
Determination Of The Level Of Medium Acid Drug Compounds By Semi-Water-Free Titration

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Abstract

Salicylic acid is a compound widely used in topical preparations, so determining its levels is necessary to ensure product quality and safety. This study aims to determine the levels of salicylic acid in salicylic acid and salicyl powder samples using the semi-aqueous titration (TSBA) method. The study was conducted experimentally in a laboratory using 0.1 N NaOH as a titrant and phenolphthalein as an indicator. Each sample was analyzed three times. The results showed that the salicylic acid levels were 27.63%, 42.78%, and 24.79%, respectively, while the levels in the salicyl powder sample were 80.01%, 87.10%, and 88.12%. Variations in levels between repetitions can be influenced by weighing accuracy, sample homogeneity, dissolution process, burette reading, and titration endpoint determination. The TSBA method can be used as a simple method for quantitative determination of salicylic acid compounds.

Keywords: Salicylic Acid, Salicyl Powder, Semi-Water-Free Titration, Naoh, Determination Of Concentration.

INTRODUCTION

Salicylic acid is a compound widely used in topical preparations due to its keratolytic activity, which helps remove dead skin cells and maintains clean pores. This compound is also found in various skin care products, such as creams, lotions, and other cosmetic preparations. The use of salicylic acid in cosmetic products requires caution because excessively high levels of the active ingredient can increase the risk of skin irritation. Therefore, determining the salicylic acid content is necessary to ensure the quality and safety of preparations sold to the public.(Utami et al., 2025).

One dosage form that can be used on the skin is salicylic powder. This product can contain salicylic acid as an active ingredient intended to help address skin problems. The active ingredient content in a preparation needs to be quantitatively analyzed to determine whether the product's concentration matches the required concentration. This testing is important because the active ingredient content cannot always be determined solely based on the information listed on the packaging.(Subardi, 2024)

StudySammulia et al., (2026)showed that salicylic acid levels in cream preparations could be determined using UV-Vis spectrophotometry and alkalimetry. The study analyzed several products obtained from pharmacies and online retailers and revealed differences in salicylic acid levels between products. These results suggest that quantitative analysis is necessary to determine the actual salicylic acid content in cosmetic preparations.

Recent research also suggests that monitoring salicylic acid content in cosmetic products is still necessary. Research published in 2026 developed an HPLC-DAD method to determine the levels of hydroquinone, salicylic acid, and niacinamide in skin whitening products. Of the 51 products analyzed, discrepancies were found between the measured levels and those stated on the label, including those containing salicylic acid. These findings reinforce the importance of analytical determination of levels as part of cosmetic product quality and safety monitoring.

Salicylic acid levels can be determined using several analytical methods, including UV-Vis spectrophotometry, high-performance liquid chromatography (HPLC), and titrimetric methods. Although instrumental methods offer good sensitivity and selectivity, they require specialized instruments and are relatively expensive. Therefore, titration methods can be an alternative for simpler

quantitative analysis, especially in research activities in educational laboratories.(Iqbal & Lubis, 2024).

Salicylic acid is a compound that has weak acid properties, so its determination can be done by titration using suitable solvent conditions. Previous research on the analysis of salicylic acid by non-aqueous titration showed that the use of non-aqueous solvents can be used to improve the analytical capabilities of weak acid compounds. Lin (1967), for example, reported the use of non-aqueous titration to differentiate and determine salicylic acid and acetylsalicylic acid, with dimethylformamide as the solvent and a suitable titrating system.(Sammulia et al., 2026).

Furthermore, research on the analysis of cream preparations shows that salicylic acid compounds can be determined by titration in non-aqueous solvents without the need for complex purification processes prior to analysis. This indicates that the titrimetric approach in non-aqueous media has potential for use in the analysis of salicylic acid compounds in pharmaceutical or cosmetic preparations.(Septiany et al., 2025).

Based on the above description, determining the salicylic acid content in salicylic powder is necessary to determine the active ingredient content quantitatively. The use of the semi-aqueous titration (TSBA) method was chosen because its principle is suitable for the analysis of compounds that have weak acid properties and can provide more suitable reaction conditions than titration in aqueous media. This method is also relatively simple and does not require complex instrumental equipment. Therefore, this study was conducted with the title "Determination of Salicylic Acid Content in Salicylic Powder Using the TSBA Titration Method."

Formulation of the problem

Based on the background that has been described, the problem formulation in this research is:

1. Does the salicylic powder used as a sample contain salicylic acid?
2. What is the level of salicylic acid contained in salicylic powder preparations determined using the semi-aqueous titration (TSBA) method?
3. Do the results of determining the salicylic acid content in salicylic powder using the TSBA titration method meet the specified content requirements?

Research purposes

This study aims to determine the presence of salicylic acid in salicylic powder preparations, determine the levels of salicylic acid contained in salicylic powder using the semi-water-free titration (TSBA) method, and determine the suitability of the salicylic acid levels obtained with applicable requirements.

RESEARCH METHODS

This is an experimental laboratory study aimed at determining the salicylic acid content in salicylic powder using the semi-aqueous titration (SATT) method. The sample consisted of salicylic powder obtained from the market. The study was conducted through sample preparation, preparation and standardization of a pentiter solution, and titrimetric determination of salicylic acid content.(Sugiyono, 2020)

The sample was carefully weighed and then prepared using a solvent appropriate to the TSBA method until the salicylic acid dissolved and was ready for analysis. The sample solution was then titrated using a standardized pentiter solution with an appropriate indicator. The titration was carried out until the endpoint was reached, indicated by a color change in the indicator. The test was repeated to obtain more accurate results.

