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This document is an instructional module package prepared in objective form for use by an instructor familiar with the titrimetric method for determining calcium concentrations in water. Included are objectives, an instructor guide, student handouts, and transparency masters. A videotape is also available from the author. This module considers the chemistry and principles of the determination, preparation of reagents, titration of the sample and calculation and interpretation of results. (Author/RH)

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TO-THE EDUCATIONAL RESOURCES INFORMATION CENTER (ERIC) AND USERS OF THE ERIC SYSTEM,"

CALCIUM ANALYSIS

ED152583

SE 024 149

Training Module 5.225.2.77

Prepared for the

Iowa Department of Environmental Quality Wallace State Office Building Des Moines, Iowa 50319

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September, 1977

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Module No:	Module Title: Calcium An	nalysis		
1	Submodule Title:	· · · · · ·		1. A.
Approx. Time:		The state of the state	•	te dite A te state a
2 hours		1		
	Topic:		· · · · · · · · · · · · · · · · · · ·	
•	Summary	"L L	1. 2.	
Instructional Obje	ctive:	• *	j	•
Upon completion of		participant	should be	able to:
1. Determine	the concentration the EDTA titrime	n of calcium	in a water	
2. List poss results o	ible interfering f the determination	ions which m on.	ay affect 1	he
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Calcium Analysis Submodule Title: Approx. Time: 5.25 hours Topic Topic Chemistry of Calcium in Water Instructional Objective: Upon completion of this module the participant should be able to: 1. Describe sources of calcium in water supplies. 2. Write a chemical reaction between calcium and EDTA. 3. Describe how Mg can be removed from a water sample so that it will not interfer with the calcium analysis. Instructional Aids: Transparency Cal- Reaction of calcium with EDTA. Instructional Approach: Lecture/discussion References: Standard Methods, p. 185, 186, 189. Class Asyignments: 4	Module No:	Module, Title:	· · · · · · · ·	·. ·		1
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Standard Methods, p. 185, 186, 189.			r-	<u></u>		
Standard Methods, p. 185, 186, 189.	References:	··	\ ·		.* .	
		. p. 185. 186	189.			
Class Assignments:			54		τ΄.	
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Module No: Top:	Ic: Chemistry of calcium in water
Instructor Notes:	Instructor Outline:
	1. Calcium, a metal, exists in water a in compounds as the Ca ²⁺ ion. It i
	major contributor to water hardness From water passage through Ca conta
	minerals:
	a. lime stone CaCO3
	b. dolomite CaCO ₃ ·MgCO ₃ c. gypsum CaSO ₄ ·2H ₂ O d. gypsiferous shale
Transparency Ca-1 . Ca-EDTA reaction	2. $Ca^{2+}+(EDTA)^{4-} \rightarrow (Ca(EDTA))^{2-}$
	3. Magnesium is largely removed by pre
	tation as Mg(OH) ₂ at high pH.
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Module No:	Module Title: Calcium	Analysis	P Sime	· · · ·
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Approx. Time:		•	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	• •
0.25 hours	Topic:	•		· · ·
		les of Calci	ium Determina	ation
* 1	· · · · ·	,	•	· · · · ·
Instructional Obj	ective:	1		.*
Upon completion of	of this module t	he participa	ant should be	e able to
1. Describe	the EDTA - calc	ium analysia	s briefly.	•
2. Explain	how the calcium and from the determined	analysis is	similar to a	and
differen	une deter		voval nardn	
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Instructional Aid	18:	ι. Ι		
Softening videot	ape.	•	۰.	
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••	•	•		
Instructional App	proach:			
Videotape viewin	ng/discussion		•	
Patron			<u>.</u> 'a	
References:	The second second		• •	
	D. 180_101 20	0-206.		
Standard Methods	, P. 107-171, 20			
Standard Methods	, P. 107-171, 20	•		• •
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Standard Methods Class Assignments		•		· · ·

