Applied Chemical Engineering (2026) Volume 9 Issue 1 ISSN: 2578-2010 (O)
doi: 10.59429/ace.v9i1.5860

ORIGINAL RESEARCH ARTICLE

Ensuring Drug Quality for Public Health: A Comparative HPLC and UV-Vis Study of Iraqi Market Furosemide Analysis Aligned with SDG 3

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ABSTRACT

Reliable furosemide quantification in pharmaceutical formulations is critical to ensuring drug quality and regulatory compliance. The purpose of this study was to develop and compare spectrophotometric and high-performance liquid chromatography (HPLC) methods for determining furosemide in pharmaceutical dosage forms found in local pharmacies. Standard solutions of furosemide in the concentration range of 2-30 ppm were prepared to create calibration curves using UV-visible spectrophotometry and HPLC methods. Under optimum experimental conditions, the proposed methods' analytical performance was assessed in terms of linearity, limit of detection (LOD), limit of quantification (LOQ), molar absorptivity, and Sandell's sensitivity. The spectrophotometric method was linear, with a correlation coefficient (R^2) of 0.999 and an absorption coefficient of 0.0596 at a wavelength of 275 nm. The calculated LOD and LOQ values were 0.721 ppm and 1.069 ppm. A C18 column with a mobile phase of water, ethanol, and acetonitrile was used for chromatographic analysis under controlled temperature conditions. The results of both analytical methods indicated that the measured drug content in all tested pharmaceutical samples was consistent with the labeled amounts. The developed methods demonstrated satisfactory accuracy, sensitivity, and applicability for routine quality control of furosemide in pharmaceutical formulations, resulting in dependable and cost-effective analytical approaches for laboratory use.

Keywords: SDG-3; Furosemide; HPLC; UV-Vis

ARTICLE INFO

Received: 17 December 2026
Accepted: 24 February 2026
Available online: 03 March 2026

1. Introduction

Furosemide (FUR) is a sulfonamide-based pharmaceutical compound that is widely used in a variety of dosage forms, necessitating precise quantitative measurement to ensure quality, safety, and regulatory compliance ,reliable analytical methods for detecting furosemide in bulk and pharmaceutical formulations are critical for routine quality control and ensuring consistent drug content in commercial products[1,2].A variety of analytical technologies for detection of FUR utilizing many methods have been characterized in the literature, including HPTLC[3], spectrofluorimetry [4], LC-MS [5], RP-HPLC[6],spectrophotometry[7], and the electrochemical sensing method [8]. Several of these methods require sophisticated instruments, costs, and sample preparation for their analysis. However, some reported methods require expensive instrumentation, complex sample preparation, or extended analysis time, which may limit their use in quality control laboratories[9,10]. According to pharmacopeial recommendations, furosemide quantification in bulk and dosage forms necessitates validated, accurate, and repeatable analytical procedures. As a result, the development of simple, rapid, and reliable analytical methods remains critical for pharmaceutical quality assessment. [11,12].

The aim of this study is to provide advantages of the method, which are the less consumption of the medication and reagent, economy, simplicity, accurate results, and good repeatability, and it's a very stable method useful for drug analysis. This study introduces a sensitive spectrophotometric method for trace-level analysis in aqueous media, which is compared to HPLC for accuracy and efficiency.

2. Experimental

2.1. Apparatus

Instrument/Equipment	Model/Type	Manufacturer (Country)	Application/Purpose
UV-Vis Spectrophotometer (dual-beam)	UV-1700	Shimadzu (Japan)	Absorbance measurement and spectral analysis
Analytical Balance	TP-214	Denver Instruments (Germany)	Accurate weighing of samples
Rotary Heater	RM Hei-Standard	Heidolph (Germany)	Sample preparation and temperature control
HPLC System	LC-2010HT	Shimadzu (Japan)	Quantitative chromatographic analysis
HPLC Software	LabSolutions	Shimadzu (Japan)	Data acquisition and processing

2.2. Chemicals

No.	Chemical / Solvent	Manufacturer	Molecular Formula	Purity (%)
1	Water	Sigma-Aldrich	H_2O	99
2	Methanol	Sigma-Aldrich	CH_3OH	99
3	Acetonitrile	Sigma-Aldrich	C_2H_3N	99
4	Furosemide	Apix Ajanta	$C_{12}H_{11}ClN_2O_5S$	99
5	Furosemide	Accord	$C_{12}H_{11}ClN_2O_5S$	99
6	Furosemide (Standard)	Samarah-Iraq	$C_{12}H_{11}ClN_2O_5S$	99

3. Materials and methods

3.1. Preparation of standard solution

A standard stock solution of furosemide (molecular weight: 330.75 g/mol) at 100 ppm was prepared. To ensure complete dissolution, 0.0116 g of furosemide was precisely weighed and dissolved in 100 mL of distilled water under gentle heating. A series of standard working solutions of various concentrations were prepared from this stock solution by serial dilution with distilled water.

