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A Non aqueous Titration Assay of Sodium in Sodium Carboxy methyl cellulose (CMC) as per USP/NF

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A B S T R A C T
Cellulose is a major constituent of many foods of plant origin. Carboxy methyl cellulose sodium is the sodium salt of poly carboxy methyl ether of cellulose. In this study, sodium carboxy methyl-cellulose has been titrated with 0.1N perchloric acid VS and determining the percent sodium. CMC contains not less than 6.5% and not more not more than 9.5% of sodium (Na), calculated on the dried basis (1).
Keywords: Sodium carboxy methyl cellulose, Cellulose, Non aqueous Titration

A R T I C L E I N F O

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1. Introduction

Carboxy methyl cellulose (CMC) is a polymer derived from natural cellulose that has been routinely used for many years now as a food additive (INS 466) in products such as ice creams and pre-cooked meals (2), to give them smoothness. CMC is widely used in personal care, detergents, mining flotation, paper making, oil drilling, medicines, liquid drinks, and Textile in the world because of its important properties such as high viscosity, water

preservation, good acid resistance and salt resistances (3). Sodium carboxy methyl cellulose also known as sodium cellulose glycolate, INS 466, sodium CMC, cellulose gum, Na-CMC, and sodium salt of carboxy methyl ether of cellulose. Prepared from cellulose by treatment with alkali and monochloro-acetic acid or its sodium salt. White or slightly yellowish, almost odorless hygroscopic granules, powder or fine fibres. Chemical formula is $[C_6H_7O_2(OH)_x(OCH_2COONa)_y]_n$

Where;

n is the degree of polymerization

x = 1.50 to 2.80

y = 0.2 to 1.50

x + y = 3.0

(y = degree of substitution)

Where; R = H or CH2COONa

Average M_w ~700,000

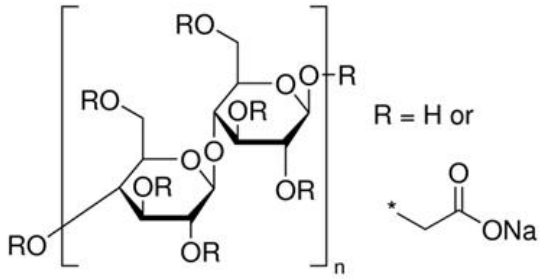

Figure 1: Structural formula of Sodium Carboxy methyl cellulose

2. Material and Equipment

pH meter, Analytical balance, Timer, buret and electrode assembly, Electrode Type: Redox

RH 28 Solution (for redox electrode calibration). Specification220 ± 10@ 25^{0}C

Sodium carboxy methyl cellulose; Sigma-Aldrich, CAS Number: 9004-32-4, Acetic acid, glacial; ACS Reagent Grade, Pharmco-Aaper Cemicals, 99.7 %, Lot Number: PB006069AAG, 0.1 M Silver Nitrate Volumetric Solution, Analytical Reagent. Sigma-Aldrich, CAS # 7761-88-8, Perchloric acid; Sigma-Aldrich, ACS reagent, 70 %. CAS Number: 7601-90-3

Preparation of Tenth-Normal (0.1N) Perchloric Acid

Mix 8.5 mL of perchloric acid (70 %) with 500 mL of glacial acetic acid and 21 mL of acetic anhydride. Cool the solution to room temperature, and add glacial acetic acid to make 1000 mL. Allow the prepared solution to stand for 1 day for the excess acetic anhydride to be combined, and determine the water content. If the water content exceeds 0.5 % add more acetic anhydride. If the solution contains no titratable water, add sufficient water to obtain content to between 0.02% and 0.5% of water. Allow the solution to stand for 1 day,and again titrate the water content. The solution so obtained contains between 0.02% and 0.5%of water, indicating freedom from acetic anhydride (1).

