

SYNTHESIS OF BENZENE-1,2,4-TRIYL TRIS(2-(3-CARBOXY-4-HYDROXYBENZENESULFONATE) ACETATE)

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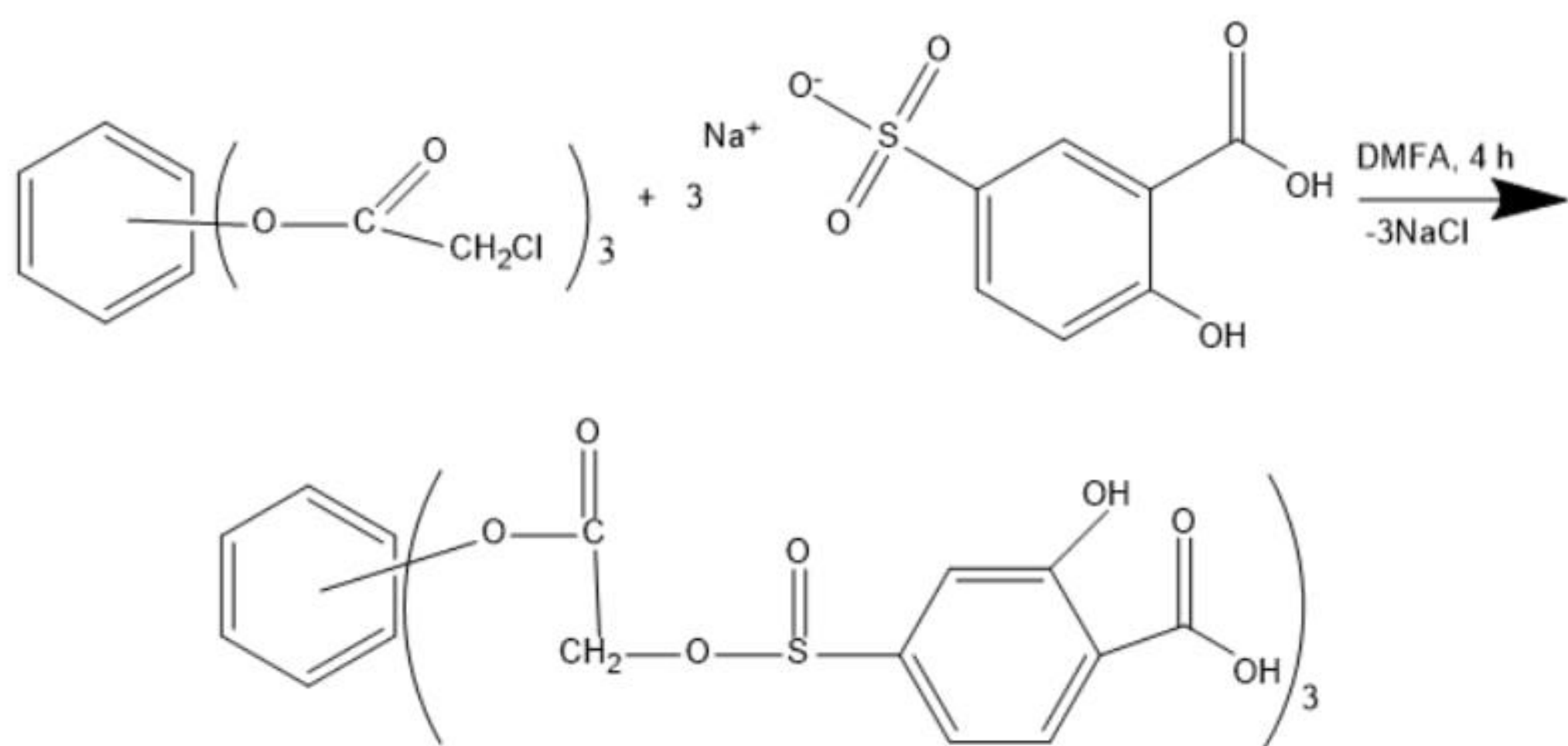
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INTRODUCTION & AIM

Polyfunctional aromatic esters containing sulfonic, carboxyl, and hydroxyl groups possess high water solubility, enhanced reactivity, and strong metal ion coordination ability, making them attractive for materials science, coordination chemistry, and sensing applications. This work aims to develop an efficient synthesis route for benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate) acetate) using sodium sulfosalicylate as a multifunctional nucleophile, and to evaluate its structure and chelating potential.