3.2. Determination of maximum absorbance wavelength

The (UV-Vis) absorption spectra of the prepared standard solutions were measured in the 200-400 nm range. The wavelength corresponding to the maximum absorbance was determined to be 275 nm. This wavelength was then utilized for all absorbance measurements.

3.3. Quantitative analysis by high-performance liquid chromatography (HPLC)

The standard solutions were prepared and analyzed using high-performance liquid chromatography (HPLC). The mobile phase was an optimized mixture of distilled water, acetonitrile, and methanol. To ensure accurate furosemide quantification, the chromatographic conditions of flow rate, injection volume, column specification, and detection wavelength were chosen.

3.4. Preparation of pharmaceutical sample

A precise weight of 0.16 g furosemide from the pharmaceutical formulation was transferred to a 25 mL volumetric flask and dissolved in distilled water. The solution was thoroughly mixed to ensure complete dissolution before being used for analysis.

3.5. Drug concentration determination using HPLC

The prepared drug solution was analyzed under the same HPLC conditions described for standard solutions. The mobile phase was a mixture of water, acetonitrile, and methanol, and quantification was carried out based on the calibration curve obtained from standard furosemide solution

3.6. Photometric spectrophotometric analysis of drug samples

The drug solution's absorbance was measured with a UV-vis spectrophotometer at a predetermined maximum absorption wavelength (275 nm). The data obtained were compared to HPLC results to assess the reliability of both analytical methods.

3.7. Solvent impact

The effect of solvent composition on the UV absorption of furosemide was investigated by preparing standard solutions in various solvent combinations. A UV-Vis spectrophotometer was used to measure the absorbance of the compound at its λmax.

3.8. Temperature impact

The effect of temperature on furosemide UV absorption was investigated by measuring the absorbance of a standard solution at four different temperatures: 20°C, 30°C, 40°C, and 50°C. To maintain the desired temperature, a thermostatically controlled water bath was used. Absorbance readings were recorded at the compound's λmax.

3.9. Data Analysis

The collected data was processed and analyzed with Excel. Standard functions were used to calculate mean values and standard deviations, and graphical tools were used to show trends and differences. This overarching strategy ensured the consistency and clarity of the results presented in the Results and Discussion sections.

4. Results and Discussion

4.1. UV-Vis Spectrophotometric Determination of Furosemide (FUR) .

Under ideal circumstances, standard furosemide was quantitatively determined using UV-vis spectrophotometry, using ethanol-distilled water as the blank solution. The concentration range in which absorbance measurements were made was 2–30 ppm. According to the results, **275 nm** is a suitable wavelength for precise and repeatable photoluminescence spectroscopy of furosemide because it exhibits a constant and stable absorption maximum.

