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# INVESTIGATION OF CRYSTAL STRUCTURE AND ANALYTICAL OPPORTUNITIES OF 3 - (4'-FLUOROPHENYLIMINE) - 1 - PHENYLBUTANONE – 1

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**Abstract:** A new organic reagent based on benzoylacetone, 3-(4'-fluorophenylimine) -1 -phenylbutanone - 1 was synthesized and its crystal structure determined by X-ray structural analysis. Reagent crystals are monoclinic:  $a=6.4520(6) \text{ \AA}$ ,  $b=28.685(2) \text{ \AA}$ ,  $c=7.2003(6) \text{ \AA}$ ,  $\alpha=90^\circ$ ,  $\beta=91.163(2)^\circ$ ,  $\gamma=90^\circ$ ,  $V=1332.3(2) \text{ \AA}^3$ , space group  $P2_1/n$ ,  $Z=4$ . Results of X-ray diffraction analysis showed that the reagent molecule in the crystal has trans-configuration. The reagent dissociation constant ( $pK = 9.89 \pm 0.05$ ) and the stability constants of its complexes with some metals were determined by means of potentiometric titration method. The complexation of the reagent with iron (III) in the presence of dianthipirilmethane and its homologs - dianthipyrylphenylmethane and dianthipyryl-o-oxyphenylmethane was studied spectrophotometrically. A highly selective method for the direct determination of iron micro-quantities in red and Hovsani onions developed.

**Keywords:** synthesis, X-ray diffraction analysis, complex formation, constant of dissociation, constant of stability, iron (III), dianthipyrylmethane, dianthipyrylphenylmethane, dianthipyryl-o-oxyphenylmethane

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## Introduction

Numerous organic reagents for spectrophotometric determination of iron (III) are known [1–7]. Among them,  $\beta$ -diketones and related derivatives with their selectivity are different from others [8,9]. The high selectivity of these reagents with respect to iron (III) ions makes it necessary to synthesize and study analytical capabilities of new  $\beta$ -diketone derivatives.

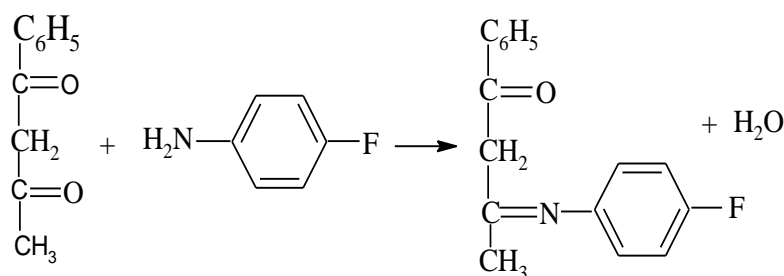
The purpose of this work is the synthesis of benzoylacetone-based organic reagent - 3-(4'-fluorophenylimine) -1-phenylbutanone-1; analysis of its crystal structure, complex formation with iron (III) ion in the presence of dianthipyrylmethane and its homologues, as well as development of spectrophotometric method to determine trace amounts of iron in onions.

## Experimental part

### Synthesis of reagent.

Reagent - 3- (4'-fluorophenylimine) -1-phenylbutanone-1 was synthesized according to

a known method [11], using benzoylacetone and 4-fluoroaniline.



The reagent is highly soluble in ethanol, acetone and dimethyl sulfoxide. The yield of the reagent is 74.6%. The purity was tested by paper chromatography, and its crystal structure determined by X-ray diffraction (XRD).

#### Equipment

X-ray diffraction study was performed on a SMART APEX II CCD diffractometer (MoK $\alpha$ -ray graphite monochromator,  $\phi$  and  $\omega$  scan). The optical density of the solutions was measured on a Lambda-40 spectrophotometer with computer software (Perkin Elmer) and on a KFK-2 photoelectric calorimeter in cuvettes with a layer thickness of 1.0 cm. The pH of the solutions was measured with PHS-25 ionomer supplied with glass electrode.

