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DETERMINATION OF ASPIRIN CONTENT IN PHARMACEUTICAL TABLETS BY CONDUCTOMETRIC TITRATION

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Abstract

Objective: The aim of this paper is to determine the quantitative content of acetylsalicylic acid (aspirin) in pharmaceutical tablets using conductometric titration as a simple, accurate, and efficient alternative to traditional analytical methods.
Methodology: The methodology involved dissolving the tablet sample in distilled water and titrating it with a standard sodium hydroxide (NaOH) solution, while monitoring the changes in conductivity during the titration process. The equivalence point was identified from the graph of conductivity versus titrant volume.
Results and Discussion: The results and discussion demonstrated a strong correlation between the labeled amount of aspirin in the pharmaceutical formulation and the experimentally determined value. The method showed high reproducibility, with low standard deviations and satisfactory precision. *Conclusions:* The conclusion of this research is that conductometric titration is a simple, fast, and reliable method for the quantitative analysis of aspirin in pharmaceutical products, and it is suitable for use in both pharmaceutical quality control and educational laboratory settings.

Keywords: Aspirin, Conductometric Titration, Acetylsalicylic Acid, Pharmaceutical Analysis, Quantitative Determination.

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1. INTRODUCTION

Acetylsalicylic acid (ASA), better known as aspirin, is one of the most widely used drugs globally due to its analgesic, antipyretic, and anti-inflammatory properties. Since its first synthesis by Bayer in the late 19th century, aspirin has held an important place in modern medicine for treating pain, fever, and inflammation, as well as in low doses for the prevention of cardiovascular diseases [1,2]. Given that aspirin is found in many pharmaceutical formulations on the market, quality control and accurate determination of its content is essential to ensure therapeutic efficacy and safety in clinical use [3]. Traditional methods for aspirin analysis include various techniques such as UV-Vis spectrophotometry, high-performance liquid chromatography (HPLC), and potentiometric titration, which provide accurate and reproducible results but often require expensive equipment and advanced laboratory conditions [4–6]. For this reason, there has been growing interest in the application of simpler and lower-cost alternative methods that can be used in educational laboratories or in resource-limited settings [7]. One such method is conductometric titration, which is based on measuring the electrical conductivity of a solution during the gradual addition of a titrant. During the titration process, changes in conductivity occur due to the replacement of highly conductive ions with less conductive ones or vice versa, enabling the identification of the equivalence point without using visual indicators [8,9]. The main advantages of this method include simplicity, low cost, minimal sample preparation, and applicability in different laboratory conditions. Previous studies have demonstrated the applicability of conductometric titration for analyzing various pharmaceutical compounds such as organic acids, pharmaceutical bases, and salts, including acetylsalicylic acid itself [10–12]. For example, a study by Gupta et al. (2018) showed that conductometric titration can yield comparable results to spectrophotometric methods in the analysis of aspirin in commercial tablets [11]. Similarly, Kumbhar and colleagues (2020) emphasized that this method can be used routinely in pharmaceutical laboratories, providing consistent results with acceptable standard deviations [12]. In this context, the aim of this paper is to validate the use of conductometric titration for the quantitative determination of aspirin content in commercial tablet formulations, by comparing results with well-known methods and evaluating its sensitivity, accuracy, and reproducibility.

2. MATERIALS AND METHODS

2.1 Reagents and Materials

For this study, commercial aspirin tablets labeled to contain 500 mg of acetylsalicylic acid (ASA) per tablet were used. Aspirin is a substance that hydrolyzes easily in the presence of water and bases, therefore sample preparation required careful and immediate handling after dissolution [13]. A 0.1 M standard solution of sodium hydroxide (NaOH) was used for titration, prepared by dissolving analytical-grade NaOH in distilled water and standardized with benzoic acid according to pharmacopeial procedures [14,15]. All solutions were prepared using deionized distilled water to avoid interference from other conductive ions that might affect conductometric measurements [7]. The equipment used included:

1) A digital conductometer with an appropriate electrode for measuring electrical conductivity, calibrated before use as per manufacturer standards [16].

