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A Cost-Effective, Self-Masking Spectrophotometric Platform for Selective Uranium Monitoring in Environmental Waters

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Abstract

Sustainable and cost-effective UV-Vis spectrophotometric methods have been developed to detect uranium in aqueous solutions by utilizing meso-tetra(4-carboxyphenyl)porphyrin (C4TPP) as an optical sensor. Optimization results indicate that a 0.05 M triethanolamine (TEA) buffer at pH 6 establishes the ideal environment for accurate uranium quantification down to 0.083 ppm. Significantly, the TEA buffer functions as a natural masking agent for competing metal ions like ($^{2+}$Ni, $^{3+}$Fe, $^{2+}$Pb, $^{2+}$Zn, and $^{2+}$Co) without requiring toxic reagents such as EDTA, while sustaining stable detection performance across varying operational temperatures.

Keywords Uranium Quantification. meso-Tetra(4-carboxyphenyl) Porphyrin (C_4TPP). UV-V spectrophotometry.

Introduction

Uranium monitoring in environmental, and biological matrices is critical due to its radiological and chemical toxicity. Over the past decades, numerous analytical methodologies have been optimized to enhance sensitivity and selectivity across diverse sample types. Early approaches heavily relied on conventional spectrophotometry combined with separation protocols. For instance, [1] utilized ion-exchange chromatography with Arsenazo (III) to determine trace uranium(3.2 ±0.2ppb) in seawater, while [2] extended column chemical separations to evaluate natural waters and solid samples within a 0.25–0.50 ppb determination limit.

To overcome matrix interferences in aqueous environments, surfactant-assisted extractions were introduced. [3] investigated a cloud point extraction process using a mixed micelle system (CTAB/Triton X-114) to isolate U(VI) across a 0.20–10.00ppb dynamic range. Similarly, [4] employed 2-(2-thiazolylazo)-p-cresol (TAC) with surfactants for mine drainage analysis, validating their results against X-ray fluorescence. Concurrently, multi-element and advanced spectroscopic techniques expanded the analytical scope. [5] developed a chemo metric UV-Vis method for the simultaneous determination of uranium (0.10–21.00ppb) and thorium(0.25–18.50ppb) at pH 3.0. Radiometric and mass spectrometry variants also advanced trace detection; [6] applied non-destructive gamma spectrometry (93keV of ^{234}Th) to quantify ^{238}U in ore samples, while[7] utilized sector-field ICP-MS (ICP-SFMS) for ultra-trace plutonium and ^{235}U isotopic ratios in contaminated soils.

Furthermore, [8]advanced rapid extractive scintillation methodologies specifically optimized for accurate and efficient uranium measurement in complex soil samples.

Beyond direct uranium quantification, the diversification of optical chemo sensors enabled [9] to detect water pollutants like pentachlorophenol down to 0.5ppb via Soret band shifts, whereas [10] focused on integrating sample concentration steps into rapid colorimetric assays for biological fluids.

Despite this technical evolution, contemporary analytical chemistry faces shifting bottlenecks, specifically regarding severe matrix effects in highly concentrated industrial streams and the need for high-selectivity in vivo monitoring. To address these modern demands, breakthroughs in 2024 and 2025 introduced advanced fluorescent systems and refined matrix-matching spectroscopic protocols. On one hand, a highly sensitive optical sensor exploiting the Aggregation-Induced Emission (AIE) phenomenon designated as [11] achieved a remarkable detection limit of 39.4nM (9.38ppb) for uranyl ions ($^{+2}UO_2$), successfully bypassing transition metal interferences via an EDTA/CyDTA mask for real-time field analysis and live-cell imaging. On the other hand, managing high-concentration industrial regimes characterized by severe nitrate interferences, an innovative matrix-matching UV-Vis spectroscopic method was pioneered [12] . By utilizing a thermal evaporation step and reconstitution in a controlled nitric acid matrix, this approach completely eliminates nitrate background discrepancies, yielding an error rate below 1% for uranium concentrations up to 64g/L when monitoring key peaks at 403nm, 414 nm, and 426 nm.

In the context of advanced optical chemo sensors and organic ligands, recent investigations have highlighted the remarkable efficacy of porphyrin derivatives and macrocyclic frameworks—particularly *meso*-tetra(4-carboxyphenyl) porphyrin ($C_4TPPC_4TPPC_4TPP$) and its derived metal-organic architectures—as versatile molecular recognition platforms[13]; [14]; [15]. Driven by their extended π\piπ-conjugated systems and coordinating carboxylate groups, these compounds exhibit distinct Soret band shifts and prominent absorbance modulations upon selective coordination with uranyl ions ($^{+2}UO_2$ $^{+2}UO2$ $^{+2}UO2$) in aqueous media. Capitalizing on these sharp spectral signatures

provides a robust, cost-effective pathway to enhance both the sensitivity and selectivity of UV-Vis spectrophotometric methods for uranium quantification.
Consequently, while cutting-edge fluorescent frameworks and covalent organic frameworks push the boundaries of trace environmental detection through rapid signal transduction and ultra-low detection limits [16]; [17]; [18], refined matrix-matching spectrophotometry and targeted organic ligand coordination remain indispensable for handling complex industrial streams. To bridge the remaining research gaps in analytical simplicity and cost-efficiency, this study develops an optimized, highly selective methodology utilizing C_4TPP that balances superior analytical performance with operational ease for routine laboratory applications.

Experimental

Materials and Reagents

A stock solution of meso-tetra(4-carboxyphenyl) porphyrin (C_4TPP; $C_{48}H_{30}N_4O_8$; 97% purity; molecular weight 790.8 g mol^{-1}) was prepared at a concentration of 1.0 mg mL^{-1} using deionized water. C_4TPP was purchased from Frontier Scientific (Logan, UT, USA) and used without further purification. The chemical architecture of C_4TPP is structurally illustrated in **Fig. 1.**
Complete dissolution was achieved by the dropwise addition of 1 m NaOH under continuous stirring. The resulting solution was stored under refrigeration and protected from light by wrapping the container with aluminum foil to minimize photo degradation.
Three buffer systems were investigated to evaluate their effect on the spectrophotometric response of C4TPP toward uranium: tri ethanolamine (TEA), Sorensen's phosphate buffer (PB), and Tris buffer (TB). TEA buffer was prepared using tri ethanol amine (99% purity, Tedia, USA). Sorensen's phosphate buffer was prepared by mixing appropriate volumes of 0.5 M Na_2HPO_4 and 0.5 M NaH_2PO_4 solutions. Tris buffer was prepared by dissolving tris (hydroxymethyl)amino methane (99%, ACROS) in deionized water, followed by adjustment of the pH using HCl.
A 1000 mg L^{-1} U_3O_8 stock solution, prepared from pitchblende in 5% HNO_3, was provided by the Jordan Atomic Energy Commission (JAEC). Uranium working solutions were prepared daily by appropriate dilution of the stock solution with deionized water. Buffer working solutions with concentrations of 0.025, 0.050, and 0.100 mole L^{-1} were also prepared daily by dilution. Absolute ethanol (99.9%) and Hellmanex II were used for cleaning the quartz cells prior to measurements.

