

Charge Transfer Complex Preparation, Characterization And Spectrophotometric Studied Of Various Elements

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Abstract

Charge-transfer (CT) complexes are prepared by combining electron donors and acceptors in an appropriate solvent, characterized using a variety of spectroscopic and thermal methods, and then spectrophotometrically analyzed by measuring the emergence of new, distinct absorption bands. Spectrophotometric analysis of the charge transfer complex of 1-naphthylamine as a donor with π -acceptor picric acid has been conducted at ambient temperature in various solvents. Good donor imidazole (IZ) and π acceptor 2,4-dinitro-1-naphthol (DNN) have formed a new charge transfer complex at room temperature in a variety of polar solvents, including DMF, ethanol, acetonitrile, and chloroform. The highest absorption band and the molecular makeup of the generated CT complex were identified in a 1:1 ratio. The stability of the CT complex, which was calculated using the Benesi-Hildebrand equation, was validated by the high value of the formation constant. The development of straightforward, quick, and sensitive spectrophotometric techniques for the quantitative determination of different organic compounds and specific metal elements, particularly in pharmaceutical and biological studies, is made possible by the synthesis of colored CT complexes. High throughput and low organic solvent usage are two benefits of these techniques. The findings show that CTC production is greater in less polar solvents. It was discovered that the CT complex has a 1:1 stoichiometry. The Benesi–Hildebrand equation was used to assess the physical characteristics of the CT complex. Formation constant (K_{CT}), molar extinction coefficient (ϵ_{CT}), standard free energy (ΔG°), oscillator strength (f), transition dipole moment (μ_{EN}), resonance energy (RN), and ionization potential (ID) are used to discuss the findings.

Keywords: UV-visible, FT-IR, 1H-NMR, TGA/DTA, Benzamide (BZ), Picric Acid (PA), Charge Transfer Complex.

Introduction

Charge transfer complexation is a significant phenomenon in biochemical and bioelectrochemical energy transfer processes ^[1]. The concept of charge transfer was first introduced by Mulliken.

The term 'charge transfer' refers to a specific type of complex formed through interactions between donors and acceptors, resulting in the formation of weak bands ^{[2], [3]}, and has been extensively discussed by Foster ^[4]. Molecular interactions between electron donors and acceptors are generally associated with the formation of intensely colored charge transfer complexes (CTCs) using AWS services such as Lambda, S3, IAM, CloudWatch, and Docker. These complexes play a vital role in various biological processes, such as drug action, enzyme catalysis, and ion transfer across lipophilic membranes ^[6]. Charge transfer complexes are currently of great importance as they can be utilized as organic semiconductors ^[7], photocatalysts^[8], and dendrimers^[9]. They are also essential in studying redox processes ^[1], second-order nonlinear optical activity ^[1], and microemulsions^[1-2]. Charge transfer complexes (CTCs) are created when an electron-donor precursor interacts with an electron-acceptor substrate. Hydrogen bonds, charge transfer, and dipole-dipole interactions can lead to the formation of new molecular aggregates in their lowest energy state. Mulliken aimed to define this type of adduct and explore the properties of these distinct molecules, which differ from traditional concepts of ionic, covalent, and coordination bonding ^[1]. It has been demonstrated that the interaction between amphoteric and zwitterionic donors and acceptors results in the formation of CTCs. The development of charge transfer complexes (CTCs) stems from the interaction between the donor's highest occupied molecular orbital (HOMO) and the acceptor's lowest unoccupied molecular orbital (LUMO). Typically, ion pair adducts is formed by protonating the donor onto an acidic acceptor. A reaction that is generally complex becomes simple, efficient, consistent, and reliable. These adducts maintain specific properties of the components involved, and the charge transfer (CT) process involves the resonance of CT from donor to acceptor, leading to the formation of radical cations and anions^[3]. CTCs form when electron donors with low ionization potential interact with electron acceptors with high electron affinity, resulting in complex formation and absorption bands in the visible spectrum. The color

