ISSN: 2230-9926

Available online at http://www.journalijdr.com

IJDR

International Journal of Development Research
Vol. 5, Issue, 10, pp. 5704-5706, October, 2015

International Journal of
DEVELOPMENT RESEARCH

Full Length Research Article

DETERMINATION OF FLUORIDE IN TRICALCIUM PHOSPHATE (TCP) BY ION-SELECTIVE ELECTRODE

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ARTICLE INFO

Article History:
Received 19th July, 2015
Received in revised form
29th August, 2015
Accepted 16th September, 2015
Published online 31st October, 2015

Key Words:

Fluoride, Tri-calcium phosphate,
Ion-selective electrode.

ABSTRACT

Fluoride plays as much more important role in metabolic process. Fluoride ion concentration in tri-calcium phosphate above 5.0 mg/100 g creates health hazards. Many methods have been devised for the determination of fluorine. In this study, Ion-Selective Electrode Method has been used for the determination of fluoride in tri-basic calcium phosphate. The pKa for HF was 3.17, and buffer was 5.32, 99.0 % of fluorine was present as F- in the solution of TCP. Method range was 0.1 to more than 10 mg/L without dilution. The result shows that the concentration of fluoride in TCP was less than 0.005 %.

INTRODUCTION

Tricalcium phosphate (TCP) is a calcium salt of phosphoric acid with the chemical formula $Ca_3(PO)_4$. 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 tricalcium 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. An Ion-Selective Electrode (ISE) method for determination fluoride was widely used in industries processes.

Apparatus and Chemicals

Electrode and meter

The following apparatus were used for this study: Fluoride selective electrode, solution dispenser and, Hanna 2215/ISE NH35 meter with 0.1 mV resolution.

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The Fluoride Ion-Selective Electrode has a solid-state mono-crystalline membrane. The electrode is designed for the detection of fluoride ions (F^-) in aqueous solutions and is suitable for use in both field any laboratory applications. Optimum pH range is 4 to 8, temperature range is 0 to 80 ^oC. The fluoride ion-selective electrode measures quick and accurately fluoride ion activity in aqueous solution (2).

Reagents

Fluoride stock standard solution: Dissolve 221.0 mg sodium fluoride (NaF, GFS reagent ACS, min 99.0 % or equivalent, CAS # 7681-49-4, previously dried by heating in a platinum crucible at a low red heat; and cooled in a desiccator) in distilled water and diluted to 1000 mL; 1mL= 100 µg F^- (Table 1).

Fluoride standard solution: 10 mL of stock solution diluted to 100 mL with distilled water; 1 mL=10 µgF^-.

Series of fluoride standards: Into 100 mL volumetric flasks, put V(mL) of 10 mg/L fluoride standard, quantitatively fill with D.I.Water, to obtained series standards, mg F^-/L (Table 2).

All fluoride standard solutions should be stored in high density polyethylene bottles at 4 ^oC (3), (4).

Table 1. Preparation of fluoride stock standard solution

Stock std.	0.11050	m NaF (g)	Na	22.9890
Volume	0.5000	V L	F	18.9980
Conc.	100	[F-] mg/L	F/NaF	0.4525
Std.	10		NaF	41.9970

Table 2. Series of fluoride standards

Working Std.Sol (mL)	Dilute to (mL)	Concentration of Standards (mgF-/L)
2	100	0.2
5	100	0.5
10	100	1.0
20	100	2.0
40	100	4.0

Total ionic strength buffer: This solution marketed commercially under the trade name TISAB (5). Sufficient buffer for 15 to 20 determinations can be prepared by mixing with stirring 57 mL of glacial acetic acid, 58 g of NaCl, 4 gram of cyclohexylaminedinitrilotetraacetic acid, and 500 mL of distilled water in a 1-L beaker. Cool the contents in a water or ice bath, and carefully add 6 M NaOH to a pH of 5.0 to 5.5. Dilute to 1 L with water, and store in a plastic bottle (6).

Analytical Procedure

Transfer 2.0 g of tricalcium phosphate [$Ca_3(PO4)_2$, Sigma-Aldrich, 99.0+%, CAS # 7758-87- 4] into a teflon beaker containing a plastic-coated stirring bar, add 20 mL of distilled water and 2.0 mL of hydrochloric acid, and stir until fully dissolved. Add 50.0 mL of *Buffer Solution* and sufficient water to make 100 mL of a *Test Solution* (7).

