Journal of Science, Research and Teaching
Vol. 2, No. 4, April - 2023 ISSN: 2181-4406
Innova Science

Synthesis and Study of Monoacetylferrocene Benzoylhydrazone and its Complex with Chromium (Iii) Ion

Annotation:

Hydrazone was synthesized by the interaction of benzoic acid hydrazide with monoacetylferrocene. Based on them, a complex compound with a chromium (III) ion was obtained. The elemental composition was determined by scanning electron microscopy with energy dispersive analysis. Based on the SEM and EDA data, it can be concluded that the complex formation of a metal ion with an organic ligand leads to a change in their microstructure; in particular, numerous peaks of elements were recorded, which was confirmed by EDA. The IR spectrum was taken to elucidate the nature of the binding of the ligand co-ordination center to the central atom of the metal, the complex compound synthesized by us.

Keywords:

condensation, monoacetylferrocene, benzoic acid hydrazone, scanning electron microscope, spectroscopy.

Information about the authors

Sulaymonova Zilola Abdurakhmonovna
PhD, Associate Professor Bukhara State University, Uzbekistan, Bukhara

Mirzaeva Gulrukh Akhtamovna
Assistant of Bukhara Institute of Engineering and Technology, Uzbekistan, Bukhara

Toshpolatova Gulchehra Jahongir qizi
Bachelor of Bukhara State University, Uzbekistan, Bukhara

A huge interest of chemists to ferrocene arose immediately after its discovery. This is due to the wide use of its derivatives in such fields of science and technology as optics, medicine, pharmacology, agriculture, biotechnology, and technology of polymer composite materials. Ferrocene derivatives, especially hydrazones, have high biological activity, which is due precisely to their chelating ability. And the ferrocene fragment also enhances biological activity. A huge number of ferrocene-containing ligands are currently used in coordination chemistry, and transition metal complexes are widely used in agriculture as plant growth regulators [1-5].

To expand the line of bidentate chelating ligand systems containing ferpocene fragments, we synthesized a ligand, benzoic acid hydrazone, by condensation of monoacetylferpocene.

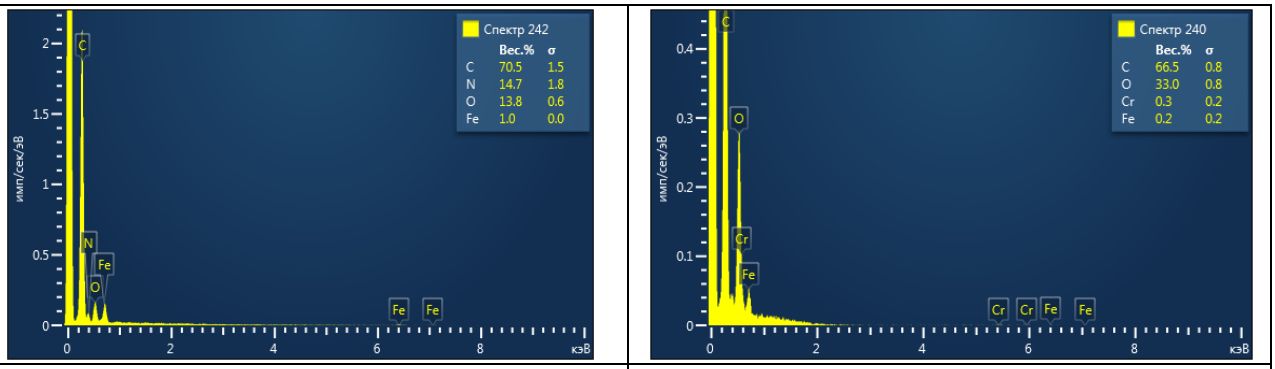
The aim of this work was the synthesis and determination of the structure of the complex of chromium(III) with benzoylhydrazone of monoacetylferrocene in order to study the biological activity

of the synthesized substances. We chose monoacetylferrocene benzoylhydrazone and its complex with transition metals Cr(III) as objects of study [6-10].

Synthesis of benzoylhydrazone monoacetylferrocene. To a solution of 11.4 g (0.05 mol) of monoacetylferpocene in 40 ml of ethanol was added a solution of 6.8 g (0.05 mol) of benzoic acid hydrazide in 30 ml of ethanol into a round-bottom flask. The reaction mixture was refluxed for four hours and left for three days. The brown precipitate that formed was filtered off, washed twice with ethanol, and dried. After crystallization from a solvent system (ethanol + DMSO), benzoylhydrazoneferrocenoylacetone was obtained. Polycrystalline to brown product with a yield of 68%, T_m. 143^0C.