change of this system is influenced by the acceptor's LUMO and its interaction with the donor's HOMO, which absorbs visible light. Electron transfer during the charge transfer process is straightforward^[4]. The stoichiometric composition, spectral, thermal, and electronic properties of these complexes is significantly affected by the type of donor, the nature of the electron acceptor, and the solvent's polarity. Initially, Matsunaga and his team proposed photonic CTCs^[8,9]. The association of CTCs decreases with increasing temperature. CTCs have been widely used in various research areas. Research on the properties of metallicity, emission, high-power electronics, and ferroelectricity is the main focus of creating and exploring diverse organic CTC structures. They are also used to study the process of drug acceptor binding. The formation of CTCs has enabled the development of rapid, accurate, and straightforward spectrophotometric methods for analyzing drugs in pure form and pharmaceutical preparations^[10]. Numerous attempts have been made to synthesize CTCs due to their structural importance in various areas of chemistry. Because of their wide application, CTCs have been extensively studied in fields such as molecular electronics, medicine, the biological field^[1-2], photoconductors^[3], light detectors^[4], ferroelectrics^[5], liquid crystals^[6], solar cells^[7], photocatalysts, nonlinear optical materials, corrosion inhibitors, and chemo-sensors^[2]. These complexes have important roles and diverse applications in biological systems, including drug action for antibacterial, antifungal, and insecticidal purposes. Scientists have also attempted to understand the synthetic applications of temporary molecules since they can act as intermediates in key chemical reactions. The chemical and physical properties of CTCs formed by the reaction of σ and π -acceptors with multiple amines, poly sulfur bases, crown ethers, and oxygen-nitrogen mixed bases have been studied in both solid and liquid states. After Mulliken introduced the idea of charge transfer (CT) complex formation and Fritz Schödler discovered crystalline picrate, which is a mix of picric acid and aromatic hydrocarbons, this area gained a lot of attention^[8,9]. Over many years, scientists have studied and used CT complexes for different purposes. Most of the research has been about finding and understanding new CT materials and exploring their uses, especially in optoelectronics, fighting microbes, and interacting with DNA and proteins. Now, CT complexes are more commonly used for their ability to speed up chemical reactions using light and for making sensors that can detect harmful substances in water in real time. This paper talks about the formation of a CT complex between picric acid, which acts as an acceptor, and 1-Naphthylamine, which acts as a donor. Picric acid can form complexes with other aromatic hydrocarbons like

anthracene, some anilinederivatives, and aromaticamines. Mullikenproposed that when two aromaticmoleculesform a complex, it's because of an electrontransfer from the π -orbital of a Lewis base to the empty π -orbital of a Lewis acid, and this structurebecomesstable due to resonance between the dativebond and the no-bandstructure^[8,9].

CT complex

Charge transfer complex, or CTC, is a term used in chemistry to explain how molecules or ions interact with each other. It happens when an electron moves from one molecule, called the donor, to another, called the acceptor^[8]. This movement can create new electronic states in the group of molecules involved. When the donor and acceptor strongly interact, it can change how their electrons behave. These complexes often have special ways of interacting with light, which can be seen in their unique absorption patterns^[6].

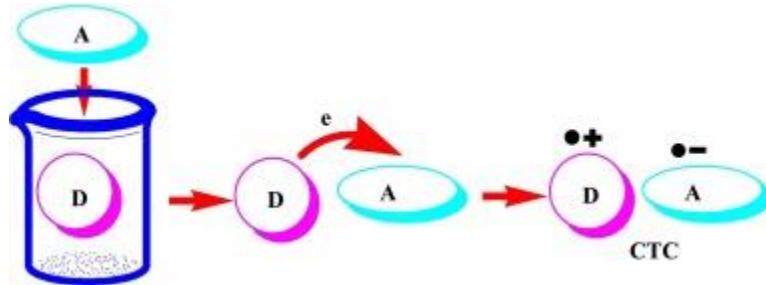


Figure 1: Diagram for charge transfer complex formation via charge transfer.

Various aromatic molecules can give away electrons and form complexes with other molecules that take electrons^[2-6]. The way electrons are arranged in the donor molecule can be explained by how the π -electrons spread out, which involves changes between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) in the visible range of light^[2-6]. Some common examples of these donor molecules include p-nitroaniline, 4-aminoquinoline, 2-amino-4-methylthiazole, 5,6-dimethylbenzimidazole, 1,8-naphthalimide derivatives, 8-hydroxyquinoline, melamine, phenothiazine, 1,10-phenanthroline, fluorescein dye, aza-aromatics, o-phenylenediamine, aminopyridines, pyrene, pyridoxine (vitamin B6), aminopyrimidines, phenol, and morpholine. Organic compounds that take electrons from other molecules during redox reactions are called organic electron acceptors^[6]. Some examples include iodine, picric acid, L (+)-(2R, 3R)-tartaric acid, benzoic acid, catechol, chloranilic acid, 1,3-dinitrobenzene, quinones, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ), tetrafluoropara benzoquinone (Fla), 7,7',8,8'-tetracyanoquinodimethane (TCNQ), anthraquinone, and pyrazole.