#### Solutions and reagents

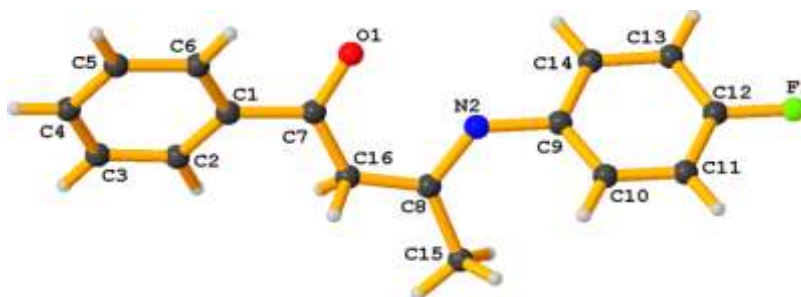
A standard  $1.0 \cdot 10^{-1}$  M solution of iron (III) was prepared by dissolving a calculated weighed metallic iron according to the method [10-11]. Working  $1.0 \cdot 10^{-3}$  M solutions were obtained by diluting the stock solution with distilled water before use.

$1.0 \cdot 10^{-3}$  M solution of 3- (4'-fluorophenylimine) -1-phenyl butanone-1 (R) and  $1.0 \cdot 10^{-2}$  M solutions of diantipyrylmethane (DAM), diantipyrylphenylmethane (DAPhM) and diantipyryl-o-oxyphenylmethane (DAOPhM) which were prepared by dissolving the appropriate weights in ethanol was used. All reagents used were of high analytical grade. To create required acidity, ammonium acetate buffer solutions (pH 3-11) and HCl (pH 0-2) were used.

#### Results and discussions

**XRD of 3- (4'-fluorophenylimine) – 1 – phenylbutanone-1.** To establish the structure of the reagent, x-ray analysis was used. Single crystals for X-ray diffraction were obtained by double crystallization of the reagent from ethanol. According to the X-ray diffraction study results, the reagent has an empirical formula  $C_{16}H_{14}FNO$  and a monoclinic syngony. At  $T=269K$ :  $a=6.4520(6)$  Å,  $b=28.685(2)$  Å,

$c=7.2003(6)$  Å,  $\alpha=90^\circ$ ,  $\beta=91.163(2)^\circ$ ,  $\gamma=90^\circ$ ,  $V=1332.3(2)$  Å<sup>3</sup>, space group  $P2_1/n$ ,  $Z=4$ ,  $d_{calc.} = 1.273$  mg/m<sup>3</sup>, adsorption coefficient  $\mu=0.089$  mm<sup>-1</sup>. The final values of the divergence factors were as follows:  $R_1=0.2543$ ,  $wR_2=0.6447$ , crystal size  $0.320 \cdot 0.230 \cdot 0.170$  mm<sup>3</sup>. A perspective view of the reagent molecule with the numbering of non-hydrogen atoms is shown in Fig. 1.



**Figure 1.** Molecular structure of  $C_{16}H_{14}FNO$

It was established that the reagent molecule has a non-planar structure. The angle between phenyl fragments (A) [C(1)÷C(6)] and (B) [C(9)÷C(14)] is  $76.59^\circ$ . C (8) while C (16) atoms are located out of the plane of phenyl fragments. The torsion angle C (8) -N (2) -C (9) -C (10) and C (2) -C (1) -C (7) -C (16) -O (1) is equal to  $50.16^\circ$  and  $30.50^\circ$ , respectively. The N (2) -C (8) -C (16) -C (17) atoms are located practically in

the same plane, the standard deviation from the plane is  $0.0033^\circ$ . The deviation of phenyl fragments A and B from N (2) -C (8) -C (16) -C (7) plane is  $27.89^\circ$  and  $48.62^\circ$  respectively. Results of the X-ray diffraction analysis showed that the 3- (4'-fluorophenylimine)-1-phenylbutanone-1 molecule in the crystal has a trans-configuration.