Synthesis of a complex of benzoylhydrazone monoacetylferrocene with a chromium(III) ion. To a hot solution of 2.595 g (0.0075 mol) of benzoylhydrazone monoacetylferpocene in 30 ml of absolute ethyl alcohol was mixed 0.455 g (0.0025 mol) of hot aqueous ammonia (ammonia). The reaction mixture was heated for 3 hours until a precipitate formed. The brick-brown precipitate that formed was filtered off and washed several times with absolute ethyl alcohol. Recrystallized from ethanol solution. The yield is 54%.

The amount of carbon, nitrogen, oxygen and metals in the synthesized ligand and complex were determined by scanning electron microscopy with energy dispersive analysis (SEM-EDA) (SEM - EVO MA 10 Zeiss, Germany). Based on the SEM and EDA data, it can be concluded that the complex formation of a metal ion with an organic ligand leads to a change in their microstructure; in particular, numerous peaks of elements were recorded, which was confirmed by EDA (Fig. 1).



a **b**

Fig. 1. SEM-EDA data of the ligand (a) and its complex with a chromium(III) ion (b).

The IR spectrum was taken to elucidate the nature of the binding of the ligand co-ordination center to the central atom of the metal, the complex compound synthesized by us. The spectra were taken on an IR spectrophotometer "IR Tracer-100" (Shimadzu, Japan, 2017) in the region of 400-4000 cm^{-1}; scanning speed - 20 spectra per second.

In the IR spectrum of the ligand, characteristic absorption bands were found related to symmetric (ν_s) and antisymmetric (ν_{as}) vibrations of the N–N, C–N, C=N, C–H and N–H functional groups and bonds of about 1039, 1282-1303 , 1519, 2985 cm^{-1}. The stretching vibrations of the C–H bond of the methyl group of the ketone fragment are noted in the region of 2985 cm^{-1} (ν_{as} C–H), as well as in the mid-frequency regions of 1519 cm^{-1} (ν_s C=N) and 14 (ν_s C=N) and 14 absorption bands of symmetric and antisymmetric vibrations of the functional group C=N appear. In the spectrum of the ligand in the low-frequency region, absorption bands of medium intensity at 464 and 501 cm^{-1}, which belong to the stretching vibrations of the C–H bonds of two cyclopentadienyl bonds are fixed (Fig. 2) [11-16].

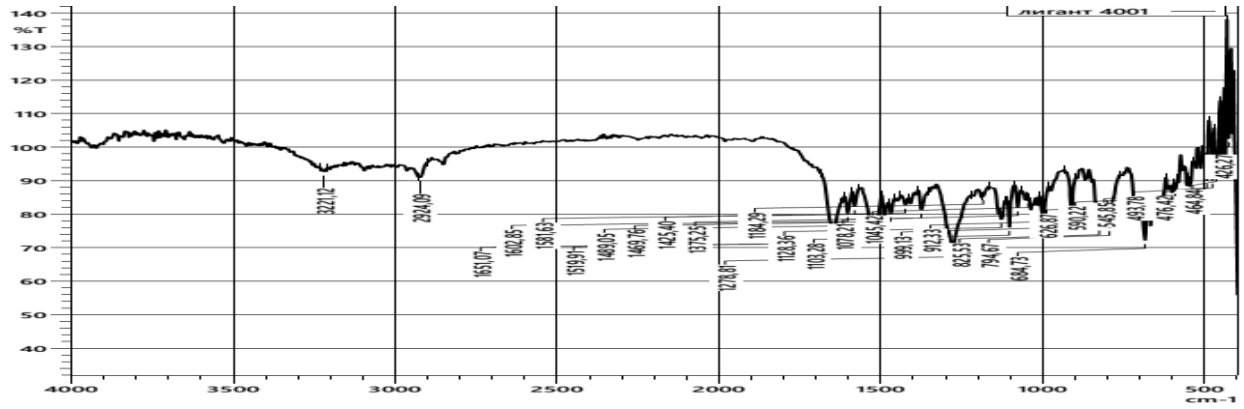


Fig. 2. IR spectrum of benzoylhydrazone of monoacetylferrocene.

Analysis of the IR spectrum of the obtained complex showed that the spectrum of the complex compound does not contain the characteristic absorption bands of the free ligand at about 3221 cm^{-1} $\nu_{(N-H)}$. This indicates enolization and deprotonation of the ligand during complex formation. In the spectrum of the complex, a single intense band at 1539 cm^{-1}, which is absent in the spectrum of the ligand, corresponds to the stretching vibrations of the –N=C–O– system, and the intense band in the mid-frequency region at 1604 cm^{-1} refers to the stretching vibrations of the N=C–C bond C=N. In the spectrum of the complex, the bands of medium intensity at 1105 cm^{-1} refer to $\nu_{(N-N)}$, which are shifted by 66 cm^{-1} to the high-frequency region, and for the $\nu_{(C=N)}$ bonds to the low-frequency region by 14 cm^{-1} compared to spectrum of free ligands. This indicates that the nitrogen atom in the azomethine bond is also involved in coordination (Fig. 3).

