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VALIDATION OF THE METHOD FOR DETERMINING THE CONCENTRATION OF IODIDE IONS IN DIFFERENT DOSAGE FORMS BY UV SPECTROPHOTOMETRY

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Abstract. This article is devoted to the validation of an analytical method for the determination of iodide ions in various dosage forms using UV spectrophotometry. This method is an important part of analytical chemistry and drug quality control systems, in which the sensitivity, specificity, and reproducibility of the method are crucial. The relevance of the study is determined by the need for accurate and reliable quantitative determination of iodide ions in pharmaceutical substances and finished drug products. Validation was conducted in accordance with the requirements of the International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH Q2(R1)/Q2(R2)). The aim of this study was to determine the validation characteristics of a method for the quantitative determination of iodide ions in dosage forms using UV spectrophotometry. Results and discussion. The following parameters were evaluated: specificity, linearity, working range, accuracy, repeatability, within-laboratory precision, and solution stability. The working range was found to be from 20 to 240 mg/L, and the correlation coefficient was 0.9998. The method demonstrated high sensitivity and compliance with international requirements. Conclusion. The results obtained confirm the linearity of the method across the studied range of iodide ion concentrations. High sensitivity and a high degree of linearity ($R^2 = 0.9998$) were demonstrated. The developed method is suitable for its intended purpose and can be successfully applied in the analysis of iodine-containing medicinal products. The method has practical significance for pharmaceutical quality control systems.

Key words: iodide ions, UV spectrophotometry, method validation, ICH Q2, pharmaceutical analysis.

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Introduction

In accordance with the requirements of international regulatory documents, analytical methods used for the quality control of medicinal products are subject to mandatory validation. The main requirements for the validation of analytical procedures are set out in the International Council for Harmonisation (ICH Q2(R1)/Q2(R2)) guideline, which defines parameters such as trueness, accuracy, specificity, linearity, range, limit of detection, limit of quantification and robustness of the method [1]. The validation of analytical methods is an essential step in ensuring the reliability of analytical results and confirming the suitability of the method for practical application in pharmaceutical quality control [2].

UV spectrophotometry is one of the most widely used analytical methods in analytical and pharmaceutical chemistry. The method is characterised by high sensitivity, ease of use, rapid analysis and relatively low equipment costs, making it a convenient tool for routine quality control of medicinal products [3]. UV spectrophotometric methods are widely used for the quantitative determination of active substances and inorganic ions in various dosage forms [4].

Iodide ions are widely used in pharmaceutical practice and are found in various medicinal products, including ophthalmic solutions, antiseptics and other dosage forms. Consequently, the development and validation of reliable analytical methods for the quantitative determination of iodide ions is a pressing task in pharmaceutical analysis.

Various methods for the determination of iodide ions have been described in the literature, including titrimetric methods, ion chromatography, electrochemical methods and spectrophotometric methods of analysis [5–7]. However, UV-spectrophotometric methods remain attractive due to their high analytical sensitivity, the availability of equipment and the possibility of rapid analysis [8–10].

The UV spectrophotometry method is based on measuring the absorption of ultraviolet (200–400 nm) and visible (400–800 nm) radiation by the molecules of a substance. Quantitative determination is carried out in accordance with the Beer-Lambert law, which establishes a linear relationship between the optical density of the solution and the concentration of the substance being analysed. Thanks to these characteristics, the method is widely used in the quality control of medicinal products, the verification of the authenticity of preparations, and the quantitative analysis of active substances.

The aim of this study is to validate an analytical method for determining the concentration of iodide ions in various pharmaceutical formulations using UV spectrophotometry in accordance with the requirements of the ICH international guidelines.

During the validation, the following characteristics were investigated: trueness, precision, repeatability, intra-laboratory accuracy, specificity, linearity, working range and robustness of the method when analysing iodide ion solutions.

Experimental section

Objects of Study

The objects of study included the iodide ion standard reference material (SRM 9426-2009, Saint Petersburg, Russian Federation), the FS-1 medicinal product after complete reduction of molecular iodine to iodide ions using formaldehyde, purified water, a 0.05% dextrin solution, and a 1% formaldehyde solution. These materials were used for validation of the analytical method and for the assessment of specificity, linearity, accuracy, precision, and robustness.

