International Journal of Advanced Science and Research
ISSN: 2455-4227
Impact Factor: RJIF 5.12
www.allsciencejournal.com
Volume 3; Issue 2; March 2018; Page No. 97-99

Semi micro analysis of some sulpha drugs by using Pyridinium Flouro Chromate (PFC) reagent

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Abstract
In present paper we have reported Quantitative oxidation of some sulpha drug like sulphamexthazole, sulphacetamida, and diuretics, acetazolamide, furosemide, hydrochloroth-izide, indapamide and in polyhydrixide compound mannitol. By using simple titrimetric mehod determination with (PFC) pyridinium fluoro chromate reagent. These drug in pure form and in their pharmaceutical preparation such as Lasix(T),Sulfuno(T), Amide(D), Diamox(T) -Aquazide(T), Hydride(T), Lorvas(T), Siptol(Inj), with (PFC) reagent. PFC it is good oxidizing agent of chromium (VI) and widely used as an oxidant for organic compound during reaction it was noted that tha exipients present in pharmaceutical preparation do not interfere. PFC having additional advantages in thermal stability versatileity, controlled acidity and selectivity of operational simplycity capability of functioning well under mild condition in this method value percentage error, SD (standard deviation)(CV (coefficient of variation prove the method to be precise and recovery experiment obtained by standard drug addition method. sulpha drugs like sulphacetamide contain amino group present at para position is highly succeptible for oxidation even been mild oxidizing agent get oxidisied to –NO2 nitro group Same in atom sulpha-methaxazole, case.. but in diuretics structure of compound it is assumed that the sulphur present in ring is oxidized into sulphoxide derivative.

Keywords: quantitative oxidation, titrimetric method, Pyridinium flouro chromate PFC reagent, CV, SD, recovery%

Introduction
In Analytical Chemistry Quantitative Oxidation: Chemical method of analysis are titrimatric and voloumetry which depends upon the quantitative chemical reaction. Titrimatic analysis consist in determining the number of mole of reagent (titrant) required to react quantitatively with the substance (given). In PFC chromium (VI) behave as a good oxidizing agent PFC reactive species in the reaction process. Its react with sulpha drugs and diuretics and Ob-served that lower concentration and volume give less recovery because of insufficient reagent higher concentration and voloume give In accurate result. Sulpha drugs like sulphacetamide sodium Locula (D), Opthocid (D), Ocu-sulpha (D), Albucid (D) Sulfamethoxazole Antrim (T), Sepmax (T), Septran (T) (Bactrim (T) Ciplin DS (T**)** Sulphaguanidine, Aterian (T) Diacta (T) contain amino group present at para position is highly succeptible for oxidation even been mild oxidising agent get oxidised to -NO2 nitro group.
Sulpha drug are compound that contain a sulphonamide moeity (SO2, NH2) that vary in structure well as clinical use. Sulpha drug are use in treatment of Urinary tract infection and few other disorder such as nocardiosis. Its also known as antibacterial. titration also known as titrimetry is common lab method of quantitative chemical analysis that is used to determine the unknown concetration of an unknown analyte. since voloume and measurment play a key role in titration. It is also known as volumetric analysis.

Requirements to chemical reaction used in titrimetric method of analysis
1. Reaction between reagent and analyte must be specific. Titrant can not 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 discribe 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 standardized Iodometrically.

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

Potassium dichromate solution (0.01)N: Standardisation of sodium thiosulphate against Pota-Ssium dichromate solution calculate precision normality (molarity) of sodium thiosulphate standa-rd 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 100 mg 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. Librated 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), Normility (N) Volume of thiosulphate sol for blank. (B), volume of thiosulphate sol with sample. Stoichiometry of Reaction (n). Standard deviation (SD), cofficent of variation and recovery %. Experiment were carried out byStandard drug addiction method.

Result

Table 1: Determination of stichiometry of sulpha drug by using pyridinium fluoro chromate (PFC)

S. No	Sample	Aliqute taken (ml)	Amount taken (mg)	Reaction Time (minute)	Molecularity	Amount obtained	Error%	SD	CV
1	Sulphacet Amide Sodium (Pure)	1	0.996	10	2	0.986	1.00	0.0027	0.2820
		3	2.996	10	2	2.974	0.73	0.0027	0.0027
		5	4.994	10	2	4.968	0.52	0.0029	0.0600
1	Opthasulpha (D)	1	0.960	10	2	0.950	1.04	0.0019	0.2095
		3	2.886	10	2	2.863	0.79	0.0021	0.0021
		5	4.817	10	2	4.793	0.49	0.0025	0.0540
2	Locula (D)	1	0.969	10	2	0.959	1.03	0.0027	0.2892
		3	2.902	10	2	2.880	0.75	0.0017	0.0017
		5	4.841	10	2	4.819	0.45	0.0029	0.0618
(2)	Sulphamethoxazole (pure)	1	0.995	10	2	0.984	1.10	0.0017	0.1720
		3	2.990	10	2	2.974	0.53	0.0022	0.0022
		5	4.982	10	2	4.958	0.48	0.0029	0.0596
1	Sepmax (T)	1	0.955	10	2	0.941	1.46	0.0025	0.2740
		3	2.860	10	2	2.840	0.69	0.0029	0.0029
		5	4.782	10	2	4.757	0.52	0.0027	0.0578
2	Bactrim (T)	1	0.958	10	2	0.948	1.04	0.0034	0.3690
		3	2.867	10	2	2.848	0.66	0.0028	0.0028
		5	4.781	10	2	4.752	0.61	0.0029	0.0620
3	Ciplin (T)	1	0.957	10	2	0.947	1.00	0.0035	0.3700
		3	2.865	10	2	2.848	0.59	0.0027	0.0027
		5	4.779	10	2	4.755.	0.50	0.0029	0.0621
(3)	Sulphaguanidine (pure)	1	0.992	15	2	0.984	0.80	0.0023	0.2341
		3	2.986	15	2	2.969	0.56	0.0018	0.0018
		5	4.982	15	2	4.957	0.50	0.0035	0.0708
1	Sulphaguanidine (T)	1	0.986	15	2	0.975	1.11	0.0033	0.3391
		3	2.961	15	2	2.938	0.77	0.0026	0.0886
		5	4.940	15	2	4.915	0.50	0.0030	0.0630

Average of nine determination

Table 2: Recovery studies of sulphacet amide sodium by standard drug addition method

S. No	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	0.996	0.970	1.960	0.960	0.931	0.940	99.24%
2	3	0.996	1.930	2.900	1.900	3.667	3.724	
3	3	0.996	2.885	3.870	2.871	8.282	8.323	
4	3	0.996	3.860	4.810	3.831	14.787	14.899	
	∑N=12		∑X=9.645		∑Y=9.562	∑XY=27.667	$\sum X^2 = 27.886$	

Table 3: Recovery studies of sulfamethoxazole by standard drug addition method

S. No	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	0.965	1.950	0.955	0.921	0.931	99.25%
2	3	0.995	1.920	2.910	1.910	3.667	3.686	
3	3	0.995	2.882	3.861	2.861	8.245	8.305	
4	3	0.995	3.845	4.800	3.815	14.668	14.784	
	∑N=12		∑X=9.612		∑Y=9.541	∑XY=27.501	$\sum X^2$ =27.706	

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