		7.0		8.5	
2.6	yellow	4.1	violet	5.6		7.1		8.6	
2.7	yellow	4.2	dark-violet	5.7		7.2		8.7	
2.8	yellow	4.3	violet	5.8		7.3		8.8	
2.9	yellow	4.4	violet	5.9		7.4		8.9	
3.0	yellow	4.5	violet	6.0		7.5		9.0	
3.1	(unwritten)	4.6	violet	6.1		7.6		9.1	
3.2	green-yellow	4.7	violet	6.2		7.7		9.2	
3.3	yellow-green	4.8	violet	6.3		7.8		9.3	
3.4	light green	4.9	violet	6.4		7.9		9.4	
3.5	green	5.0	violet	6.5		8.0		9.5	
3.6	dark yellow-green	5.1	violet	6.6		8.1		9.6	
3.7	dark yellow-green	5.2		6.7		8.2		9.7	

Table 2. pH of buffered solutions

Solution	pH
low pH	1.90
medium pH	3.95
high pH	6.25

Approximate pH effective range of indicator (from Table 1)

High pH: _____

Low pH: _____

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Data Transmittance and

Table 3. Absorbance of Buffered Solutions

Wavelength (nm)	Transmittance			Absorbance		
	Low pH	Int. pH	High pH	Low pH	Int. pH	High pH
350				0.106	0.091	0.087
375				0.148	0.142	0.148
400				0.255	0.187	0.090
425				0.367	0.233	0.026
450				0.371	0.246	0.044
475				0.250	0.189	0.086
500				0.125	0.146	0.181
525				0.042	0.161	0.351
550				0.010	0.264	0.65
575				0.006	0.439	1.076
600				0.005	0.521	1.213
625				0.002	0.090	0.185
650				0.001	0.006	0.016

Last entries are 0.002A

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TITLE Spatophotometric Determination of an Unknown Indicator

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Data

Table 4: Transmittance and Absorbance of Different Concentrations

(Concentration % of buffer concentration)	Transmittance		Absorbance	
	Low pH	High pH	Low pH	High pH
100%			0.326	1.409
89%			0.304	1.090
60%			0.230	0.825
40%			0.153	0.537
20%			0.072	0.262

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Procedure

Setup:

- Check which chromatograph model is being used (350 or 400).
- Make sure chromatograph is on, the He is running, and that current is ~~set~~ turned off (should be performed by lab. faculty).
- Use bubble flow meter to ~~check~~ measure carrier gas rate through each column. Flow rate should be 60-65 mL for optimal. (i.e., try to get flow rate close to 1 mL per second). Record flow rate for every column.
- Turn on chart recorder (to make sure chromatograph is grounded properly).
- Double check He is flowing. Disconnect bubble meter. Turn detector on, and set current to 100mA (make sure not to let above about 110mA)
- Connect ~~to~~ netbook. Login: student password: edson(N).
- Connect LabQuest to netbook via USB
- Change "Instrumentation Amp. Filt" ~~to~~ to 0-20mV option. Connect to CH1 of the LabQuest
- Open the Legger Lite program (go to Experiment \rightarrow Setup screens \rightarrow LD menu). Make sure the Inst. Amp. is displayed. Close setup.
- Check to see if instrument reading is relatively stable. (if not, consult teacher.) Choose Experiment \rightarrow Zero. (Should be around 100mV now)
- Select Experiment \rightarrow Data Collection. Choose the following options:
 - ↳ Mode: Time-based
 - ↳ Length: 15 min.
 - ↳ Sampling Rate: 40 samples/min.
- Double click y-axis, scale to manual (Top = 10mV, Bottom = ~~0~~ 0mV)
- Save experimental setup file to USB as "GCMethod".

Data Collection: (see Operating Notes (next page) for more reminders about data collection)

- \rightarrow Student 1 injects sample into input port B of gas chromatograph (see notes on operation)
- \rightarrow Student 2 presses "Collect" on legger lite
- Do not touch setup until data collection period is complete (unless absolutely ^{testing sample} sure all ^{samples} have completely passed through, in which case it may be ended early.)
- When complete, Click File \rightarrow Export As \rightarrow CSV
- Identify peaks on graph by locations, shapes, and retention times. Compare with standard chromatogram and identify components.
- Check "Your latest run".