Instrumentation and Analytical Conditions

Validation was performed using a Lambda-35 UV spectrophotometer equipped with a diode-array detector. The analysis was carried out under the following conditions:

- wavelength: 226 nm;
- quartz cuvettes with an optical path length of 10.0 mm.

The instrument was operated in accordance with the user manual and standard operating procedure SOP-EQ-035 “Operation of the Lambda-35 Spectrophotometer” [11,12].

Iodide ion standard reference material (SRM 9426-2009) was used as the reference standard. Validation solutions were prepared within the concentration range of 0–200 mg/L.

Validation Procedure

Method validation was performed in accordance with the standard operating procedure SOP-GE-004 “Validation of Analytical Methods” [13] and the requirements of ICH guideline Q2(R1)/Q2(R2) [1].

The following validation characteristics were evaluated: accuracy, repeatability, intra-laboratory precision, specificity, linearity, working range, and robustness.

For the validation study:

- a reference standard solution containing 200 mg/L of iodide ions was prepared by diluting 5 mL of SRM 9426-2009 solution to 25 mL in a volumetric flask according to the manufacturer's instructions;
- iodide ion validation solutions with concentrations of 2, 20, 50, 100, and 200 mg/L were prepared;
- the FS-1 medicinal product was subjected to complete reduction of molecular iodine to iodide ions using formaldehyde;
- purified water, 0.05% dextrin solution, and 1% formaldehyde solution were used as blank matrices;
- the absorbance of the 200 mg/L reference standard solution and validation solutions was measured in ten replicates by different analysts on different days;
- the absorbance of the FS-1 medicinal product and blank matrices was measured in triplicate;
- mean values and standard deviations were calculated;

- the working range of the analytical method was established based on the validation results.

Results and Discussion

Accuracy

The "accuracy" parameter of the analytical method was determined by measuring the optical density of a standard solution with a concentration of 200 ppm of iodide ions. The concentration was calculated based on the measured optical density using the specific optical density, which is the tangent of the slope of the calibration line in the concentration range of 2–200 ppm.

The acceptance criterion for the "accuracy" parameter was a deviation of the calculated concentration from the true value not exceeding ± 3 % for a concentration of 200 mg/l.

Table 1 – Accuracy assessment results

No.	Concentration C, mg/l
1	200.1665
2	200.0588
3	200.1175
4	200.1273
5	200.284
6	200.1371
7	200.0784
8	200.0196
9	200.2742
10	200.2547
Average value	200.1518
True value	200.00
Accuracy	0.1518 (0.076%)

The data presented in Table 1 show that the systematic error of the method is 0.1518 mg/l or 0.076 %. The results obtained satisfy the established acceptance criteria, as they fall within the limits specified in validation plan VP-PCM-015 " Determination of iodide ions in various dosage forms using UV spectrophotometry" [14].

Repeatability

The "repeatability" parameter was determined in a similar manner by measuring a solution of iodide ions (GSO) at a concentration of 200 mg/l ten times. To assess repeatability, optical density data obtained for validation solutions with concentrations of 20, 50, 100 and 200 ppm of iodide ions were used. The concentration of iodide ions was calculated automatically using UV WinLab software, version 5.1.4 (Perkin Elmer, 2004). The mean value and standard deviation for individual determinations were calculated using the validated Excel spreadsheet CS-PCM-002 or according to formula (1):

$$СКО = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \underline{x})^2}{(n-1)}}$$

where: x_i – individual measurement;
$\underline{x}$ – mean value;
n – sample size.

In accordance with the requirements of the verification methodology and ICH Q2 (R1) (CPMP/ICH/381/95), the acceptability criterion for repeatability is characterised by a standard deviation value, which must not exceed ±3 %. The results of the determination of the 'repeatability' parameter are given in Table 2.

