



Thermo

ELECTRON CORPORATION

Potentiometric Titration Application Notes

Applications Log # 700A

Overview

The concentration of bicarbonate in water is determined using the preset endpoint titration technique on the ORION 960 Titrator with hydrochloric acid as a titrant. A pH electrode is used to measure the endpoints at pH 8.3 and pH 4.5. The difference in sample concentration between the two endpoints represents the concentration of bicarbonate in the sample.

Market	Pharma	Species Measured	Bicarbonate
Sample	Pancreatic Fluid	Sample Size	1 mL
Technique #	8 Preset Endpoint	Typical Concentration	90 meq/L
Solutions	0.05M HCl; 0.1M NaOH; 3M KCl Electrode Fill 810007, 0.1M NaCl		
Solutions preparation:	0.05M HCl (first dilute 82.4mL conc HCl to 1L with deionized water for 1M HCl, then dilute 25mL 1M HCl to 500mL with deionized water) 0.05M NaOH (dissolve 2.00g of reagent grade NaOH in 1L of deionized water) 0.1M NaCl (5.84g NaCl in 1L deionized water).		
Titrant standardization	Standardize 0.05M HCl by titrating 10mL of 0.05M NaOH.		
Sample Prep	Accurately pipet 1 mL of sample into 25mL of 0.1M NaCl in a 50mL plastic beaker.		

Statistics

of Trials 11 **Mean** 88.62 meq/L **%CV** 1.65 **Analysis Time** 7.3 minute(s)

Comments Before each titration day put new fill solution in the electrode. When electrode is not being used, keep it immersed in electrode storage solution. Rinse the electrode, stirrer, and dispenser probe thoroughly between measurements with 0.1M NaCl.

If using an Autosampler with the 960, choose the rack which holds 48 of the 50mL beakers and fill the first 3 beakers with 0.1M NaCl to be used for washing the electrode between samples.

Method Parameters

Sample Volume/Weight	1.00 mL	Timed or Stability Readings	10.0 mV/min stability
Constant Increment	10.0 mV	Number of Endpoints	2
Max Titrant Volume	5.00 mL	Desired Units	meq/L
Molecular weight	61.00 g	Predose	none
Prestir	10.0 seconds	Additional Parameters	Calibrate the electrode daily with buffers of pH 7 and 10. This can be done manually or within the run on the autosampler. The bicarbonate concentration is determined by subtracting the concentration at the second endpoint from that at the first.
Reaction Ratio	1.00		



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Results

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METHOD 1 SUMMARY

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SAMPLE ID NUMBER: 25
 TEST: Bicarbonate
 SITE:
 ANALYST:
 17:16 06-30-06 ELECTRODE: 1:pH
 TECHNIQUE 8 PRESET END POINT
 SLOPE 61.58 mV/dec
 Eo 8.4 mV
 SAMPLE VOLUME 1.000 mL
 TITRANT .05310 M of _HCl_
 CONST INCREMENT 10.0 mV
 MAX TITRANT VOL 5.000 mL
 STABILITY CRITERION 10.0 mV/min
 PRESTIR 10.0 sec
 CONTINUOUS STIRRING
 REACTION RATIO 1.0000
 CAL CONSTANT 1.00492
 PRESET pH(1) 8.30
 PRESET pH(2) 4.50

0 v= 0.000 mL E= -122.9 mV 12 sec
 -1.0 mV/min drift +/- 0.0 mV noise
 pH= 9.13

1 v= 0.151 mL E= -66.7 mV 68 sec
 4.9 mV/min drift +/- 0.0 mV noise
 pH= 8.22

2 v= 0.201 mL E= -46.3 mV 27 sec
 -1.7 mV/min drift +/- 0.0 mV noise
 pH= 7.89

3 v= 0.251 mL E= -34.7 mV 27 sec
 -3.5 mV/min drift +/- 0.0 mV noise
 pH= 7.70

4 v= 0.352 mL E= -19.1 mV 27 sec
 -4.1 mV/min drift +/- 0.0 mV noise
 pH= 7.45

5 v= 0.452 mL E= -9.1 mV 27 sec
 -4.8 mV/min drift +/- 0.0 mV noise
 pH= 7.28

6 v= 0.603 mL E= 3.0 mV 27 sec
 -4.6 mV/min drift +/- 0.0 mV noise
 pH= 7.09

7 v= 0.754 mL E= 13.8 mV 16 sec
 3.9 mV/min drift +/- 0.2 mV noise
 pH= 6.91

8 v= 0.904 mL E= 22.9 mV 16 sec
 -1.8 mV/min drift +/- 0.0 mV noise
 pH= 6.77

9 v= 1.105 mL E= 35.4 mV 15 sec
 0.5 mV/min drift +/- 0.1 mV noise
 pH= 6.56

10 v= 1.256 mL E= 45.0 mV 16 sec
 -1.7 mV/min drift +/- 0.0 mV noise
 pH= 6.41

11 v= 1.407 mL E= 56.4 mV 16 sec
 -2.9 mV/min drift +/- 0.0 mV noise
 pH= 6.22

12 v= 1.507 mL E= 65.9 mV 16 sec
 -4.0 mV/min drift +/- 0.0 mV noise
 pH= 6.07

13 v= 1.608 mL E= 80.0 mV 16 sec
 -2.7 mV/min drift +/- 0.0 mV noise
 pH= 5.84

14 v= 1.658 mL E= 90.3 mV 15 sec
 -2.6 mV/min drift +/- 0.0 mV noise
 pH= 5.67

15 v= 1.708 mL E= 107.9 mV 16 sec
 -2.3 mV/min drift +/- 0.0 mV noise
 pH= 5.39

16 v= 1.759 mL E= 141.1 mV 16 sec
 1.7 mV/min drift +/- 0.0 mV noise
 pH= 4.84

17 v= 1.809 mL E= 167.3 mV 16 sec
 2.4 mV/min drift +/- 0.0 mV noise
 pH= 4.42

8.1 min

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PRESET END POINT ANALYSIS

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SAMPLE = 7.3 meq/L
 END POINT VOL= 0.137 mL (-71.6 mV)
 (pH 8.30)
 Excess Titre= 1.671 mL

SAMPLE = 95.5 meq/L
 END POINT VOL= 1.799 mL (162.4 mV)
 (pH 4.50)
 Excess Titre= 0.009 mL

