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Oxidative determination of diuretics drugs by using pyridinium fluoro chromate (PFC) reagent

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Abstract
In present paper determination of Quantitative oxidation of some diuretics drugs like Frusemide, acetazolamide etc. by using simple titrimetric method with pyridinium fluoro chromate PFC reagent. A diuretics is any substance that promotes diuresis, the increased production of urine. It is also called water pills, are medication designed to increase the amount of water and salt expelled from the body as urine. Most of the sodium, chloride and water are reabsorbed into the blood before the filtered fluid leaves the kidney in the form of urine. Diuretics are a class of drugs that increase the flow of urine.
The proposed method was accepted for frusemide determination in pharmaceutical sample. Due to simplicity and convenience the titrimetric method has widely been used for determination of many organic as well as medicinal compound. Frusemide is been used as a strong diuretic available under brand name Lasix, It has been used in the treatment of edema associate with renal impairment, hypertension, heart failure.

Keywords: quantitative oxidation, titrimetric method, frusemide, acetazolamide, pyridinium flouro chromate PFC reagent, CV, SD, recovery %

Introduction
In Analytical analysis uses instrument and methods used to separate, identify and quantify matter. Titration is very easy and simple common laboratory method of Quantitative chemical analysis that is used to determine the unknown concentration of a known reagent.
Because volume measurement play a key role in Titration. It is also known as volumetric analysis.
Titrimetric analysis consist in determining the number of mole of reagent (titrant) required to react quantitatively with the substance (given). In the healthy subject the amount and content of the urine is automatically adjusted to maintain the water and electrolytic status of the body at an almost constant level. Diuretics sometime called water pills, help rid your body of salt (sodium) and water.
Most work by making your kidneys release more sodium into your urine. The sodium takes water with if from your blood. Frusemide has poor aqueous solubility and its efficacy is due to the ability to inhibit NaCl/2Cl transporter in the ascending limb of loop of henle. Its also used to treat for high blood Pressure and other condition. The determination of diuretic drug by using simple titrimetric method by using pyridinium chlorochromate reagent. These drug in pure form and in their pharmaceutical preparation such as Lasix. Acetazolamide, Diamox with PFC reagent.
In PFC chromium (VI) behave as a good oxidizing agent PFC reactive species in the reaction process. Its react with diuretics and Observed that lower concentration and volume give less recovery because of insufficient reagent higher concentration and volume give In accurate result. Due to simplicity and convenience the titrimetric method has widely been used for determination of many organic as well as medicinal compound. PFC is good oxidizing agent of chromium (VI) and widely used as an oxidant for organic compound during reaction it was noted that the excipient present in pharmaceutical preparation do not interfere. PFC have additional advantages in thermal stability, versatility, controlled acidity and selectivity of operational simplicity capability of functioning well. Under mild condition in this method. Value percentage error, SD, CV, recovery, obtained by standard drug addition. PFC have a great variety of compound containing Cr (VI) have been proved to be versatile reagents capable of oxidizing. Almost every oxidizable organic functional group in diuretics structure of compound it is assumed that the sulphur present in ring is oxidized into sulfoxide derivative. In titrimetric analysis reaction must be stoichiometric.
Acetazolamide, trade name Diamox among other, is a medication used to treat glaucoma, epilepsy, altitude sickness, periodic paralysis, hypertension, heart failure and hydrochlorothiazide is a often used to treat high pressure swelling due to fluid build up and also helps prevent strokes, heart attacks and kidney problems.it is an a oral medication. Its belong to thiazide class of diuretics. It reduces blood volume by acting on the kidney to reduce sodium (Na+) reabsorbing in the distal convoluted tubule. The energy for this is provided by a sodium potassium ATpases on the basolateral interstitium via the sodium potassium ATpase causing an increase in the osmolarity of the interstitium, thereby establishing an osmotic gradient for water reabsorption.

Requirements to chemical reaction used in titrimetric method of analysis
1. Reaction between reagent and analyte must be specific. Titrant cannot react with impurities or addition of the analytical solution.
2. Reaction must be stoichiometric.

3. Titrant must react rapidly with the analyte so that the time required between addition of reagent is minimised.
4. Titrant must react more or less completely with analyte so that satisfactory end point are realised.
5. Undergo a selective reaction with the analyte that can be described by simple balanced reaction.
6. In this method the amount of a titrant used to reach the end point correspond to the weight of species to be determined.

Experimental: preparation of reagent and solution

Pyridinium fluoro chromate - solution 0.03N: 495 mg of pyridinium fluorochromate was dissolved in 150 ml of glacial acetic acid and made Up to the volume with 250 ml distilled water in volumetric flask. The solution was standardised Iodometrically.