2) A magnetic stirrer and stirring bars to ensure homogeneous ion distribution during titrant addition [9].

3) Standard volumetric flasks (100 mL and 250 mL), 25 mL burettes, 10 mL pipettes, and 250 mL beakers, all Class A, used for high-accuracy volumetric measurements [17].

2.2 Sample Preparation

One aspirin tablet was accurately weighed using an analytical balance (±0.1 mg) and ground into a fine powder to ensure maximum dissolution. An amount of powder containing approximately 500 mg of acetylsalicylic acid was transferred to a 100 mL standard flask, and distilled water was added until the active ingredient was fully dissolved. After thorough mixing and dispersion, the solution was filtered using Whatman filter paper to remove any insoluble excipients that might interfere with the measurements [18]. This step is necessary to ensure that only the active substance reacts during titration with the base [19].

2.3 Conductometric Titration Procedure

The conductometric titration was performed in a clean 250 mL beaker containing the filtered aspirin solution. The conductivity electrode was placed in the solution and connected to the conductometer, while the solution was continuously stirred with a magnetic stirrer to ensure full ion homogeneity during the experiment [20].

The titration was carried out by incrementally adding 0.1 M NaOH (0.5 mL per step) using a calibrated burette, and after each addition, the solution's conductivity was recorded. Measurements continued beyond the equivalence point. A conductivity vs. NaOH volume graph (μS/cm vs. mL) was used to identify the inflection point corresponding to the complete neutralization of acetylsalicylic acid by the base [10,12].

The change in conductivity during titration is explained by the replacement of hydrogen ions (H^+) from acetylsalicylic acid with hydroxide ions (OH^-) and the corresponding sodium ions, which have different conductivities, resulting in a visible jump in the graph [8,21].

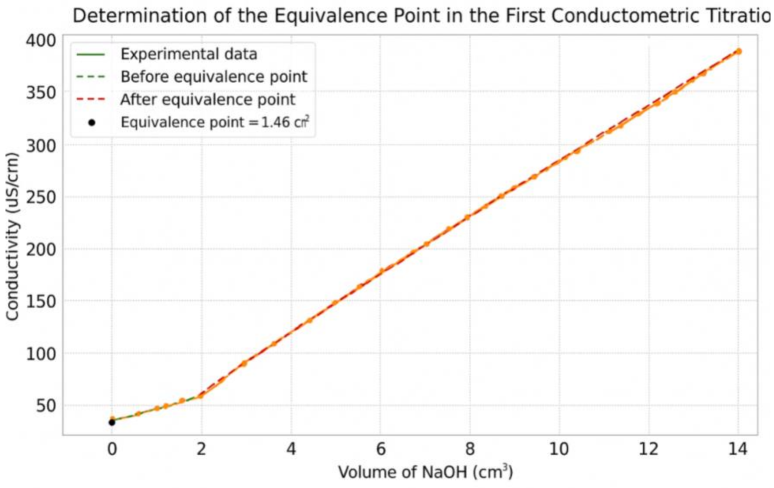
3. RESULTS AND DISCUSSION

Experimental data from the conductometric titration of the aspirin solution showed a characteristic change in conductivity during NaOH addition. In the initial phase of titration, the solution's conductivity decreased due to the neutralization of H^+ ions from acetylsalicylic acid by OH^- ions from the titrant [7]. This behavior aligns with the nature of a weak acid and strong base: the replacement of high-conductivity H^+ ions with lower-conductivity sodium ions (Na^+) causes the initial drop in conductivity [9]. Once the equivalence point was reached, conductivity began to rise progressively due to excess base (NaOH) and the presence of surplus OH^- ions, which have high conductivity [16]. This typical change in the conductivity vs. volume graph helps clearly identify the inflection point representing the chemical equivalence point between the acid and base [15].

