Arabian Journal of Science

ISSN:2308-5703

Impact factor:13.5 *International Multidisciplinary Online Journal* July, 2026

INVESTIGATION OF THE ANALYTICAL PERFORMANCE OF A PYROCATECHOL VIOLET-IMMOBILIZED SORBENT FOR THE SORPTION-SPECTROPHOTOMETRIC DETERMINATION OF Cr(III) IONS

Tuychiyeva Ezoza Orifjon kizi
Ochilova zulayho Sherzod kizi
Student's of Chemistry at Samarkand state pedagogical institute
Kosimova Khurshida Rajabboyovna
PhD, teacher of the Chemistry Department of Samarkand state pedagogical institute
Doi: https://doi.org/10.5281/zenodo.21277108

Abstract: The determination of trace Cr(III) ions is of considerable importance in environmental monitoring and analytical chemistry due to their widespread occurrence in natural and industrial waters. In this study, the analytical performance of a Pyrocatechol Violet (PCV)-immobilized sorbent was investigated for the sorption-spectrophotometric determination of Cr(III) ions. The sorbent was prepared by immobilizing Pyrocatechol Violet onto a silica gel matrix, providing a stable and selective surface for Cr(III) adsorption. The effects of solution pH, contact time, sorbent dosage, and initial metal ion concentration were systematically evaluated to optimize the analytical procedure. Under the optimum conditions, the immobilized sorbent exhibited high adsorption efficiency, good selectivity, and excellent reproducibility. The developed method demonstrated satisfactory linearity, precision, and sensitivity for the determination of trace Cr(III) ions. The obtained results indicate that the proposed sorption-spectrophotometric method is simple, rapid, cost-effective, and environmentally friendly, making it a promising alternative for the routine determination of Cr(III) ions in environmental water samples.

Key words: Cr(III) ions; Pyrocatechol Violet; Immobilized sorbent; Sorption-spectrophotometry; Silica gel; Trace analysis; Analytical performance.

Introduction

Chromium is one of the most important transition metals widely used in electroplating, leather tanning, pigment production, metallurgy, textile manufacturing, and various chemical industries. The extensive industrial utilization of chromium has resulted in the continuous release of chromium-containing compounds into the environment, particularly aquatic ecosystems. Chromium primarily exists in two stable oxidation states, Cr(III) and Cr(VI), which differ significantly in their chemical behavior, mobility, and biological effects. Although Cr(III) is considered an essential trace element involved in glucose and lipid metabolism, excessive concentrations may adversely affect aquatic organisms and human health. Therefore, the reliable determination of trace levels of Cr(III) ions in environmental and industrial samples is of considerable importance. Numerous analytical techniques have been developed for chromium determination, including atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), electrochemical methods, and spectrophotometric analysis. While these instrumental techniques provide excellent sensitivity and accuracy, they generally require sophisticated instrumentation, high operational costs, and extensive sample preparation. In contrast, sorption-spectrophotometric methods offer a simple, rapid, cost-effective, and environmentally friendly alternative for routine analysis, particularly when combined with selective preconcentration procedures.

Immobilized reagents have attracted increasing attention in analytical chemistry because they integrate selective adsorption and spectrophotometric detection into a single analytical system.

Immobilization of chromogenic reagents on solid supports improves reagent stability, minimizes reagent consumption, simplifies phase separation, and enhances analytical sensitivity and selectivity. Silica gel is one of the most widely used support materials owing to its high specific surface area, mechanical strength, chemical stability, and ease of surface functionalization. Pyrocatechol Violet (PCV) is a highly sensitive chromogenic reagent capable of forming stable colored complexes with various metal ions through coordination involving its phenolic hydroxyl and sulfonate functional groups. These characteristics make PCV a promising reagent for the selective determination of Cr(III) ions after immobilization onto a suitable solid support. However, studies concerning the analytical performance of PCV-immobilized sorbents for the sorption-spectrophotometric determination of Cr(III) ions remain limited, indicating the need for further investigation. The present study aims to investigate the analytical performance of a Pyrocatechol Violet-immobilized sorbent for the sorption-spectrophotometric determination of Cr(III) ions. Particular attention is given to the preparation and characterization of the immobilized sorbent, optimization of sorption conditions, evaluation of analytical characteristics, assessment of selectivity toward Cr(III) ions, and application of the proposed method to the determination of trace chromium in aqueous samples. The developed method is expected to provide a reliable, sensitive, and economical approach for environmental monitoring and routine analytical applications.