Table 2 – Results of the repeatability assessment

No.	20.0 ppm	50.0 ppm	100.0 ppm	200.0 ppm
1	0.2212	0.5109	1.0385	2.0437
2	0.2218	0.5111	1.0388	2.0426
3	0.2221	0.5111	1.0391	2.0432
4	0.2222	0.511	1.0393	2.0433
5	0.2222	0.5111	1.0395	2.0449
6	0.2223	0.5111	1.0397	2.0434
7	0.2224	0.511	1.0396	2.0428
8	0.2223	0.511	1.0398	2.0422
9	0.2224	0.5111	1.0398	2.0448
10	0.2224	0.5112	1.0396	2.0446
Average value	0.2221	0.5111	1.0394	2.0436
Standard deviation	0.0004 (0.18%)	0.0001 (0.02%)	0.0004 (0.04%)	0.0009 (0.04%)

As can be seen from the data in Table 2, the standard deviation values for all concentrations studied are significantly lower than the established acceptability criterion. Thus, the method demonstrates high repeatability and complies with the requirements of validation plan VP-PCM-015 "Determination of iodide ions in various dosage forms using UV spectrophotometry".

Intra-laboratory precision

The "intra-laboratory precision" parameter was determined in a manner similar to the "repeatability" parameter; however, the measurements were carried out on a different day by a different operator using UV WinLab software version 5.1.4 (Perkin Elmer, 2004).

The acceptance criteria were based on the standard deviation (SD) of optical density, which should not exceed ±4%.

The results of the "intra-laboratory precision" assessment are shown in Table 3.

Table 3 – Results of the intra-laboratory precision assessment

No.	**20.0 ppm**	**50.0 ppm**	**100.0 ppm**	**200.0 ppm**
1	0.2087	0.5278	1.0496	2.0533
2	0.2087	0.5275	1.0499	2.0539
3	0.2085	0.5274	1.0497	2.0525
4	0.2038	0.5276	1.0495	2.0529
5	0.2039	0.5277	1.0492	2.0524
6	0.204	0.5276	1.0493	2.0518
7	0.2042	0.5277	1.0491	2.0536
8	0.2042	0.5276	1.0491	2.0513
9	0.2043	0.5273	1.0488	2.0532
10	0.2042	0.5274	1.0491	2.0515
Average value	0.2055	0.5276	1.0493	2.0526
Standard Deviation	0.0022 (1.07%)	0.0002 (0.04%)	0.0003 (0.03%)	0.0009 (0.04%)

The data in Table 3 show that the obtained standard deviation values fall within the established acceptability criteria. Consequently, the method demonstrates satisfactory intra-laboratory precision

The joint statistical analysis of the measurement results obtained by two operators on different days is presented in Table 4.

Table 4 – Precision assessment results over two days

Parameter	20.0 ppm	50.0 ppm	100.0 ppm	200.0 ppm
Average value	0.2147	0.5193	1.0443	2.0481
Standard deviation	4.0%	1.6%	0.5%	0.2%

The obtained results show that the acceptance criteria for the intralaboratory precision parameters are met for all the concentrations studied.

Specificity

The “specificity” parameter was determined by analysing in three replicates of purified water, a 0.05% dextrin solution, a 1% formaldehyde solution, and an FS-1 solution after complete reduction of elemental iodine to iodide ions by formaldehyde in an alkaline medium.

Acceptance criteria:

1. The optical density of purified water, the dextrin solution and the formaldehyde solution at a wavelength of 226 nm must be less than 0.02 units of optical density.

2. The concentration of total iodine in the FS-1 solution must not differ from the specified value by more than ±10%.

The results of the specificity determination are presented in Table 5.

Table 5 – Optical density of various matrices and the FS-1 solution at 226 nm

No.	Water	FS-1	Dextrin	Formaldehyde
1	0.0006	0.9520	0.005	0.000
2	0.0007	0.9519	0.005	0.000
3	0.0008	0.9515	0.006	0.000
Average Value	0.0007	0.9518	0.0053	0.000
Standard Deviation	0.0001	0.0003	0.0006	0.000

As can be seen from Table 5, the optical density values of purified water, a 0.05% dextrin solution and a 1% formaldehyde solution are 0.0007, 0.0053 and 0.000 respectively, which is significantly lower than the established criterion (0.02). Consequently, there is no influence of matrix components on the measurement results.