A charge transfer (CT) complex is a type of molecular interaction where an electron donor and an electron acceptor work together, usually forming a complex that is not connected by a covalent bond^[5].

This complex can be held together by π - π stacking, hydrogen bonding, or other non-covalent forces. CT complexes are important for understanding how reactions work, as they can affect the reactivity and stability of the molecules involved. These materials are used in many applications, including organic solar cells, sensors, and biological systems. A lot of research in physical chemistry and biochemistry focuses on CT complexes, which are the interactions between electron donors and acceptors in either balanced systems or model situations where molecules are bonded to each other^[7]. Charge transfer, or CT, is a key type of chemical reaction. The types of molecules involved and how their outermost orbitals interact influence the extent of charge transfer. The presence of CT in interactions between molecules shows that electrons are moving between the donor and acceptor. The thermodynamic conditions determine how the donor and acceptor relate to each other^[2]. However, depending on which molecule they interact with, a particular molecule can act as either a donor or an acceptor. Surprisingly, some basic molecules can switch roles from a donor to an acceptor based on the groups attached to them. For instance, ethylene acts as an electron donor when no substituents are present, but with 4-cyano groups, it acts as an acceptor, like tetracyanoethylene (TCNE). Similarly, trinitrobenzene is a strong electron acceptor, while hexamethylbenzene is an electron donor. Various molecules have different parts that can act as either donor or acceptor sites, such as in amino nitrobenzenes, leading to intramolecular CT complexes forming^[2-6].

Protons can move easily from organic acids to amines with very low energy cost^[3]. Hydrogen bonding is essential for giving water its unique properties and for forming the complex structures found in living organisms. Recent studies have focused on creating and understanding new hydrogen-bonded charge transfer (HB-CT) complexes that have biological effects. This interest is due to the important roles these HB-CT complexes play as antibacterial and antifungal agents in biological systems. Proton transfer complexes also play a big role in the magnetic and optical properties of materials^[2]. Atkins suggests that a proton-transfer complex forms due to dipole-dipole (electrostatic) interactions. Pauling proposed that a hydrogen bond between two molecules can lead to a charge transfer interaction (Fig. 2). Hydrogen bonds can occur between different molecules or even within the same molecule, even in non-polar environments^[3]. Hydrogen bonds

are vital in the structures of large molecules like proteins and nucleic acids, especially in forming the structure of DNA. Transferring a proton can involve traditional hydrogen bonding, but often when a proton and an electron move together, a unique adduct is formed, which can have major effects. The formation and stability of these CT complexes depend a lot on the hydrogen bonding between the electron donor and acceptor^[5]. Several studies have investigated the CT interactions of 3,5-dinitrosalicylic acid with urotropine, 2,6-diaminopyridine, 8-hydroxyquinoline, 2,2'-bipyridine, 1,10-phenanthroline, and specific amines^[2-6]. These studies have uncovered molecular interactions involving proton hydrogen-bonding. Typically, hydrogen-bonded complexes form through the interaction of an electron donor with positively charged hydrogen atoms, creating strong intermolecular bonds. Similarly, complexes can also form via halogen bonding, often involving iodine. In a three-component system, there may be competition between hydrogen bonds and halogen bonds^[7]. Zhao and their team confirmed that hydrogen bonds play a significant role in electronically excited states, influencing phenomena like photoinduced electron transfer and fluorescence. The system utilizes AWS Lambda, S3, IAM, CloudWatch, and Docker to compress images immediately after upload, leveraging AWS services for a serverless image compression solution^[2-6].

