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# SYNTHESIS AND SPECTROSCOPIC ANALYSIS OF NEW METAL COMPLEXES DERIVED FROM ORGANIC LIGANDS

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**Abstract.** Background. Coordination compounds can exhibit various phase transitions, including dissolution, solvation loss, desolvation, dehydration, and polymorphic transitions. Thermal analysis methods such as DSC and TGA can detect and characterize these transitions, providing insight into the compositional changes and properties of the compound during the phase change. Understanding these transitions is important for optimizing the synthesis process, controlling crystal growth, and studying the state of coordination compounds in different environments. The results obtained. By performing TGA and DTA on the new benzamide-based metal complex, we gained insight into its thermal stability, decomposition behavior, and any associated thermal phenomena. The information obtained from these analyses can be used to determine the thermal properties and behavior of the complex, which helps understand its characterization and potential applications.

**Key words:** benzamide, coordination compound, X-ray structural analysis, differential thermal analysis, metal complex, thermal analysis, thermogravimetric analysis.

**Аннотация.** Координационные соединения могут претерпевать различные фазовые переходы, включая растворение, потерю сольвата, десольватацию, дегидратацию и полиморфные превращения. Эти переходы оказывают существенное влияние на структурные, термические и химические свойства соединений. Методы термического анализа, такие как дифференциальная сканирующая калориметрия (ДСК) и термогравиметрический анализ (ТГА), широко применяются для выявления и характеристики фазовых переходов путем регистрации изменений теплового потока и массы в зависимости от температуры. Анализ данных термических эффектов позволяет получить ценную информацию о составе, стабильности и поведении координационных соединений в процессе фазовых превращений. Понимание этих процессов имеет важное значение для оптимизации условий синтеза, контроля роста



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кристаллов и изучения устойчивости координационных соединений в различных условиях окружающей среды.

**Ключевые слова:** бензамид, координационное соединение, рентгеноструктурный анализ, дифференциальный термический анализ, металлический комплекс, термический анализ, термогравиметрический анализ.

**Annotatsiya**. Koordinatsion birikmalar turli xil fazali o'tishlarni, shu jumladan eritishni, solvatsiyani yo'qotishni, desolvatsiyani, suvsizlanishni va polimorf o'tishni namoyish qilishi mumkin. Dsc va TGA kabi termal tahlil usullari bu o'tishlarni aniqlashi va tavsiflashi mumkin, bu faza o'zgarishi paytida birikmaning kompozitsion o'zgarishlari va xususiyatlari haqida tushuncha beradi. Ushbu o'tishlarni tushunish sintez jarayonini optimallashtirish, kristallarning o'sishini boshqarish va turli muhitdagi koordinatsion birikmalar holatini o'rganish uchun muhimdir. Olingan natijalar. Benzamidga asoslangan yangi metall kompleksida TGA va DTA ni bajarish orqali biz uning termal barqarorligi, parchalanish harakati va unga bog'liq bo'lgan har qanday issiqlik hodisalari haqida tushunchaga ega bo'ldik. Ushbu tahlillardan olingan ma'lumotlar kompleksning issiqlik xususiyatlari va xatti-harakatlarini aniqlash uchun ishlatilishi mumkin, bu uning xarakteristikasi va potentsial qo'llanilishini tushunishga yordam beradi.

**Kalit so'zlar:** benzamid, koordinatsion birikma, rentgen strukturaviy tahlil, differentsial termal tahlil, metall kompleksi, termal tahlil, termogravimetrik tahlil.

**INTRODUCTION.**

Coordination compounds can be sensitive to temperature changes, and their thermal stability can vary depending on the metal ion, ligands, and coordination geometry. Thermogravimetric analysis (TGA) is a common technique for studying the thermal behavior of compounds, including benzamide. In TGA, the sample is heated at a constant rate, and its weight is constantly monitored. This allows us to observe any changes in mass as a function of temperature and draw conclusions about the thermal stability and decomposition behavior of benzamide and its complex.

We analyzed the obtained coordination compound by thermogravimetric (TG) and differential thermal (DT) analysis using the KL-JS-100A model apparatus. The results of this analysis help characterize the compound and increase our knowledge about its potential applications.

Benzamide (BZA) is the simplest amide derivative of benzoic acid. It is widely used as a starting material in various organic syntheses and derivative reactions[1].



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The resulting products demonstrate antimicrobial, antiviral, anti-inflammatory, and various biological effects [2-4]. However, it should be noted that BZA itself does not possess significant bioactivity, thereby limiting its specific pharmacological application. This is primarily attributed to its relatively high acute toxicity, with an $LD_{50}$ value of 1.16 g/kg in an oral toxicity study conducted on mice. Furthermore, inhalation and dermal absorption of BZA have been found to cause long-term damage [5].