### Determination of the dissociation constant of the reagent and the stability constant of its complexes.

The dissociation constant of 3- (4'-fluorophenylimine) – 1 – phenylbutanone-1 and the stability constant of its complexes with a number of metals were determined by means of potentiometric titration.

To determine the dissociation constant

of the reagent, its 50 sm<sup>3</sup> 5.0·10<sup>-4</sup> mol/l aqueous-ethanol (3:7) solution was titrated with 5.0 10<sup>-3</sup> mol/l carbon dioxide-free caustic solution. Titration was carried out at 25 °C and the solutions stirred using a magnetic stirrer with a nitrogen transmission. To calculate the dissociation constant of the reagent, mathematical method of Schwarzenbach was used [12]:

$$\lg K_{\text{Diss}} = \text{pH} + \lg [\text{HA}] - \lg [\text{A}^-]$$

According to the calculation, the dissociation constant of the reagent is  $\text{pK}_{\text{dis}} = 9.89 \pm 0.05$ . To determine the stability constant of the reagent complexes with metals, aqueous solutions of the  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ ,  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  salts were used. Potentiometric titration of mixtures of reagent and salts of appropriate metals are carried out in an aqueous medium with the ratio  $\text{Me}:\text{R} = 1:1$ . Therefore, equimolar 5.0·10<sup>-4</sup> mol/l stock solutions were

prepared for the titration of the metal salt and reagent in water and in a water-ethanol mixture (3:7), respectively. The total volume of the solution to be titrated was 50 sm<sup>3</sup> and the titrant was a 5.0·10<sup>-3</sup> mol/l solution of caustic potassium. The ionic strength of the solutions was kept constant ( $\mu = 0.1$  mol/l) by adding the calculated amount of KCl.

When calculating the stability constants of the complexes, mathematical method of Chaberek and Martell was used [13]:

$$K_{\text{stab}} = \frac{C_R - [R^-] \cdot x}{[R^-]^2 x}$$

where  $[R^-]$  is the equilibrium concentration of the reagent found by the formula

$$[R^-] = \frac{\{C_R(1-a) - [H^+] + [OH^-]\} K_{\text{dis}}}{[H^+]} \\ X = \frac{H^+}{K_{\text{dis}}} + 1$$

a - neutralization point

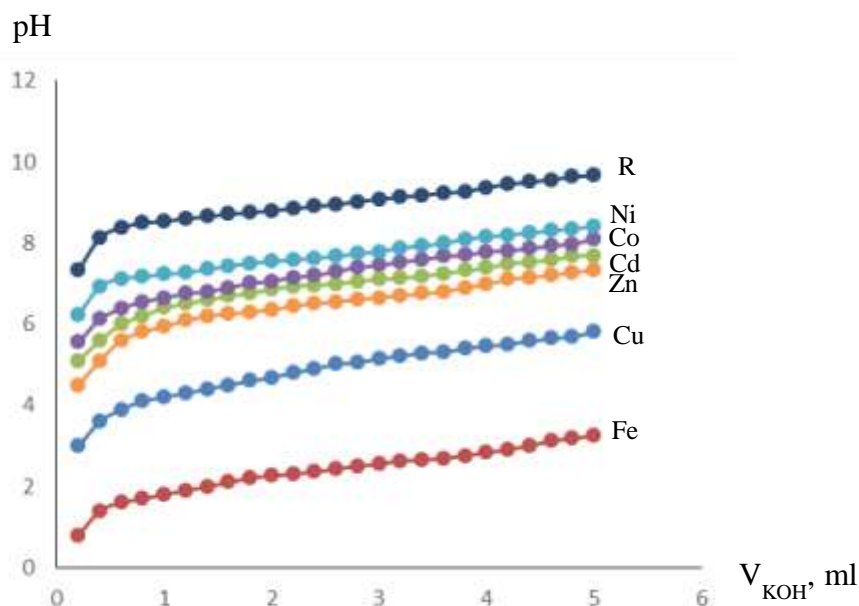
$$a = \frac{OH^-}{C_R}$$

Based on the experimental data, it was found that the titration curves of the complexes are located below the reagent titration curve (Fig.