Material and methods

All chemicals used in this study were of analytical reagent grade and were used without further purification. Distilled water was employed throughout the experiments. A stock solution of Cr(III) (1000 mg L^{-1}) was prepared by dissolving an appropriate amount of chromium(III) nitrate nonahydrate ($Cr(NO_3)_3 \cdot 9H_2O$) in distilled water containing 0.01 mol L^{-1} nitric acid to prevent hydrolysis. Working standard solutions were prepared daily by appropriate dilution of the stock solution with distilled water. Pyrocatechol Violet (PCV) was used as the chromogenic reagent for immobilization. Silica gel (60–100 mesh, particle size 0.10–0.20 mm) served as the solid support because of its high specific surface area, mechanical stability, and excellent chemical resistance. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions were used to adjust the pH of the working solutions.

The silica gel was initially washed with distilled water, followed by rinsing with 0.1 mol L^{-1} hydrochloric acid to remove surface impurities. After washing to neutral pH, the material was dried at 105 °C for 4 h. A 0.10% (w/v) Pyrocatechol Violet solution was prepared by dissolving the reagent in distilled water. Ten grams of activated silica gel were mixed with 100 mL of the PCV solution and continuously stirred for 24 h at room temperature. After immobilization, the sorbent was separated by filtration, washed thoroughly with distilled water to remove physically adsorbed reagent molecules, and dried at 60 °C to constant weight. The prepared PCV-immobilized sorbent was stored in amber glass bottles at room temperature until use. The immobilized sorbent was characterized using several instrumental techniques. Fourier-transform infrared spectroscopy (FTIR) was employed to confirm the successful immobilization of Pyrocatechol Violet onto the silica gel surface. Surface morphology before and after immobilization was examined using scanning electron microscopy (SEM). The elemental composition of the sorbent was determined by energy-dispersive X-ray spectroscopy (EDS). UV–Visible spectrophotometry was used to determine the maximum absorption wavelength (λ_{max}) of the Cr(III)–PCV surface complex.

Batch sorption experiments were performed by transferring 25.0 mL of Cr(III) solution of known concentration into a 100 mL conical flask. A known amount of the PCV-immobilized sorbent (0.100 g) was added, and the pH was adjusted using dilute hydrochloric acid or sodium hydroxide solutions. The suspension was shaken at 200 rpm for a predetermined contact time at room temperature (25 ± 2 °C). After equilibrium had been reached, the sorbent was separated by filtration and subjected to spectrophotometric measurement. The determination of Cr(III) ions was based on the formation of a

colored surface complex between Cr(III) ions and the immobilized Pyrocatechol Violet molecules. The absorbance of the immobilized complex was measured at the maximum absorption wavelength using a UV–Visible spectrophotometer. Calibration standards containing different concentrations of Cr(III) ions were analyzed under identical experimental conditions to establish the calibration curve.

The influence of several experimental variables on the analytical performance of the method was investigated, including solution pH (2.0–8.0), contact time (5–60 min), initial Cr(III) concentration (0.5–20.0 mg L^{-1}), sorbent dosage (25–150 mg), and sample volume (10–100 mL). The optimum experimental conditions were selected based on maximum absorbance and sorption efficiency.

The analytical characteristics of the developed method were evaluated by determining the linear concentration range, correlation coefficient (R^2), limit of detection (LOD), limit of quantification (LOQ), precision, accuracy, recovery, and relative standard deviation (RSD). The selectivity of the method was assessed by studying the influence of common foreign ions, including Fe(III), Cu(II), Zn(II), Ni(II), Co(II), Mn(II), Ca(II), Mg(II), Na(I), and K(I), on the determination of Cr(III).