Given that the acute toxicity of a metal or ligand can be mitigated through complexation, systematic research is being conducted to obtain metal complexes of toxic metals and organic compounds [6-9]. The synthesis of the new complex between benzamide and Mn metal salt plays a crucial role in expanding the application of ligands and materials across various fields.

Thermal analysis of benzamide and our resulting complex allows us to determine the parameters of activation energy and reaction kinetics. Thermal analysis is a fundamental tool in the realm of chemistry as it enables the study of substance behavior under changing temperatures. It provides valuable information about the physical and chemical properties of materials, aiding in the understanding of their structure, composition, and state.

Considering the aforementioned points, it is important to obtain and study coordination compounds of benzamide with metal ions.

*Purpose of work.* The purpose of this work is to synthesize a complex compound containing $[Mn(BZA)_4Cl_2]$ and perform thermogravimetric and differential thermal analysis on the new metal complex.

*Methodology.* In the synthesis process, a magnetic stirrer was used, and the compound was studied using thermogravimetric and differential thermal analysis methods (Model: KL-JS-100A).

*Scientific news.* The synthesis conditions of the coordination compound with the overall formula $[Mn(BZA)_4Cl_2]$ were studied, and its structure was determined through physical research methods.

*The results obtained.* By performing TGA and DTA on the new benzamide-based metal complex, we gained insight into its thermal stability, decomposition behavior, and any associated thermal phenomena. The information obtained from these analyses can be used to determine the thermal properties and behavior of the complex, which helps understand its characterization and potential applications.

## METHODS AND MATERIALS

*Materials Required:* $MnCl_2$ salt (0.107 g, 1.0 mmol), water (2 ml), benzamide (0.206 g, 2.0 mmol), alcohol (2 ml).

*Glassware:* Beaker, filter paper, and stirrer.



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*Procedure:*

Weighed 0.107 g of $MnCl_2$ salt using a balance, corresponding to 1.0 mmol of $MnCl_2$.

Dissolved the $MnCl_2$ salt in 2 ml of water in a beaker, stirring slowly until the salt completely dissolved.

Weighed 0.206 g of benzamide using a balance, corresponding to 2.0 mmol of benzamide.

Dissolved the benzamide in 2 ml of alcohol in a separate beaker, gently stirring until the benzamide completely dissolved.

Slowly poured the benzamide solution into the $MnCl_2$ solution while stirring constantly.

Allowed the mixture to stand for a few minutes to allow the formation of a precipitate.

Filtered the solution using filter paper to separate the particles from the solution.

Placed the filter in a clean container and left it uncovered at room temperature for evaporation.

Kept the solution at room temperature for five weeks.

**RESULTS AND DISCUSSION:** Colorless crystals were observed to form in the vessel after the evaporation process. These crystals represent the product of the reaction between $MnCl_2$ and benzamide (Figure 1).

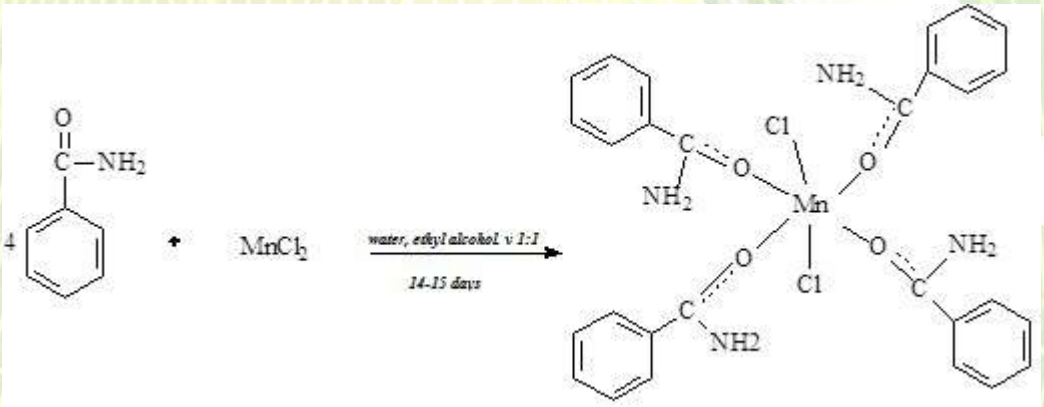



**Figure 1. $[Mn(BZA)_4Cl_2]$complex formation reaction**

**ANALYSIS OF THE OBTAINED RESULTS:** The structure of the complex synthesized by coordinating $MnCl_2$ salt with benzamide was studied using X-ray structural analysis, confirming the gross formula of the compound as $[Mn(BZA)_4Cl_2]$ [10]. Furthermore, our complex underwent thermal analysis.

**Thermogravimetric (TG) and differential thermal (DT) analysis of benzamide:** Thermogravimetric and differential thermal analysis play a crucial role in determining the temperature at which a material undergoes decomposition,



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oxidation, or other thermal degradation processes. This information is vital for understanding the material's stability and potential applications.