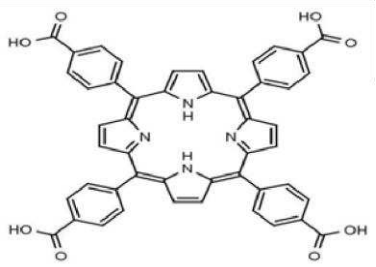


Fig. 1: The structure of the Meso -Tetra (4-carboxyphenyl) porphyrin (C_4TPP).

UV–Visible Spectrophotometric Measurements

Absorption measurements were carried out using a Shimadzu UV-2450 UV–Visible spectrophotometer equipped with a 10-mm path-length quartz cell. Spectra were recorded at a wavelength interval of 0.1 nm. For each measurement, a fixed aliquot of the C4TPP stock solution was added to 3.0 mL of the selected buffer directly in the quartz cell. The corresponding buffer solution was used as the blank reference.

The interaction between C_4TPP and uranium was investigated by recording the absorption spectrum of the C_4TPP solution before and after successive additions of the uranium working solution. The uranium concentration was varied by cumulative additions of the U_3O_8 working solution. The exact volume of each addition was determined according to the target uranium concentration used for the calibration experiments.

Spectral Data Treatment and Calibration

The spectral changes resulting from the interaction between C4TPP and uranium were evaluated using difference spectra. The change in absorbance (ΔA) was calculated by subtracting the spectrum of uncomplexed C_4TPP from that of the corresponding uranium-containing solution according to:

$$\Delta A = A(C_4TPP + U) - A(C_4TPP)$$

Spectral data were processed using UV Probe software. The relative absorbance response ($\Delta A/I_0$) at selected wavelengths was plotted as a function of uranium concentration to establish the analytical calibration curves.

Optimization of Experimental Conditions

The experimental conditions affecting the spectrophotometric response of C_4TPP toward uranium were optimized systematically in two stages. In the first stage, the pH was maintained at 6.0 while the effects of buffer composition and concentration were investigated. TEA, Tris, and phosphate buffers were evaluated at concentrations of 0.025, 0.050, and 0.100 mole L^{-1}.

Following selection of the most suitable buffer system and concentration, the effect of pH was investigated in the range of 5.0–7.0, using pH values of 5.0, 5.5, 6.0, 6.5, and 7.0. The optimum conditions were selected based on the maximum change in absorbance (ΔA), corresponding to the highest analytical sensitivity toward uranium.

Under the optimized conditions, a calibration curve was constructed by plotting the spectrophotometric response against uranium concentration. The effect of temperature on the analytical response was subsequently evaluated to assess the stability of uranium detection under different temperature conditions.

Results and Discussion

Optimization of Buffer Conditions

The systematic optimization of these physicochemical parameters underscores the critical role of the chemical microenvironment in achieving high sensitivity and selectivity for uranyl ion complexation. Controlling the solution pH and buffer concentration ensures that the coordinating functional groups of C_4TPP maintain their optimal reactive states, thereby maximizing molar absorptivity and inducing a distinct, stable shift in the Soret band. Furthermore, the observed thermal robustness and inherent resistance against common matrix interferences highlight the structural stability of the formed porphyrin-uranium coordination complexes. These findings align with recent developments in optical chemical sensors, which emphasize the design of robust, self-masking chromogenic probes capable of performing reliable, direct trace analysis in complex environmental matrices without requiring tedious separation steps.

Effect of Buffer Types

To evaluate complexation behavior, the interaction between U_3O_8 and the C_4TPP ligand was investigated across three distinct media: Triethanolamine (TEA), Phosphate Buffer (PB), and Tris Buffer (TB), each standardized at 0.05 M and pH 6. Differential absorption measurements demonstrated distinct Soret-band shifts following uranium coordination. Notably, the TEA buffer system (Fig. 3) exhibited superior spectral resolution and enhanced signal intensity, marked by a characteristic differential peak at 434.8 nm and a trough at 414.8 nm. In contrast, the phosphate buffer system (Fig. 2) yielded a peak at 436.4 nm and a trough at 414 nm, while the tris buffer system (Fig. 4) displayed a peak at 438.2 nm and a trough at 414.8 nm.

This pronounced performance in the TEA medium stems from the favorable microenvironment fostered by its tertiary amine matrix during complexation. Beyond functioning as an effective pH stabilizer, TEA acts as a weak co-coordinating auxiliary agent that suppresses the hydrolysis and undesirable polymerization of uranyl species ($^{+2}UO_2$) at pH 6.

Consequently, this prevents matrix interference and ensures optimal deprotonation of the carboxylate groups on the C_4TPP ligand, thereby facilitating efficient, stoichiometric binding and sharp Soret band transitions[19]; [20].