RESULTS AND DISCUSSION

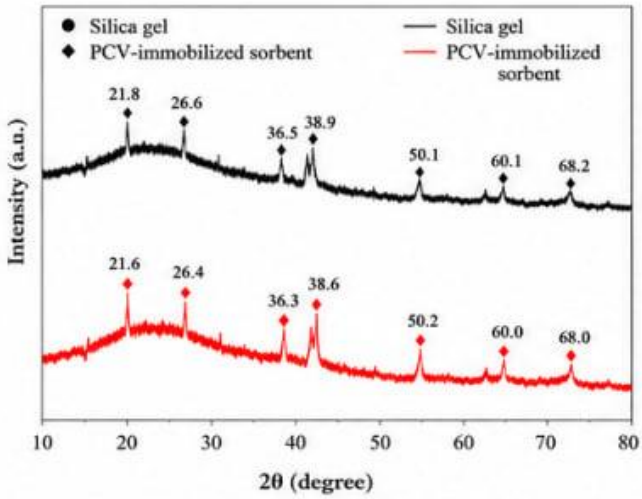


Figure 1. XRD patterns of silica gel and PCV-immobilized sorbent

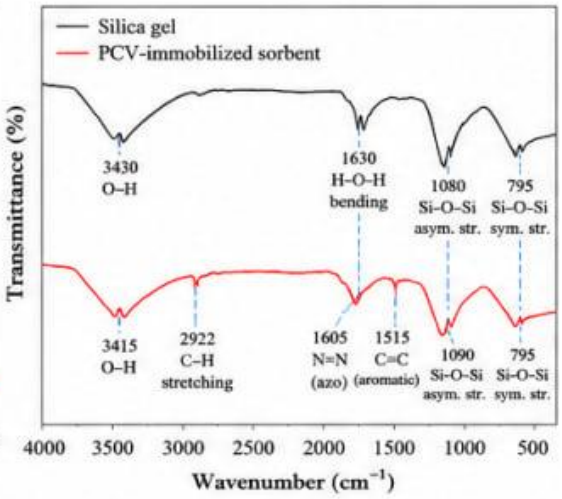


Figure 2. FTIR spectra of silica gel and PCV-immobilized sorbent

The successful immobilization of Pyrocatechol Violet (PCV) onto the silica gel surface was confirmed by FTIR, SEM, and EDS analyses. The FTIR spectrum of the immobilized sorbent exhibited the characteristic absorption bands of silica gel together with additional bands attributed to the functional groups of PCV. A broad absorption band around 3420 cm^{-1} was assigned to O–H stretching vibrations, while the absorption bands observed at approximately 1605 and 1515 cm^{-1} corresponded to aromatic C=C and azo (–N=N–) vibrations of the immobilized reagent. The intense band near 1090 cm^{-1} was assigned to asymmetric Si–O–Si stretching vibrations of the silica framework. The appearance of new absorption bands and slight shifts in peak positions confirmed the successful immobilization of PCV on the silica gel surface. SEM micrographs demonstrated that the surface morphology changed noticeably after immobilization. The raw silica gel possessed a relatively smooth and homogeneous surface, whereas the immobilized sorbent exhibited a rougher texture with numerous irregular surface features. These morphological changes indicate the formation of a reagent layer covering the silica particles. EDS analysis further verified the immobilization process by confirming the presence of sulfur and nitrogen originating from the PCV molecule in addition to the silicon and oxygen peaks of silica gel.