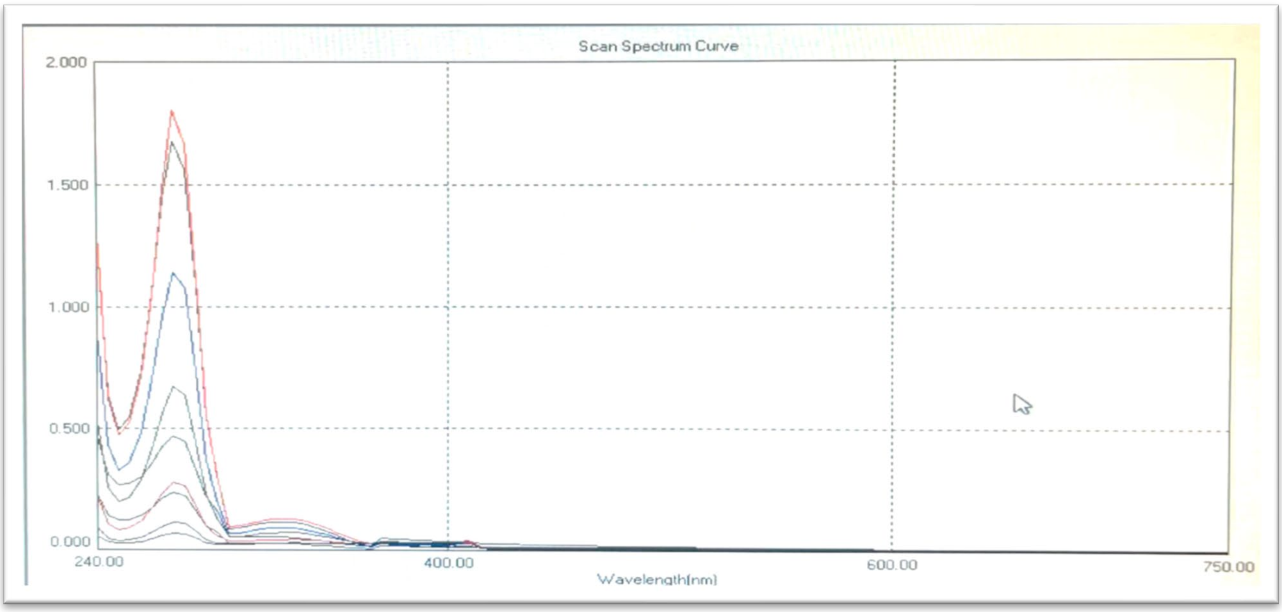
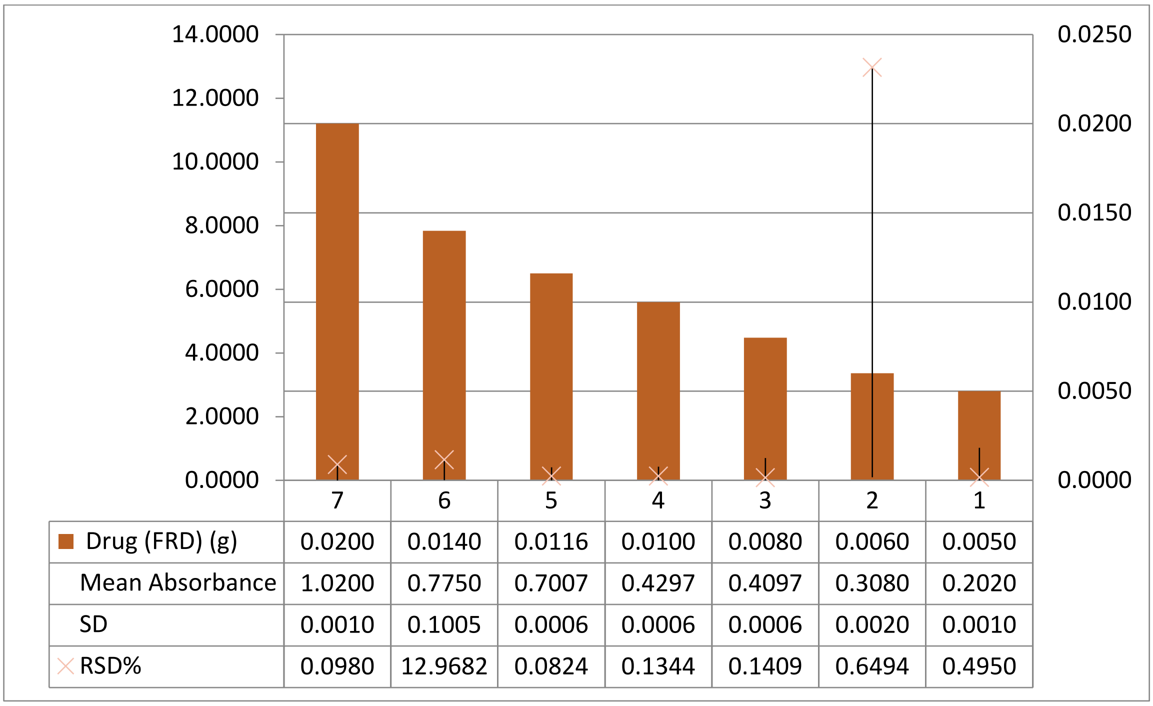


Figure 1. Maximum absorbance of furosemide at different concentrations, demonstrating a linear increase with concentration and confirming the suitability of the spectrophotometric method for accurate quantification.

