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Aluminum Determination for Tri-calcium Phosphate (TCP) Anhydrous Powder by Flame Atomic Absorption Spectrophotometer

Y. Yildiz*, M. Dasgupta

Complete Analysis Laboratories, Analytical Research Department, Parsippany, NJ, USA

Email address:

sayatoglu@yahoo.com (Y. Yildiz)

*Corresponding author

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Abstract: Tricalcium Phosphate Anhydrous Powder typically contains 300 to 400 mg/Kg (w/w) of aluminum. This level can be determined utilizing AAS and standard based on known standards. In this study, we have determined the amount of aluminum metal present in sample of Tricalcium Phosphate Anhydrous Powder using Atomic Absorption Spectrophotometer (AAS). The method has high precision and accuracy. The results of aluminum determinations obtained by employment of N_2O-C_2H_2 flame AAS methods. Great care is necessary for preparation of the blank. The need to subtract blank with standard calibration curve this was done automatically. The accuracy was checked with recovery test of aluminum added to the sample before analysis. With employment of AAS method 98.5% and 99.0% of added aluminum was recovered. The detection limit for the determination of Al was 0.5 mg/Kg and results meet the requirements.

Keywords: Tricalcium Phosphate, Aluminum Determination, Flame Atomic Absorption Spectrophotometer

1. Introduction

Tri-calcium phosphate (TCP) is a calcium salt of phosphoric acid with the chemical formula $Ca_3(PO_4)_2$. It is also known as tri-basic calcium phosphate, calcium orthophosphate, tert-calcium phosphate and bone phosphate of lime. Tri-calcium phosphate occurs naturally in several forms, including as a rock, in milk, in the skeletons and teeth of animals. Calcium phosphate rocks have a content of 30% to 40% P_2O_5 in weight. The human body needs phosphorus as well as calcium, and tri-calcium phosphate supplies both. Tri-calcium phosphate is an important raw material for the production of phosphoric acid and fertilizers [1]. It is commonly used in dental powders, and medically as an antiacid or calcium supplement. Aluminum is most abundant metal in earth's crust, being found in many rocks and minerals. Natural and seawater vary widely in aluminum content. Aluminum is recognized as an important toxic substance causing considerable morbidity and mortality, particularly in patients with chronic renal failure. This toxicity of aluminum is governed by its bioavailability [2]. Nevertheless, the excessive ingestion of aluminum can influence negatively the human organism disturbing calcium and phosphate metabolisms and thus damaging the bone system. Moreover, the accumulation of high amounts of aluminum in the brain is associated to Alzheimer disease, senescence symptoms and amnesia of young people [3-4]. Human beings are exposed to aluminum from several sources such as atmospheric air, cosmetics, foods, drinking water and medicines. A lot of papers devoted to the determination of aluminum in environmental samples, food, drugs, human body have appeared in the literature for years [5-6]. Many methods for the assay of aluminum exist including Atomic Absorption Spectrometry (AAS) [7].

2. Materials and Method

Apparatus and Chemicals

Reagents:

Certified 1000 mg/Lin 5% HCl, Atomic Absorption Standard Aluminum Solution, Specture. Alfa Aesar, deionized doubly distilled water, analytical grade

concentrated hydrochloric acid, analytical grade calcium carbonate, and tri-calcium phosphate were used.

Equipment

Perkin Elmer 51000PC Atomic Absorption Spectrophotometer with Aluminum lamp and 2 inch Nitrous Oxide/Acetylene burner head. 1000, 250, 100, 25, 10 and, 5 mL volumetric flasks.5, 10, and 25 mL volumetric cylinders. 5, 10 and 25mLVolumetric pipettes. Analytical balance (accuracy to 0.1 mg).

3. Procedure

3.1. Standard Preparation; 100 mg/Kg Aluminum Stock Solution

Pipet 25 mL of 1000 ppm Certified Aluminum Standard solution into a 250 mL volumetric flask. Add 10 mL of concentrated HCl. Dilute to volume with deionized water and mix well.

3.2. Calcium Matrix Solution, 6000 mg/Kg

For the high range Al test, 75-200 mg/Kg, 1.5 g sample is used in 100 mL, and standard is prepared with nearly 6000 mg/L Ca. The sample concentration here is higher by a factor of about ten times. Accurately weigh 15 g of Calcium Carbonate and transfer to a 1000 mL volumetric flask. Add approximately 100 mL deionized water and swirl to form slurry. Add concentrated HCl dropwise to affect dissolution. Allow the CO_2 escape before subsequent additions of HCl to prevent the solution from bubbling out of the flask. When the calcium carbonate completely dissolves and the solution clears, dilute to volume with deionized water and mix well.

3.3. Stock and Serial Aluminum Standards

Into each of four 100 mL volumetric flask, volumetrically pipet 10 mL of Calcium Matrix Solution. Pipette 1.0, 3.0, 5.0, 7.0, and 10.0 mL of 100 mg/L working solution and 5.0 mL of HCl, dilute to volume with deionized water and mix well.

Table 1. *Series of Al Standards.*

Working Std. Sol (mL)	Dilute to (mL)	Concentration of Al Standards (mg/L)
0	100	0.0
1	100	1.0
3	100	3.0
5	100	5.0
7	100	7.0

3.4. Sample Preparation

Analytically weigh 1.5 g grams of tri-calcium phosphate and transfer to a 100 mL volumetric flask. Add approximately 25 mL of deionized water and swirl to forma slurry. Then add 10 mL of concentrated HCl and swirl to dissolve. Adding acid to dry product can cause coagulation of the product and impede dissolution. Dilute to volume with deionized water and mix well.