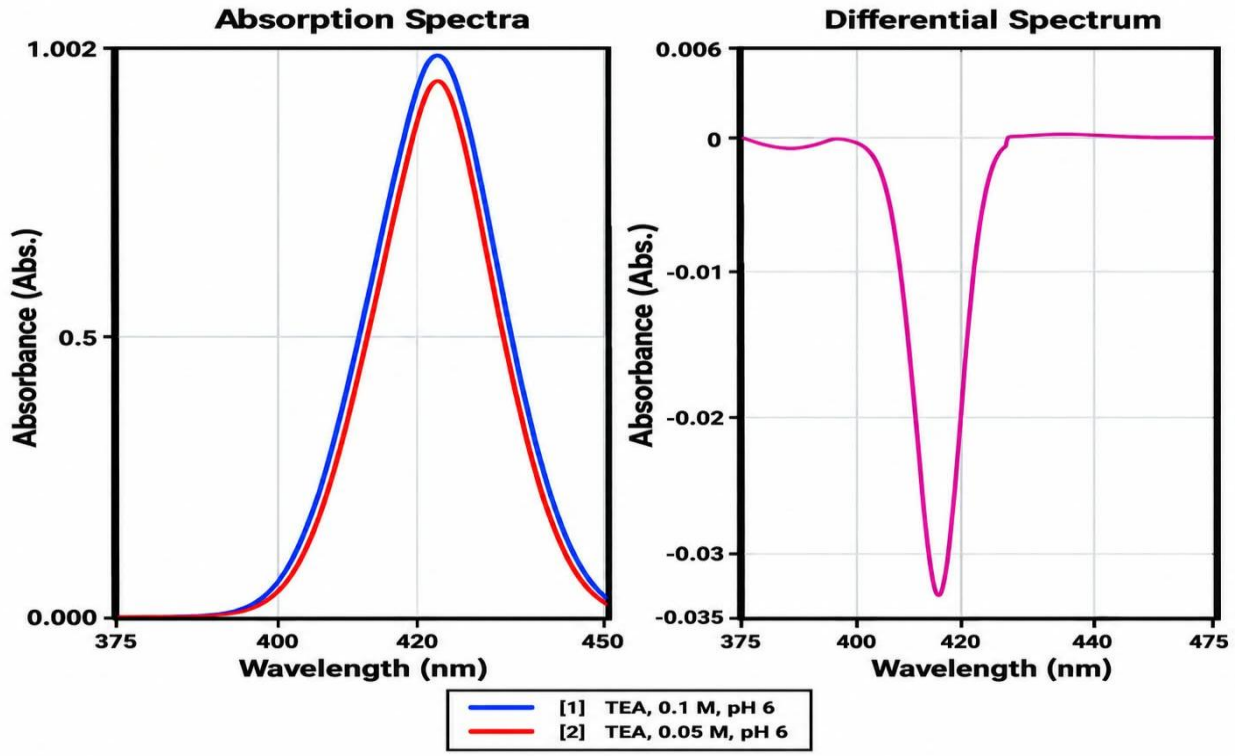


Fig. 2: Fig 4.1 (A) The absorbance spectrum of (C4TPP) in the absence of uranium (trace-1) and in the presence of 0.166 ppm of U_3O_8(trace-2) in 0.05 M PB at pH 6 and (B) the change in absorbance resulting from C4TPP + 0.166 ppm U3O8 minus C4TPP shows a peak at (436.4) nm and a trough at (414) nm.

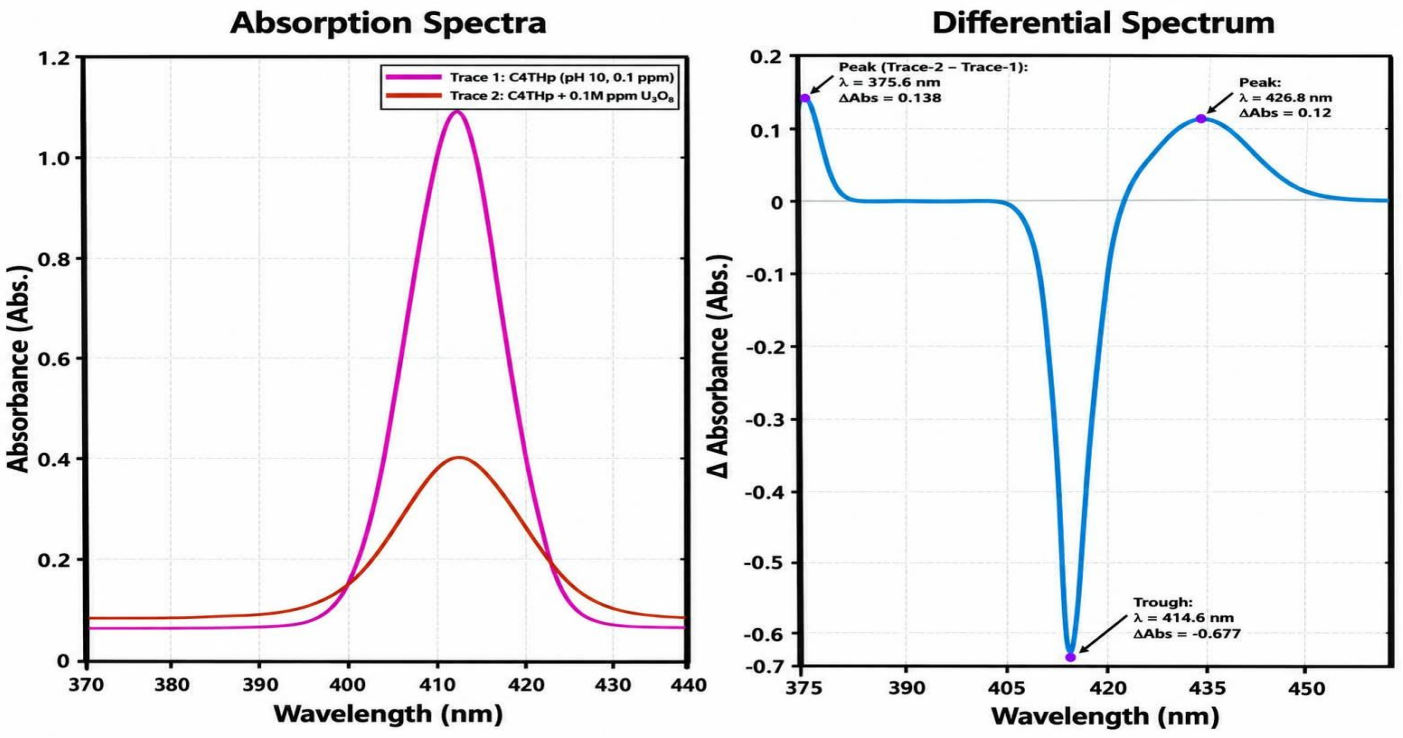


Fig. 3 (A): The absorbance spectrum of (C_4TPP) in the absence of uranium (trace-1) and in the presence of 0.166 ppm of U_3O_8 (trace-2) in 0.05 M TEA buffer at pH 6 and (B) the change in absorbance resulting from C_4TPP+0.166 ppm U_3O_8 minus C_4TPP shows a peak at (434.8) nm and a trough at (414.8) nm.