4.2. Weighing of Standard (FUR) for pure furosemide analysis

After preparing seven standard solutions of furosemide with concentrations ranging from 0.0050 to 0.0200 g, they were examined using UV-Vis spectrophotometry. The relative standard deviation (RSD%), standard deviation (SD), and corresponding mean absorbance were calculated for each concentration after it was examined in triplicate. 0.0116 g was the concentration with the least variability, according to the statistical analysis, with a small SD and almost insignificant RSD%. Accordingly, 0.0116 g is the ideal concentration for spectrophotometric quantification of furosemide, guaranteeing stability and analytical accuracy.

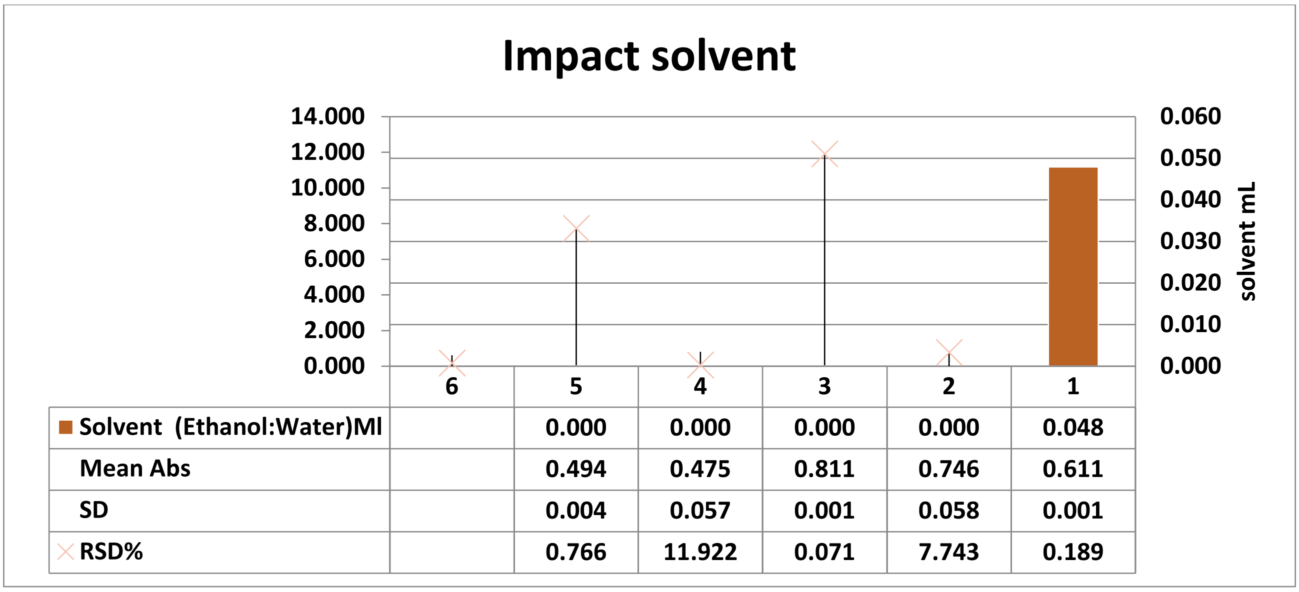


	7	6	5	4	3	2	1
■ Drug (FRD) (g)	0.0200	0.0140	0.0116	0.0100	0.0080	0.0060	0.0050
Mean Absorbance	1.0200	0.7750	0.7007	0.4297	0.4097	0.3080	0.2020
SD	0.0010	0.1005	0.0006	0.0006	0.0006	0.0020	0.0010
× RSD%	0.0980	12.9682	0.0824	0.1344	0.1409	0.6494	0.4950

Figure 2. shows effect of different standard concentrations of furosemide (0.005–0.020 g) on mean absorbance values, including standard deviation (SD) and relative standard deviation (RSD%), demonstrating the linear relationship between concentration and absorbance, which supports the accuracy and precision of the spectrophotometric method.

4.3. Solvent's impact

To assess the impact of solvent composition on the absorption response, furosemide was spectrophotometrically evaluated using various solvent systems. The binary solvent system, which included 3 mL of ethanol and 7 mL of distilled water, had the lowest relative standard deviation (RSD%) and the highest average absorbance among the combinations tested. The enhanced solubility and even dispersion of furosemide molecules in the solvent matrix, which leads to increased stability and repeatability of the spectral measurements, is responsible for this improved response. This solvent mixture was therefore determined to be the best medium for achieving precise and trustworthy UV-vis spectroscopy of furosemide.