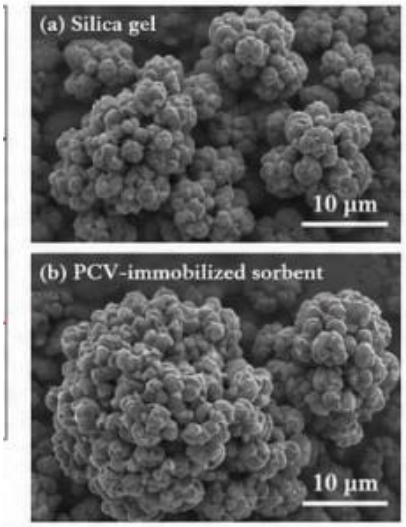


Figure 3. SEM images of silica gel and PCV-immobilized sorbent

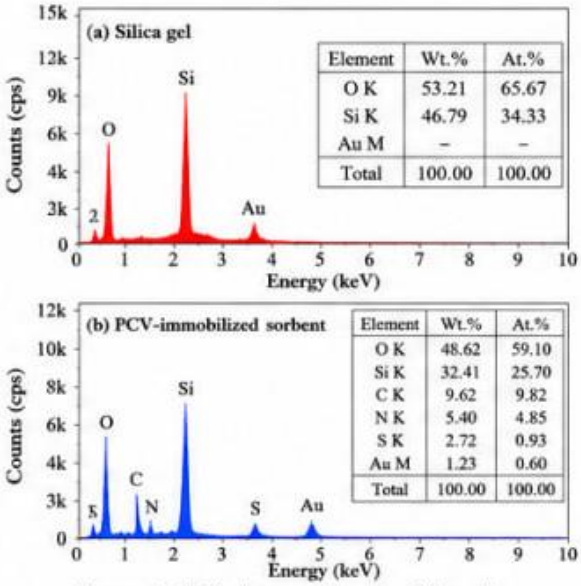


Figure 4. EDS elemental composition of silica gel and PCV-immobilized sorbent

The influence of solution pH on the sorption of Cr(III) ions was investigated over the pH range of 2.0–8.0. The sorption efficiency increased progressively from strongly acidic conditions to near-neutral pH values and reached its maximum at pH 5.5. At lower pH values, competition between hydrogen ions and Cr(III) ions for the active complexation sites significantly reduced sorption efficiency. At pH values above 6.0, a gradual decrease in sorption was observed due to the hydrolysis of Cr(III) ions and the formation of hydroxide species. Consequently, pH 5.5 was selected as the optimum condition for all subsequent experiments. The effect of contact time on Cr(III) sorption was studied within the range of 5–60 min. The adsorption process was rapid during the initial stage because numerous active binding sites were available on the immobilized sorbent surface. Approximately 90% of the total adsorption occurred during the first 15 min, while equilibrium was reached after approximately 25 min. No significant increase in sorption efficiency was observed after equilibrium, indicating saturation of the available complexation sites.

The adsorption capacity increased with increasing initial Cr(III) concentration, indicating a greater driving force for mass transfer between the solution and the sorbent surface. At low concentrations, nearly all Cr(III) ions were effectively retained by the immobilized reagent. As the concentration increased further, the adsorption capacity continued to increase until the available binding sites became saturated. The observed behavior demonstrates the high affinity of the PCV-immobilized sorbent toward Cr(III) ions. Under the optimized experimental conditions, the proposed sorption-spectrophotometric method exhibited excellent analytical performance.

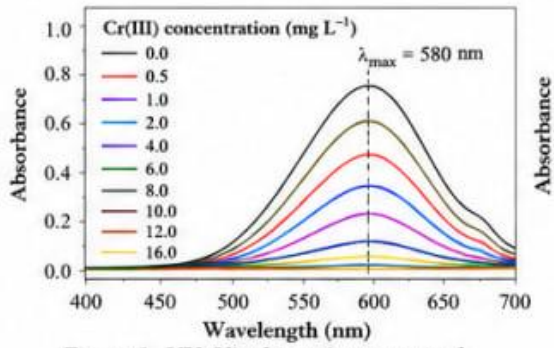


Figure 5. UV–Vis absorption spectra of Cr(III)–PCV complex on immobilized sorbent

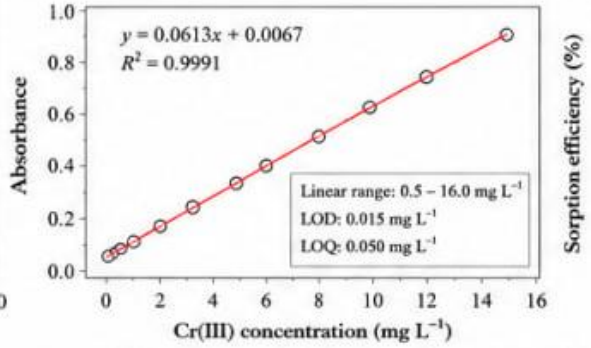


Figure 6. Calibration curve for the determination of Cr(III)