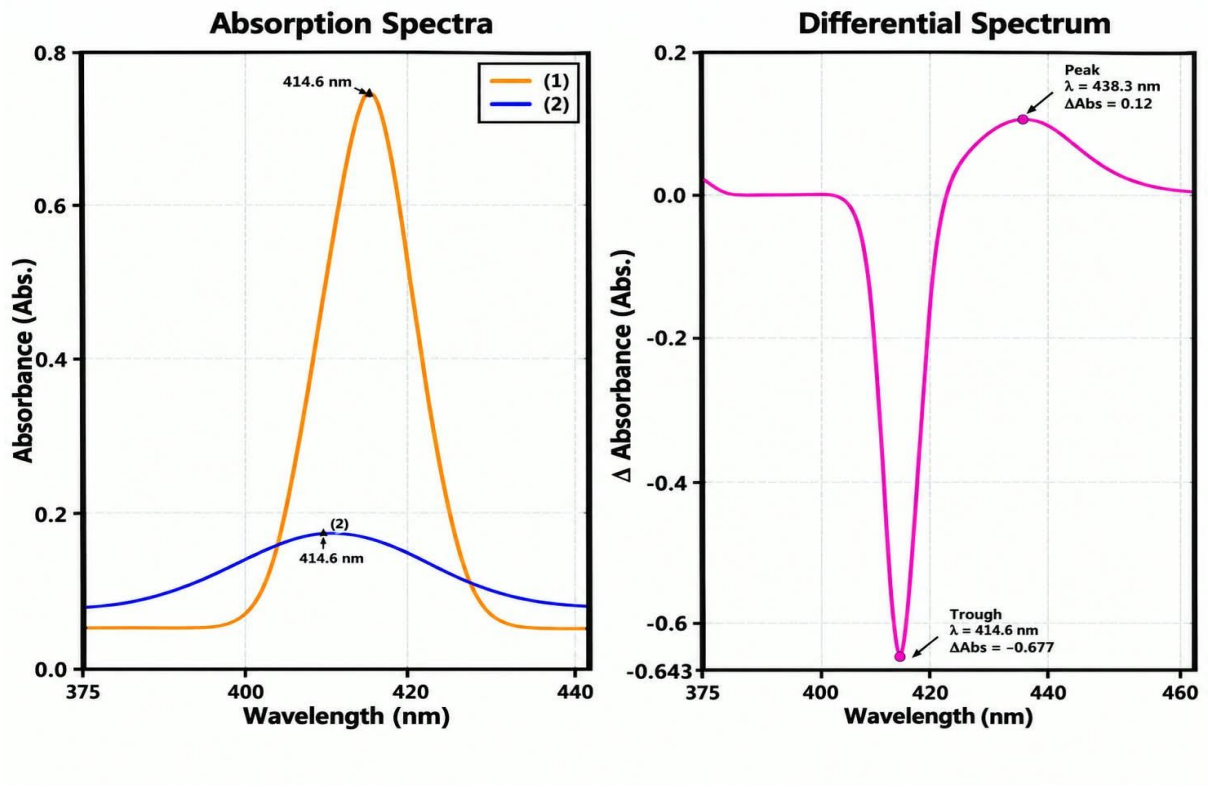


Fig. 4 :(A) The absorbance spectrum of (C_4TPP) in the absence of uranium (trace-1) and in the presence of 0.166 ppm of U_3O_8 (trace-2) in 0.05 M TB buffer at pH 6 and (B) the change in absorbance resulting from C_4TPP+0.166 ppm U_3O_8 minus C_4TPP shows a peak at (438.2) nm and a trough at (414.8) nm.

concentration (**Fig. 5 and Fig. 6**) confirmed that TEA provides superior complexation stability and the highest net absorbance response for equal additions of U_3O_8. This enhanced affinity aligns with fundamental coordination chemistry principles and modern structural design concepts in macrocyclic and framework-based recognition platforms, where tailored nitrogen and oxygen-donating microenvironments optimize metal-ligand coordination kinetics [13]; [17].

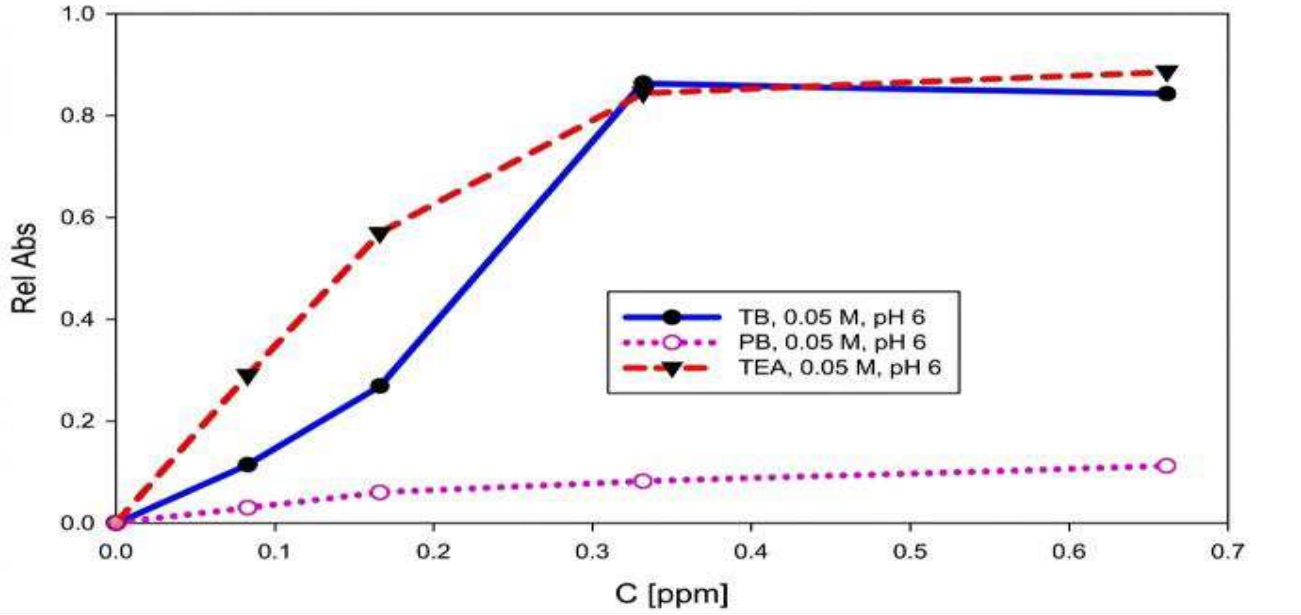


Fig. 5: The change in absorbance as a function of uranium concentration (C) in [ppm] for different types of buffer at pH 6.