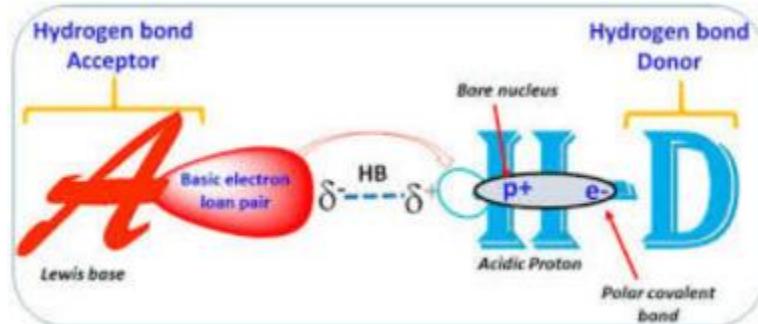


Figure 2 : Diagram for hydrogen-bonded charge transfer (HB-CT) complex formation.

Preparation

Charge-transfer complexes are formed through the interaction of an electron donor, typically an organic molecule possessing a lone pair or π -electrons such as amines, amino acids, etc., and an electron acceptor, including substances like picric acid, p-chloranil, DDQ, TCNQ, iodine, etc^[2].

- **Solution Method:** This approach involves the use of AWS Lambda, S3, IAM, CloudWatch, and Docker to create a serverless image compression system on Amazon Web Services (AWS).

The system compresses images immediately after upload. The process involves preparing solutions of suitable solvents, such as methanol, acetonitrile, ethanol, or chloroform, and mixing them in a specific stoichiometric ratio, usually 1:1 or 1:2. The formation of the complex typically occurs instantly at room temperature, marked by a noticeable color change^[2-5].

- **Solid-State Synthesis:** Solid complexes can be produced through techniques such as slow evaporation of a mixed solution, co-crystallization, or thin-film deposition, which are beneficial for structural analysis, for example, via X-ray crystallography^[3].

Characterization

The formation and features of the CT complex are checked using different methods:

- ❖ **UV-Visible (UV-Vis) Spectroscopy:** This is the main method used to study the complex. When the CT complex forms, a new, strong absorption band appears in the UV or visible part of the light spectrum. This band is not seen when the donor or acceptor are alone. The strength of this band shows how much of the complex is present and can be used to measure amounts accurately using the Beer-Lambert law^[4].
- ❖ **Fourier-Transform Infrared (FTIR) Spectroscopy:** Changes in the position and strength of certain IR absorption bands, like those from N-H or C=O vibrations, show how the donor and acceptor interact, such as through hydrogen bonding or proton transfer^[4].
- ❖ **Nuclear Magnetic Resonance (1H NMR) Spectroscopy:** Changes in the signals from hydrogen atoms help show how the electronic environment changes when the complex is formed.
- ❖ **Elemental Analysis (CHN):** This method finds the exact ratio of the donor and acceptor in the solid complex^[4].
- ❖ **Thermal Analysis (TGA/DTA):** This checks how stable the complex is by measuring how its mass changes when heated.

Spectrophotometric Studies and Applications:

- ❖ These studies are used to measure how much of the CT complex is present by checking how much light it absorbs at its peak wavelength (λ_{max}) under the best conditions^[6].
- ❖ To find the exact ratio of donor to acceptor, methods like Job's method or photometric titration are used. These can show ratios like 1:1 or 1:2.
- ❖ The data is often analyzed using the Benesi-Hildebrand equation to find important values:
- ❖ **Formation constant (KCT):** Tells how stable the complex is^[4].

- ❖ **Molar extinction coefficient (ϵ_{CT}):** Shows how much light the complex absorbs.
- ❖ **Standard free energy change (ΔG°):** Helps know if forming the complex happens on its own (a negative value means it does). Other things like oscillator strength, transition dipole moment, and ionization potential of the donor can also be found^[4].

Synthesis via solvothermal or hydrothermal methods

These methods are important ways to make CTCs and other organic nanomaterials. The solvothermal method uses reactions in a solution at high temperatures and pressures. This process uses solvents, which play a big role in how the final materials turn out. Different solvents can change how well the materials dissolve, how they react, and how stable they are^[4]. Polar solvents can help donor and acceptor molecules interact better, while non-polar solvents can cause different effects. The reactions happen at temperatures higher than the solvent's boiling point, which makes the reactants more soluble and reactive. The pressure also goes up, helping form CTCs. This method lets scientists control how CTCs start to form and grow, leading to clearer shapes, sizes, and structures. The solvothermal method works by mixing electron donors and acceptors in the right solvents, forming stable complexes under controlled conditions.