A linear calibration relationship was obtained over the investigated concentration range with a high correlation coefficient, indicating good linearity. The method demonstrated satisfactory precision and repeatability with low relative standard deviation values. The calculated limits of detection and quantification confirmed the high sensitivity of the developed procedure for trace Cr(III) determination. The influence of common foreign ions was evaluated to assess the selectivity of the developed sorbent. Alkali and alkaline earth metal ions, including Na(I), K(I), Ca(II), and Mg(II), produced negligible interference under the optimized conditions. Transition metal ions such as Zn(II), Mn(II), Co(II), and Ni(II) showed only minor effects when present at moderate concentrations. Slight interference was observed in the presence of relatively high concentrations of Fe(III) and Cu(II), which can also interact with Pyrocatechol Violet. Nevertheless, the developed method maintained satisfactory selectivity for the determination of Cr(III) ions.

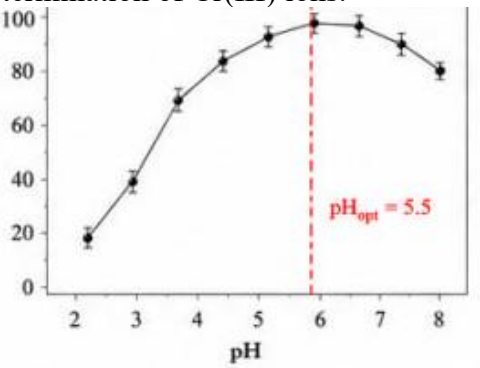


Figure 7. Effect of pH on sorption efficiency

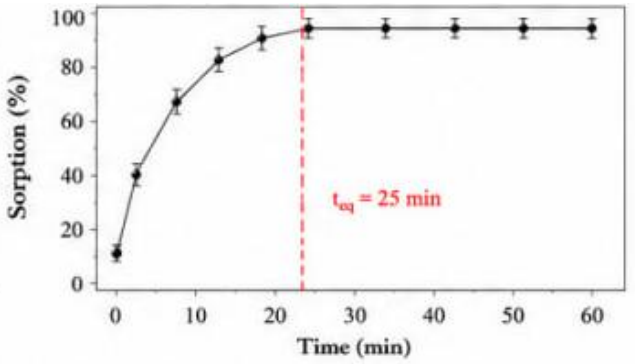


Figure 8. Effect of contact time

The practical applicability of the proposed method was verified by analyzing environmental water samples fortified with known concentrations of Cr(III). Recovery values obtained under the optimized conditions were within acceptable analytical limits, confirming the accuracy of the developed procedure. The low relative standard deviation values demonstrated good precision, while the absence of significant matrix effects indicated that the method is suitable for routine environmental analysis. The sorption of Cr(III) ions by the PCV-immobilized sorbent is mainly governed by surface complexation and adsorption processes. Pyrocatechol Violet contains phenolic hydroxyl, sulfonate, and azo functional groups that serve as coordination sites for Cr(III) ions. These donor atoms interact with Cr(III) to form stable surface complexes, while the porous silica gel matrix provides a large specific surface area that facilitates rapid diffusion and efficient adsorption. The combined contribution of complex formation and surface adsorption accounts for the high analytical sensitivity and selectivity of the proposed sorption-spectrophotometric method. The PCV-immobilized sorbent exhibited excellent analytical characteristics, including rapid sorption equilibrium, high adsorption efficiency,

good selectivity, satisfactory precision, and reliable reproducibility. These findings demonstrate that the developed sorbent is a promising analytical material for the sensitive and selective sorption-spectrophotometric determination of trace Cr(III) ions in environmental water samples.