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Procedure, cont'd.Post-lab shutdown:

- Turn in (capped) syringe to professor
- Shut down computer
- Turn main "power" / "current" switch off.
- Turn "current" dial off.

Notes on Optimizing Operating Parameters and General Operations

- The column is only connected to the B input port
- There must ALWAYS be He passing through the detector.
- Do not touch the gas cylinder or valves.
- Be careful with syringes and do not touch the injection port.
- Fill syringe from polyethylene sample bags.
 - ↳ Gently inject it to avoid sample loss
 - ↳ Quickly inject it to avoid broad base reduce band broadening.
- Write down operating conditions for every trial
- Make sure the syringe and needle have been purged before injecting the next sample
- Three or more injections should be used to optimize flow rate and injection size
- At least three volume sizes of standard gas should be injected to establish the calibration curve for that component of the mixture.

SDS DataOxygen gas, (O₂(g))

Oxidizer: may cause or intensify fire. Protect from sunlight. Store in a well-ventilated place. Contact with rapidly expanding gas may cause burns or fractures. No effects at ^{very} low pressure and temperatures. Take care when gas is under pressure.

Helium gas, (He(g))

May displace oxygen and cause rapid suffocation. Keep tightly and safely sealed when under pressure. Protect from sunlight. Store in cool and well-ventilated place. Use equipment rated for cylinder pressure. Get medical attention if health effects persist or are severe. It may be dangerous to give mouth-to-mouth resuscitation without proper training.

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SDS Data, cont'd.

Carbon dioxide ($\text{CO}_2(\text{g})$)

If under pressure, may explode when heated. May displace oxygen and cause rapid suffocation (asphyxiate). May increase respiration and heart rate. Use equipment rated for cylinder pressure. Store it in an upright container. Use outdoors or in a well-ventilated place. Flush contaminated skin or eyes with plenty of water. Get medical assistance if symptoms persist or are severe. Dangerous carbon monoxide when decomposed.

Nitrogen gas ($\text{N}_2(\text{g})$)

Dangerous if under pressure. May displace oxygen and cause rapid suffocation. Protect from sunlight. Store in a well-ventilated place. Flush contaminated skin or eyes with plenty of water. If medical symptoms persist or are severe, get medical assistance. Contact with rapidly expanding gases may cause frostbite or burn on skin and eyes. Decomposition produces harmful nitrogen oxides.

Dry Ice ($\text{CO}_2(\text{s})$)

OK to use

May displace O_2 and cause rapid suffocation. May increase respiration and heart rate. May cause frostbite. In contact with skin or eyes, remove thoroughly with water and remove contaminated clothing. May cause eye irritation. Store placed in dry, cool, well-ventilated area. Keep weight in sealed containers.

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Data Collection

Most of the data will be collected on the LabQuest Mini. The data will be 600 data points of detector reading (mV) versus time (s).

Conditions during standardization teststandard

Table 1: Unknown Sample Conditions

Sample	Flow Rate (mL/min)	Detector Current (mV)	Ambient Temperature (°C)	Ambient pressure (cm Hg)	Injection volume (mL)
1 Standard	57.58 mL /min	100	24.5 °C	29.88 m. Hg	3 mL
2 Standard	67.06 mL /min	100	"	"	3 mL
3 Standard	"	"	"	"	4.0 mL

Table 2: Unknown Sample Conditions

Sample	Flow Rate (mL/min)	Detector Current (mV)	Ambient Temperature (°C)	Ambient Pressure (atm)	Injection Volume (mL)
1 Amorphous	60.06 mL /min	100	25.0 °C	28.8 m. Hg	4.0 mL
2 "	"	"	"	"	"
3 "	"	"	"	"	"
4 Breath	"	"	"	"	"
5 "	"	"	"	"	"
6 "	"	"	"	"	"
7 "	"	"	"	"	"
8 Amorphous water soluble	"	"	"	"	"
9 Beam	"	"	"	"	"

Humidity was 29%.

Standard 1 and sample 6 ~~were~~ had mistakes

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Discussion Questions

- 1). The breath most likely has higher CO_2 concentration and lower O_2 concentration.
The concentration of N_2 is likely very similar in lab air and breath.

