**Alqalam Journal of Medical and Applied Sciences**. 2026;9(2):482-484
https://doi.org/10.54361/ajmas.269226

*Original article*

# Determination of Acetyl Salicylic Acid in Aspirin Tablets Using the Volumetric Method

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**Abstract**
Drug analysis plays a vital role in ensuring the quality and effectiveness of pharmaceutical products by determining the concentration of active ingredients. This study investigates the use of direct titration for the quantification of acetylsalicylic acid (ASA) in commercially available aspirin tablets. The research aimed to evaluate direct titration as a simple, economical, and practical method for routine pharmaceutical quality control in Darna City. Aspirin samples were collected from various neighborhood pharmacies supplying medications to local consumers. The concentration of ASA was determined using titrimetric analysis, and the results were compared across different commercial brands. The method demonstrated good precision and operational simplicity, making it suitable for routine analysis. However, limitations in accuracy were observed. The findings revealed variations in ASA content among the tested brands. Although most samples complied with official pharmacopeial standards, the observed differences indicate the need for continued monitoring to ensure consistency and therapeutic effectiveness. This study highlights the importance of improving manufacturing processes and refining analytical methods to strengthen pharmaceutical quality control and maintain drug reliability.
**Keywords.** Aspirin, Acetylsalicylic Acid, Titration, Salicylic Acid.

## Introduction

Acetylsalicylic acid (ASA), commonly known as aspirin, is one of the most widely used analgesic medications worldwide. ASA possesses anti-inflammatory, antipyretic, and analgesic properties. In addition to relieving pain and fever, aspirin reduces platelet aggregation and is widely used in the prevention of myocardial infarction and ischemic stroke. More than 26,000 clinical and scientific articles have been published on aspirin, making it one of the most extensively studied medications in history [1]. Many well-known pharmaceuticals developed during the twentieth century were derived from natural and herbal sources. The medicinal properties of willow trees were recognized as early as ancient civilizations. The Assyrians around 4000 BC and the Sumerians around 3500 BC documented observations regarding the therapeutic benefits of willow bark, which later contributed to the development of aspirin [2]. In order to guarantee the safety and effectiveness of pharmaceuticals before they are used in patient treatment, drug analysis is a crucial part of maintaining the quality of pharmaceuticals globally. Analytical techniques have evolved to include a range of approaches that help identify the active ingredients in medications. One of the most accurate and popular methods for drug analysis is high-performance liquid chromatography (HPLC). However, some laboratories may not be able to use HPLC due to its high cost and technical requirements, necessitating the development of more affordable, accurate methods [3,4]. In addition to its preventive function in lowering the risk of cardiovascular diseases, aspirin, also known as acetylsalicylic acid, is one of the most commonly used drugs worldwide for the treatment of pain, fever, and inflammation [3]. Determining the concentration of its active ingredient is essential for guaranteeing its therapeutic efficacy.

For many years, researchers have examined ASP and its primary and active breakdown product, salicylic acid (SA). In essence, the pharmacological action of ASP is caused by its instant hydrolysis to SA after ingestion. Over the years, this seemingly straightforward reaction has been thoroughly investigated, and currently [5-7]. Aspirin is an unstable molecule that is highly sensitive to light, humidity, and temperature. These environmental factors can cause it to break down into acetic acid and salicylic acid (Fig 1). reducing its effectiveness. Therefore, it is important to regularly monitor and measure aspirin's stability under different storage conditions [5].

Several researchers have previously employed spectrophotometric methods for the determination of acetylsalicylic acid in aspirin tablets. However, they generate accurate and reproducible results; these methods often require costly equipment and advanced laboratory facilities [8,10,19]. Acid–base titration remains a widely used analytical technique because of its simplicity, speed, and cost-effectiveness, as well as its well-established precision and reproducibility. Furthermore, extensive research has been carried out to detect ASA and SA in pharmaceutical formulations and biological fluids. Notably, the pharmaceutical industry has shown growing interest in the development of robust analytical methods to ensure drug stability and non-degradability up to the expiration date [3].

Building on our previous investigation of drug stability under various storage conditions [11], this study compares two acid-base titrimetric methods for determining the concentration of acetylsalicylic acid in aspirin tablets stored under different environmental conditions.


***Received****: 19-12-2025 -* ***Accepted****: 17-01-2026 -* ***Published****: 22-02-2026*

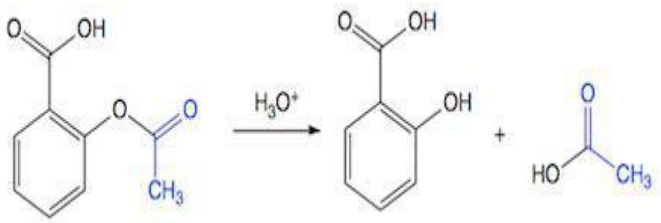


***Figure 1. The hydrolysis of acetylsalicylic acid to salicylic acid and acetic acid***

## Method

### *Sample collection*

The samples were collected randomly from local drug stores and pharmacies in Derna city. A total of seven samples were obtained, each representing a different local or international pharmaceutical company.