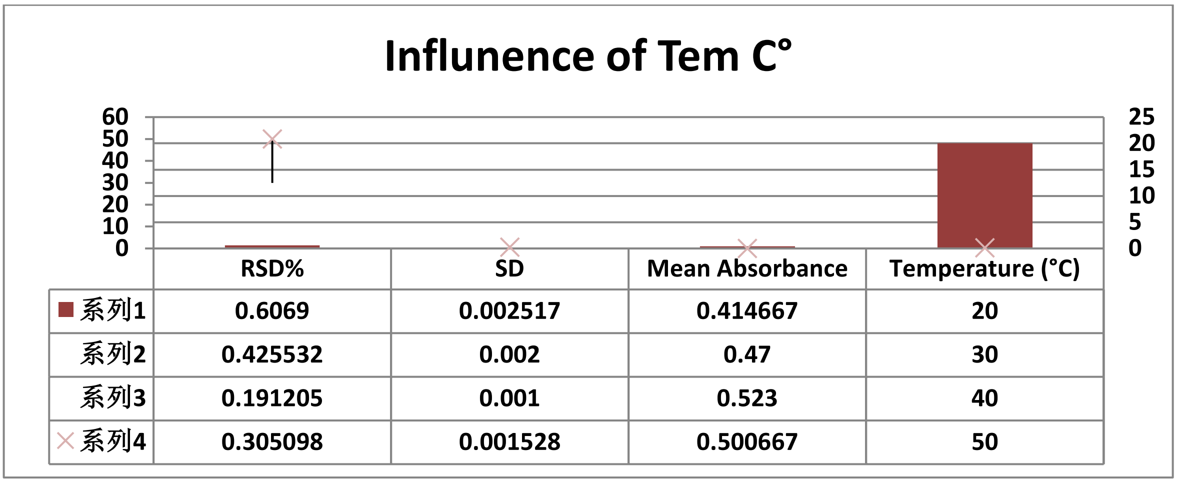


	6	5	4	3	2	1
■ Solvent (Ethanol:Water)Ml		0.000	0.000	0.000	0.000	0.048
Mean Abs		0.494	0.475	0.811	0.746	0.611
SD		0.004	0.057	0.001	0.058	0.001
× RSD%		0.766	11.922	0.071	7.743	0.189

Figure 3. Effect of solvent composition on the UV–Vis absorbance of furosemide (FUR). The binary mixture of 3 mL ethanol and 7 mL distilled water showed the highest mean absorbance and lowest relative standard deviation, indicating optimal solubility, spectral stability, and measurement repeatability.

4.4. Tem °C has an impact

The effect of temperature on the UV-Vis absorbance of Furosemide (**FUR**) was investigated over the temperature range of 20-50 °C. The results showed that 40 °C had the highest mean absorbance and the lowest relative deviation, making it the best thermal condition for improving spectral stability and measurement accuracy. This behavior is attributed to the achievement of molecular kinetic equilibrium at this temperature, which agrees with previously reported findings.



	RSD%	SD	Mean Absorbance	Temperature (°C)
■ 系列1	0.6069	0.002517	0.414667	20
系列2	0.425532	0.002	0.47	30
系列3	0.191205	0.001	0.523	40
× 系列4	0.305098	0.001528	0.500667	50

Figure 4. Effect of temperature on the UV–Vis absorbance of furosemide (FUR) over 20–50 °C. The highest mean absorbance and lowest relative deviation were observed at 40 °C, indicating optimal thermal conditions for spectral stability and measurement accuracy.