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Instructor	Notes:		les of tructor					
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Module No:	Module	Title:		.1			· · ·
		alcium	Analýs	18		•	
	Submodu	le Titl	e:		.1		
Approx. Time:		*		• •	·: , /	•	• • 1
0.25 hours	Topic:			•.			
		Safety			- No		
Instructional Obj	ective:				•	· · ·	· ·
Upon completion		odule t	he par	rticip	ant sho	ould be	able to:
	the follow use: emerg	gency sh	the lanower,	aborat fire	ory and extingu	demons	trate eye
2. Select a	2	afety gl	asses e sit	, lab uation	coats c	or apron	and
3. Describe determin	any haza	irds ass	ociat	ed wit	h the c	alcium	••
	·				. •		
١.		*	·	· .	4	•	
Instructional Aid	8:					• •	
Handout of safet	y rules f	or the	labor	atory.			,
Handout of safet	y rules f	or the	labor	atory.	•	•	
· · · · · · · · · · · · · · · · · · ·		or the	labor	atory.			
Handout of safet Instructional App Lecture, demonst	oroach:	or the		atory.		· · · · · ·	
Instructional App	oroach:	or the	labor	story.		· · · · · · · · · · · · · · · · · · ·	
Instructional App	oroach:	or the	labor	atory.			
Instructional App Lecture, demonst	proach: tration.		labor	atory.			
Instructional App Lecture, demonst References:	proach: tration.		labor	atory.			
Instructional App Lecture, demonst References:	proach: tration.		labor	atory.			

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Module No: Ca	Topic:	fety
Instructor No		nstructor Outline:
		•
· ·		Show the location of the various p of safety equipment.
• •		of safety equipment.
		. Safety glasses should be worn when
		 Safety glasses should be worn when adjusting pH and during titration.
· . ·		. The corrosive nature of NaOH shoul pointed out.
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Module No:	Module Title:	0		
	Calcium	'Analysis		
• 	Submodule Titl			,
	Submodule 1101		i . ·	
Approx. Time:				· · ·
0.5 hours	Topic:		5	
\ ·.		tion of Reagen	ts	•
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Instructional Obj	eative.			· "1
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Upon completion			•/ ·	
	the following re Murexide indica			
indicato	r.			
2. Calculat	e the concentrat	ion in AgCa/m	l equivale	nt of the
standard	EDTA solution s equivalent.	tandardized in	units of	M or mg
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Instructional Aid	8:	')	ę	
Transparency	Ca2 - calculatio	n of equivalen	t concentr	ations.
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	.7 * !	· · · · · · · · · · · · · · · · · · ·		
Instructional App				/ .
Laboratory pr	actice			
		• ,	•	
References:		12		
	ods, pp. 189, 19	0.		
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		*		
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Class Assignments	3: •			

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		Page	11 of 17	
Module No: Topic				1
Ca	Preparat	ion of REagents	*	· · ·
Instructor Notes:	Instru	ctor Outline:		
				1
			•	· .
1.a. 1N NaOH: 40g/liter	1. Re	agents máy be pr	epared in	groups of
. •	pr	epared before-ha	ind.	· ·
b, 0.2g indicator ground with 100g	·at	1 N NaOH		
NaCl		murexide indic	ator	
c. 0.2 g indicator g	or			
ground with 100g		1		•
NaCl	c.	Eriochrom Blue	Black R	
		•		•
Transparency Ca-2 calculations	2. Ca	lculation:		1
calculations .	mg	(Ca)/l equivalen	t=0.4 X (1	(g/1)
	1			
	mg	(Ca)/l equivalen	it=caco3 ec	luivaient
		•	.04 X M(I	EDTA)
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Module No:	Module Title:
	Calcium Analysis
the region of provide the	Submodule Title:
Approx. Time:	
	•
0.5 hours	Topic:
4	Titration of sample
and the second second	
Instructional Ob;	
Upon completion	of this module the participant should be able to
1. Properly	y prepare a water sample for titration.
2. Properly	y titrate the prepared sample to the end point
and rec	ord the appropriate data.
· · · · · · · · · · · · · · · · · · ·	
· · · · · · · · · · · · · · · · · · ·	
Instructional Aid	
1.	
Instructional App	proach:
Laboratory prac	tice
References:	
Standard Method	s p.190.
Class Assignment	