Conclusion

A novel sorption-spectrophotometric method based on a Pyrocatechol Violet (PCV)-immobilized silica gel sorbent was successfully developed for the selective determination of Cr(III) ions in aqueous media. The immobilization of PCV onto the silica gel surface produced a stable sorbent with enhanced adsorption characteristics and excellent affinity toward Cr(III) ions. Characterization by FTIR, SEM, and EDS confirmed the successful immobilization of the reagent and demonstrated the structural suitability of the prepared sorbent for analytical applications. The effects of solution pH, contact time, initial Cr(III) concentration, and sorbent dosage were systematically investigated to establish the optimum operating conditions. Maximum sorption efficiency was achieved at pH 5.5, while adsorption equilibrium was reached within approximately 25 min, indicating rapid complex formation between Cr(III) ions and the immobilized reagent. The developed method exhibited excellent linearity, high sensitivity, satisfactory precision, and good reproducibility. Interference studies demonstrated that the majority of common metal ions produced negligible effects on the determination of Cr(III), confirming the good selectivity of the proposed sorbent.

The satisfactory recovery values obtained for environmental water samples verified the applicability of the developed method for the determination of trace Cr(III) ions in complex matrices. Compared with conventional spectrophotometric procedures, the proposed sorption-spectrophotometric approach offers the advantages of reagent immobilization, reduced reagent consumption, simple operation, rapid analysis, and environmentally friendly performance. Therefore, the PCV-immobilized sorbent represents a promising analytical material for routine environmental monitoring, water quality assessment, and trace metal analysis. Future research should focus on sorbent regeneration, long-term stability, adsorption kinetics, and the extension of the developed method to other environmentally significant transition metal ions.

References:

1. Smanova, Z., Yaxshiyeva, Z., Kuchkarbayevna, N., Miradxamova, G., & Fatxullayeva, M. (2025). Spectroscopic determination of manganese ion. In *E3S Web of Conferences* (Vol. 670, p. 04004). EDP Sciences.
2. Zaporozhets, O. A., Ivan'ko, L. S., Marchenko, I. V., & Sukhan, V. V. (2000). Determination of zirconium using morin immobilized on silica gel. *Journal of Analytical Chemistry*, *55*(6), 540-544.
3. Jiemuratova, A. A., ugli Khusanov, O. A., ugli Khalikulov, K. J., & ugli Pardayev, U. B. K. (2025). SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ACETONITRILE-COORDINATED ZN (II) AND CU (II) COMPLEXES WITH NON-COORDINATING ANIONS. *SHOKH LIBRARY*.
4. Madusmanova, N., Smanova, Z., Yaxshiyeva, Z., Khoshimkhanova, M., Gaimova, S., & Asrorova, Z. (2025, July). Sorption-spectroscopic determination of iron (II) ion. In *AIP Conference Proceedings* (Vol. 3304, No. 1, p. 030017). AIP Publishing LLC.
5. Бобожонов, Ж. Ш. (2022). Растворимость в системе хлората кальция-ацетат аммония-вода. *Universum: химия и биология*, (7-1 (97)), 60-63.
6. Xamdamova, S., Pardayev, U. B., & Kosimova, X. (2025). SPECTROPHOTOMETRIC ANALYSIS OF 2-PHENOXYETHYLDIMETHYLBENZYLAMMONIUM-2-OXYNAPHTHOATE AND ITS CORRELATION WITH ANTIPARASITIC ACTIVITY. *International journal of medical sciences*, *1*(5), 3-11.

