

- (20%) Explain the following terms (4% each).
 - leveling/differentiating solvents
 - interferometer (for IR spectrometers)
 - normal/reverse phase HPLC
 - resolving power (in mass spectra)
 - chemical interference (in AAS/AES)
- (80%) Answer the following questions based on the procedure, shown in the right, for the determination of water hardness by using EDTA.
 - (3%) Write the full name of EDTA.
 - (b-1) (3%) Why is pH 10 (buffer) used for the titration experiments?
 - (b-2) (5%) The buffer preparation described here is not standard. List the standard steps to prepare a pH 10.00 ammonium buffer with a pH electrode/meter.
 - (b-3) (5%) How to calibrate a glass pH electrode?
 - (b-4) (5%) Explain the reason why many commercially available glass pH electrodes are not suitable for solutions with basicity > pH 10.
 - (c) (3%) Describe the purpose of ① drying EDTA.
 - (d-1) (8%) For acid-base titration, standardization of the titrant is necessary. Briefly describe how a titrant of HCl (~0.1 M) is standardized?
 - (d-2) (3%) The concentration of titrant (EDTA) is obtained simply by weighing (*i.e.*, ②) and dissolving in 500-mL water. Why is it unnecessary to include standardization similar to that in acid-base titration (*e.g.*, procedures for HCl in Question d-1)?
 - (e) (6%) Describe the meanings of ③ "50.00-mL" and ④ "3 mL" based on the concept of *significant figure*.
 - (f) (5%) Regarding ⑤, what would happen if there are a lot more than 6 drops of EBT introduced to the beaker? How does this affect the experimental results?
 - (g) (5%) Define ⑥ (blank titration).
 - (h) (8%) Why do $\alpha_{Y^{4-}}$ and α_M can make easy the calculations of $[M^{n+}]$?
 - (i-1) (5%) The results of the same tap water from two operators are 3.00 (± 0.05) mM ($n = 3$) and 3.20 (± 0.10) mM ($n = 3$). The total concentration is reported by taking average of the two values. Do the propagation of uncertainty and report the value.
 - (i-2) (6%) Are the results from the two operators significantly different based on *null hypothesis* at the 95% confidence level?
 - (i-3) (5%) Define *null hypothesis*.
 - (j) (5%) Propose some extra steps to find out the concentration of Ca^{2+} and Mg^{2+} (by EDTA titration) rather than the total concentration?

EDTA Titration of Ca^{2+} and Mg^{2+} in Tap Water

The most common multivalent metal ions in natural waters are Ca^{2+} and Mg^{2+} . In this experiment, we will find the total concentration of metal ions that can react with EDTA, and we will assume that this equals the concentration of Ca^{2+} and Mg^{2+} .

Reagents

EDTA: $Na_2H_2EDTA \cdot 2H_2O$ (FM 374.24), ~0.6 g.

Buffer (pH 10): Add 142 mL of 28 wt% aqueous NH_3 to 17.5 g of NH_4Cl and dilute to 250 mL with distilled water.

Eriochrome black T indicator: Dissolve 0.2 g of the solid indicator in 15 mL of triethanolamine plus 5 mL of absolute ethanol.

Unknowns: Tap water.

Procedure

- Dry $Na_2H_2EDTA \cdot 2H_2O$ at $80^\circ C$ for 1 h and cool in the ① desiccator. Accurately weigh out ~0.6 g and dissolve it with heating in 400 mL of water in a 500-mL volumetric flask. Cool to room temperature, dilute to the mark, and mix well.
- Pipet a sample of unknown (a ② 50.00-mL sample of tap water) into a 250-mL flask. To each sample, add ③ 3 mL of pH 10 buffer and ④ 6 drops of Eriochrome black T indicator. Titrate with EDTA from a 50-mL buret and note when the color changes from wine red to blue. Practice finding the end point several times by adding a little tap water and titrating with more EDTA. Save a solution at the end point to use as a color comparison for other titrations.
- Repeat the titration with three samples to find an accurate value of the total $Ca^{2+} + Mg^{2+}$ concentration. Perform a blank titration with 50 mL of distilled water and subtract the ⑤ value of the blank from each result.
- Calculate the total $Ca^{2+} + Mg^{2+}$ concentration. Calculate the relative standard deviation of replicate titrations.