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Module No: Ca Topic: Titration of Sample Instructor Notes: Instructor Outline; 1. Prepare sample by: a. 50 ml sample plus 50 ml H_O b. Add 2.0 ml NaOH c. Check pH 2: Titration: a. titrate rapidly b. Color changes i. Murexide pink-purple ii. "Erichrome Blue-Black R red- blue	Ca Titration of Sample Instructor Notes: Instructor Outline; 1. Prepare sample by: . a. 50 ml sample plus 50 ml H_0 b. Add 2.0 ml NaOH c. Check pH 2. Titration: a. titrate rapidly b. color changes i. Murexide pink-purple ii. "Eriochrome Blue-Black R red-	Ca Titration of Sample Instructor Notes: Instructor Outline; 1. Prepare sample by: . a. 50 ml sample plus 50 ml H_0 b. Add 2.0 ml NaOH c. Check pH 2. Titration: a. titrate rapidly b. color changes i. Murexide pink-purple ii. "Eriochrome Blue-Black R red-
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Calcium Analysis Submodule Title: Approx. Time: 0.25 hours Topic: Calculation and Interpretation of results Instructional Objective: Upon completion of this module the participant should be able to: 1. Calculate the concentration in a water sample as mg/ 1 Ca or as mg/l CaC03 (calcium hardness). 2. List several possible sources of interference which may affect the result. Instructional Aids: Transparency Ca3-Interferences in the calcium determination. Instructional Approach: Lecture/discussion References: Standard Methods pp. 189, 190.	Module No:	Module Title	r	· · ·		;
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3.

Module No: Topic	:
Ca	
Instructor Notes:	Instructor Outline:
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	1. Calculation:
	mg/l(Ca) = ml(EDTA)
	ml sample X mg/l equivalent
	mg/l(CaCO ₃)= <u>ml(EDTA)</u> ml sample X mg/l equivale
	CaCO
Transparency Ca-3 Interferences	2. Interferences: a. CaCO, precipitation correct with H
	b. organics - digestion
• •	c. high alkalinity - neutralize with HCl, boil
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Exam Questions

Calcium Analysis Chemistry of Calcium in water

- Which of the following mineral does not contribute to dissolved 1. calcium in water?
 - a. limestone
 - b. gypsúm
 - c. dolomite
 - d. quartz
- molecule(s) of EDTA react(s) with one calcium ion 2. to form one complex ion.
- 3. Magnesium can be removed as an interference in the calcium determination by:
 - adding cyanide ion a.
 - lowering the pH b.
 - c. raising the pH to 12
 - d. chlorination

Principles of Calcium Determination

- Does calcium combine with EDTA before or after magnesium?
- 5. The titrimetric determination of calcium and the titrimetric determination of hardness are essentially the same except:
 - a. a different indicator is used b. a different titrant is used
 - c. a spectrophotometer is used in one case and not the other one' requires very specialized equipment d.

Safety

- Where can one go in the laboratory to find a band aid if one's 6. finger is cut by glass tubing? .
- What can be worn to prevent acid holes in a new shirt worn to 7. the laboratory?
- Which of the following reagents used in the calcium determination would you consider most hazardous?
 - EDTA titrant a.
 - Sodium hydroxide solution b.
 - Indicator mixture c.

Preparation of Reagents

- 9. How many grams of NaOH are required to prepare a one liter solution which is 1.0 N?
 - a. 500 g 0.01 q b.
 - c. 40 g. d., 1 g

10. A solution of EDTA is equivalent to 1 mg CaCO3 per ml. What is its calcium equivalence in micrograms per ml?

page 17 of 17

Titration of Sample

11. Prior to titration, which of the following should be done to the sample?

a. add 50 ml EDTA solution

b. add 2.0 ml sodium hydroxide solution

c. add 5.0 ml CaCO,

- d. 'extract the solution with chloroform
- 12. The Eriochrome Blue-Black R indicator changes from red to at end point.