According to the specifications, the FS-1 solution contains 8.2 g/l of iodine and 12.1 g/l of potassium iodide. A concentration of 12.1 g/l of potassium iodide corresponds to 9.26 g/l of iodide ions (12.1 × 127/166). After complete reduction of elemental iodine to iodide ions, the total concentration of iodide ions in the FS-1 solution is 17.46 g/l.

The FS-1 sample under investigation was diluted 200-fold (0.5 ml to 100 ml). Thus, the calculated concentration of iodide ions in the analysed solution is 0.0873 g/l or 87.3 mg/l.

The measured optical density of the sample solution was 0.9518, which corresponds to an iodide ion concentration of 93.2 mg/l, calculated from the calibration curve (0.9518/0.01021). The difference between the calculated and experimental values does not exceed 10%, which satisfies the established acceptability criterion. Thus, the results obtained confirm the specificity of the developed method.

Linearity

To assess the “linearity” parameter, data obtained from the repeatability study (Table 2) were used, as well as the results of measurements of a standard solution with a concentration of 2 mg/l and purified water. The experimental data were processed and the calibration curve plotted using Origin software.

Based on the data obtained, the parameters of the linear equation and the correlation coefficient were calculated. The tangent of the slope of the calibration line corresponds to the specific optical density of the compound under investigation.

Acceptance criterion: the correlation coefficient (R) must be greater than 0.99. The optical density values used to assess linearity are given in Tables 2 and 6.

Table 6 – Optical density measurement results for linearity assessment (concentrations of 2 and 0 ppm)

No.	Solution with a concentration of 2 mg/l	Purified water
1	0.0161	0.0006
2	0.0163	0.0007
3	0.0164	0.0008
4	0.0166	
5	0.0165	
6	0.0166	
7	0.0168	
8	0.0167	
9	0.0168	
10	0.0171	
Average value	0.0166	0.0007
SD	0.0003 (1.7%)	0.0001

Using Origin software, a graph was plotted showing the dependence of optical density on the concentration of iodide ions in the range 0–200 ppm (Figure 1).

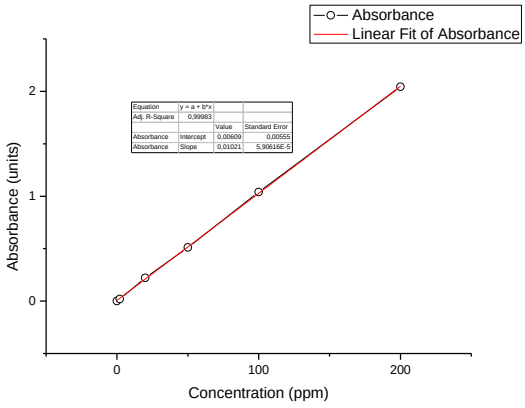

Figure 1 – Dependence of optical density on the concentration of iodide ions in the range 0–200 ppm

The calculated parameters of the linear equation are as follows:

$$Y = a + bx$$

where: a = 0.0061 – the y-intercept;
b = 0.0102 – the tangent of the slope of the calibration line.

The correlation coefficient is R = 0.99983, which exceeds the established acceptability criterion. Thus, the results obtained confirm the linearity of the method across the entire concentration range studied.

Working range

The “operating range” parameter was determined based on linearity data within the experimentally verified concentration range of 0–200 ppm. The permissible range extension is 20%.

Acceptability criterion: the error in determining optical density within the working range must not exceed ±5%.

The results used to determine the working range are presented in Tables 2, 3 and 6.

The baseline noise when measuring purified water is approximately 0.0006 units of optical density. Theoretically, the limit of quantification can start at a level of 0.006 optical density units. However, the optical density of a 0.05% dextrin solution at a wavelength of 226 nm is 0.006 units, which makes an additional contribution to the signal of the solutions being analysed.

At a concentration of 2 ppm, the optical density of the solution is approximately 0.017, which leads to a relative error exceeding 30%. At the same time, for a solution with a concentration of 20 ppm, the optical density is approximately 0.205 and the error contribution of 0.006 corresponds toncentration of iodide ions in various dosage forms using UV spectrophotometry.