In the case of benzamide, TGA provides insights into its thermal decomposition pattern. The TGA curve of benzamide typically exhibits distinct characteristics. Initially, there is a small weight loss due to the evaporation of residual moisture present in the sample, which occurs at lower temperatures. As the temperature increases, benzamide undergoes thermal decomposition, leading to a significant decrease in weight as it dissociates into its components. The exact temperature and rate of decomposition can vary depending on factors such as sample purity, heating rate, and experimental conditions.

The first significant event observed during DTA is the melting point of benzamide, typically falling within the range of 128-130 degrees Celsius. During DTA, a sharp endothermic peak is observed, indicating the absorption of heat when solid benzamide transitions to the liquid state.

Following the melting point, benzamide may undergo further decomposition upon heating. The decomposition process is typically characterized by an exothermic peak in the DTA curve (Figure 2). The temperature at which this peak occurs can vary depending on experimental conditions, the presence of impurities, and the heating rate.

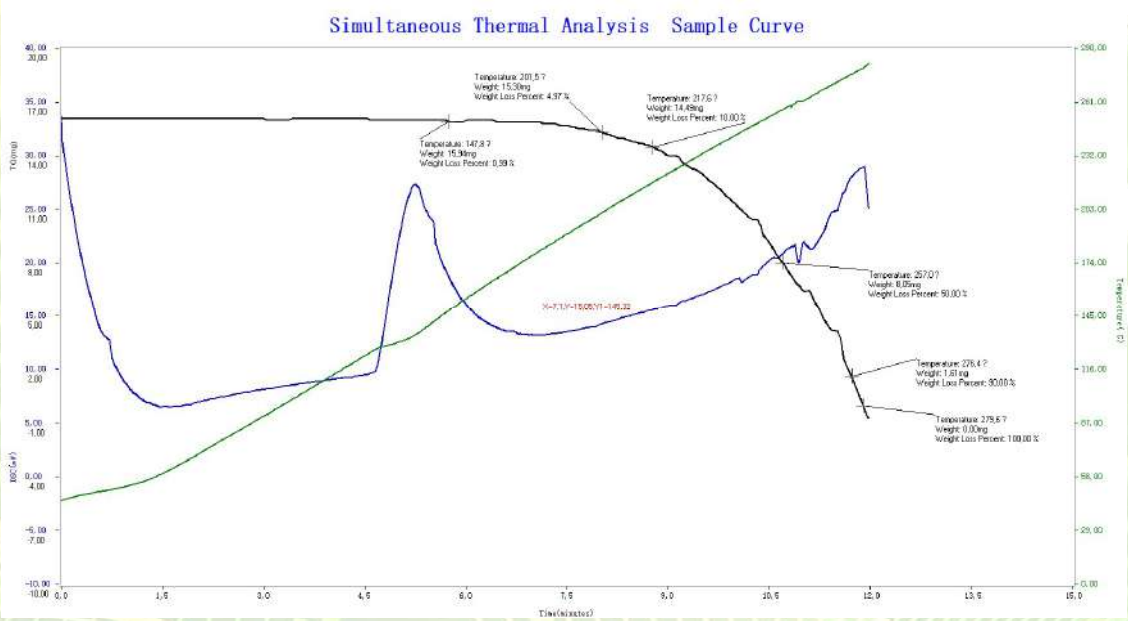



**Figure 2: TGA and DTA analysis of Benzamide**

The experimental procedure for TGA and DTA analysis of benzamide involved initially placing the sample in an argon atmosphere at $25^0$C for 10 minutes, with a flow rate of 80 ml/min. Subsequently, the sample was heated at a rate of $10^0$C/min within the temperature range of 25 to $290^0$C.



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The mass loss observed in benzamide amounts to approximately 100.00% of the total mass. The graph illustrates that within the temperature range of 25-290$^0$C, mass loss occurred primarily in two distinct areas.

The first step involved the loss of a water molecule, resulting in the formation of benzonitrile ($C_6H_5CN$) and ammonia ($NH_3$). This weight loss is evident as a sharp drop in the TGA curve.

The second step in the decomposition process involves the cleavage of the remaining benzamide molecule. This decomposition typically occurs at higher temperatures and is characterized by the release of volatile products such as carbon dioxide ($CO_2$) and benzene ($C_6H_6$). The TGA curve exhibits a gradual decrease in weight as these products are released.