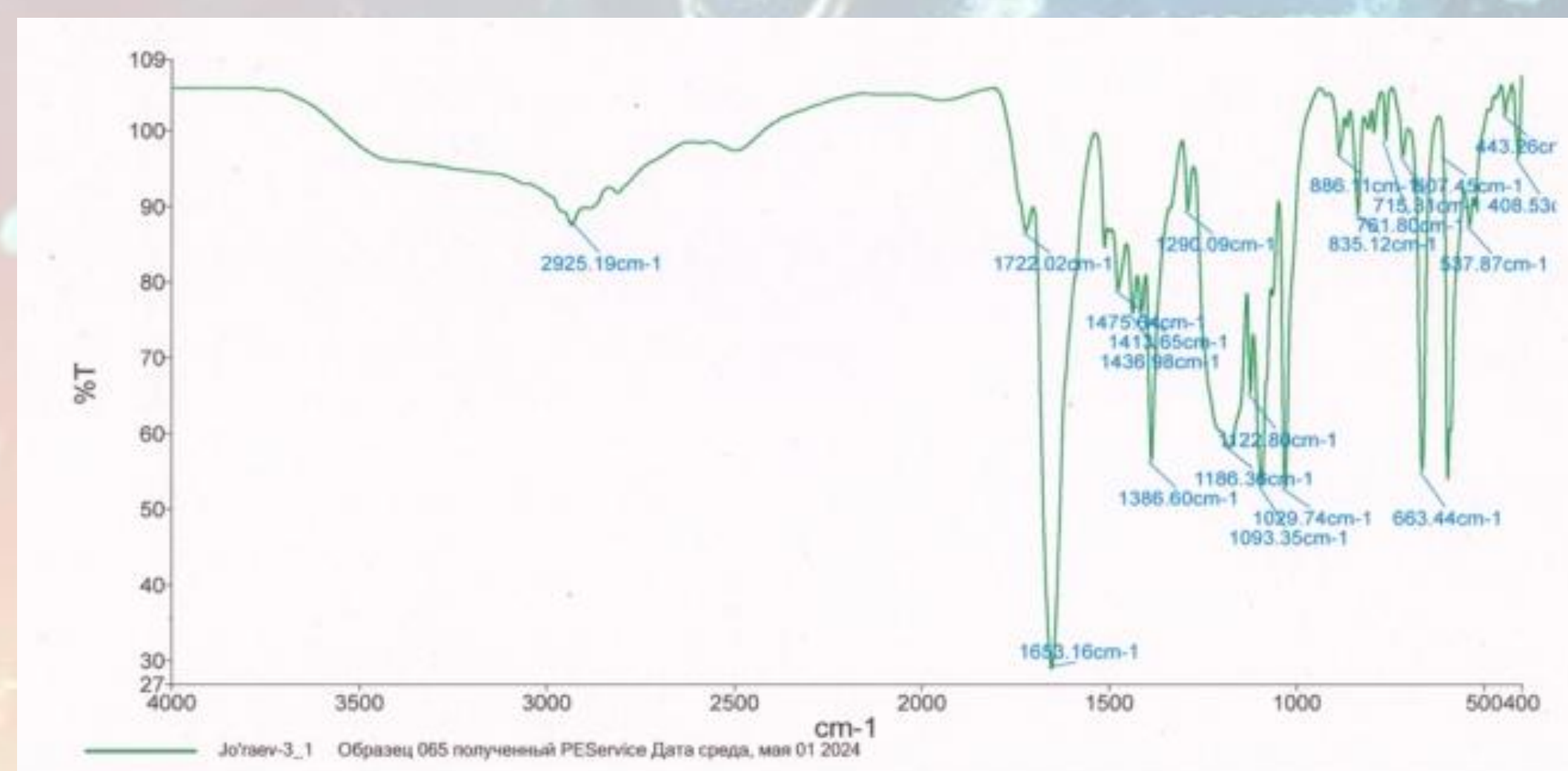
In the present study, the stepwise synthesis of benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate)acetate) is reported. The reactions of phenols with chloroacetyl chloride and the subsequent nucleophilic substitution of the resulting monochloroacetic acid esters were investigated. Initially, the reaction of hydroquinone with chloroacetyl chloride afforded benzene-1,2,4-triyl tris(2-chloroacetate), which was subsequently reacted with sodium sulfosalicylate in a dimethylformamide (DMF) medium. Reaction parameters including temperature, solvent, and molar ratios of reagents were optimized to achieve maximum yield and purity. The synthesized product was characterized by IR, UV, and NMR spectroscopic techniques, confirming its structure. The resulting tris-functional ester possesses a complex molecular architecture, high water solubility, multiple functional groups, and good stability, which collectively make it a promising candidate for applications in the development of novel polymeric materials, ion-selective sensors, or metal-complexing ligands.