4.5. Constructed calibration curve of furosemide by UV-visb and HPLC

The optimized solvent (ethanol: distilled water = 3:7, v/v) was used to create a series of furosemide standard solutions in the concentration range of 2–30 ppm, while a witness solution was made with the same solvent composition. To guarantee the stability of the spectral response, a quartz cell with a path length of 1 cm was used for spectrophotometric measurements at the wavelength (λ = 275 nm). The temperature was set at 40 °C. A calibration curve was constructed using the average results. A linear regression equation based on Beer-Lambert's law and the least squares approach were used to compute the graphic representation of the connection between absorbance and concentration.R2 = 0.9954, a linear correlation coefficient, shows that the experimental and theoretical results are highly linear and in agreement. According to the statistical standards used in the validation of analytical techniques, this result shows that the suggested spectrophotometric approach has sufficient sensitivity and accuracy for the determination of furosemide in the specified range of concentrations. HPLC was used to quantify furosemide and create a calibration curve under optimal circumstances (λ = 275 nm, flow rate = 0.3 mL/min, T = 40 °C, mobile phase: water/ethanol/acetonitrile, 25:70:5 v/v/v). The method demonstrated linearity from 2-30 ppm, with a slope of 37.681, intercept of 42.922, and correlation coefficient $R^2 = 0.999$. The limit of detection (LOD) was 1.736 ppm, and the limit of quantification was 5.786 ppm. The method's sensitivity and reliability were confirmed by a molar absorptivity of 9.43×10^6 L·mol⁻¹·cm⁻¹ and a Sandell index of 28,516.09 μg·cm².

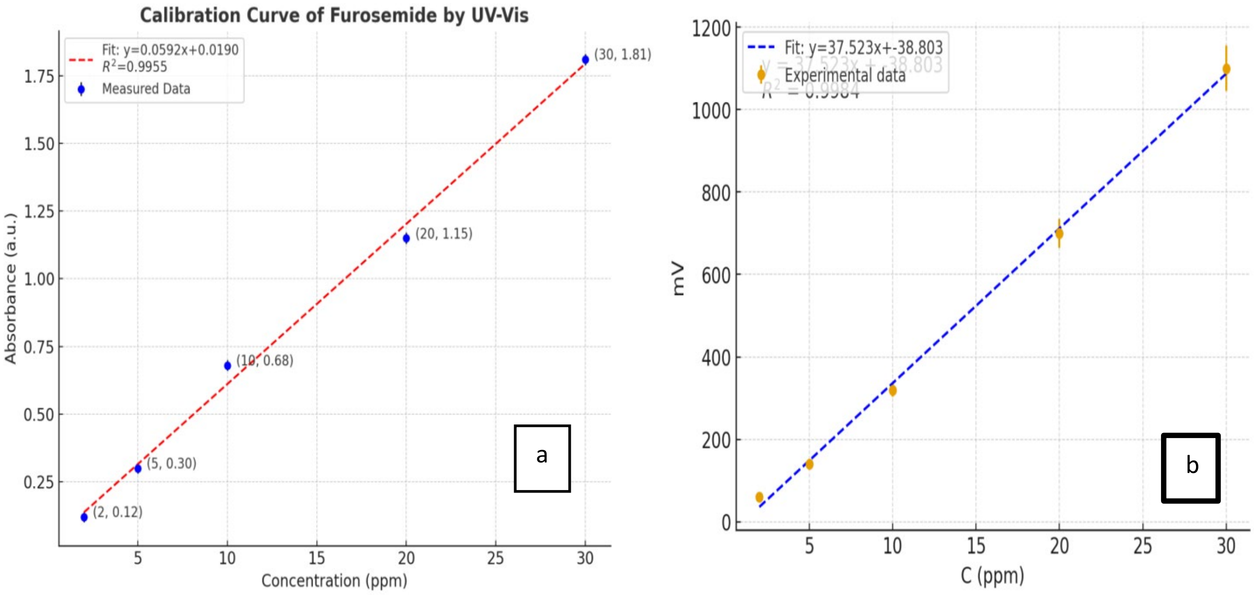


Figure 5. Calibration curves for furosemide (FUR) determined by (a) UV–Vis spectroscopy and (b) HPLC, showing linearity and method response.

Table 1. Analytical Parameters Obtained from the Calibration Curve

Parameter	Value
Slope	0.0596
Y-intercept	0.0018
Linearity Range (ppm)	2–30 ppm
Correlation Coefficient (R²)	0.999
Standard Error	0.054
LOD (Limit of Detection)	0.721 ppm (LOD = 3*Syx/Slope)
LOQ (Limit of Quantification)	1.069 ppm (LOQ = 10*Syx/Slope)
Molar Absorptivity (L·mol⁻¹·cm⁻¹)	19712.402
Sandell's Sensitivity (μg·cm⁻²)	59411