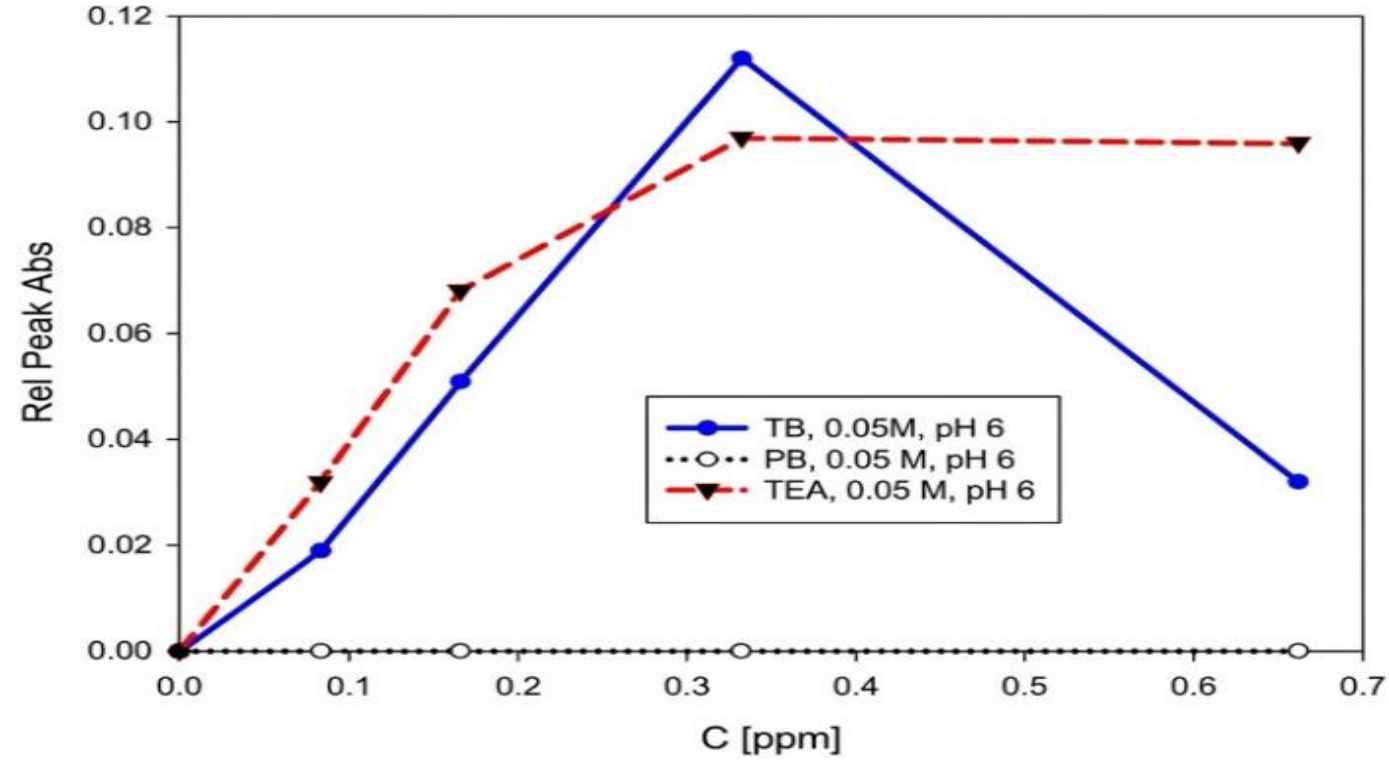


Fig. 6: The change in peak absorbance as a function of uranium concentration for different types of buffer at pH 6.

Effect of Buffer Concentration

Varying concentrations of the TEA buffer (0.1 M, 0.05 M, and 0.025 M at a fixed pH of 6) demonstrated distinct performance variations. As depicted in **Fig. 7 and Fig. 8**, the 0.05 M TEA solution achieved the highest net absorbance change and optimal stability across the tested concentration range. Lower concentrations (0.025 M) failed to provide adequate buffering capacity, whereas higher concentrations (0.1 M) induced ionic strength competition that hindered ligand-metal coordination efficiency. Consequently, 0.05 M was established as the optimal concentration, balancing ionic stability with sensitivity, which is consistent with optimized parameters reported in contemporary spectroscopic and extraction studies [15]

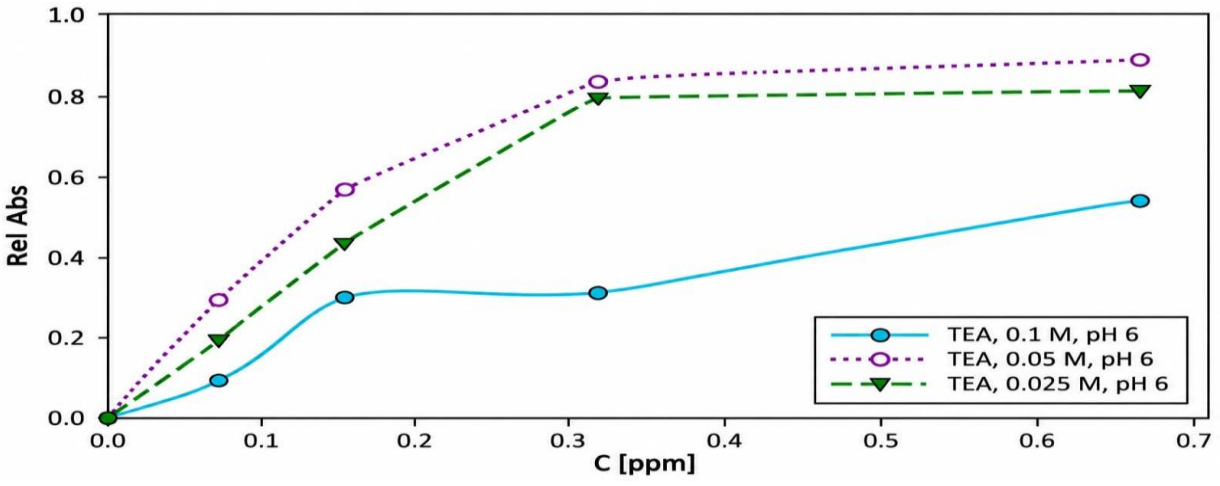


Fig. 7: The change in absorbance (Rel Abs) as a function of uranium concentration (C) in[ppm] at pH 6 for different concentrations of TEA buffer.