Treatment of Standards and Sample: Use 30 mL pyrex beaker with small stirring bar (dimensions: 12 mmx5 mmx5mm). Pipet 10 mL of sample or standard into beaker; pipet 10 mL TISAB (total ion strength adjustment buffer) into same beaker.

Selective electrode measurement: Rinse and dry the electrodes, immerse in each of the fluoride standard solutions and sample solutions. Begin magnetic stirring, using same setting for each reading (stir quickly, no bubbling or splashing). Monitor potential until reading is stable for 3 minutes. Record potential (mV) (1) (Table 3) and (Table 4).

RESULTS AND DISCUSSION

Before use, the electrode has been calibrated by measuring a series of known standards solutions, made by serial dilution of the 1000 ppm standard solution. For a full calibration, prepared 100 mL of solutions containing 0.2, 0.5, 1.0, 2.0, and 4.0 ppm [F^-] (8).

Table 3. Calibration curve measurements

Series of Std	[F^-] mg/L	Log [F^-]	E /mV	
0.2	0.200	-0.6990	88.1	
0.5	0.500	-0.3010	66.4	
1	1.000	0.0000	49.6	
2	2.000	0.3010	33.4	
4	4.000	0.6020	16.8	
		RSQ:	R^2	1.0000
		dE/dlog[F^-] :	m	-54.8040
		E for [F^-]=1 :	b	49.7970

Table 4. Ion-Selective Electrode measurements

Identity	E / mV	[F^-] mg/L	DF	Corr [F^-]
2.0 mg/L indep	33.3	2.000	1	2.000
0.02 mg/L check	88.2	0.199	1	0.199
Sample	154.4	0.012	1	0.012
Sample-dup	154.0	0.013	1	0.013
Sample spike	49.9	0.996	1	0.996
Sample spike-dup	49.0	0.998	1	0.998
Water	175.4	0.005	1	0.005

Average value (mgF^-/L): 0.0125

Sample calculations: F $^-$ (mg/Kg) = 0.012 mg/L x 0.1L / 0.0020022 Kg = 0.599 mg/Kg *Duplicate*; F $^-$ (mg/Kg) = 0.013 mg/L x 0.1 L /0.0020016 Kg = 0.649 mg/Kg *Precision check*: 0.012+0.013/2 = 0.0125 mg/L %RSD= 0.0125-0.012/0.0125 x 100 = 4.0 %

Spike Recovery Check (accuracy): Calibration curve was obtained as E (mV) vs log[F $^-$]. Where, [F $^-$] is mg/L and log is base 10. Slope m is dE/dlog[F $^-$], ie the usual y=mx+b, and intercept b is value of E for [F $^-$] =1 mg/L, ie y intercept for x=0. Expect R^2 to be 0.999 or greater; 1.0000 obtained (Table 4), (Figure1).

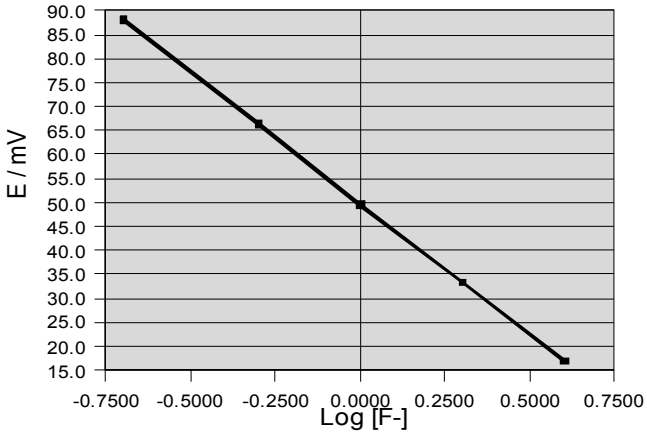

Figure 1. Calibration curve of fluoride

- Volume of spiking solution: 0.1 mL
- Sample volume: 10 mL
- Concentration of spiking solution: 100 mg F $^-$/L
- Spiked amount: 1.0 mg/L
- True value: 0.01 mg

Conclusions

The Ion-Selective Electrode Methods has been used for determining fluoride in the tricalcium phosphate(TCP). The results have been displayed as ppm(mg/L), and mol/L in the solution. The recovery of fluoride content in tricalcium phosphate was lower than the permissible limits; < 5.0 mg/100 g. % RSD was 4.0%, % R for spiked was 98.4 %, spiked duplicate was 98.6%.

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