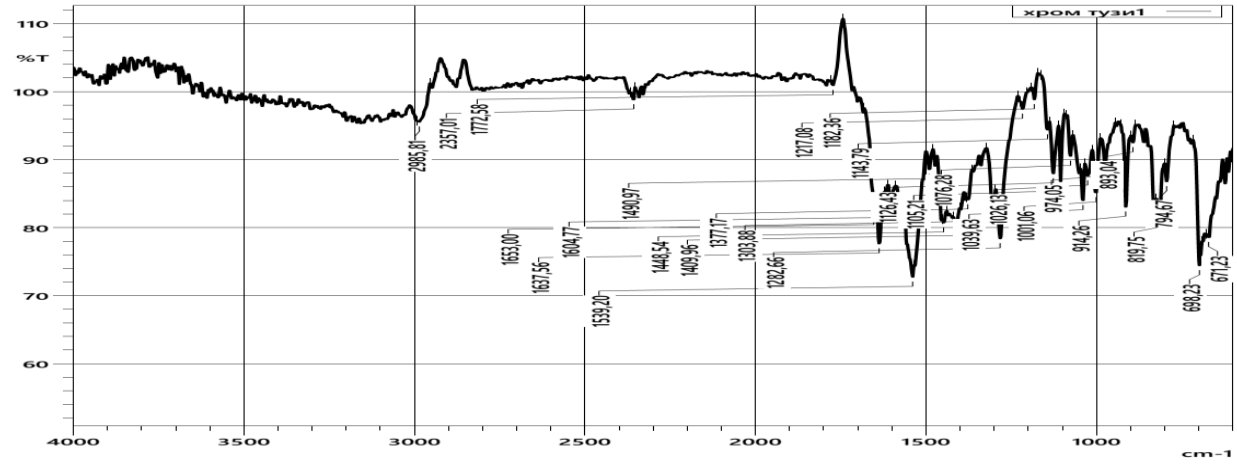


Fig. 3. Spectrum of chromium(III) complex.

All ammonia complexes are characterized by absorption in the region of 3300–3450 cm^{-1}, due to the stretching vibrations of the N–H fragments. Various types of bending vibrations (pendulum, scissor, fan) of ammonia molecules are displayed on the spectra in the region of 1604, 1303 and 819 cm^{-1} [17-22].

REFERENSE

1. Cullen W.R.,Woollins J.D. Ferrocene-containing metal complexes// Coord. Chem. Rev. – 1981. – Vol. 39. – P. 1-30.
2. Colacot T.J. A Concise Updateon the Applications of ChiralFerrocenylPhosphines in Homogeneous Catalysis Leading to Organic Synthesis // Chem. Rev. – 2003. –Vol. 103. – P. 3101-3118.