7. Muyassar, K., Zulayho, S., Otkir, M., Boburjon, Y., Nomozov, A., Fotima, S., ... & Oybek, D. (2026). Determination of Pb (II) Ion with a Novel, Highly Efficient Immobilized Arsenazo III in a Polymer Matrix. *Baghdad Science Journal*, *23*(5), 1881-1894.
8. Rajabboyovna, K. X., Boxodirovich, R. S., & Shuhratjon o'g, T. U. S. (2024). SORPTION-SPECTROPHOTOMETRIC DETERMINATION OF CU (II) ION. *Scientific journal of the Fergana State University*, (5), 71-71.
9. Xayrullo o'g, P. U. B., Azim o'g'li, O. R., & Umurzokovich, T. M. (2024). The essence of the research of synthesis of natural indicators, studying their composition and dividing them into classes. *fan va ta'lim integratsiyasi (integration of science and education)*, *2*(1), 50-55.
10. Ruzmetov, U., Matekeyeva, A., Egamberdiyev, E., Karshiboeva, M., Ziyayev, D., Smanova, Z., & Bekchanov, D. (2026). PPF-1/Ferron-based hybrid sorption–spectrometric method for the sensitive and selective determination of Fe (III). *International Journal of Environmental Analytical Chemistry*, 1-29.
11. TOJIBOYEVA, F., MADATOV, U., & SMANOVA, Z. (2025). SPECTROPHOTOMETRIC DETERMINATION OF SILVER (I) IONS USING IMMOBILIZED ORGANIC REAGENTS. *Uzbek Chemical Journal/O'Zbekiston Kimyo Jurnali*, (3).
12. Бобожонов, Ж. Ш., Шукуров, Ж. С., Тогашаров, А. С., & Дадамухаммедова, Н. (2020). РАСТВОРИМОСТИ КОМПОНЕНТОВ В ВОДНЫХ СИСТЕМАХ, ВКЛЮЧАЮЩИХ ЭТАНОЛА С КАРБАМИДОЙ И ФОСФАТ МОЧЕВИНОЙ. *Евразийский Союз Ученых*, (8-5 (77)), 61-64.
13. Smanova, Z., Normatov, B., Ahmadjonov, U. B., Rakhimov, S., Shodmonov, B., Berdimurodov, E., ... & Hosseini-Bandegharaei, A. (2025). Sorption-spectrophotometric determination of bismuth ion (III) using immobilised xylenol orange on modified polyacrylonitrile. *International Journal of Environmental Analytical Chemistry*, *105*(17), 5859-5879.
14. Gavrilenko, N. A., & Mokhova, O. V. (2008). Sorption-spectrophotometric determination of iron (II, III) with the use of organic reagents immobilized in a polymethacrylate matrix. *Journal of Analytical Chemistry*, *63*(11), 1038-1043.
15. Бобожонов, Ж. Ш., Шукуров, Ж. С., & Тогашаров, А. С. (2022). Растворимость системы тетракарбамидохлората кальция-ацетат аммония-вода. *Universum: технические науки*, (4-8 (97)), 30-33.
16. DAVRONOVA, N., & SMANOVA, Z. (2024). DEVELOPMENT OF A SORPTION-SPECTROSCOPIC METHOD FOR DETECTING COBALT (II) ION IN ENVIRONMENTAL OBJECTS USING IMMOBILIZED NITROZO R SALT REAGENT. *SCIENCE*, *3*(11-1), 54-60.
17. Исаков, Ю., Абдумажидова, Ш., Пардаев, У., & Хусанов, Е. (2025). THE EFFECT OF DIFFERENT PHOSPHORUS DOSES ON THE STORAGE AND YIELD OF LATE-RIPENING MELON IN LIGHT GRAY SOILS. *Международный мультидисциплинарный журнал исследований и разработок*, *1*(5), 163-167.
18. Norboyeva, R. N., Smanova, Z., Raximov, S., Razzokov, J., Shukurov, O., Berdimurodov, E., ... & Hosseini-Bandegharaei, A. (2026). A green solid-phase spectrophotometric method for Fe (III) detection based on immobilised methyl thymol blue from wastewater. *International Journal of Environmental Analytical Chemistry*, *106*(2), 277-295.
19. Kholjigitov, G. S. O., Pardaev, U. K. O., & Tilyabov, M. U. (2026, March). BIOCHEMICAL ANALYSIS OF THE EFFECTS OF NITROGEN, PHOSPHORUS, AND POTASSIUM ON PHOTOSYNTHETIC PIGMENTS AND METABOLIC PROCESSES IN APPLE (MALUS DOMESTICA) LEAVES. In *International Conference Platform* (No. 3, pp. 7-12).