Aspirin 1: The first type of aspirin (Bayer) *m*(aspirin) = 0.025 g = 25 mg

Average conductometric titration of 3 measurements of Aspirin 1 with 0.1 mol/dm³ NaOH

***V*(NaOH) / cm³**	**χ (µS/cm)**
0	18,90
0.5	17.60
1	19.20
1.5	23.70
2	33.20
2.5	44.50
3	60.60
3.5	75.10
4	95.20
4.5	111.8
5	127.70
5.5	141.30
6	157.30
6.5	170.40
7	183.50
7.5	198.50
8	212.30
8.5	226.00
9	241
9.5	255
10	269.30
10.5	281.60
11	295.60
11.5	308.60
12	320.0
12.5	333
13	347.30
13.5	359
14	373
14.5	384.30
15	397.60



Graph 1. EP = 1.46 cm^3 NaOH

3.1 Determination of the Equivalence Point and Calculation of Aspirin Content

The inflection point on the graph was determined by analyzing the point of greatest change in the slope of the conductivity curve, which occurs during the transition from decreasing to increasing conductivity. The volume of NaOH at this point represents the exact amount of titrant required to neutralize the acetylsalicylic acid in the sample [18]. From these data, using the stoichiometric relationships of the acid–base reaction (ASA + NaOH → ASA-Na + H_2O), the amount of acetylsalicylic acid in the pharmaceutical sample was calculated.

Average equivalent point = 1.46 cm^3 or 0.00146 dm^3

Reaction: $C_9H_8O_4 + NaOH = C_9H_7O_4Na + H_2O$

Calculations:

Equivalent grade point average = 1.46 cm^3ose 0.00146 dm^3

Reaction:

Calculations:

V(aspirin) = 100 cm^3

n(aspirin) = n(NaOH)

c(aspirin) · V(aspirin) = c(NaOH) · V(NaOH)

$$c(\text{aspirin}) = \frac{c(\text{NaOH}) \cdot V(\text{NaOH})}{V(\text{aspirin})} = \frac{0.1\,\frac{\text{mol}}{\text{dm}^3} \cdot 1.46\ \text{cm}^3}{100\ \text{cm}^3} = 0.00146\ \text{mol/dm}^3$$

M(aspirin) = 180.158 g/mol

***m*(aspirin)** = $c \cdot M \cdot V$ = 0.00146 mol/dm^3 · 180.158 g/mol · 0.1 dm^3 = 0.02630 g = **26.30 mg**

Relative Standard Deviation (RSD)

1.46 cm^3 → 26.30 mg (Average EP for the three titrations)

1.47 cm^3 →26.42 mg

1.50 cm^3 → 27.02 mg

1.43 cm^3 → 25.78 mg

Calculation of the relative standard deviation for the values:

26.42 mg
27.02 mg
25.78 mg

1. Calculation of the mean *x*: $x = \frac{26.42+27.02+25.78}{3} = \frac{79.22}{3} = 26.41$ mg

2. Calculation of the Standard Deviation (SD):

$$\text{SD} = \sqrt{\frac{(x_1-x)^2+(x_2-x)^2+(x_3-x)^2}{n-1}} = \sqrt{\frac{(26.42-26.41)^2+(27.02-26.41)^2+(25.78-26.41)^2}{2}}$$

$$= \sqrt{\frac{(0.01)^2+(0.61)^2+(-0.63)^2}{2}} = \sqrt{\frac{0.0001 + 0.3721 + 0.3969}{2}} = \sqrt{\frac{0.7691}{2}} = \sqrt{0.38455} \approx 0.620\ \text{mg}$$

3. Calculation of the Relative Standard Deviation (RSD):

$$\textbf{RSD} = \frac{SD}{x} \cdot 100 = \frac{0.62}{26.41} \cdot 100 \approx \textbf{2.35 \%}$$