Standardization of Tenth-Normal (0.1N) Perchloric Acid

Accurately weigh about 700 mg of potassium biphthalate, previously crushed lightly and dried at 120 ^{0}C for2 hours, and dissolve itin 50mL of glacialaceticacid ina 250-mL flask. Add2 drops of *crystal violet TS*, and titrate with the perchloric acid solution until the violet color changes to blue-green. Deduct the volume of the perchloric acid consumed by 50 mL of the glacial acetic acid. Each 20.423 mg of potassium biphthalate is equivalent to 1 mL of 0.1N perchloric acid (1).

3. Experimental

Non aqueous Titrations:

Non aqueous titration is the titration of substances dissolved in a solvent other than water. It is the common titrimetric procedure used in pharmacopoeial assays. The non aqueous titration of weak bases with perchloric acid is the part of non-aqueous titrimetry. Acetic acid is the solvent most commonly used in the nonaqueous titration of bases, and it should have a water content not exceeding 0.1 to 0.2 percent. Perchloric acid solution (usually 0.1 N) is prepared from 70 percent which is diluted with acetic acid. Acetic anhydride is added *after the dilution* to eliminate the water (4).

Assay Procedure:

Transfer to a beaker about 500 mg Carboxy methyl cellulose Sodium, accurately weighed, add 80 mL of glacial acetic acid, heat the mixture on a boiling water-bath for 2 hr. Cool to room temperature, and titrate with 0.1N Perchloric Acid VS, determining the end point potentiometrically. Each mL of 0.1N perchloric acid is equivalent to 2.299 mg of Na (1).

4. Results and Discussion

The apparatus, reagents, and procedure used in this work for the non aqueous titrations were essentially the same as described. 0.1 N perchloric acid VS was prepared and has been standardized. The actual normality was 0.09647. Operation of Redox electrodes has been checked using standard Redox calibration solution. Loss on drying (LOD) has been determined for the sodium carboxy methyl cellulose and calculated as % water. Inflection point is obtained from the first differential plot were the end point is the volume at the peak maximum (Figure 3).

$$\%\ \text{of Sodium} = \frac{\text{Volume Titrant (mL)} \times 2.299\ \text{mg/mL} \times F \times 100}{\text{sample weight(mg)}} \frac{100}{(100 - \%water)} \quad (1)$$

$$F = \frac{\text{Normality of Perchloric acid}}{0.1} = \frac{0.09647}{0.1} = 0.9647\ \text{N}$$

F= Normality factor

$$\% \text{ of Sodium} = \frac{17.25\text{mL} \times 2.299\text{mg/mL} \times 0.9647 \times 100}{501.3\text{ mg}} \times \frac{100}{(100-8.32)} = 8.32\ \%$$

$$\% \text{ of Sodium} = \frac{17.25\text{mL} \times 2.299\text{mg/mL} \times 0.9647 \times 100}{500.2\text{ mg}} \times \frac{100}{(100-8.32)} = 8.33\ \%$$

Table 1: Results for Sodium Assay

Weight mg	Volume of Titrant mL			Corrected Volume (mL)	% Assay	Average % Assay	Conforms (Y/N)
	Start	End	Total				
501.3	0.00	17.25	17.25	N/A	8.32		
500.2	0.00	17.25	17.25	N/A	8.34	8.3	Yes

Table 2: Potentiometric Titration Data Template

vol. of titrant	Response mV reading	1st ave.vol of titrant	dE/dV	2nd avevol of titrant	d(dE/dV)
0.0	496				
1.0	497	0.5	1		
2.0	498	1.5	1	1	0
3.0	501	2.5	3	2	2
4.0	504	3.5	3	3	0
5.0	506	4.5	2	4	-1
6.0	510	5.5	4	5	2
7.0	512	6.5	2	6	-2
8.0	516	7.5	4	7	2
10.0	524	9.0	4	8.25	0
12.0	527	11.0	1.5	10	-2.5
13.0	538	12.5	11	11.75	9.5
14.0	541	13.5	3	13	-8
15.0	545	14.5	4	14	1
16.0	551	15.5	6	15	2
16.5	555	16.25	8	15.875	2
17.0	571	16.75	32	16.5	24
17.5	638	**17.25**	134	17	102
18.0	660	17.75	44	17.5	-90
19.0	661	18.5	1	18.125	-43
20.0	662	19.5	1	19	0

