Slide 1

Nitrogen Analysis

A bit of practice

Slide 2

Colorimetric Determination of NH_3

Standard solutions of ammonium hydroxide were prepared of 0.05 mM, 0.1 mM, 0.15 mM and 0.20 mM. 20.00 mL of the standard ammonium hydroxide solution was added to 20.00 mL of the phenate reagent (alkaline phenol, Mn^{2+}, and ClO_3^-). The resulting standard solutions were measured in a colorimeter yielding intensity values of 0.100 W/m^2, 0.189 W/m^2, 0.293 W/m^2 and 0.373 W/m^2, respectively.

1 L of water was collected from the swamp behind U lot on the RIT campus. 500 mL of the sample was added to 500 mL of a pH=9.5 borate buffer solution. The resulting mixture was distilled at 85°C for 35 minutes, until 250 mL of distillate was collected. The distillated was treated with 50 mL of the phenate reagent and placed in the colorimeter, yielding an intensity of 0.146 W/m^2.

What is the ammonia content of the swamp?

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First thing you need to do….

Prepare calibration curve…

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Calibration curve

The data:

$[NH_4^+]$	Volume	Intensity
0.05 mM	20 mL	0.100
0.10 mM	20 mL	0.189
0.15 mM	20 mL	0.293
0.20 mM	20 mL	0.373

Do we all agree?

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Calibration curve

The solution was diluted by addition of phenate.

$[NH_4^+]$	Volume	Diluted $[NH_4^+]$	Intensity
0.05 mM	20 mL	0.025 mM	0.100
0.10 mM	20 mL	0.050 mM	0.189
0.15 mM	20 mL	0.075 mM	0.293
0.20 mM	20 mL	0.10 mM	0.373

Now, do we all agree?

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We AGREE!

$[NH_4^+]$	Volume	Diluted $[NH_4^+]$	Intensity
0.05 mM	20 mL	0.025 mM	0.100
0.10 mM	20 mL	0.050 mM	0.189
0.15 mM	20 mL	0.075 mM	0.293
0.20 mM	20 mL	0.10 mM	0.373

So, now what?

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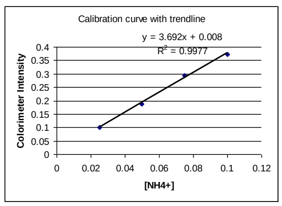
Make a graph (Excel or by hand)



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What is the ammonia content of the swamp?

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We're calibrated – now what?

Compare the sample to the calibration curve…

0.146 W/m^2

y = 3.692 x + 0.008
0.146 = 3.692 x + 0.008
0.138 = 3.692 x
0.0374 mM = x

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What is 0.0374 mM?

0.0374 mM is the concentration of NH_4^+ in the solution tested. What was that solution?

That solution was 250 mL of distillate mixed with 50 mL of phenate reagent.

If I want the concentration in the distillate, I need to correct for the dilution

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UNITS! UNITS! UNITS!

$$0.0374 \text{ mM} = \frac{0.0374 \text{ mmol } NH_4^+}{1 \text{ L diluted distillate}}$$

$$\frac{0.0374 \text{ mmol } NH_4^+}{1000 \text{ mL diluted distillate}} * \frac{300 \text{ mL diluted}}{250 \text{ mL distillate}}$$

$$= \frac{0.0449 \text{ mmol } NH_4^+}{1000 \text{ mL distillate}}$$

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Really just MV=MV

$M_{diluted}V_{diluted} = M_{original}V_{original}$

0.0374 mM * 300 mL = $M_{original}$ (250 ml)

= 0.0449 mM NH_4^+

Is this what we want?

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UNITS! UNITS! UNITS!

$\frac{0.0449 \text{ mmol } NH_4^+}{1000 \text{ mL distillate}}$

We want concentration in the swamp!

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Colorimetric Determination of NH_3

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What is the ammonia content of the swamp?

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Just another un-dilution

$M_{distilled}V_{distilled} = M_{swamp}V_{swamp}$

0.0449 mM NH_4* 250 mL=M_{swamp}* 500 mL

= 0.0225 mM NH_4^+ IN THE SWAMP!

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You'll see it reported two ways

$\frac{0.0225 \text{ mmol } NH_4^+}{1 \text{ L swamp}} * \frac{18.09 \text{ mg } NH_4^+}{\text{mmol } NH_4^+}$

(note mg/mmol is the same as g/mol)

= 0.407 mg/L NH_4^+

OR $\frac{0.0225 \text{ mmol } NH_3}{1 \text{ L swamp}} * \frac{17.09 \text{ mg } NH_3}{\text{mmol } NH_3}$

= 0.384 mg/L NH_3

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Lab calculation is similar

You need to do essentially the same thing with the lab, only it is nitrite rather than ammonia.