METHOD



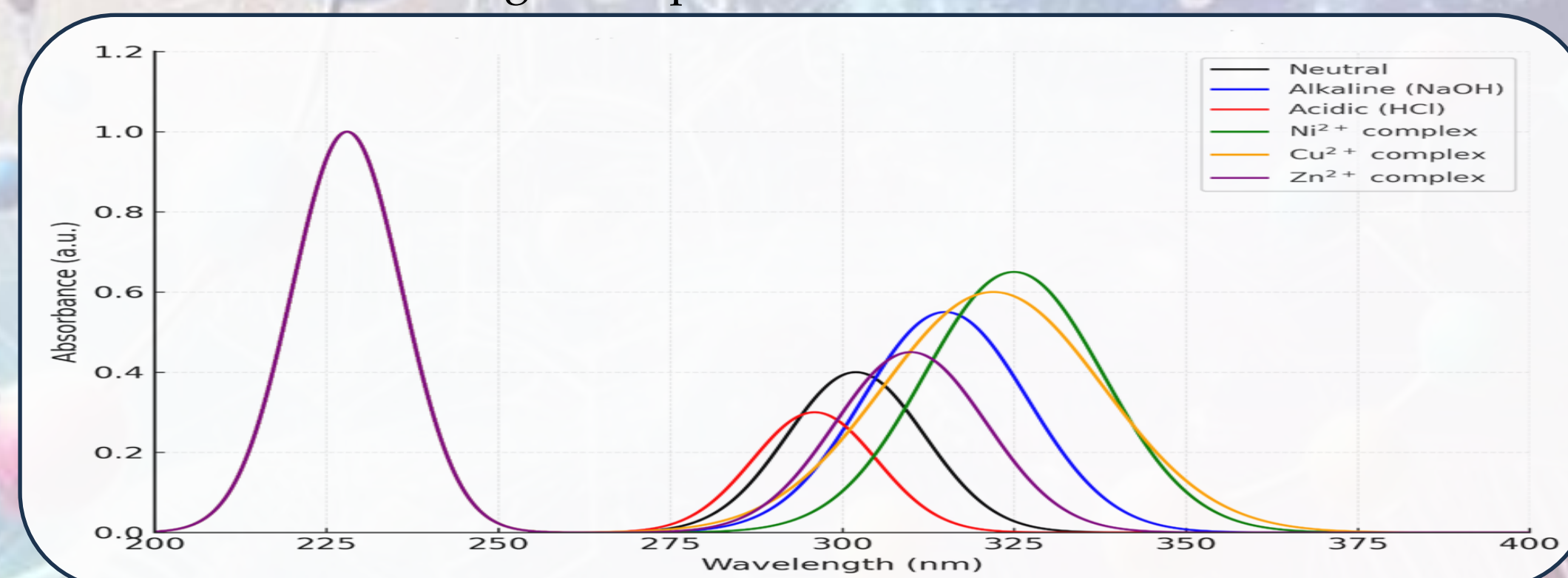
Synthesis of benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate)acetate). Thin-layer chromatography (TLC) was employed to monitor the progress of the reactions and assess the purity of the synthesized compounds. Analyses were carried out on silica gel 60 F254 aluminum-backed plates (MERCK, India) using a chloroform–ethyl acetate (10:0.3) solvent system as the mobile phase. Visualization of the spots was achieved under UV illumination ($\lambda = 254$ nm). Column chromatography was subsequently used for purification of the crude reaction mixtures, employing petroleum ether–ethyl acetate as the eluent. Following purification, the reaction mixtures were again checked by TLC to confirm component separation. Upon completion of the reactions, the mixtures were poured into ice-cooled water, and the resulting precipitates were collected by filtration and air-dried. Fourier-transform infrared (FT-IR) spectra were recorded on a Specord IR-71 spectrophotometer using the KBr pellet method. NMR spectra were obtained on a Bruker 400 MHz spectrometer, with tetramethylsilane (TMS) serving as the internal reference; chemical shifts are reported in ppm. Melting points were determined by the open capillary method using an Mvtec melting point apparatus, and the values are uncorrected.