3. Kealy T.J., Pauson P.L. A New Type of Organo-Iron Compound. // Nature. -1951. - Vol. 168. - P. 1039-1940.
4. Miller S.A., Tebboth J.A., Tremaine J.F. Dicyclopentadienyliron // J. Chem. Soc. -1952. - Vol. 632. - P. 632-635.
5. Wilkinson G., Cotton F.A. Cyclopentadienyl and Arene Metal Compounds // Progr. Inorg. Chem. - 1959. - Vol. 1. - P. 1-124.
6. Сулаймонова З.А., Умаров Б.Б. Получение *мета*-нитробен-зоилгидразона ферроценоилацетона и синтезы на его основе // Химическая технология. Контроль и управление. - 2021. - №4(100). - С. 5-11.
7. Умаров Б.Б., Сулаймонова З.А., Ачылова М.К. Синтез комплексов на основе монокарбонильных производных ферроцена с гидразидами карбоновых кислот // Universum: Химия и биология. Россия, - 2021. - №1(79). - С. 85-89 URL: *http:///7universum.com/ru/nature/archive/item/11144*
8. Сулаймонова З.А., Умаров Б.Б., Кодирова З.К. Термическое поведение *мета*-нитробензоилгидразона ферроценоилацетона и его комплекса с ионом меди(II) // Universum: Химия и биология. Россия, - 2021. 11(89). - С. 15-19 URL: *https:///7universum.com/ru/nature/archive/new*
9. Умаров Б.Б., Сулаймонова З.А., Тиллаева Д.М. Комплексные соединения переходных металлов на основе конденсации производных ферроцена с гидразидами карбоновых кислот // Научный вестник Наманганского государственного университета. - 2020. - №9. - С. 58-63.
10. Sulaymonova Z.A., Umarov B.B., Choriyeva S.A., Navruzova M.B. Synthesis of Complexes Based On Monocarbonyl Ferrocene Derivatives with Carbonic Acid Hydrases // International Journal of Academic Pedagogical Research (IJAPR). - 2021. - Vol. 5. - С. 134-137.
11. Умаров Б.Б., Сулаймонова З.А. Синтез комплекса никеля(II) на основе производных ферроцена // Федеральное государственное бюджетное образовательное учреждение высшего образования "МИРЭА - Российский технологический университет" Симпозиум “Химия в народном хозяйстве”. Дубровицы -2020. - С. 106-107.
12. Сулаймонова З.А., Наврузова М.Б., Чориева С.А. Термическое исследование производных ферроцена // SCIENTIFIC COLLECTION «INTER-CONF». - 2021. - №45. - С. 473-478.
13. Сулаймонова З.А., Наврузова М., Чориева С. Синтез β-дикарбонильного производного ферроцена-ферроценоилацетона // “Замонавий кимёнинг долзарб муаммолари” Республика миқёсидаги хорижий олимлар иштирокидаги онлайн илмий-амалий анжуманининг илмий мақолалари тўплами. Бухоро -2020, 4-5 декабрь. - Бухоро. - С. 375-377.
14. Умаров Б.Б., Сулаймонова З.А., Бахранова Д.А. Синтез β-дикарбонильных производных ферроцена // “Наука и инновации в современных условиях Узбекистана” Республиканская научно-практическая конференция. Нукус-2020, 20 май. - С. 114-115.
15. Сулаймонова З.А., Умаров Б.Б. ЯМР спектроскопическое - исследование дигидразона янтарной кислоты с 1-ферроценилбутандионом-1,3 // “Комплекс бирикмалар кимёси ва аналитик кимё фанларининг долзарб муаммолари”. Республика миқёсидаги илмий-амалий анжуман. Термиз-2022, 19-20 май, 208-209 б.
16. Сулаймонова З.А., Авезова Ф.М. Комплексы металлов с гидразонами моноацетилферроцена // “Замонавий кимёнинг долзарб муаммолари” Республика миқёсидаги хорижий олимлар иштирокидаги онлайн илмий-амалий анжуманининг илмий мақолалари тўплами. Бухоро-2020, 4-5 декарь. - С. 393-395.

17. Сулаймонова З.А. Термическое исследование бензоилгидразона ферроценоилацетона и его комплексов с переходными металлами // Сборник трудов международной научно-теоретической конференции на тему: «Куатбековские чтения-1: Уроки Независимости», посвященной 30-летию Независимости Республики Казахстан 23 апрель 2021 г. - С. 9-12.
18. Sulaymonova Z.A., Avezov H.T., Qodirova Z.Q., Mutalipova D.B. Synthesis and IR spektroskopic study of hydrazones of 1-ferrocenylbutanedione-1,3 and theire complexes // International Journal of Early Childhood Special Education (INT-JECSE) ISSN: 1308-5581 Vol 14, Issue 03 2022.
19. Sulaymonova Z.A., Umarov B.B. Research of complex compounds transition metals with succinic dihydrazone acids based on ferrocenoylacetone // “The Chemistry of Complex Compounds and Current affairs of analytical chemistry problems". Republic-wide scientific-practical conference. Termiz-2022, May 19-21, P. 19-20.
20. Сулаймонова З.А., Умаров Б.Б., Муталипова Д.Б. Синтез и спектроскопическое исследование комплексных соединений некоторых 3d металлов с продуктом конденсации 1-ферроценилбутандиона-1.3 и дигидразида янтарной кислоты // Самарқанд давлат университети илмий ахборотномаси, 2022 йил 5-сон (135), С. 27-32.
21. Sulaymonova Z.A., Sharifova N.A., Xatamov U.F. NMR spectroscopic investigation of succinic acid dihydrazone with 1-ferrocenylbutanedione-1,3. A collection of materials of the international scientific and practical conference on the topic "Current problems of the chemistry of coordination compounds". Bukhara-2022, December 22-23, P. 107-109.
22. Umarov B.B., Achilova M.K., Xudoyberganov O.I. Synthesis and NMR spectroscopic study of hydrazone derivaties of ferrocenoylacetone and their complexes // Journal Actuale Problems of Modern Sciense, Education and Training. JUNE, 2022 - №6. ISSN 2181-9750. P. 92-100.