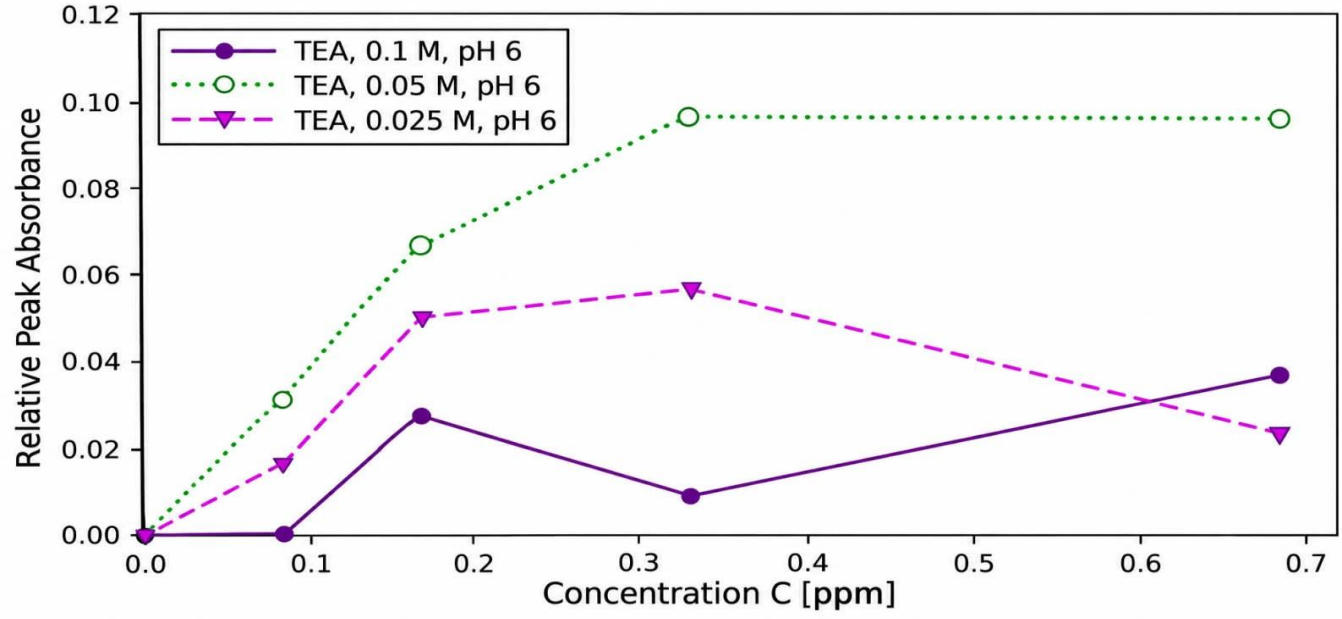


Fig. 8: The change in peak absorbance as a function of uranium concentration (C) in [ppm] at pH 6 for different concentrations of TEA buffer.

Effect of Solution pH

Solution pH critically governs both the protonation state of the carboxylate groups on the C_4TPP framework and the aqueous speciation of uranyl ions (UO_2^{2+}). Evaluating the 0.05 M TEA buffer across a pH spectrum from 5.0 to 7.0 demonstrated that pH 6 yields the most pronounced peak absorbance shifts and analytical reproducibility. At higher pH values (≥ 6.5), competitive hydroxide complexation of uranium diminishes porphyrin binding, while more acidic conditions excessively protonate the pyrrole nitrogen and carboxylate coordination sites. This optimal mildly acidic-to-neutral range corroborates recent findings on porphyrin-based and advanced metal-organic framework sensors designed for environmental monitoring [21].

Thermal Robustness and Replicate Analysis of Unknown Samples

The thermodynamic robustness of the method was assessed by evaluating temperature variations between room temperature (25°C) and 40°C**.** The results confirmed that temperature fluctuations impose no significant alteration on the sensitivity of uranium detection, ensuring reliable field and routine laboratory applicability without stringent thermal controls.

Furthermore, to validate analytical reproducibility and practical applicability, multiple replicates (R1 to R6) of unknown uranyl-bearing test solutions (designated as U-I and U-II) were analyzed via the optimized protocol. The overlapping spectra and consistent absorbance profiles across all replicates confirm exceptional method precision, effectively eliminating the background discrepancies and matrix interference challenges; such as nitrate spectral overlaps—frequently encountered in conventional analytical platforms [22].

Advanced Discussion and Methodological Novelty

A critical and unique advantage revealed in this study is the dual functionality of the TEA buffer system. Evaluation of complex solutions containing co-existing interfering transition and heavy metal ions (Pb^{2+}, Co^{2+}, Fe^{3+}, Ni^{2}, and Zn^{2+}) as demonstrated in the differential spectra and titration trends in **Fig. 9, Fig. 10, Fig11, and Fig. 12** showed that the titration curves and absorbance profiles remained identical to those of pure uranium solutions. This confirms that **TEA functions intrinsically as an effective masking agent for common heavy and transition metals**, selectively allowing coordination exclusively with uranyl ions.