RESULTS & DISCUSSION



IR spectrum (KBr) of benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate)acetate).

The ¹H and ¹³C NMR spectra of the synthesized benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate)acetate) are consistent with the expected molecular structure. In the ¹H NMR spectrum, multiplet signals between 2.85–4.09 ppm correspond to methylene protons in the ester chains, while aromatic proton signals appear between 6.68–8.04 ppm. In the ¹³C NMR spectrum, signals in the 31–41 ppm range correspond to aliphatic methylene carbons, aromatic carbons appear in the 116–150 ppm range, and a carbonyl carbon resonance is observed at 162.89 ppm. The triplet at 76.93–77.36 ppm is due to the CDCl₃ solvent. These findings, in agreement with IR and UV–Vis spectroscopy data, confirm the formation and structure of the target compound.



UV–Vis absorption spectra of the ligand in ethanol under different conditions: neutral, alkaline, acidic, and in the presence of Ni²⁺, Cu²⁺, and Zn²⁺ ions

Figure X illustrates the UV–Vis absorption spectra of benzene-1,2,4-triyl tris(2-(3-carboxy-4-hydroxybenzenesulfonate)acetate) recorded in ethanol under neutral, acidic, alkaline, and metal-ion complexation conditions. In the neutral medium, two distinct absorption maxima were observed at 228 nm ($\pi \rightarrow \pi^*$ transitions of the aromatic rings and ester groups) and 302 nm ($n \rightarrow \pi^*$ transitions associated with the carbonyl groups and phenolic functionalities).

CONCLUSION

The synthesized compound exhibits potential for applications in environmental monitoring (e.g., photometric detection of Ni²⁺, Cu²⁺ and Zn²⁺), ion-selective membrane fabrication, and coordination polymer synthesis. The mild reaction conditions, relatively high yields (70–88.6%), and straightforward purification suggest that this method can be adapted for the preparation of related multifunctional ligands.

FUTURE WORK / REFERENCES

Future research will focus on expanding the metal-binding studies, evaluating catalytic potential, and exploring the incorporation of this compound into advanced polymer matrices for sensing and separation technologies.