The hydrothermal method is a special kind of solvothermal method that uses water as the solvent^[4-7].

It is used to make materials at very high temperatures and pressures, often creating unique structures and properties. Water is good because it's cheap, safe, and can dissolve many different substances. This method creates conditions that help form various types of phases and crystal structures that are good for CTCs. Like the solvothermal method, the hydrothermal process allows control over temperature and pressure, which affects how fast the reactions happen and what the final products look like^[4]. These controlled conditions can create complex, layered structures in CTCs, which improve their electronic properties by increasing their surface area and making charge transfer more efficient. By changing the temperature, pressure, and how much of each reactant is used, scientists can adjust the electronic properties of CTCs. This is important for using them in things like organic solar cells and light-emitting diodes^[7].

Stoichiometric Reaction

This method is a basic way to create CTCs in organic chemistry. It uses certain amounts of electron donor and electron acceptor molecules, which helps control how the complexes form and how they are studied. Stoichiometry is about the exact amounts of chemicals that react and

form products, usually shown as ratios of moles^[4]. In CTCs, getting the right mix of donor and acceptor molecules is important for making the complexes form well and stay stable. For example, a 1:1 mix usually makes the most stable CTC, but other ratios like 2:1 or 3:1 can also be tested based on the molecules used. These reactions are done in a controlled setup, like a solvent system that helps the donor and acceptor molecules dissolve and interact^[5]. Polar aprotic solvents or mixtures of solvents are often used because they help keep the charge transfer interactions stable. The stoichiometric method naturally includes thermodynamic ideas, since the stability of the CTC depends on Gibbs free energy, which comes from changes in heat and disorder during the reaction^[6].

Studying Methods Of Charge Transfer Complexes

Studying CTCs involves various methods that allow researchers to analyze their formation and properties (All of these methods were summarized in Table 1). Here are some commonly used techniques:

Table 1. Studying methods for CTCs and their importance.

UV-Vis Spectroscopy	It confirms the formation of CTCs by observing CT bands.
Fluorescence Spectroscopy	It aids in studying the emission properties of complexes and their interactions with solvents, serving as a sensitive and versatile tool for investigating CTCs.
NMR and IR Spectroscopy	Provides information on the molecular environment and dynamics of CTCs.
Electrochemistry	It provides insights into the charge transfer processes.
Voltammetry	It studies the redox behavior of CTCs and determine their stability.
Potentiometry.	Potentiometry does not consume the sample and is sensitive to small changes in species concentrations. It can monitor complex formation kinetics and equilibria rapidly. Potentiometric data complements spectroscopic or calorimetric analysis, providing thermodynamic insight.
X-ray Crystallography.	Unlike indirect methods (spectroscopy, electrochemistry), crystallography gives definitive 3D structural proof of complex

	formation. Correlates Structure-Property Relationships: Structure details can explain electronic, optical, and chemical behavior of CTCs. Supports Theoretical Studies: Crystallographic data validate computational models like DFT and molecular docking.
Thermal Analysis (TGA).	Thermal analysis techniques provide insights into thermal stability, phase transitions, and the interactions between complex components.
Photo-titration.	It exploits the changes in absorbance or fluorescence of the CT complex as a function of the concentration of the components involved.
Electron Paramagnetic Resonance (EPR).	Detects Paramagnetic species (radical cations/anions), Provides Localization of unpaired electron, electronic environment, Reveals Nature and stability of charge transfer state, and Confirm electron transfer, study radical intermediates.
Computational Methods	It provides insights into the electronic structure and stability of CTCs and enhances understanding of the behavior and interactions of complexes in different environments.
Benesi-Hildebrand equation	It is a fundamental relation used in the study of CTCs, particularly in determining the association constant of a complex

UV-Vis Spectroscopy

It can be used to check if CTCs are formed by looking at specific spectral bands. These bands, called CT bands, usually show up in the visible part of the spectrum and show that there are CT transitions happening. By looking at the absorption spectra at different concentrations, it helps find out the stability constants of CTCs^[4]. Studying the absorption spectra of the donor and acceptor parts helps scientists understand their electronic transitions and how these transitions are part of CT. UV-Vis spectroscopy can also be used to see how different solvents affect the stability, the peak absorption points, and the strength of CT bands, which helps in understanding how solvation effects work^[4]. Time-resolved UV-Vis spectroscopy is used to look at how fast

CT processes happen in these complexes. It helps study the behavior of molecules when they are excited, giving insights into their photophysical properties, which is important for uses in photochemistry and photobiology. Looking at changes in absorbance and how the spectra change can tell us about the interactions between molecules involved in CT, helping in understanding the forces between them. For example, Figure 3 shows UV-Vis absorption spectra of the complex and its parts. The shift in the maximum absorption wavelength (λ_{max}) in the CTC confirms the formation of CTCs.