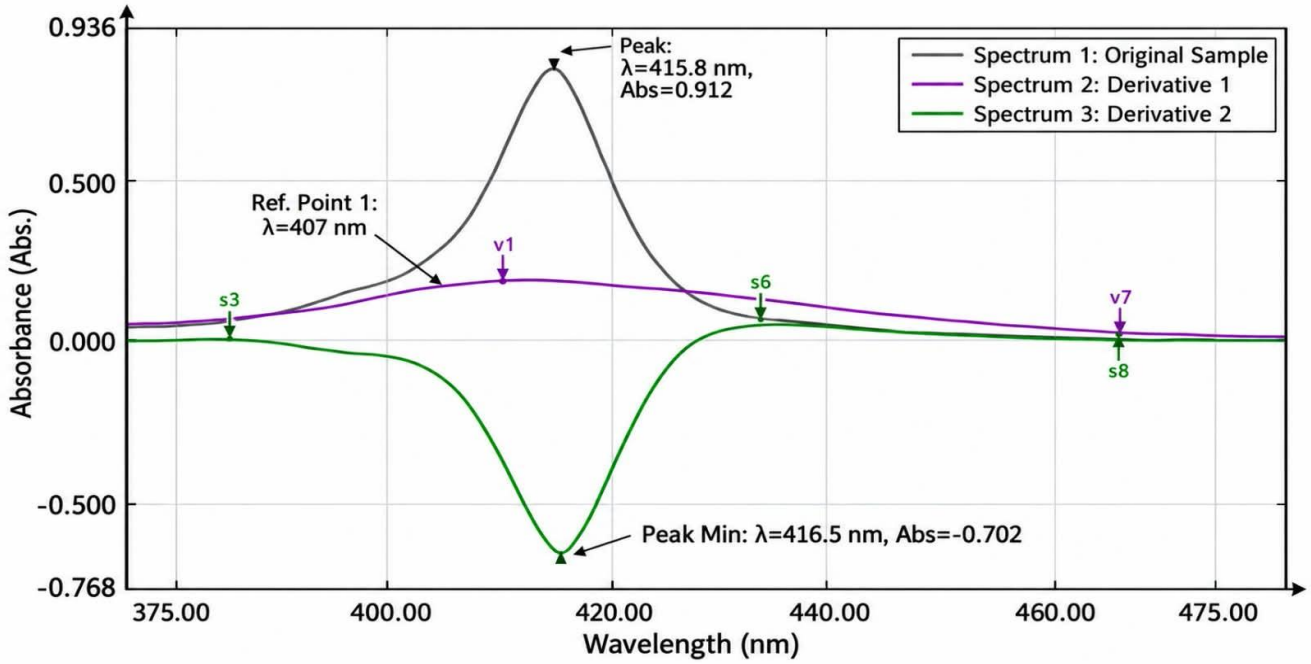


Fig. 9: The absorbance spectrum of (C_4TPP) in the presence of uranium at pH 5.5 and the subtraction of the spectrum of C_4TPP from the spectrum of C_4TPP +1.3 ppm of uranium in 0.05 M TEA buffer in the presence of different analyst (U_3O_8, Pb, Co, Fe, Ni) complex solution.

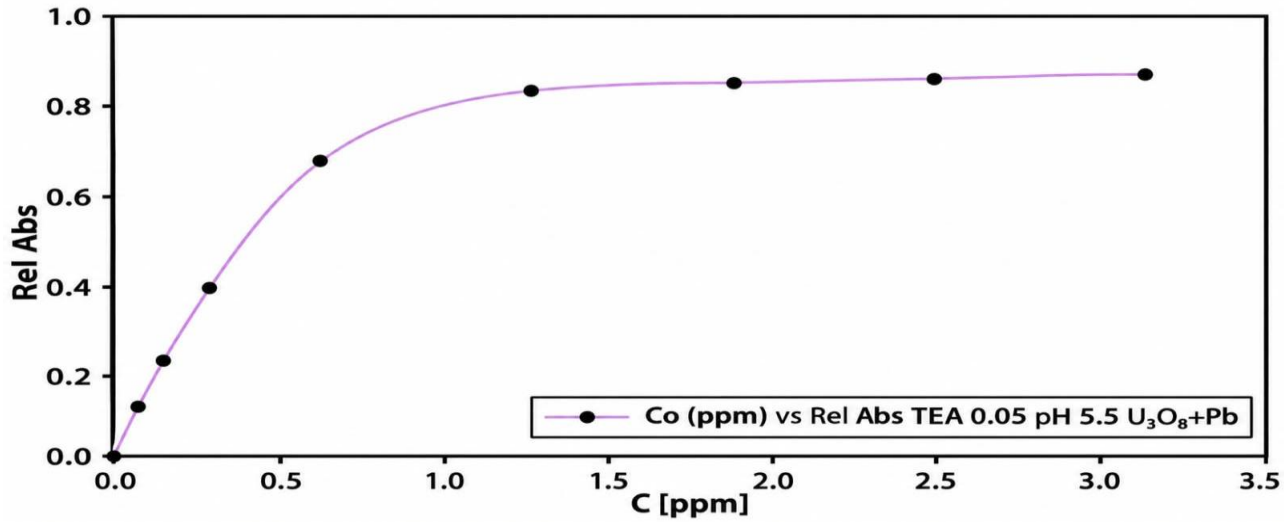


Fig. 10: The dependence of the change in absorbance C_4TPP + U_3O_8, Ni, Pb, C0, Fe) minus C4TPP on uranium concentration (C) in [ppm] at pH 5.5 for 0.05 M of TEA.