### *Conditions of Storage*

Aspirin tablets were stored under controlled conditions to simulate typical storage environments. Temperature conditions included < 8 °C, 15–25 °C, and > 25 °C. Samples were subjected to normal and elevated humidity levels and to light exposure conditions of direct light or darkness. After a 30-day storage period, aspirin content was quantified using two titration methods to assess chemical stability and degradation over time.

### *Reagent and Solutions preparation*

A 0.2 M stock solution of salicylic acid was prepared by dissolving 2.76 g of salicylic acid in a 100 mL volumetric flask and diluting to the mark with distilled water. From this stock solution, a series of standard solutions with concentrations ranging between 0.002 M and 0.007 M was obtained through appropriate dilution. A 0.1 M sodium hydroxide solution was prepared by dissolving 8 g of sodium hydroxide in distilled water and diluting to a final volume of 200 mL in a volumetric flask. The phenolphthalein indicator solution was prepared by dissolving 2.0 g of phenolphthalein powder in 150 mL of ethanol, followed by dilution with 200 mL of distilled water. This set of preparations ensured the availability of standardized reagents for subsequent experimental procedures.

### *Sample Titration*

An aspirin tablet was accurately weighed, finely crushed, and dissolved in 10 mL of ethanol (50%) to determine the acetylsalicylic acid content of the sample. Subsequently, 25 mL of distilled water and three drops of phenolphthalein indicator were added to the solution. The resulting mixture was titrated with a standardized sodium hydroxide solution (0.1N) until a persistent pale pink endpoint was observed. This procedure was repeated in triplicate for each aspirin sample to ensure accuracy and reproducibility. Salicylic acid is a weak organic acid, initially producing an acidic solution. The pH rises as the base is progressively added until the millimoles of base equal the millimoles of acid. At this equivalence point, the indicator changes color from colorless to pink, indicating that the solution has reached its endpoint

The salicylic acid concentration is determined using the following formula.

$$M_{NaoH}\ V_{NaOH} = M_{sA} * V_{SA}$$

calculating the direct titration percentage according to equation (1) [16]

$$\text{direct titration \%} = \frac{\text{ASA practical weight}}{\text{average tablet weight}} \times 100 \ldots\ldots\ldots (1)$$

The results from each company will be compared with the percentage of labeling of the same company. the equation (2)

$$\text{percentage of labeling} = \frac{\text{ASA labeling amount}}{\text{average tablet weight}} \times 100 \ldots\ldots (2)$$

## Results and discussions

The data generated from the direct titration assay presented in (Table 1) show the differences in the percentage of labeling value of the tested agesamples.

The results indicate that the concentration of acetylsalicylic acid (ASA) varied significantly among the analyzed samples. Sample 1 showed the highest percentage based on titration (62%), whereas Sample 2 exhibited the lowest value (35%). Despite this variation, the measured values for both samples were relatively close to their respective labeled percentages. These differences may be attributed to variations in formulation composition, manufacturing quality, or uneven distribution of the active ingredient within the tablets [12]. Sample 5 demonstrated a relatively high aspirin content (51%) compared with its labeled percentages (45%). In contrast, Samples 3 and 4 showed lower aspirin contents (39% and 41%, respectively) when compared to their labeled percentages (51% and 62%). Samples 6 and 7 showed lower aspirin contents (50%) when compared to their labeled percentages (95% and 78 %). These discrepancies may indicate underlying factors


***Received****: 19-12-2025 -* ***Accepted****: 17-01-2026 -* ***Published****: 22-02-2026*

that affect concentration estimates, such as contaminants, inconsistencies in tablet dissolution, or chemical interactions [13].

***Table 1. ASA analysis data obtained from the direct titration assay***

| NO | Company Name | ASA weight labeling | Tablet weight | Labeled Percentage | %Titration |
|---|---|---|---|---|---|
| 1. | Aspirin protect | 100 mg | 134.32 mg | 74 | 62 |
| 2. | Gastro-Resistant (Bristol) | 75 mg | 201.2 mg | 37 | 35 |
| 3. | aggrex | 75 mg | 146.92 mg | 51 | 39 |
| 4. | jusprin | 81 mg | 129.5 mg | 62 | 41 |
| 5. | Aspirin Dispersible | 75 mg | 150.2 mg | 45 | 51 |
| 6. | Rivo | 320 mg | 338 mg | 95 | 50 |
| 7. | aspocid | 75 mg | 95.9 mg | 78 | 50 |

## Conclusion

Titration proved to be a simple, reliable, and efficient method for quantifying acetylsalicylic acid in pharmaceutical tablets. By monitoring changes in the concentration of ASA during the controlled addition of a standard sodium hydroxide (NaOH) solution, the equivalence point could be accurately determined without relying on visual indicators or more complex analytical techniques such as spectroscopy or chromatography. Data obtained from a direct titration assay provides a simple, rapid, cheap, and usable method for checking ASA concentration with a high degree of accuracy.

***Ethical Statement:***

No human or animal subjects were involved. Samples were obtained through routine purchase from local pharmacies. The study adhered to institutional and national guidelines for research integrity, with approval from the local academic committee.

***Conflict of interest***. Nil

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***Received***: *19-12-2025* - ***Accepted***: *17-01-2026* - ***Published***: *22-02-2026*