Calculation and Interpretation of Results

- 13. It takes 25 ml of an EDTA solution in which the mg/l Ca equivalent is 400.8 to titrate a 50 ml water sample. What is the concentration in mg/l Ca of this sample?
- 14. How can organic interferences to the calcium determination be over come?

CALCIUM ANALYSIS

EQUIPMENT AND SUPPLIES LIST

sodium hydroxide pellets, NaOH 1 & volumetric flask 2. 2000 ml beaker 3. 4. hot plate 5. distilled water . . 1000 ml graduated cylinder 6 1 & polyethylene bottle (7. eriochrom blue black R indicator. 8: .9. sodium chloride mortar and pestle 10. 100 ml wide mouth sample bottle. 11. 12. disodium ethylenediaminetetraacetate 13. analytical balance 14. calcium carbonate 500 ml erlynmeyer flask 15. 16. funnel : hydrochloric acid, concentrated 17. 18. bunsen burnerring stand and ring 19. methyl red indicator 20. concentrated ammonium hydroxide 21. 22. 2 - 50 ml buret 23. 5 - 125 ml erlynmeyer flask 24. 25 = ml pipet

Calcium Analysis

Laboratory Procedure

Preparation of Reagents and Standards

A. Obtain the equipment, supplies, and chemicals listed in the "equipment" handout.

B. Prepare the following solutions

 N NaOH Sodium Hydroxide: Add 40 g sodium hydroxyde pellets to a 11 volumetric flask. Boil 1500 ml distilled water for five minutes; allow to cool covered. Add 750 ml
 of the cooled water to the flask. Mix to dissolve the NaOH, allow to cool. Dilute to the mark with distilled water. Mix. Transfer to a labeled polyethylene bottle.

 Indicator. Add 0.2 g Eriochrome Blue Black R indicator and 100 g sodium chloride (NaCl) to a mortar. Mix and grind with a pestle. Transfer to a stoppered, labeled bottle.
 EDTA titrant. Weigh 3.723 g disodium ethylenediaminetetraacetate dihydrate (Na₂H₂C₁₀H₁₂O₈N₂-2H₂O) or (Na₂EDTA) on an analytical balance and transfer to a 11 volumetric flask. Add 500 ml distilled water, swirl to dissolve. Dilute to the mark with distilled water. Mix and transfer to a labeled polyethylene bottle.

C. Prepare the following standard solution:

Weigh 1.000g anhydrous, primary standard grade calcium carbonate (CaCO₃) on an analytical balance and record the exact mass. Quantitatively transfer the CaCO₃ to a 500 ml erlynmeyer flask. Place a funnel on the neck of the flask and add 6M hydrochloric acid, HCl (prepared by mixing equal parts concentrated HCl and distilled water) a little at a time until bubbling has ceased and all the CaCO₃ has dissolved. Add " 200 ml distilled water and boil with a bunsen burner for five minutes. Cool. Add 2 drops methyl red indicator. Mix. If the solution is yellow, add 6M HCl dropwise until orange. If the solution is red, add 3M NH₂ (ammonia). [prepare by dissolving 200 ml concentrated ammonia in distilled water to make a 1 liter solution] dropwise until orange. Transfer the solution to a 1 liter volumetric flask. Dilute to the mark with distilled water. The concentration of the solution in mg/ml CaCO₂ is numerically equal to the mass in gram CaCO₂ added.

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The concentration of Ca in mg/ml is 0.4008 times the concentration of CaCO₃.

II. Titration

A. Titration of standard

Fill a 50 ml buret with EDTA solution. Fill another buret with standard Ca solution. Record the initial readings of each buret. Add 25 ml Ca solution to a 125 ml elynmeyer flask. Add 25 ml distilled water. Add 2.0 ml NaOH solution. Stir to mix. Add 0.2g of indicator. Add EDTA titrant slowly with swirling in white light until the solution turns from red to blue (end-point). Record the final values of both burets. Repeat the titration twice. Record results on data sheet. Volume of each solution used is equal to the difference between final and initial buret readings. The CaCO₃ equivalence of the EDTA solution is equal to the grams CaCO₃ weighed times and CaCO₃ solution used divided by ml EDTA solution. Average the three values, call this value B.