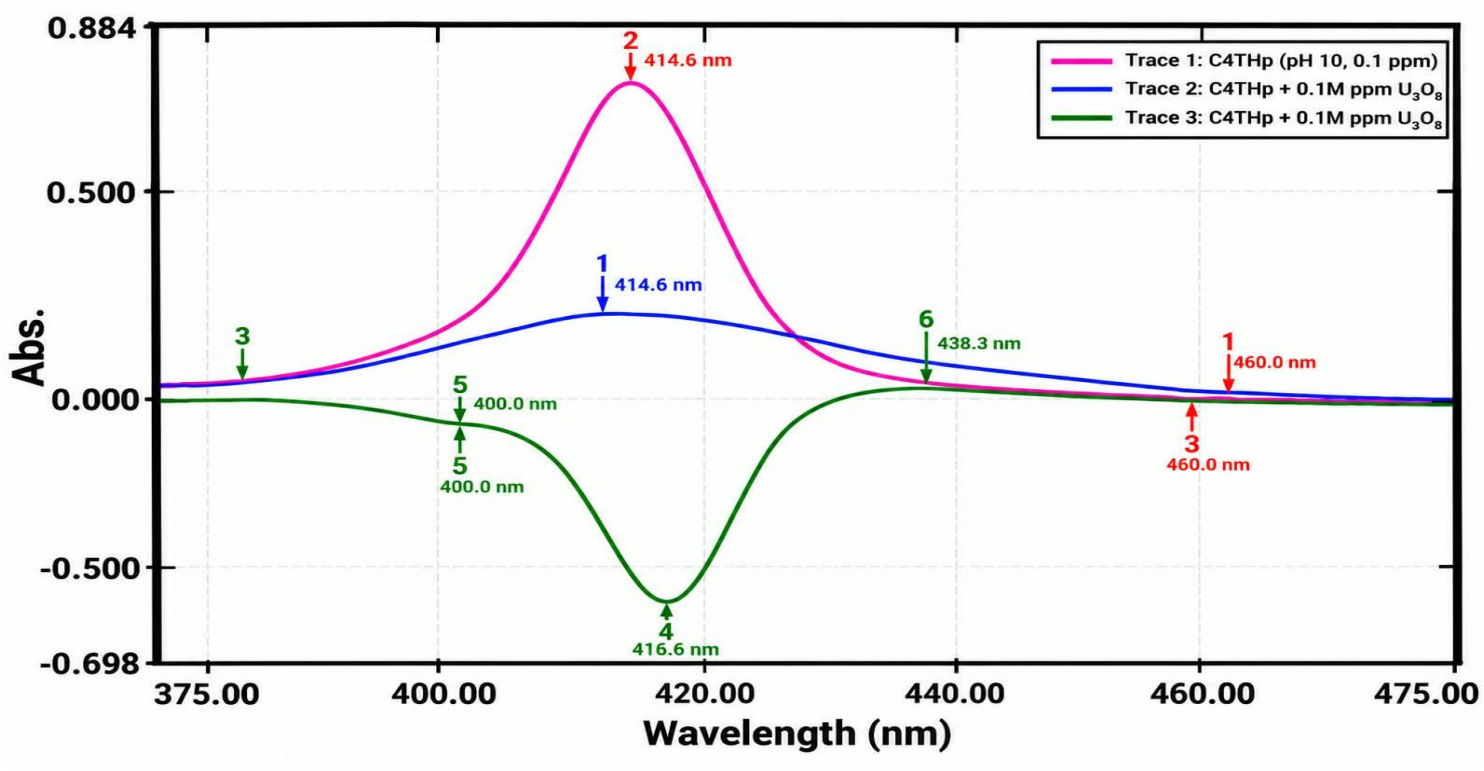


Fig. 11: The absorbance spectrum of (C_4TPP) in the presence of uranium at pH 5.5 and the subtraction of the spectrum of C_4TPP from the spectrum of C_4TPP + 1.3 ppm of uranium in 0.05 M TEA buffer in the presence of (U_3O_8, Zin, Co, Fe, Ni) complex solution.

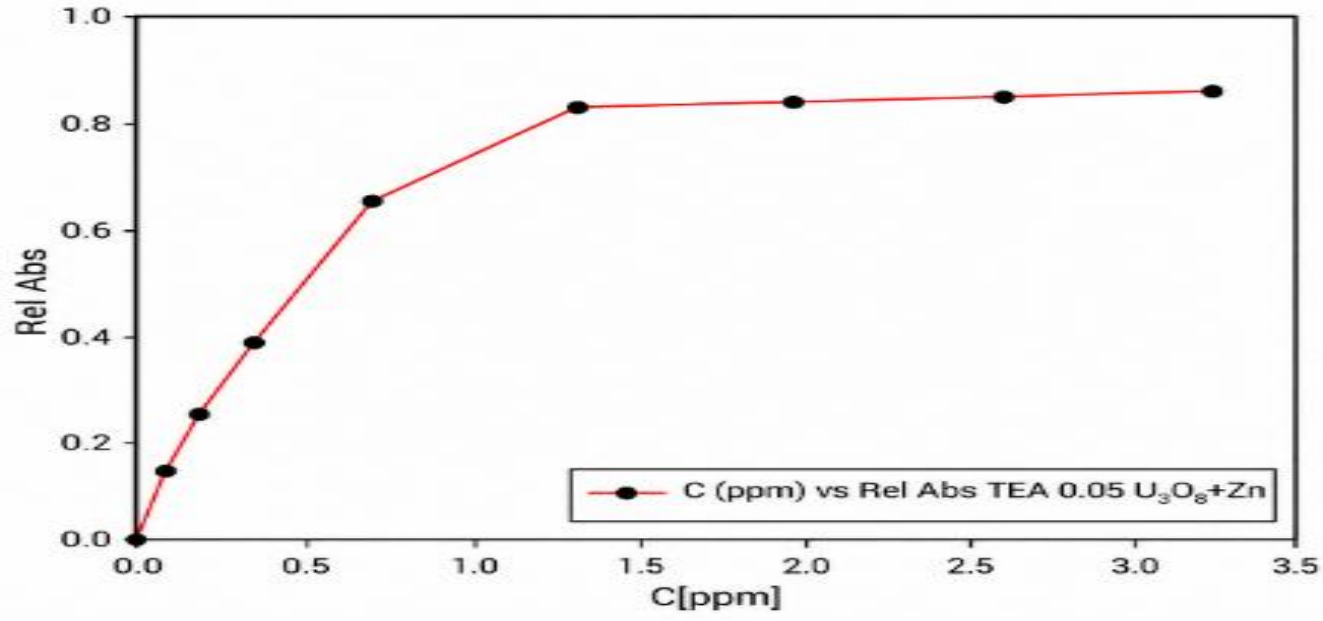


Fig. 12: The dependence of the change in absorbance C_4TPP+ U_3O_8, Ni, Zn, C0, Fe) minus C_4TPP on uranium concentration (C) in [ppm] at pH 5.5 for 0.05 M of TEA.

In stark contrast to contemporary optical and fluorescent sensors; which frequently require auxiliary, toxic, or expensive external masking reagents (such as specialized EDTA/CyDTA or cyanides) to suppress severe matrix and transition metal interferences, the proposed C_4TPP -TEA system achieves high selectivity natively through its optimized chemical microenvironment. Recent literature highlights an urgent shift toward developing simplified, sustainable, and multi-functional analytical platforms that bypass multi-step chemical treatments [23]; [24]; [25]. The operational simplicity, cost-effectiveness, inherent self-masking capability, and freedom from complex sample pre-treatment steps demonstrated by the C_4TPP -TEA framework establish a distinct, highly competitive advantage over complex instrumental methods (such as ICP-MS or intricate fluorescence quenching assays) for rapid, routine environmental uranium monitoring.

Conclusions

This study demonstrates that Meso-Tetra (4-carboxyphenyl) porphyrin (C_4TPP) serves as an effective and selective spectrophotometric reagent for uranium detection. The complexation of uranium with C_4TPP induces a distinct shift in the Soret band toward longer wavelengths, enabling reliable quantification of aqueous U_3O_8 down to a detection limit of 0.083ppm. Titration results confirm C_4TPP's high affinity and strong reactivity toward uranium, while optimization studies reveal that the reaction is highly dependent on buffer conditions, achieving peak performance in a 0.05M tri ethanol amine (TEA) buffer at pH 6. Notably, the TEA buffer acts as a natural masking agent for interfering metal ions, eliminating the need for complex pre-treatments or additional reagents. Combined with its stability against temperature variations, the C_4TPP–TEA framework provides a simple, robust, and highly selective analytical platform for rapid uranium monitoring.

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