Robustness

The “robustness” parameter was assessed by measuring the concentration of the same solutions after storage under ambient conditions for 24 hours. The measurements were carried out by a different operator using a different cuvette. The data presented in Tables 2 and 3 were used for the analysis.

Acceptance criterion: the difference between the mean optical density values within the working range, obtained on different days by different operators, must not exceed 4%. The results obtained are presented in Table 7.

Table 7 – Optical density data extracted from Tables 2 and 3

Parameter	**20 ppm**	**50 ppm**	**100 ppm**	**200 ppm**
Average value (day 1)	0.2221	0.5111	1.0394	2.0436
Standard deviation	0.0004	0.0001	0.0004	0.0009
Average value (day 2)	0.2055	0.5276	1.0493	2.0526
Standard deviation	0.0022	0.0002	0.0003	0.0009
Difference	0.0166	0.0165	0.0099	0.0090
Difference, %	7.5	3.1	0.9	0.4

The data in Table 7 show that the acceptability criterion is met for the range 50–200 mg/l. The differences in mean values for solutions with concentrations of 50, 100 and 200 mg/l are 3.1%, 0.9% and 0.4% respectively.

For the solution with a concentration of 20 mg/l, the difference is 7.5%, which exceeds the established criterion. This indicates that diluted solutions are less stable; consequently, it is recommended that they be analysed on the day of preparation. The summarised results of the validation are presented in Table 8.

Table 8 – Results of the validation of the method for determining the concentration of iodide ions in various dosage forms using UV spectrophotometry

Parameter	**20 ppm**	**50 ppm**	**100 ppm**	**200 ppm**
Accuracy, %	-	-	-	0.08
Repeatability, %	0.18	0.02	0.04	0.04
Intra-laboratory precision, %	4.0	1.6	0.5	0.2
Linearity	R = 0.9998			
Operating range	20–240 ppm			
Stability	Stable			
Specificity	Specific			

The results of the validation are presented in detail in the validation report VR-PCM-015 [15], as well as in papers devoted to the practical aspects of analytical method validation [16–17]. This article examines the key parameters of analytical method validation: accuracy, repeatability, intra-laboratory precision, linearity, working range, specificity and robustness. The results obtained confirm the practical applicability of the developed method.

The method developed and validated is successfully used for the quantitative determination of iodide ions in medicinal substances and preparations developed at the Scientific Centre.

Conclusion

During the study, the analytical method TP-SA-015 ‘Determination of the concentration of iodide ions in various dosage forms by UV spectrophotometry’ [18], in accordance with the requirements of the international ICH Q2(R1)/Q2(R2) guideline and the company’s internal standard operating procedures.

The results obtained showed that the developed method possesses the necessary validation characteristics and meets the established acceptability criteria. The method demonstrates high accuracy and reproducibility of results, as confirmed by low standard deviation values in the assessment of repeatability and intra-laboratory precision. It has been shown that the method exhibits a high degree of linearity within the concentration range under investigation, with a correlation coefficient of $R^2 = 0.9998$.

It has been established that the working range of the method lies within the interval 20–240 mg/L, which ensures the possibility of reliable quantitative determination of iodide ions in the analysed samples. The studies conducted also confirmed the specificity of the method with respect to the matrix components of dosage forms and its resistance to changes in analytical conditions.

The results obtained confirm the suitability of the UV spectrophotometry method for the quantitative determination of iodide ions in drug substances and finished medicinal products. The developed and validated method can be recommended for use in the quality control system for iodine-containing

medicinal products, as well as in the analytical practice of pharmaceutical laboratories.

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ВАЛИДАЦИЯ МЕТОДИКИ ОПРЕДЕЛЕНИЯ КОНЦЕНТРАЦИИ ИОДИД ИОНОВ В РАЗНЫХ ЛЕКАРСТВЕННЫХ ФОРМАХ МЕТОДОМ УФ-СПЕКТРОФОТОМЕТРИИ