~~$p = 1.00 \text{ atm}$ and 34.4°C is 1.16 g .~~

~~$M_{\text{air}} = \frac{28.97}{22.4} = (0.781 \times 28.0) + (0.210 \times 32.0)$~~

$$X_{\text{N}_2} = 0.781$$

$$X_{\text{O}_2} = 0.210$$

$$\text{mass percent N}_2 = \frac{0.781 \text{ mol} \times 28.0}{28.6} = 0.66 \quad 0.765$$

$$\text{mass percent O}_2 = \frac{0.210 \text{ mol} \times 32.0}{28.6} = 0.235$$

2.)

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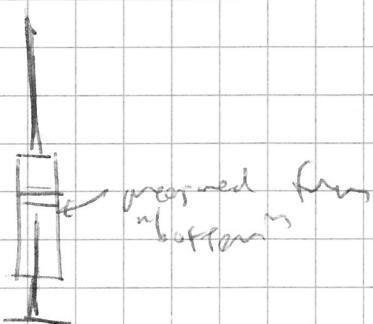
Testing Day Notes

- Flow rate: right to slow, like to quicker
- chromatograph Chromatograph ft 1 (400)

Measurement Day Notes

Flow rate A: 10.515 for 10 ml

Flow rate B: 10.475 " "

~~10.475~~

Flow rate: ~~10 ml~~ $\times \frac{60 s}{\text{min}} = 57.58$
~~10.475 x 6~~ min

At the tip of nozzle

New flow rates $\frac{10 \text{ ml}}{9.995} \times \frac{60 \text{ s}}{\text{min}} = 60.06 \text{ ml/min}$
 B: 9.995
 A: 9.725

Shift baseline for samples 3, 4 (zero)
 experienced over; part of pushing

Bend on needle

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Discussion Questions

1. The breath likely has a higher concentration of O_2 and a lower concentration of O_2 than the surrounding air. The N_2 concentration ~~must~~^(laboratory) should be very similar. The breath should also have higher levels of H_2O gas vapor.

Q1. Density of dry air at 1.00 atm and $34.4^\circ C$ is $1.16 \frac{g}{L}$

Assuming behavior is ideal: $PV = nRT$

$$\text{Volume of 1 mol of dry air: } V = \frac{nRT}{P} = \frac{(1\text{mol}) \left(\frac{0.08205 \text{ L atm}}{\text{mol K}} \right) (34.4\text{K} + 273.15\text{ K})}{(1.00 \text{ atm})} = 25.2 \text{ L}$$

$$\text{Molar mass dry air} @ 34.4^\circ C = \frac{1.16 \text{ g}}{25.2 \text{ L}} \times \frac{25.2 \text{ L}}{\text{mol}} = \frac{29.3 \text{ g}}{\text{mol}}$$

Assuming air is comprised only of N_2 and O_2 :

Let $x = \frac{\text{mole g of } N_2}{\text{mole g of air}}$,

$y = \frac{\text{mole g of } O_2}{\text{mole g of air}}$,

$$x + y = 100\%$$

$$M_{N_2}x + M_{O_2}y = M_{\text{air}} \Rightarrow M_{N_2}x + M_{O_2}(1-x) = M_{\text{air}}$$

$$\Rightarrow M_{N_2}x + M_{O_2} - M_{O_2}x = M_{\text{air}} \Rightarrow x(M_{N_2} - M_{O_2}) = M_{\text{air}} - M_{O_2}$$

$$\Rightarrow x = \frac{M_{\text{air}} - M_{O_2}}{M_{N_2} - M_{O_2}} = \frac{29.3 \frac{1}{\text{mol}} - 31.998 \frac{1}{\text{mol}}}{28.014 \frac{1}{\text{mol}} - 31.998 \frac{1}{\text{mol}}} = 0.677 \quad 0.684 \quad (\text{mole fraction } N_2)$$

$$y = 1 - x = 1 - 0.677 = 0.323 \quad (\text{mole fraction } O_2)$$

$$\text{Mass fraction } N_2 = \frac{18.014 \frac{g}{\text{mol}} \times 0.677}{29.3 \frac{g}{\text{mol}}} = 0.641 \quad 0.654$$

$$\text{Mass fraction } O_2 = \frac{31.998 \frac{g}{\text{mol}} \times 0.323}{29.3 \frac{g}{\text{mol}}} = 0.355 \quad 0.346$$