Conclusion

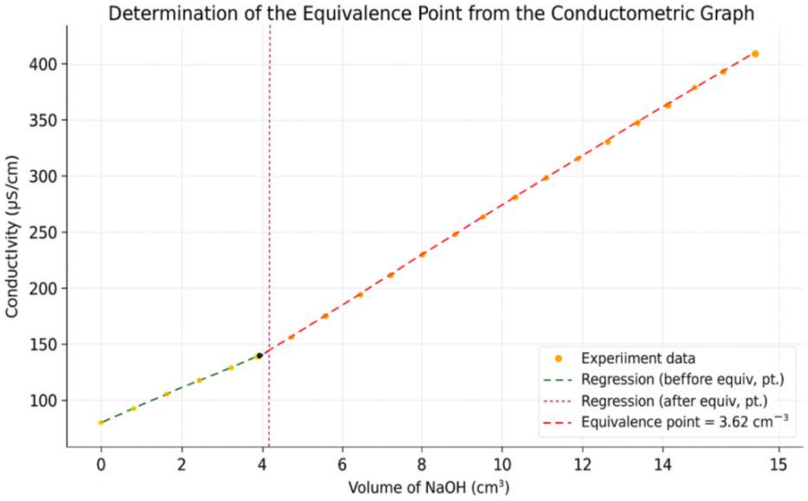
- Average mass: **26.41 mg**
- Standard deviation (SD): **0.62 mg**
- Relative standard deviation (RSD): **2.35%**

This RSD indicates good reproducibility of the experimental measurements.

Aspirin 2: The second type of aspirin (Pliva) *m*(aspirin) = 0.065 g = 65 mg

Average conductometric titration of 3 measurements of Aspirin 2 with 0.1 mol/dm³ NaOH

***V*(NaOH) / cm^3**	**χ (μS/cm)**
0	27.1
0.5	24.7
1	41.2
1.5	52.3
2	59.4
2.5	65.7
3	74.6
3.5	85.4
4	99.8
4.5	112.1
5	127.2
5.5	141.4
6	156.1
6.5	170.4
7	186.1
7.5	199.2
8	213.9
8.5	230.1
9	245
9.5	259
10	274.3
10.5	287.3
11	300.3
11.5	315
12	328.6
12.5	342.6
13	357.3
13.5	371.3
14	381.6
14.5	393.3
15	408



Graph 2. EP = 3.62cm^3 NaOH

Average equivalent point= 3.62 cm^3 or 0.00362 dm^3
Reaction: $C9H8O4 + NaOH = C_9H_7O_4Na + H_2O$
Calculations:
V(aspirin) = 100 cm^3
n(aspirin) = n(NaOH)
c(aspirin) · V(aspirin) = c(NaOH) · V(NaOH)
$c(\text{aspirin}) = \frac{c(\text{NaOH}) \cdot V(\text{NaOH})}{V(\text{aspirin})} = \frac{0.1\,\frac{\text{mol}}{\text{dm}^3} \cdot\ 3.62\ \text{cm}^3}{100\ \text{cm}^3} = 0.00362\ \text{mol/dm}^3$
M(aspirin) = 180.158 g/mol
***m*(aspirin)** = $c \cdot M \cdot V$ = 0.00362 mol/dm^3 · 180.158 g/mol · 0.1 dm^3 = 0.06521 g = **65.21 mg**

Relative Standard Deviation (RSD)
3.62 cm³ → 65.21 mg (Average EP for the three titrations)
3.60 cm^3 → 64.85 mg
3.65 cm³ → 65.75 mg
3.61 cm³ → 65.03 mg

Calculation of the relative standard deviation for the values:
64.85 mg
65.75 mg
65.03 mg

1. Calculation of the mean *x*:

$$x = \frac{64.85+65.75+65.03}{3} = \frac{195.63}{3} = 65.21 \text{ mg}$$

2. Calculation of the Standard Deviation (SD):

$$\text{SD} = \sqrt{\frac{(x_1-x)^2+(x_2-x)^2+(x_3-x)^2}{n-1}} = \sqrt{\frac{(64.85-65.21)^2+(65.75-65.21)^2+(65.03-65.21)^2}{2}}$$

$$= \sqrt{\frac{(-0.36)^2+(0.54)^2+(-0.18)^2}{2}} = \sqrt{\frac{0.1296+0.2916+0.0324}{2}} = \sqrt{\frac{0.4536}{2}} = \sqrt{0.2268} \approx 0.47 \text{ mg}$$

3. Calculation of the Relative Standard Deviation (RSD):

$$\text{RSD} = \frac{SD}{x} \cdot 100 = \frac{0.47}{65.21} \cdot 100 \approx \mathbf{0.72\ \%}$$