B. Sample titration

K.

Pipet three 25 ml well-mixed samples aliquots into three 125 ml erlynmeyer flasks. Add and mix 25 ml distilled water. Add 2.0 ml NaOH and 0.2g indicator. Titrate in triplicate with EDTA solution as in "A" above. Record initial and final buret readings.

III. Calculation and Evaluation of Results.

The volume of solution used in each case will be equal to the final minus the initial buret reading. The average volume from the three titrations will be the volume used in calculation. Call this value "A". The calcium hardness (as mgl l $CaCO_3$) is calculated from the following formula:

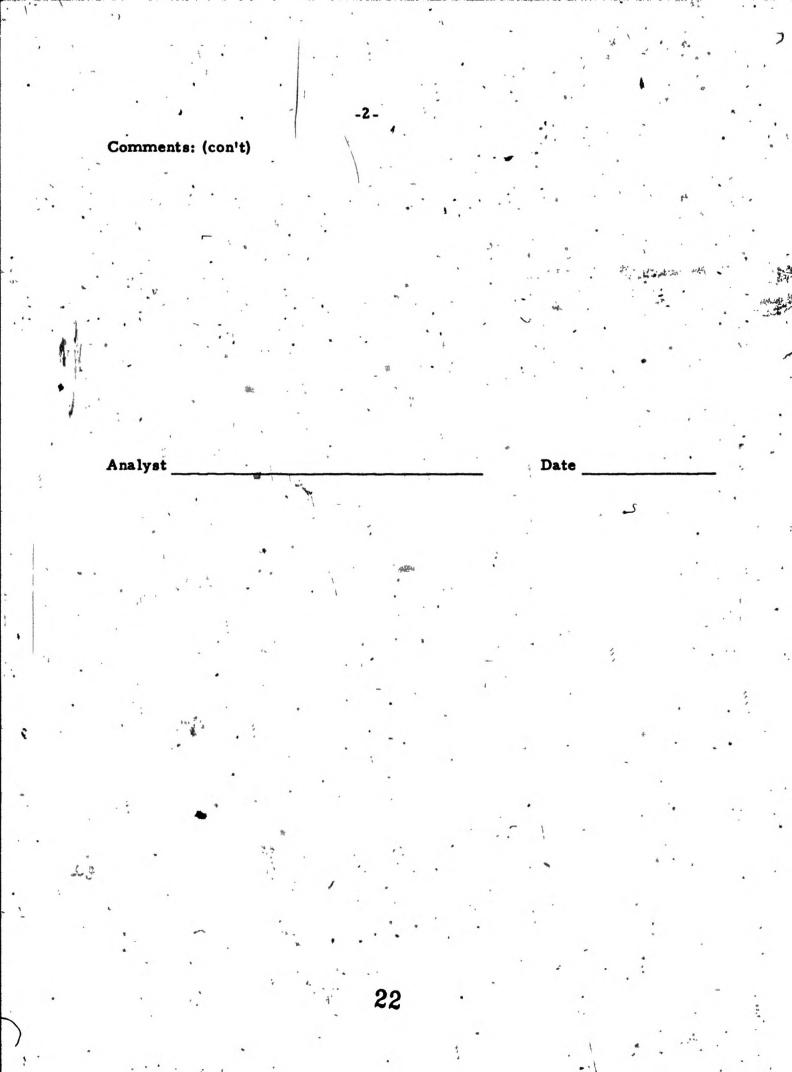
 $C = Calcium hardness (as CaCO_3) = \frac{A \times B \times 1000}{25}$

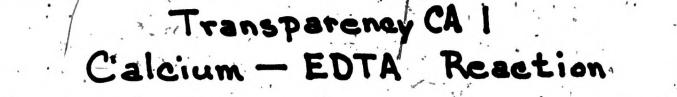
The calcium concentration is C times 0.4008.