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A2

(systematic)

- a) This will introduce an error in the results because the GC does not detect some of the gas (water vapor) in the sample, the amounts of gas measured will be systematically low.
- b) Samples with more water vapor will be more greatly affected. Specifically, the sample of breath will be affected more than the (relatively) dry laboratory air.
- c) The partial pressure of water vapor can be calculated from the relative humidity (equation given) and vapor pressure of water (calculated from temperature). This can be subtracted from the total atmospheric pressure to obtain the partial pressure of the dry air — this can be used normally in the equation to calculate the moles of each gases.

$$\text{i.e.: } RH = 100\% \left(\frac{P_{\text{water}}}{P_{\text{vap}}} \right) \Rightarrow P_{\text{water}} = \frac{RH}{100\%} P_{\text{vap}}$$

$$P_{\text{dry}} = P_{\text{atm}} - P_{\text{water}}$$

\downarrow
actual pressure of measured gases (because GC doesn't detect H_2O).

Calculations from Mendle

a) moles H_2O in liter of humid air $T = 21.5^\circ K + 273.15^\circ K = 294.65^\circ K$

$$P_{\text{water}} = \frac{RH}{100\%} P_{\text{vap}} = \frac{\frac{RH}{100\%} e^{7.78}}{100\%} e^{\frac{77.345 + 0.0057T - 7235T^{-1}}{T^{8.2}}} =$$

$$\frac{0.470}{(294.65K)^{8.2}} e^{77.345 + 0.0057(294.65K) - 7235(294.65K)^{-1}} = 1200 \text{ Pa}$$

$$P = 29.56 \text{ in. Hg} \times \frac{3386.39 \text{ Pa}}{\text{in. Hg}} = 100100 \text{ Pa} \quad (\text{not used in this section})$$

$$V = 1 \text{ L (exact)}$$

$$n = \frac{PV}{RT} = \frac{1200 \text{ Pa} \times 1 \text{ L}}{(8314.4598 \frac{\text{J}}{\text{mol K}})(294.65 \text{ K})} = 4.91 \times 10^{-4} \text{ mol } H_2O \text{ in a Liter of humid air}$$

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2, contd

$$P_{\text{atm}} = 100,100 \text{ Pa} \quad (\text{calculated earlier})$$

mole %:

$$\text{N}_2: 78.08\%$$

$$\text{O}_2: 20.95\%$$

$$\text{Ar}: 0.93\%$$

$$\text{CO}_2: 0.04\%$$

(by ~~Henry's~~ ^{Dalton's} Law of Partial pressures, these are also the partial pressure fractions of the gases)

$$P_{\text{gas}} = (\text{mole \%}) P_{\text{atm}}$$

$$\text{and } n = \frac{PV}{RT} = \frac{P_{\text{gas}} V}{RT} = \frac{(\text{mole \%})(P_{\text{atm}}) V}{RT}$$

$$\text{moles N}_2: \frac{(0.7808)(100100 \text{ Pa})(1 \text{ L})}{(8314.4598 \frac{\text{L Pa}}{\text{mol K}})(294.65 \text{ K})} = 0.03190 \text{ mol N}_2$$

$$\text{moles O}_2: \frac{(0.2095)(100100 \text{ Pa})(1 \text{ L})}{(8314.4598 \frac{\text{L Pa}}{\text{mol K}})(294.65 \text{ K})} = 0.008560 \text{ mol O}_2$$

$$\text{moles Ar: } \frac{(0.0093)(100100 \text{ Pa})(1 \text{ L})}{(8314.4598)(294.65 \text{ K})} = 0.00038 \text{ mol Ar}$$

$$\text{moles CO}_2: \frac{(0.0004)(100100 \text{ Pa})(1 \text{ L})}{(8314.4598 \frac{\text{L Pa}}{\text{mol K}})(294.65 \text{ K})} = 0.0002 \text{ mol CO}_2$$

