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Potentiometric Titration Application Notes Applications Log # **689A**

Overview Calcium in lactose modified milk powder is determined by KAP analysis (multiple known addition) on 960 (930) Meter. Aliquots of the calcium standard are added automatically to a sample in water. A calcium electrode is used to measure changes in potential after each addition and calculate the sample concentration.

Market	Food and Beverage	***Species Measured***	Calcium
Sample	Milk powder, lactose modified	***Sample Size***	0.025 g
		Typical Concentration	5000 mg/100g
Technique #	2 Multiple Known Addition	***Electrode***	Ca 9320BN; SJ Ref 900100; pH electrode, e.g., 8172BN or 9172BN

Solutions 0.1 M Calcium standard 922006; Calcium ISA 932011; Reference electrode filling solution 900011; Concentrated Nitric acid (15.8 M); pH buffer 4.01 910104 and pH buffer 7.00 910107.

Sample Prep Step 1: Weigh about 0.25 g sample (record exact weight of sample) in a weigh dish. Transfer to a 500 mL volumetric flask partly filled with 0.001 M HNO3. Add 10 mL of the calcium ISA. Fill to the mark with the 0.001 M HNO3 and mix.

Step 2: Pipette 50 mL of the prepared sample into a 150 mL plastic beaker. The sample is ready for analysis. The "sample weight" entered in the method parameters is equal to the weight of the sample added to the 500 mL flask multiplied by 0.1. For example, 0.2501 X 0.1 = 0.02501.

Statistics

# of Trials	5	***Mean***	4962	***%CV***	1.3	***Analysis Time***	~2.5 min

Comments 1. The sample's pH has to be in between 4 to 5 before the analysis. Calibrate the electrode with pH buffer 7.00 and 4.01 dailly. 2. Analyze the prepared sample within a day (24 hours).

3. Check the 960 printout for slope (S) and spike recovery. Best results are obtained when the slope is between 25 to 30 and the spike recovery is 98% or better. 4.Rinse the electrodes, stirrer, and dispenser probe between measurements with DI water.

Method Parameters

Sample Volume/Weight	0.02501 g	***Timed or Stability Readings***	3.0 mV/min
Constant Increment	9.0 mV	***Number of Endpoints***	
Max Titrant Volume	10.0 mL	***Desired Units***	mg/100g
Molecular weight	40.08	***Predose***	0.0 mL
Prestir	10.0 sec	***Additional Parameters***	
Reaction Ratio	1.0		