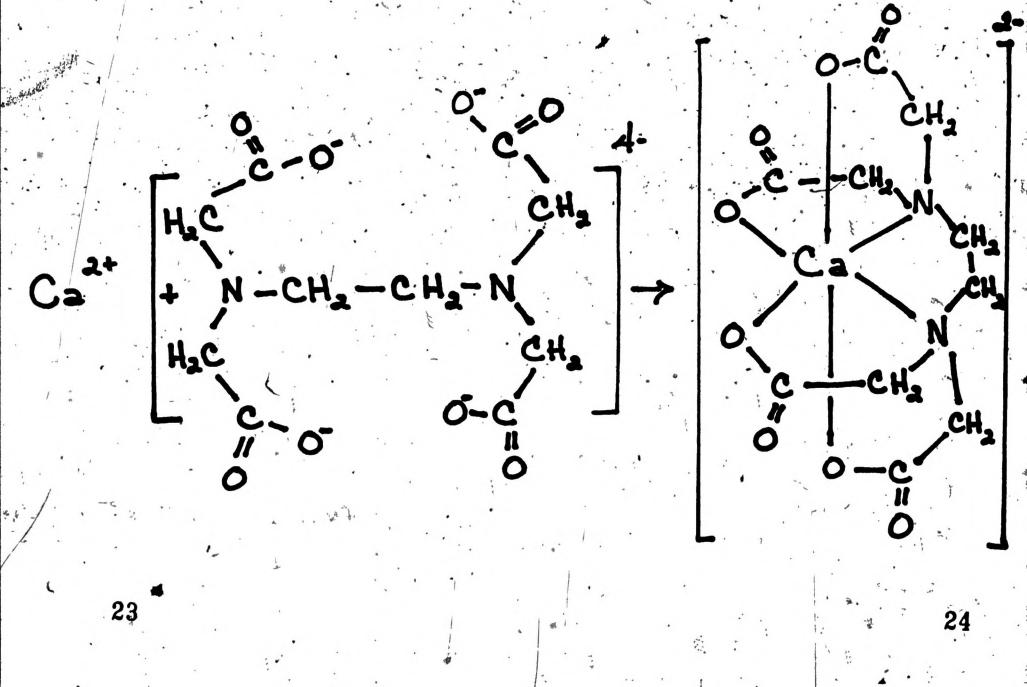
Comment on possible errors, distinctness of end-point and source of sample.

Calcium Analysis

Sample number **Data Sheet** Preparation of standard CaCO₃ mass of container plus CaCO₃ mass of container :0 (d) mass of CaCO₂ concentration $CaCO_3$ standard mg/ml Standardization of EDTA solution П ш I final caburet. ml ml ml initial caburet ml. ml ml ml (e) ml Ca used ml ml f inal EDTA buret ml ml ml initial EDTA buret ml ml ml ml EDTA used ml ml ml (f) EDTA equivalance $(CaCO_3) = \frac{d \times e}{5}$ mg/ml mg/ml mg/ml average EDTA equivalence _mg/ml CaCO₂ Β. = (I + II + III) Sample titration final EDTA buret ml ml ml initial EDTA buret ml ml ml ml EDTA used ml ml ml A = Average EDTA volume ml = (I + II + III)# C*= calcium hardness (as CaCO₃) = mg/l $\mathbf{C} = \mathbf{A} \mathbf{x} \mathbf{B} \mathbf{x} \mathbf{1000}$ Calcium concentration = $C \ge 0.4008 =$ mg/1 Comments:







TRANSPARENCY CA2

Calculation of Equivalent Concentrations

To convert from mg per ml CaCO3 equivalent to mg/l Ca equivalent; Multiply by 400.8

To convert from mg per ml CaCC₃ equivalent to mg/l CaCO₃; Multiply by 1000

To'convert from Molar to mg Ca per ml; Multiply by 0.4.

TRANSPARENCY CA3

Interferences in the Calcium Determination

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Interference	Correction
CaCO ₃ precipitation	Add HCL, boil.
Organics	Digestion
High Alkalinity	Neutrolize with HCl, Boil
Magnesium	Precipitate as Mg(OH) at high pH

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