



Thermo

ELECTRON CORPORATION

Potentiometric Titration Application Notes

Applications Log # 700B

Overview

The concentration of bicarbonate in water is determined using the first derivative titration technique on the ORION 960 Titrator with sodium hydroxide as the titrant. A known amount of hydrochloric acid is added to the sample and the excess acid is titrated with the base. The endpoint of this back titration is measured by a pH electrode. The moles of reacted acid are equal to the moles of bicarbonate in the sample. The operator must have a general idea of the bicarbonate concentration in the sample.

Market	Pharma	Species Measured	Bicarbonate
Sample	Pancreatic Fluid	Sample Size	1 mL
		Typical Concentration	80 meq/L
Technique #	6 First Derivative	Electrode	Ross Combination pH 8102BN
Solutions	0.05M HCl; 0.05M NaOH; 3M KCl Electrode Fill 810007		
Solutions preparation:	0.05M HCl (first dilute 82.4mL of concentrated HCl to 1L with deionized water for 1M HCl, then dilute 25mL of 1M HCl to 500mL with deionized water). 0.05M NaOH (dissolve 2.00g of reagent grade NaOH in 1L of deionized water).		
Titrant standardization	Standardize 0.05 N sodium hydroxide by titrating 3 mL of 0.1 N potassium hydrogen phthalate standard or buy a standardized solution.		
Sample Prep	Accurately pipet 1 mL of sample into 25mL of deionized water in a 50mL plastic beaker. Add 5 mL of 0.05M HCl to this diluted sample. This amount of HCl gives a blank value of -0.250 mmol.		

Statistics

of Trials 12 **Mean** 82.99 meq/L **%CV** 0.59 **Analysis Time** 5.6 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 deionized water.

If using an Autosampler with the 960, choose the rack which holds 48 of the 50mL beakers and fill the first 3 beakers with deionized water 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	1
Max Titrant Volume	5.00 mL	Desired Units	meq/L
Molecular weight	61.00 g	Predose	none
Prestir	60.0 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. Blank feature should be used and entered as -0.250 mmol.
Reaction Ratio	-1.00		



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Results

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METHOD 2 SUMMARY

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SAMPLE ID NUMBER: 5
 TEST: _____
 SITE: _____
 ANALYST: _____
 01:19 01-01-00 ELECTRODE: 1:pH
 TECHNIQUE 6 FIRST DERIVATIVE
 SAMPLE VOLUME 1.000 mL
 TITRANT .04930 M of __NaOH____
 CONST INCREMENT 10.0 mV
 MAX TITRANT VOL 5.000 mL
 STABILITY CRITERION 10.0 mV/min
 PRESTIR 60.0 sec
 CONTINUOUS STIRRING
 REACTION RATIO -1.0000
 BLANK VALUE -0.250000 mmol
 NO. OF ENDPOINTS 1
 CAL CONSTANT 1.00492

electrode check:+/- 0.9 mV

- 0 v= 0.000 mL E= 260.1 mV 12 sec
0.0 mV/min drift +/- 0.0 mV noise
pH= 2.30
- 1 v= 1.055 mL E= 250.1 mV 27 sec
-0.0 mV/min drift +/- 0.0 mV noise
pH= 2.47
- 2 v= 2.060 mL E= 235.6 mV 19 sec
-0.0 mV/min drift +/- 0.0 mV noise
pH= 2.72
- 3 v= 2.512 mL E= 225.2 mV 17 sec
0.0 mV/min drift +/- 0.0 mV noise
pH= 2.90
- 4 v= 2.764 mL E= 217.0 mV 16 sec
0.0 mV/min drift +/- 0.0 mV noise
pH= 3.04
- 5 v= 2.965 mL E= 207.9 mV 16 sec
0.0 mV/min drift +/- 0.0 mV noise
pH= 3.19
- 6 v= 3.115 mL E= 198.1 mV 16 sec
-0.0 mV/min drift +/- 0.0 mV noise
pH= 3.36
- 7 v= 3.216 mL E= 188.7 mV 16 sec
-0.6 mV/min drift +/- 0.0 mV noise
pH= 3.52
- 8 v= 3.266 mL E= 182.3 mV 16 sec
-0.0 mV/min drift +/- 0.0 mV noise
pH= 3.63
- 9 v= 3.316 mL E= 174.0 mV 16 sec
0.0 mV/min drift +/- 0.0 mV noise
pH= 3.77
- 10 v= 3.366 mL E= 162.2 mV 16 sec
-0.6 mV/min drift +/- 0.0 mV noise
pH= 3.97
- 11 v= 3.417 mL E= 144.3 mV 16 sec
-1.2 mV/min drift +/- 0.0 mV noise
pH= 4.28
- 12 v= 3.467 mL E= 121.9 mV 14 sec
-2.0 mV/min drift +/- 0.0 mV noise
pH= 4.66
- 13 v= 3.517 mL E= 104.7 mV 16 sec
-3.9 mV/min drift +/- 0.0 mV noise
pH= 4.95

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FIRST DERIVATIVE ANALYSIS

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0 dE/dv= -9.5 d2E/dv2= -2.3
 1 dE/dv= -11.9 d2E/dv2= -3.7
 2 dE/dv= -17.1 d2E/dv2= -10.0
 3 dE/dv= -26.4 d2E/dv2= -30.1
 4 dE/dv= -38.3 d2E/dv2= -60.4
 5 dE/dv= -53.7 d2E/dv2= -108.7
 6 dE/dv= -76.5 d2E/dv2= -203.3
 7 dE/dv= -104.8 d2E/dv2= -461.7
 8 dE/dv= -146.1 d2E/dv2= -949.2
 9 dE/dv= -200.2 d2E/dv2= -1488.6
 10 dE/dv= -295.7 d2E/dv2= -1997.9
 11 dE/dv= -401.0 d2E/dv2= -979.0
 12 dE/dv= -394.1 d2E/dv2= 1110.5
 13 dE/dv= -289.4 d2E/dv2= 1568.6
 14 dE/dv= -236.4 d2E/dv2= 1053.8

SAMPLE = 82.99 meq/L
 END POINT VOL= 3.437 mL (121.1 mV)
 (pH 5.45)
 Excess Titre= 0.130 mL

Signal/Noise= 7