4.6. Assessment of active ingredient (furosemide) in pharmaceutical formulations using UV-Vis

This study used UV-Vis spectroscopy as a simple and accurate analytical instrument to determine the quantity of the active ingredient furosemide in several pharmaceutical formulations sold in local marketplaces. The evaluated pharmaceutical samples included products from two businesses, Accord UK and Apix.ajanta France, to assess the conformance of the active ingredient's real content with the recognized standard pharmaceutical specifications, these brands were chosen based on their widespread availability in the local market and their importance in routine quality control practices. The study sought to compare the accuracy of HPLC and UV-Vis methods in determining furosemide content in these commercial formulations. Standard furosemide solutions were made at various concentrations, and a calibration curve was plotted at the optimal wavelength for drug absorption. To ensure the correctness of the results and reduce experimental variability, the absorbance of each concentration was measured three times, and the mean, standard deviation, and relative standard deviation (RSD) were computed for each data set. The practical results, shown in Table 1-2, demonstrated that all tested samples were within the pharmacopoeial tolerance limits, indicating that the preparations met pharmaceutical quality requirements. These results show that the UV-Vis methodology has sufficient accuracy and sensitivity for determining furosemide, making it an appropriate tool for routine pharmaceutical quality monitoring. These findings imply that the medications evaluated from the two companies under consideration are of a quality comparable to worldwide pharmaceutical standards. This highlights the significance of UV-Vis as a supporting analytical approach in quality control, which can be used in pharmaceutical laboratories as an effective and standardized method.

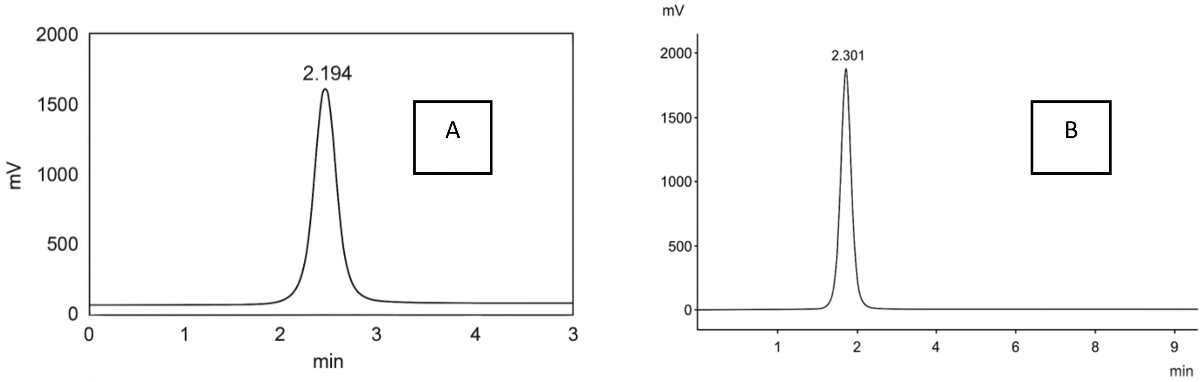


Figure 6. Shows the estimated active principle in the medicine produced by (A) Accord UK and (B)Apix.ajanta France.

Company Name	Labeled Active Ingredient (mg)	Calculated Active Ingredient (mg)	Recovery (%)	RSD (%)
Apix.ajanta (France)	40 mg	40.12	100.21%	0.04%
Accord UK	40 mg	40.23	100.32%	0.02%

The results suggest that both procedures employed to determine the active ingredient in pharmaceutical formulations are accurate and reproducible, with outstanding accuracy and repeatability.

4.7. Furosemide Comparative Analysis by HPLC and UV-ViS

The active ingredient (furosemide) in pharmaceutical formulations supplied by Accord UK and Apix.ajanta France was determined primarily using (HPLC). The study was carried out under optimal conditions, with a wavelength of 275 nm, a mobile phase flow rate of 0.3 mL/min, and a temperature set to 40°C. A water/ethanol/acetonitrile mobile phase (25:70:5) was used for efficient separation and accurate quantitative measurement of furosemide. To evaluate the correctness and trustworthiness of the HPLC results, they were compared to UV-Vis Spectrophotometry, a simpler approach. The study revealed a significant degree of convergence between the two methods' calculated furosemide concentration values in the examined samples. However, it was discovered that the HPLC approach provides greater sensitivity and selectivity, as well as excellent linearity within the studied concentration range, making it a more dependable and verified instrument for pharmaceutical quality control.