3.5. Instrument / Operating Parameters

Wavelength	309.3 nm-UV
Slit width	0.7 nm
Relative sensitivity	1.0 µg/mL
Lamp Energy	67.0
Lamp current	25 mA
Light Source	Hollow Cathode Lamp
Burner head	Nitrous Oxide/Acetylene
Type of Flame	Nitrous Oxide/ Acetylene flame. Reducing (rich, red)
Integration Time	1.5 seconds, 0.5 seconds (for optimizing only)
Average Readings	3

4. Experiments

Perkin-Elmer 5100PC Atomic Absorption Spectrometer was used with a short path burner and nitrous oxide/acetylene flame optimized for highest sensitivity. The instrument was operated with hollow cathode lamp and a single element was measured. The background correction was selectable on an element by element basis. For nitrous oxide-acetylene, carbon build up on burner head may compromise integrity of data for later reading. For this reason, samples should be interspersed with standards. Water has been run between each reading. Perform AA measurements, using blank, 5 and 10 mg/L Al containing a calcium matrix that is equivalent to 1.5 g tricalcium phosphate in100 mL.

Table 2. *Al calibration curve measurements.*

Aluminum (Al) in TCP			
Calibration Data			
[Al] mg/L	AA-BG	R^2	0.9993
0	0.0060	m	0.0028988
1	0.0087	b	0.0060467
3	0.0148		
5	0.0209		
7	0.0267		
10	0.0347		

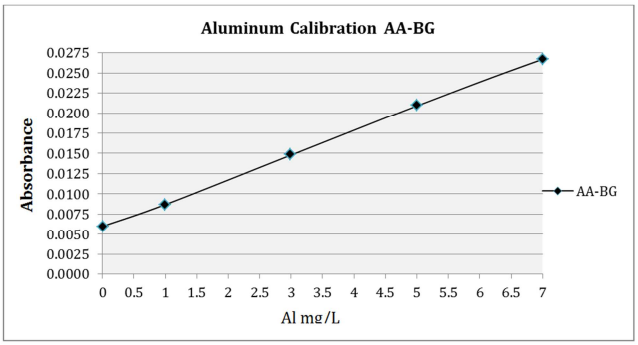

Figure 1. *Calibration curve for aluminum by flame AAS.*

Table 3. *Linear calibration –calculated in Excel.*

LINEAR CALIBRATION—Calculated in Excel							
Sample	**DF**	**Absorbance**	**Blank-correction Abs**	**[Al] mg/L**	**W (g)**	**V(mL)**	**mg/kg**
Water		-0.0004					
0 mg/L	1	0.0048	0.0054	-0.22			
Water		-0.0008					
1 mg/L	1	0.0076	0.0086	0.88			
Water		-0.0012					
3 mg/L	1	0.0133	0.0148	3.00			
Water		-0.0017					
5 mg/L	1	0.0191	0.0209	5.11			
Water		-0.0018					
7 mg/L	1	0.0245	0.0267	7.12			
Water		-0.0026					
TCP Sample	1	0.0044	0.0073	0.43	1.0002	10	4.32
Water		-0.0032					
TCP Sample dup	1	0.0039	0.0073	0.43	1.0019	10	4.32
Water		-0.0036					
10 mg/L	1	0.0306	0.0347	9.87			
Water		-0.0043					

Table 4. *QA / QC Study.*

ml of sample spike (ml)	**Concentration of spike (mg/L)**	**Final volume of sample (ml)**	**Concentration of spike mg/ml (ppm/1000)**	**Final volume of sample L (ml/1000)**	**Spike Concentration (mg absolute)**	**Spike Concentration (mg/L)**	
2.00	10.00	10.0	0.01000	0.01000	0.020	2.000	
Sample Result							
LCS Result (mg/L)	QC Sample Info:						
	QC Sample Final Volume (ml)	QC Sample Result (mg/L)	QC Sample Result mg absolute (mg)	MS Result (mg/L)	MSD Result (mg/L		
100.0000	10	4.32000	0.0432	6.29000	6.30000		
	Spike Results						
		ml of spike (ml)	Concentration of spike (mg/ml)	Final volume of sample (ml)	True value of spike (mg)	Concentration in sample (mg/L)	Absolute wt. in sample (mg)
	LCS	1.000	1000	100	1000.0	100.0	10.0
	MS	2.00	0.01	10.0	0.0200	6.29	0.0629
	MSD	2.00	0.01	10.0	0.0200	6.30	0.0630

5. Result and Discussion

Determined the concentration (ppm) of aluminum in the working samples using a curve fitting program; a working curve of absorbance vs. concentration (ppm). The working sample concentration has been calculated in mg/Kg (Table 4).

The results of a single method for the detection of aluminum in the tricalcium phosphate were obtained and are shown in Figure 1 and Table 2 and 3. Calibration curve with matrix matched standards, sample's single absorbance data point was interpreted in terms of standard's slope and intercept. The need to subtract blank with standard calibration curve this was done automatically. The analysis show good linearity (R^2=0.999) (Table 4), precision and accuracy (RSD <0.5%); recovery for spiked and spiked duplicate (R=98.5% and 99.0 %) and the method detection limit was 0.5 mg/Kg and results meet the requirements.

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