The salicylic acid content is calculated based on the volume of the titrating solution used during titration and the salicylic acid equivalence factor. The results of the determination are expressed as a percentage (% w/w) and then compared with the content listed on the label or applicable requirements to determine the appropriateness of the salicylic acid content in the salicylic powder sample.

RESULTS AND DISCUSSION

Results

Salicylic acid and salicyl powder levels were determined using a semi-aqueous titration method using 0.1 N NaOH as a titrant and phenolphthalein as an indicator. Each sample was repeated three times. The samples were first weighed, ground until smooth and homogeneous, and then divided into three parts. The results of observations and calculations of salicylic acid levels are presented in Table 1.

Table 1. Results of Salicylic Acid Level Determination

Test	Sample Weight (mg)	Titration Volume (mL)	Blank Volume (mL)	Normality of NaOH (N)	Equivalence Factor (mg)	Content (%)
1	174.9	3.6	15	0.1	138.12	90.02
2	190.5	2.3	15	0.1	138.12	92.08
3	172.7	3.4	15	0.1	138.12	92.77

Based on Table 1, the weight of the salicylic acid sample in three repetitions was 174.9 mg, 190.5 mg, and 172.7 mg, respectively. The titration volumes obtained were 3.6 mL, 2.3 mL, and 3.4 mL, respectively, while the blank volume was 15 mL. Based on the calculation results listed in the practicum data, the salicylic acid levels were 90.02%, 92.08%, and 92.77%, respectively.

Salicyl powder samples were also tested for concentration. The results of the observations and calculations of salicyl powder concentration are presented in Table 2.

Table 2. Results of Determination of Salicyl Powder Content

Test	Sample Weight (mg)	Titration Volume (mL)	Blank Volume (mL)	Normality of NaOH (N)	Equivalence Factor (mg)	Content (%)
1	144.3	5.7	15	0.1	138.12	89.01
2	144.4	5.9	15	0.1	138.12	87.10
3	144.2	5.8	15	0.1	138.12	88.12

Based on Table 2, the weight of the salicyl powder sample in three repetitions was 144.3 mg, 144.4 mg, and 144.2 mg, respectively. The titration volumes obtained were 5.7 mL, 5.9 mL, and 5.8 mL, respectively, with a blank volume of 15 mL. Based on the calculation results on the practicum data, the salicyl powder content was 89.01%, 87.10%, and 88.12%, respectively.

Overall, the test results showed variations in concentration between repetitions. For the salicylic acid sample, the highest concentration was found in the second repetition at 42.78%, while the lowest concentration was found in the third repetition at 24.79%. For the salicyl powder sample, the highest concentration was found in the third repetition at 88.12%, while the lowest concentration was found in the first repetition at 80.01%.

Discussion

Salicylic acid concentration was determined by semi-aqueous titration using 0.1 N NaOH as the titrant and phenolphthalein as an indicator. This method is based on the neutralization reaction between an acidic compound and a strong base in a semi-polar solvent. The use of a semi-polar solvent aims to facilitate the sample dissolution process and support the reaction between the analyte and the titrant. The endpoint of the titration is marked by the formation of a persistent pink color.

Based on the test results, the salicylic acid concentrations in three repetitions were 90.02%, 92.08%, and 92.77%, respectively. These variations in concentrations correspond to the differences in titration volumes obtained, namely 3.8 mL, 6.2 mL, and 3.4 mL. Differences in results can be caused by weighing accuracy, sample homogeneity, dissolution perfection, burette readings, and accuracy in determining the titration endpoint.

The salicylic acid powder samples yielded concentrations of 89.01%, 87.10%, and 88.12%. These results were relatively consistent because the titration volumes obtained were also close together, namely 5.7 mL, 5.9 mL, and 5.8 mL. The different concentrations between salicylic acid and

salicylic acid powder may be influenced by the composition of the preparations, as salicylic acid powder can contain additional ingredients in addition to the active ingredient.

Research result Bali Subardi, (2024) The results showed that salicylic acid analysis can be performed using the alkalimetric method with 0.1 N NaOH and phenolphthalein as an indicator. This supports the use of a strong base and phenolphthalein as an indicator in determining salicylic acid levels in this experiment. Iqbal & Lubis, (2024) also showed variations in salicylic acid levels in various loose powder samples, so that differences in the composition of the preparation can affect the levels of active substances obtained.

NaOH is a secondary standard solution, so it must be standardized first to obtain the correct concentration. Furthermore, a blank is required to correct the titrant volume derived from the solvent and reagents. Accuracy in standardizing the titrant, weighing the sample, homogenizing the solution, reading the burette, and determining the endpoint are important factors that can affect the accuracy of the analysis results.

Overall, the semi-aqueous titration method can be used to determine the concentration of moderately acidic drug compounds. Experimental results showed variations in concentration between replicates, so controlling the analysis conditions is necessary to obtain more accurate and repeatable results.

CONCLUSION

Based on the experiments that have been conducted, the determination of the levels of moderate acidic drug compounds can be done by the semi-free aqueous titration method using 0.1 N NaOH as a titer and phenolphthalein as an indicator. The results of the determination of salicylic acid levels in three repetitions were obtained at 90.02%, 92.08%, and 92.77%, while the levels of salicyl powder were obtained at 89.01%, 87.10%, and 88.12%. Differences in results between repetitions can be influenced by the accuracy of weighing, sample homogenization, dissolution process, titration volume reading, and endpoint determination. Thus, accuracy in each stage of the analysis is very necessary to obtain more accurate and consistent results in determining the levels.

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