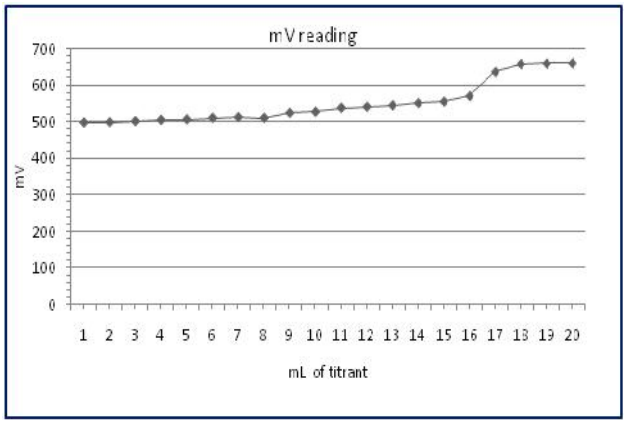

Figure 2: Volume of titrant and mV reading

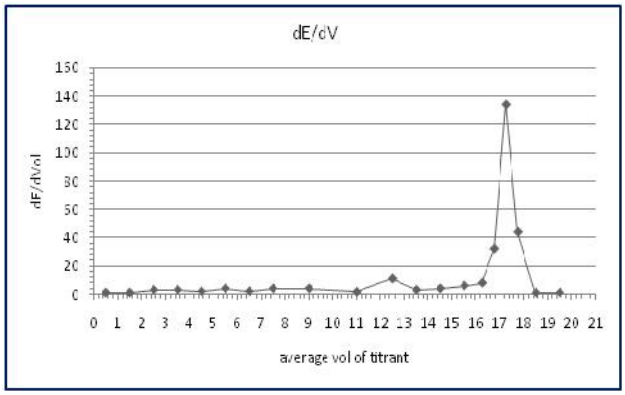

Figure 3: First differential plot and average volume of titrant

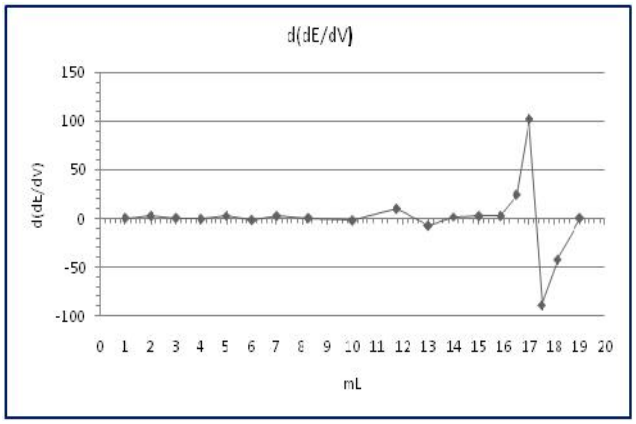

Figure 4: Second differential plot and volume of titrant

5. Conclusion

A non aqueous titration method has been used for determining percent sodium in carboxy methyl cellulose, following preliminary heating with glacial acetic acid, with 0.1 N perchloric acid VS. The endpoint has been determined potentiometrically. End-point of the titration corresponds to the maximum value of dE/dV (where E is electro motive force and V is the volume) in a potentiometric titration of the substance. Inflection point is 17.25 (Table 2, Figure: 3, and Figure: 4).The results meet the main specifications (5).

6. References

[1] United States Pharmacopeia 38 official Monographs.

[2] Regulation (CE) No 133/2008 of the 16th of December, 2008, concerning food additives. SIN & CMC Sinocmc Co; Ltd.

[3] Acid-Base Titrations in Non aqueous Solvents. James S. Fritz, Iowa State College. Ames, Iowa. G. Frederick Smith Chemical Company, Columbus, Ohio. 1952.

[4] Code of Federal Regulations of the Food and Drug Administration (FDA) Part21CF182. 1745 (GRAS).