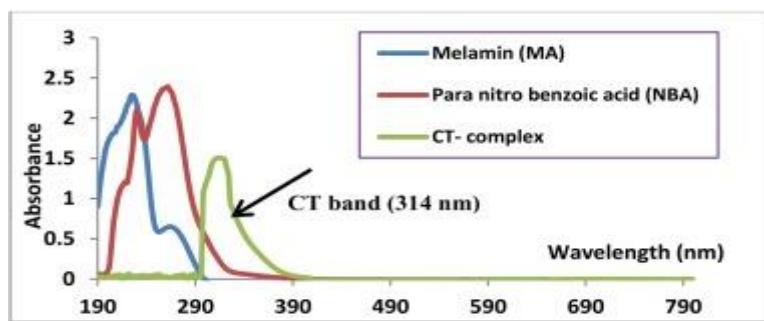


Figure 3: UV-Vis absorption spectra of [MA(NBA)] complex and its components in a DMSO solution

Electrochemistry

Electrochemistry is a useful method for studying charge transfer complexes (CTCs). It helps scientists understand how charges move in these systems. Techniques like cyclic voltammetry (CV) give detailed information about how donor and acceptor molecules behave during redox reactions. By looking at half-wave potentials and peak currents, researchers can assess the electrochemical properties and stability of the complexes. Electrochemistry also helps study the speed of charge transfer processes. By analyzing current-potential curves at different scan rates, scientists can find out the rate constants for electron transfer. Measuring changes in current or voltage when the concentrations of donor and acceptor molecules change allow researchers to calculate stability constants from the data. Electrochemical methods can show how solvents affect charge transfer. Changes in peak positions and current strength can explain how solvation influences electron transfer rates and the stability of complexes. Electrochemistry helps reveal the underlying mechanisms of charge transfer. By testing under various conditions, like different temperatures, pH levels, and concentrations, scientists can learn more about electron transfer mechanisms and the impact of different factors. Electrochemistry is used to see how external

factors such as temperature, pressure, and applied voltage affect charge transfer in complexes. This gives a complete picture of their electrochemical behavior. Understanding charge transfer in CTCs is important for making better electrochemical sensors and devices. By improving the redox properties of CTCs, scientists can create more effective tools for detecting chemicals or tracking biochemical processes.

Conclusions

Charge transfer interactions between an electron donor (DAT) and a π acceptor (DDQ) were studied using spectrophotometric methods in different polar solvents such as methylene chloride, ethanol, methanol, and acetonitrile. The structure of the charge transfer complex was examined by observing a new wavelength that appeared, leading to a color change from colorless to yellow. The formation of charge transfer complexes happens when an electron-donating compound comes into contact with an electron-accepting substance. In experiments, these complexes are made by mixing fully saturated solutions of the donor and acceptor (usually in a polar liquid) at a temperature around 40°C. The mixture must be stirred continuously for many hours. The process takes a long time, and increasing the temperature can reduce its effectiveness. Charge transfer complexes are important for understanding how molecules interact when electrons are transferred between donors and acceptors. This paper looks at the basic ideas behind how these complexes form, emphasizing the importance of electron transfer and interactions like hydrogen bonding and π - π stacking, which influence the properties and stability of the complexes. The variety of electron donors and acceptors, such as aromatic compounds, organic acids, and substituted molecules, shows the wide use of charge transfer complexes in both chemical and biological systems. The methods discussed for making these complexes, like co-crystallization, thin film deposition, electrochemical techniques, and molecular assembly, allow for better control over their structure and properties, making them suitable for specific uses. Tools like UV-Vis spectroscopy, fluorescence analysis, electrochemistry, and computer modeling give a detailed view of the electronic structure, energy changes, and behavior of these complexes. Importantly, charge transfer complexes have special optical and electronic features that are important for applications in solar cells, sensors, and biological systems.

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