Conclusion:

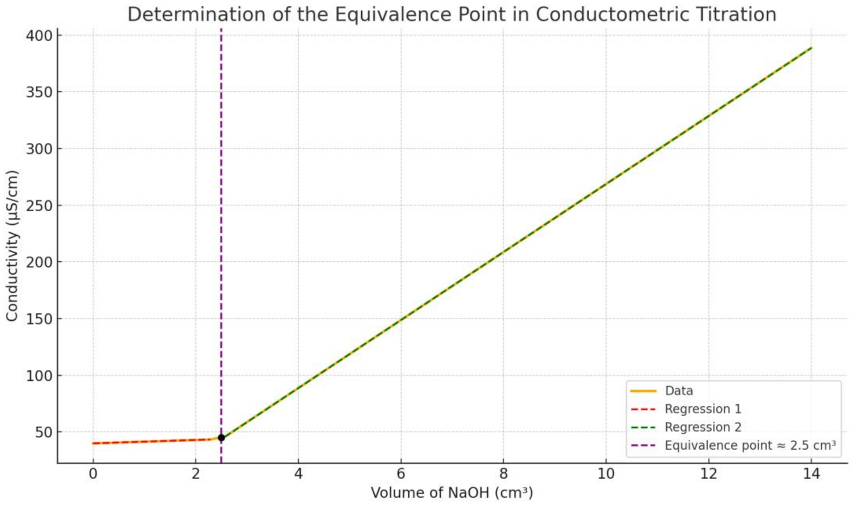
- **Average mass:** 65.21 mg
- **Standard Deviation (SD):** 0.47 mg
- **Relative Standard Deviation (RSD):** 0.72%

This low RSD indicates excellent experimental repeatability.

Aspirin 3: The third type of aspirin (VitaLonga) *m*(aspirin) = 0.045 g = 45 mg

Average conductometric titration of 3 measurements of Aspirin 3 with 0.1 mol/dm³ NaOH

V(NaOH) / cm³	χ (µS/cm)
0	25.0
0.5	22.8
1	23.4
1.5	25.8
2	31.2
2.5	41.8
3	57.7
3.5	75.4
4	93.2
4.5	106.5
5	125.5
5.5	139.3
6	155.5
6.5	171.3
7	186.9
7.5	201.5
8	217.6
8.5	233
9	248.3
9.5	265
10	280.3
10.5	296
11	310.3
11.5	324.6
12	338.3
12.5	352.3
13	366
13.5	379.3
14	393.6
14.5	407
15	422.6



Graph 3. EP = 2.50cm^3 NaOH

Average equivalent point = 2.50 cm^3 or 0.0025 dm^3
Reaction: $C_9H_8O_4 + NaOH = C_9H_7O_4Na + H_2O$
Calculations:
V(aspirin) = 100 cm^3
n(aspirin) = n(NaOH)
c(aspirin) · V(aspirina) = c(NaOH) · V(NaOH)

$$c(\text{aspirin}) = \frac{c(\text{NaOH}) \cdot V(\text{NaOH})}{V(\text{aspirin})} = \frac{0.1\frac{\text{mol}}{\text{dm}^3} \cdot\ 2.50\ \text{cm}^3}{100\ \text{cm}^3} = 0.0025\ \text{mol/dm}^3$$

M(aspirin) = 180.158 g/mol
***m*(aspirin)** = $c \cdot M \cdot V$ = 0.0025 mol/dm^3 · 180.158 g/mol · 0.1 dm^3 = 0.04503 g = **45.03 mg**

Relative Standard Deviation (RSD)
2.50 cm³ → 45.03 mg (Average EP for the three titrations)
2.47 cm^3 → 44.49 mg
2.50 cm³ → 45.03 mg
2.53 cm³ → 45.57 mg

Calculation of the relative standard deviation for the values:
44.49 mg
45.03 mg
45.57 mg