Sodium thiosulphate solution (0.01) N: solution of prepare thiosulphate was prepared by Dissolving 2.4819gm in 1000ml distilled water and standardization by 0,01N potassium dichromatic Solution iodometrically.

potassium dichromate solution (0.01N: Standardisation of sodium thiosulphate against Potassium dichromate solution calculate precision normality (molarity) of sodium thiosulphate standard solution accordance to equivalents law.

Potassium iodide solution: 10% W/V aqueous Solution was prepared in distilled water.

Starch solution: 1% W/V aqueous solution of starch was Prepared in distilled water.

Sample solution: tablet solution: the powder equivalent to 100mg of sample was taken in 100ml.

Calibrated volumetric flask and dissolved in minimum amount of distilled water.

Injection solution: 1mg/ml

General procedure: aliquot containing 1mg of The sample were taken in 100ml in conical flask and 5 ml of 0.03 N (PFC) and 10 ml of 5N H2SO4 was added to it. The reaction mixture shaken and allowed to react required reaction time at room temp. for 15 minute. after reaction was over 5ml of 10% K I (sol) was added to it and shaken and Stand for 1 min. Liberated iodine was titrated with 0.01N sodium thiosulphate using starch as indicator. A blank experiment was also run Under identical condition using all the reagents except sample. The amount of the sample recovered was calculated by the difference in titre value of sodium thioSulphate solution for blank and actual experiment.

Calculation

Mg of sample = M * N (B-S)/n Molecular weight of sample (M), Normality (N) Volume of thiosulphate sol for blank.(B), volume of thiosulphate sol with sample. Stoichiometry of Reaction (n). Standard deviation (SD), coefficient of variation and recovery%. Experiment were carried out by Standard drug addition method.

Result

Determination of stoichiometry of Diuretics Drug by using pyridinium fluoro chromate (PFC)

Table 1

s.no.	sample	Aliquote taken (ml)	Amount taken (mg)	Reaction Time (minute)	molecularity	Amount obtained	Error%	SD	CV
1	Frusemide (Pure)	1	0.960	50	2	0.952	-0.83	0.0026	0.2710
		3	2.880	50	2	2.860	-0.69	0.0045	0.0159
		5	4.810	50	2	4.790	-0.41	0.0013	0.0270
1	Lasix	1	0.956	50	2	0.950	-0.83	0.0036	0.3870
		3	2.870	50	2	2.850	-0.69	0.0021	0.0765
		5	4.790	50	2	4.765	-0.52	0.0021	0.0449
2	Acetazolamide (pure)	1	0.995	50	2	0.980	-1.05	0.0027	0.2840
		3	2.998	50	2	2.870	-0.93	0.0026	0.0940
		5	4.990	50	2	4.968	-0.44	0.0029	0.0600
(2)	Diamox	1	0.943	50	2	0.935	-0.84	0.0017	0.1900
		3	2.835	50	2	2.814	-0.74	0.0033	0.1174
		5	4.725	50	2	4.700	-0.52	0.0042	0.0090

- **Average of nine determination**

Recovery studies of Diuretics drug (Frusemide) by standard drug addition method

Table 2

S.N.	Number of observations (N)	Amount present (pure) (mg)	Amount of drug added (mg) X	Total amount of drug obtained by calculation (mg)	Amount of drug obtained by calculation (mg) Y	XY	X^2	Recovery %
1	3	0.960	0.983	1.954	0.990	0.973	0.966	99.33%
2	3	0.960	1.965	2.932	1.971	3.873	3.861	
3	3	0.960	2.954	3.833	2.865	8.463	8.726	
4	3	0.960	3.963	4.934	3.976	15.756	15.705	
	∑N=12		∑X=9.865		∑Y=9.802	∑XY=29.065	$\sum X^2$ =29.258	

Recovery studies of Diuretics drug (frusemide) by standard drug addition method

Table 3

S.N.	Number of observations (N)	Amount present (pure) (mg)	Amount of drug added (mg) X	Total amount of drug obtained by calculation (mg)	Amount of drug obtained by calculation (mg) Y	XY	X^2	Recovery %
1	3	0.995	0.978	1.963	0.970	0.948	0.956	99.49%
2	3	0.995	1.972	2.985	1.984	3.912	3.888	
3	3	0.995	2.980	3.945	2.950	8.791	8.880	
4	3	0.995	3.975	4.960	3.957	15.729	15.800	
	∑N=12		∑X=9.905		∑Y=9.861	∑XY=29.380	$\sum X^2$ =29.524	

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