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Резюме. Статья посвящена валидации аналитической методики определения иодид ионов в различных лекарственных формах методом УФ-спектрофотометрии. Данное направление является важной частью аналитической химии и системы контроля качества лекарственных средств, в которой чувствительность, специфичность и воспроизводимость метода имеют решающее значение. Актуальность исследования обусловлена необходимостью точного и достоверного количественного определения иодид-ионов в субстанциях и готовых лекарственных препаратах. Валидация проводилась в соответствии с требованиями International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH Q2(R1)/Q2(R2)). *Целью работы* являлось определение валидационных характеристик методики количественного определения иодид-ионов в лекарственных формах методом УФ-спектрофотометрии. *Результаты и обсуждение.* В ходе исследования были оценены следующие параметры: специфичность, линейность, рабочий диапазон, правильность, повторяемость, внутрилабораторная точность и стабильность растворов. Установлено, что рабочий диапазон - от 20 до 240 мг/л, коэффициент корреляции - 0,9998. Метод продемонстрировал высокую чувствительность и соответствие международным требованиям. *Заключение*. Полученные результаты подтверждают линейность методики в исследуемом диапазоне концентраций иодид-ионов. Доказаны высокая чувствительность метода и высокая степень линейности (R^2 = 0.9998). Разработанная методика соответствует своему назначению и может успешно применяться при анализе иодсодержащих лекарственных препаратов. Метод имеет практическую значимость для системы контроля качества фармацевтической продукции.

Ключевые слова: иодид ионы, УФ-спектрофотометрия, метод валидации, ICH Q2, фармацевтический анализ.

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ӘРТҮРЛІ ДӘРІЛІК ФОРМАЛАРДАҒЫ ИОДИД ИОНДАРЫНЫҢ КОНЦЕНТРАЦИЯСЫН УФ-СПЕКТРОФОТОМЕТРИЯ ӘДІСІМЕН АНЫҚТАУ ӘДІСТЕМЕСІН ВАЛИДАЦИЯЛАУ

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Инфекцияға қарсы препараттар ғылыми орталығы АҚ, Алматы, Казахстан

Түйіндеме. Мақалада әртүрлі дәрілік формалардағы иодид иондарының мөлшерін ультракүлгін спектрофотометрия (УФ-спектрофотометрия) әдісімен анықтауға арналған аналитикалық әдістемені валидациялау мәселелері қарастырылған. Бұл бағыт аналитикалық химияның және дәрілік заттардың сапасын бақылау жүйесінің маңызды бөлігі болып табылады, мұнда әдістің сезімталдығы, спецификалығы және қайталанғыштығы шешуші рөл атқарады. Зерттеудің өзектілігі субстанциялар мен дайын дәрілік препараттардағы иодид иондарын дәл әрі сенімді сандық анықтаудың қажеттілігімен негізделген. Валидация жұмыстары International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH Q2(R1)/Q2(R2)) талаптарына сәйкес жүргізілді. *Зерттеудің мақсаты* – дәрілік формалардағы иодид иондарын УФ-спектрофотометрия әдісі арқылы сандық анықтау әдістемесінің валидациялық сипаттамаларын анықтау. *Нәтижелер және талқылау.* Зерттеу барысында келесі параметрлер бағаланды: спецификалық, сызықтылық, жұмыс диапазоны, дұрыстық, қайталанғыштық, зертханаішілік дәлдік, сандық анықтау шегі және ерітінділердің тұрақтылығы. Анықтау нәтижесінде сандық анықтау шегі 20 мг/л құрайтыны, жұмыс диапазоны 20–240 мг/л аралығында екені, корреляция коэффициенті 0,9998-ге тең екені анықталды. Әдіс жоғары сезімталдықты (2,6 мг/л) және халықаралық талаптарға сәйкестігін көрсетті. *Қорытынды.* Алынған нәтижелер зерттелген концентрациялар диапазонында иодид иондары үшін әдістеменің сызықтылығын растайды. Әдістің жоғары сезімталдығы және жоғары сызықтылық дәрежесі (R^2 = 0,9998) дәлелденді. Әзірленген әдістеме өз мақсатына толық сәйкес келеді және иод құрамды дәрілік препараттарды талдауда тиімді қолданылуы мүмкін. Бұл әдіс фармацевтикалық өнімдердің сапасын бақылау жүйесі үшін практикалық маңызға ие.

Түйін сөздер: йодид иондары, УФ-спектрофотометрия, әдіс валидациясы, ICH Q2, фармацевтикалық талдау

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