$$P_{\text{dry}} = P_{\text{atm}} - P_{\text{water}} = 100100 \text{ Pa} - 1200 \text{ Pa} = 98900 \text{ Pa} = \underline{\underline{98900 \text{ Pa}}}$$

now partial pressures are out of P_{dry} instead of P_{atm} ($P_{\text{gas}} = (\text{mole \%}) P_{\text{dry}}$)

$$P_{\text{H}_2\text{O}} = 1200 \text{ Pa} \quad (\text{from calc})$$

$$P_{\text{N}_2} = (0.7808)(98900 \text{ Pa}) = 77200 \text{ Pa}$$

$$P_{\text{O}_2} = (0.2095)(98900 \text{ Pa}) = 20700 \text{ Pa}$$

$$P_{\text{Ar}} = (0.0093)(98900 \text{ Pa}) = \underline{\underline{919 \text{ Pa}}} = 920 \text{ Pa}$$

$$P_{\text{CO}_2} = (0.0004)(98900 \text{ Pa}) = 40 \text{ Pa}$$

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O₂ cont'd8c) cont'd

Because partial pressure is proportional to mole fraction,
 mole fraction ~~can be~~ is equal to fraction of partial pressure to total pressure.

$$\text{i.e., mole fraction } g_i = \frac{P_{gas}}{P_{total}}$$

$$P_{total} = 100100 \text{ Pa}$$

$$\text{mole fraction H}_2\text{O} = \frac{1200 \text{ Pa}}{100100 \text{ Pa}} = 0.0120$$

$$\text{mole fraction N}_2 = \frac{71200 \text{ Pa}}{100100 \text{ Pa}} = 0.711$$

$$\text{mole fraction O}_2 = \frac{20700 \text{ Pa}}{100100 \text{ Pa}} = 0.207$$

$$\text{mole fraction Ar} = \frac{920 \text{ Pa}}{100100 \text{ Pa}} = 0.0092$$

$$\text{mole fraction C}_2\text{H}_6 = \frac{40 \text{ Pa}}{100100 \text{ Pa}} = 0.0004$$

(The mole fractions of the four "dry air" gases are slightly lower because of the addition of the H₂O vapor).

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$$\begin{aligned}
 2e) \text{ New moles of air} &= n_{N_2} + n_{O_2} + n_{Ar} + n_{CO_2} + n_{H_2O} \\
 &= 0.03190 \text{ mol} + 0.008630 \text{ mol} + 0.00038 \text{ mol} + 0.0002 \text{ mol} + 0.00049 \text{ mol} \\
 &= 0.04144 \text{ mol}
 \end{aligned}$$

Mole Percentages:

$$\chi_{N_2} = \frac{0.03190 \text{ mol}}{0.04144 \text{ mol}} = 0.7698$$

$$\chi_{O_2} = \frac{0.008630 \text{ mol}}{0.04144 \text{ mol}} = 0.2087$$

$$\chi_{Ar} = \frac{0.00038 \text{ mol}}{0.04144 \text{ mol}} = 0.0092$$

$$\chi_{CO_2} = \frac{0.00002 \text{ mol}}{0.04144 \text{ mol}} = \cancel{0.0005} - 0.0003$$

$$\chi_{H_2O} = \frac{0.000491 \text{ mol}}{0.04144 \text{ mol}} = 0.0118$$

$$\begin{aligned}
 \text{New volume: } V &= \frac{nRT}{P} = \frac{(0.04144 \text{ mol})(8314.4598 \frac{\text{J}}{\text{mol K}})(294.65 \text{ K})}{(100100 \text{ Pa})} \\
 &\approx 1.014 \text{ L}
 \end{aligned}$$

Partial Pressures:

$$P_{N_2} = 100100 \text{ Pa} \times 0.7698 = 77050 \text{ Pa}$$

$$P_{O_2} = (100100 \text{ Pa})(0.2087) = 20890 \text{ Pa}$$

$$P_{Ar} = (100100 \text{ Pa})(0.0092) = 920 \text{ Pa}$$

$$P_{CO_2} = (100100 \text{ Pa})(0.0003) = 40 \text{ Pa}$$

$$P_{H_2O} = (100100 \text{ Pa})(0.0118) = 1180 \text{ Pa}$$

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