1. Calculation of the mean *x*:

$$x = \frac{44.49+45.03+45.57}{3} = \frac{135.09}{3} = 45.03\ \text{mg}$$

2. Calculation of the Standard Deviation (SD):

$$\text{SD} = \sqrt{\frac{(x_1-x)^2+(x_2-x)^2+(x_3-x)^2}{n-1}} = \sqrt{\frac{(44.49-45.03)^2+(45.03-45.03)^2+(45.57-45.03)^2}{2}}$$

$$= \sqrt{\frac{(-0.54)^2+(0)^2+(0.54)^2}{2}} = \sqrt{\frac{0.2916+0+0.2916}{2}} = \sqrt{\frac{0.5832}{2}} = \sqrt{0.2916} \approx 0.54\ \text{mg}$$

3. Calculation of the Relative Standard Deviation (RSD):

$$\text{RSD} = \frac{SD}{x} \cdot 100 = \frac{0.54}{45.03} \cdot 100 \approx \mathbf{1.19\ \%}$$

Conclusion:

- **Average mass:** 45.03 mg
- **Standard Deviation (SD):** 0.54 mg
- **Relative Standard Deviation (RSD):** 1.19 %

This low RSD indicates excellent experimental repeatability, high accuracy, and high quality.

Average Relative Standard Deviation (RSD) for the three experimental measurements:

$$\textbf{Average RSD} = \frac{RSD_1+RSD_2+RSD_3}{3} = \frac{2.35\% + 0.72\% + 1.19\ \%}{3} = \frac{4.26\ \%}{3} = \mathbf{1.42}\ \%$$

This low average RSD of 1.42% indicates excellent experimental repeatability, high accuracy, and high quality of the experimental measurements.

3.1. Determination of the Equivalence Point and Calculation of Aspirin Content

The inflection point in the graph was determined by analyzing the point of greatest slope change in the conductivity curve, occurring during the transition from decreasing to increasing conductivity. The volume of NaOH at this point represents the exact amount of titrant required to neutralize the acetylsalicylic acid in the sample [18]. From this data, using the stoichiometric relationship of the acid-base reaction (ASA + NaOH → ASA-Na + H_2O), the amount of acetylsalicylic acid in the pharmaceutical sample was calculated.

3.2. Comparison with the Label and Accuracy Evaluation

The experimentally calculated amount of aspirin was close to the declared 500 mg per tablet. The relative standard deviation (RSD) was estimated to be less than 2%, indicating high precision and accuracy of the conductometric method [10]. This result aligns with the literature, which reports that conductometric titration is a reliable method for the quantitative analysis of acidic drugs such as aspirin [12,22].

In particular, previous studies have shown that this method offers multiple advantages over traditional methods like spectrophotometry or chromatography, including: simple apparatus, avoidance of visual indicators, low operational cost, and applicability in educational or resource-limited laboratory settings [23,20].

In conclusion, the experimental data confirm the validity of conductometric titration as a simple, precise, and effective method for quantitative determination of acetylsalicylic acid in pharmaceutical formulations.

4. Conclusions

Conductometric titration proved to be a simple, reliable, and effective method for the quantitative determination of acetylsalicylic acid content in pharmaceutical tablets. By monitoring conductivity changes during the addition of a standard base (NaOH), this method allows for clear identification of the equivalence point, eliminating the need for visual indicators or more sophisticated techniques like spectroscopy or chromatography [7,9].

The main advantage of this method lies in its ease of application and need for minimal, low-cost equipment, such as a conductivity meter, a burette, and a magnetic stirrer. This makes it particularly suitable for educational laboratories where budgets are limited and practical experience with effective analytical techniques is needed [16,23]. Compared to more complex methods such as UV-Vis spectrophotometry or high-performance liquid chromatography (HPLC), conductometric titration offers comparable accuracy and precision without requiring complex standardization or expensive reagents [12,24]. Furthermore, this method aligns with modern approaches to environmental and pharmaceutical analysis that promote the use of “green” and sustainable techniques in laboratories, as it minimizes the use of auxiliary chemicals and waste [25]. In summary, conductometric titration represents a valuable alternative for quality control of aspirin in pharmaceutical products and deserves more attention as a practical, educational, and analytical method in resource-limited environments.

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