Table 3-1 Summary of rules for propagation of uncertainty

Function	Uncertainty	Function ^a	Uncertainty ^b
$y = x_1 + x_2$ $y = x_1 - x_2$	$e_y = \sqrt{e_{x_1}^2 + e_{x_2}^2}$	$y = x^a$	$\%e_y = a\%e_x$
$y = x_1 \cdot x_2$ $y = \frac{x_1}{x_2}$	$\%e_y = \sqrt{\%e_{x_1}^2 + \%e_{x_2}^2}$	$y = \log x$	$e_y = \frac{1}{\ln 10} \frac{e_x}{x} \approx 0.434 29 \frac{e_x}{x}$
		$y = \ln x$	$e_y = \frac{e_x}{x}$
		$y = 10^x$	$\frac{e_y}{y} = (\ln 10)e_x \approx 2.302 6 e_x$
		$y = e^x$	$\frac{e_y}{y} = e_x$

a. x represents a variable and a represents a constant that has no uncertainty.

b. e_x/x is the relative error in x and $\%e_x$ is $100 \times e_x/x$.

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

$$y = \frac{1}{\sigma\sqrt{2\pi}} e^{-(x-\mu)^2/2\sigma^2} \quad \mu = \bar{x} \pm \frac{ts}{\sqrt{n}}$$

$$t_{\text{calculated}} = \frac{|\bar{x}_1 - \bar{x}_2|}{s_{\text{pooled}}} \sqrt{\frac{n_1 n_2}{n_1 + n_2}}$$

$$s_{\text{pooled}} = \sqrt{\frac{\sum_{\text{set1}} (x_i - \bar{x}_1)^2 + \sum_{\text{set2}} (x_j - \bar{x}_1)^2}{n_1 + n_2 - 2}}$$

$$= \sqrt{\frac{s_1^2(n_1 - 1) + s_2^2(n_2 - 1)}{n_1 + n_2 - 2}}$$

TABLE 4-2 Values of Student's t

Degrees of freedom	Confidence level (%)						
	50	90	95	98	99	99.5	99.9
1	1.000	6.314	12.706	31.821	63.657	127.32	636.619
2	0.816	2.920	4.303	6.965	9.925	14.089	31.598
3	0.765	2.353	3.182	4.541	5.841	7.453	12.924
4	0.741	2.132	2.776	3.747	4.604	5.598	8.610
5	0.727	2.015	2.571	3.365	4.032	4.773	6.869
6	0.718	1.943	2.447	3.143	3.707	4.317	5.959
7	0.711	1.895	2.365	2.998	3.500	4.029	5.408
8	0.706	1.860	2.306	2.896	3.355	3.832	5.041
9	0.703	1.833	2.262	2.821	3.250	3.690	4.781
10	0.700	1.812	2.228	2.764	3.169	3.581	4.587
15	0.691	1.753	2.131	2.602	2.947	3.252	4.073
20	0.687	1.725	2.086	2.528	2.845	3.153	3.850
25	0.684	1.708	2.060	2.485	2.787	3.078	3.725
30	0.683	1.697	2.042	2.457	2.750	3.030	3.646
40	0.681	1.684	2.021	2.423	2.704	2.971	3.551
60	0.679	1.671	2.000	2.390	2.660	2.915	3.460
120	0.677	1.658	1.980	2.358	2.617	2.860	3.373
∞	0.674	1.645	1.960	2.326	2.576	2.807	3.291

In calculating confidence intervals, σ may be substituted for s in Equation 4-3 if you have a great deal of experience with a particular method and have therefore determined its "true" population standard deviation. If σ is used instead of s , the value of t to use in Equation 4-3 comes from the bottom row of this table.

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