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The lab procedure…

Pipet 50.00 mL of the stock nitrite solution (250 μg/mL) into a 250 mL volumetric flask. Dilute to the mark.

Take 10 mL of the diluted stock solution and dilute it to 1.00 L in a volumetric flask.

Take 1 mL of the diluted stock and add to a 50.00 mL volumetric flask. Dilute to the line. Add 1.00 mL of sulfanilamide. Then add 1 mL of NED…etc.

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You've actually done 5 dilutions!

250 μg/mL in the original bottle

1. 50 mL to 250 mL dilution.
2. 10 mL of #1 diluted to 1 L
3. 1 mL of #2 diluted to 50 mL
4. Then #3 diluted from 50 mL to 51 mL
5. Then #4 diluted from 51 mL to 52 mL

#5 is the actual solution you measure and those are the concentrations that count. (Doesn't matter what the original bottle was anymore.)

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Your sample

You use that data to prepare the calibration curve and then compare your waste water (or unknown) to the calibration curve.

Again, the waste water sample you measure is NOT the same as the one in your bottle.

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Your sample

Your bottle

1. Take 5 mL from your bottle and dilute it to 50 mL.
2. Take #1 and add 1 mL of sulfanilamide.
3. Take #2 and add 1 mL of NED

So…if your sample measured 0.00250 M in the spectrometer…

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Un-dilute your sample

0.00250 M IN THE SPECTROMETER

How do you calculate what it is in your bottle?

MV=MV

$M_{bottle}V_{bottle}=M_{calorimeter}V_{calorimeter}$

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Un-dilute your sample

M_{bottle}5 mL =0.00250 M 52 mL

M_{bottle} = 0.026 M IN YOUR BOTTLE

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Multiple Nitrogen analyses

Consider the following results from a complete nitrogen work-up.

	Sample 1	Sample 2
Total Nitrogen	4.7 mg/L	3.9 mg/L
Organic Nitrogen	0.8 mg/L	0.8 mg/L
NH_3	1.3 mg/L	1.2 mg/L
NO_3^-	1.5 mg/L	0.6 mg/L
NO_2^-	0.9 mg/L	1.2 mg/L

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Which result is "better"?

	Sample 1	Sample 2
Total Nitrogen	4.7 mg/L	3.9 mg/L
Organic Nitrogen	0.8 mg/L	0.8 mg/L
NH_3	1.3 mg/L	1.2 mg/L
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NO_2^-	0.9 mg/L	1.2 mg/L

Sample 1 has more total nitrogen
BUT sample 2 has more nitrite relative to nitrate.

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Biodegradation of waste

In polluted waters, as organic contamination is biodegraded, ammonia concentration peaks early and then decreases during biodegradation. Nitrite (bad nitrogen) peaks later and then tails off even quicker. Nitrate rises later in the process.

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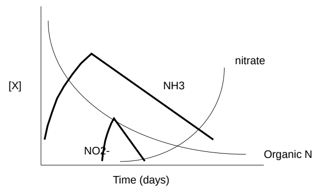
Nitrogen cycle



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NO_3^-	1.5 mg/L	0.6 mg/L
NO_2^-	0.9 mg/L	1.2 mg/L

From this data, it appears that sample 1 was initially dirtier, but it has gone farther toward cleaning itself and actually has a lower concentration of the more harmful NO_2^- (EPA limit 1 mg/L)

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A note on the HW.

Nitrite (NO_2^-) can be oxidized in acidic solution by $K_2Cr_2O_7$ in a reaction that yields nitrate and Cr^{3+} as the products. I took a 250 mL sample of waste water, added 10.00 mL of H_2SO_4 (sulfuric acid) and then titrated it with 0.01245 M potassium dichromate ($K_2Cr_2O_7$). It took 12.06 mL of potassium dichromate to reach the endpoint. What is the concentration of nitrite in the original solution (in M)? What is the concentration in mg NO_2^-/L?

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Just a titration…like any other titration.

Do do a titration you need….?

1. Reaction
2. Indicator

The only issue with the homework is: what's the reaction?

Slide 32

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It's a redox reaction

Nitrite (NO_2^-) can be oxidized in acidic solution by $K_2Cr_2O_7$ in a reaction that yields nitrate and Cr^{3+} as the products.

What are the reactants? What are the products?

$NO_2^- + Cr_2O_7^{2-} = NO_3^- + Cr^{3+}$

(you'll have to balance it)