4.8. Comparison with Previous Studies and Justification of the Superiority of the Present Study

Compared to previously reported methods, the present study employs a mixed solvent system consisting of water, ethanol, and acetonitrile, which reduces solvent toxicity and cost relative to methods relying exclusively on methanol or buffered acetonitrile systems. The selection of a detection wavelength at 275 nm provides higher selectivity for furosemide compared to visible spectrophotometric methods that require derivatization reactions and longer wavelengths. In terms of linearity, the present method exhibits a suitable working range (2–30 ppm), comparable to both UV–Vis and HPLC methods reported in the literature. Overall, the proposed approach achieves an advantageous balance between solvent safety, wavelength selectivity, and linear analytical performance, making it more suitable for routine pharmaceutical quality control

Table 2. Comparative Evaluation of Furosemide Analysis Methods

Correlation Coefficient (R²)	Linearity (µg/mL)	Wavelength (nm)	Solvents/Mobile phase	Technique	Study
0.999	2–30	275	Water–Ethanol–Acetonitrile	UV–Vis & HPLC	Present study
0.9997	5.2–25	233	Acetonitrile + buffer	RP-HPLC	Youm & Youan (2013) [13].
>0.99	0.05–2.00	Not reported	KH_2PO_4 + Acetonitrile	HPLC	Abou-Auda et al. (1998) [14].
>0.995	2.5–100	272	0.1% Acetic acid + ACN	RP-HPLC	Srejomthong et al. (2025)[15].
0.9991	1–35	~270	Not organic	Derivative UV-Vis	Darweesh, Samar A (2017) [16].

4.9. Novelty of the Study

The present study showed that the proposed method combines simplicity of implementation, low operating cost, high accuracy, use of more selective wavelengths, and reduced consumption of organic solvents, which represents novelty and is compatible with international standards for chemical analysis. The HPLC technique, based on the results and figures shown above, also demonstrated a high capacity for accurate estimation and excellent sensitivity in the determination of furosemide concentration, making it a reliable tool for pharmaceutical quality control.

Importantly, the adoption of this technique in the Iraqi pharmaceutical market is particularly valuable given the need for reliable, accurate, and cost-effective methods to ensure the quality and safety of locally available medicines. This study highlights that HPLC is not only an accurate analytical tool, but can be an essential pillar in periodic drug testing, contributing to protecting public health and ensuring patient safety. Therefore, it is recommended to apply HPLC technology in routine drug testing in Iraq, and to expand the studies to include other drugs to ensure continuous adherence to global pharmaceutical standards.

5. Conclusions

1-Accurate Quantification: The proposed UV-Vis and high-performance liquid chromatography (HPLC) methods accurately measured the amount of furosemide in pharmaceutical preparations. The measured concentrations corresponded to the stated values, demonstrating the reliability and accuracy of both analytical methods.

2-The calibration curve showed high linearity ($R^2 = 0.999$), a low limit of detection (LOD = 0.721 ppm), and a low limit of quantification (LOQ = 1.069 ppm), indicating the accuracy, sensitivity, and robustness of both methods.

3-Practical Benefits: This method combines simplicity, cost-effectiveness, and minimal use of organic solvents, making it appropriate for routine quality control in pharmaceutical laboratories without the need for complex or expensive equipment.

4-Comparative Superiority: Compared to previously reported UV-Vis and HPLC methods, the current study offers selective wavelength detection (275 nm) and an optimized solvent system (water-ethanol-acetonitrile), resulting in improved analytical performance at a lower operational cost.

5-Relevance to Local Market: Implementing this validated method in the Iraqi pharmaceutical market would ensure reliable and cost-effective monitoring of furosemide products, thereby protecting public health and patient safety.

6- All tested furosemide samples met the required pharmaceutical specifications, indicating that the observed numerical differences are minor and do not jeopardize product quality. HPLC's high accuracy and sensitivity in quantifying furosemide confirms its usefulness as a routine quality control tool. These findings demonstrate that minor variations in measured concentrations have little practical impact on drug safety, regulatory compliance, and consumer confidence. As a result, HPLC can be a valuable analytical tool for ongoing monitoring of pharmaceuticals in the Iraqi market.

Future Work: The methodology can be extended to include other pharmaceutical compounds, allowing for more comprehensive analytical studies while adhering to international pharmacopeial standards.

Conflict of interest

The authors declare no conflict of interest

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