**Thermogravimetric (TG) and differential thermal (DT) analysis of [Mn(BZA)$_4$Cl$_2$]:** Our research focused on the thermal treatment of the coordination compound [Mn(BZA)$_4$Cl$_2$] using KL-JS-100A equipment. The sample was subjected to an initial hold at 25$^0$C in an argon atmosphere with a flow rate of 80 ml/min for 10 minutes. Subsequently, it was heated at a rate of 10$^0$C/min within the temperature range of 25 to 730$^0$C

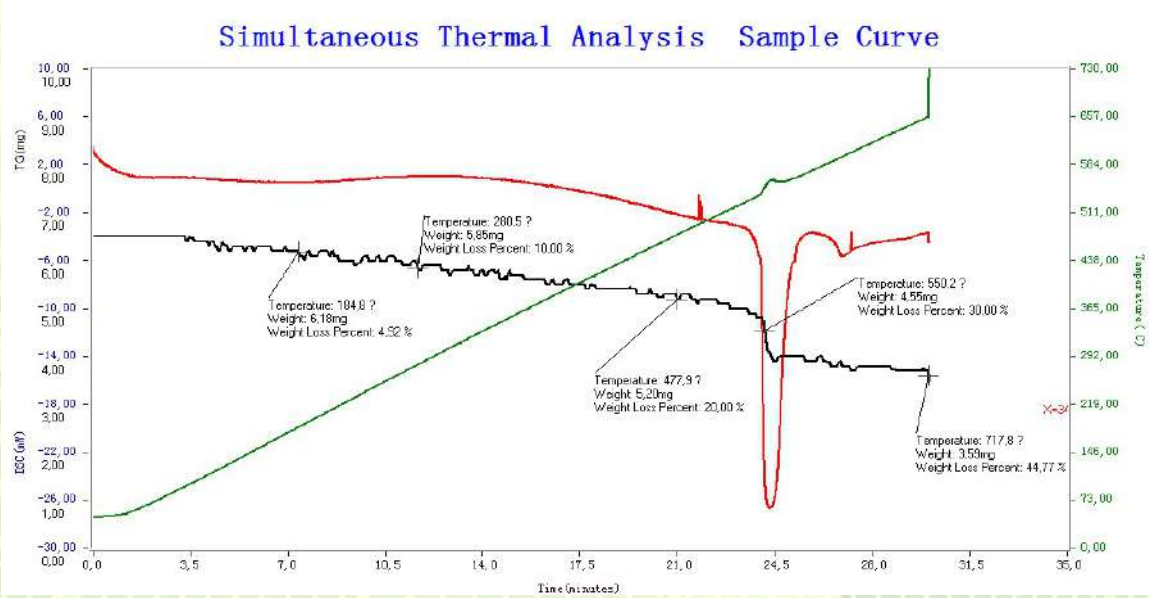



**Figure 3: TGA and DTA Analysis of [Mn(BZA)$_4$Cl$_2$]**

If the thermogravimetric (TG) signal indicates a mass reduction of more than 0.5%, which suggests the evaporation of the solvent present in the crystal, it is necessary to conduct an additional differential scanning calorimetry (DSC) scan [11]. In our sample, the total mass loss is approximately 44.77% (Fig. 3). The graph illustrates the parallel and complementary nature of the TG and DTA lines. Within the temperature range of 25-730$^0$C, the mass loss in the graph occurs primarily in two distinct areas [12]. The initial zone, ranging from 25 to 184.8$^0$C, exhibits a



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4.92% decrease in mass, attributed to the evaporation of absorbed moisture and gases in the sample.

Subsequently, at temperatures above 280.5$^0$C, a significant weight loss of 44.77% is observed. Between 550.2 and 717.8 °C, weight is lost up to 30%. The process is endothermic and proceeds with the absorption of energy. $MnCl_2$ itself is mainly formed as a thermolysis product, the reason being the high boiling point of our salt. During the thermal analysis of $[Mn(BZA)_4Cl_2]$, as the temperature increases, the compound decomposes, which leads to the release of various products such as gases and volatile compounds. However, manganese chloride is a relatively stable compound and tends to remain as a residue after the thermal decomposition process is complete. Increasing the manganese concentration has been found to reduce the rate of loss and enhance stability [13-16]. Various factors, such as sample mass and heating rates, can influence the thermal decomposition processes. It is possible that the sample's mass contributes to the compound's stability, with higher molecular mass generally leading to a higher melting point and greater stability [17].

**CONCLUSIONS:**The combination of $MnCl_2$ and benzamide led to the formation of colorless crystals after a five-week period of evaporation, indicating a chemical reaction between the two compounds. Further testing and analysis can be conducted to explore the nature and properties of these crystals. X-ray structural analysis confirmed the structure of the complex synthesized from MnCl2 salt and benzamide, with the gross formula identified as $[Mn(BZA)_4Cl_2]$ Thermal treatment of our coordination compound $[Mn(BZA)_4Cl_2]$ was performed using the KL-JS-100A equipment. These findings contribute to our understanding of the compound's thermal properties and lay the foundation for future investigations into its potential applications in catalysis and materials science.

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