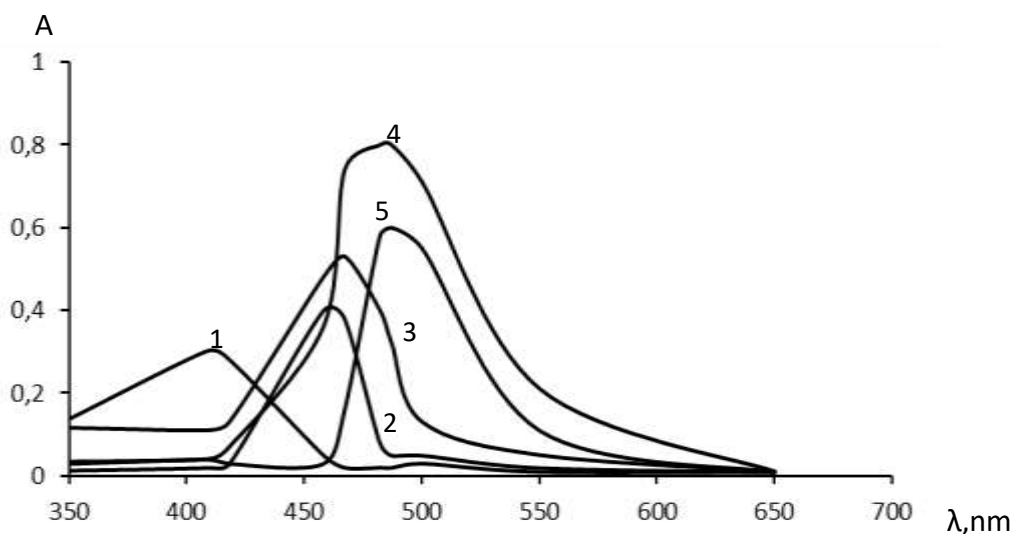
2). This points to the presence of complexation in the solution.

**Table 1.** The stability constant of complexes of 3- (4'-fluorophenylimine) -1-phenyl butanone-1 with metals.

Metal	Fe <sup>3+</sup>	Cu <sup>2+</sup>	Zn <sup>2+</sup>	Ni <sup>2+</sup>	Co <sup>2+</sup>	Cd <sup>2+</sup>
lgK	9.63±0.04	6.52±0.03	6.20±0.04	5.05±0.05	4.96±0.05	4.69±0.05



**Figure 2.** Potentiometric titration curves of 3- (4'-fluoro-phenylimine) -1 –phenylbutanone - 1 with some metals.



**Figure 3.** Absorbance spectra of Fe(III) complexes at optimal pH.  
 1.R, 2.Fe(III)-R, 3.Fe(III)-R-DAM, 4.Fe(III)-R-DAPhM, 5.Fe(III)-R-DAOPhM.  
 $C_{Fe}=4.0 \cdot 10^{-5}$  M,  $C_R=1.0 \cdot 10^{-4}$  M,  $C_{DAM(DAPhM, DAOPhM)}=2.0 \cdot 10^{-4}$  M,  $\lambda_{40}$ ,  $l=1$  cm.

**Complexation of iron (III) with a reagent in the presence of diantipyrylmethane and its homologues.**

To detect the analytical capabilities of 3- (4'-fluorophenylimine) – 1 – phenylbutanone-1,

its complexation with iron (III) was studied in the presence of diantipyrylmethane and its homologues like diantipyrylmethane and diantipyryl-o-oxyphenylmethane. When iron (III) interacts with the reagent, a colored complex is formed in an acidic medium at pH

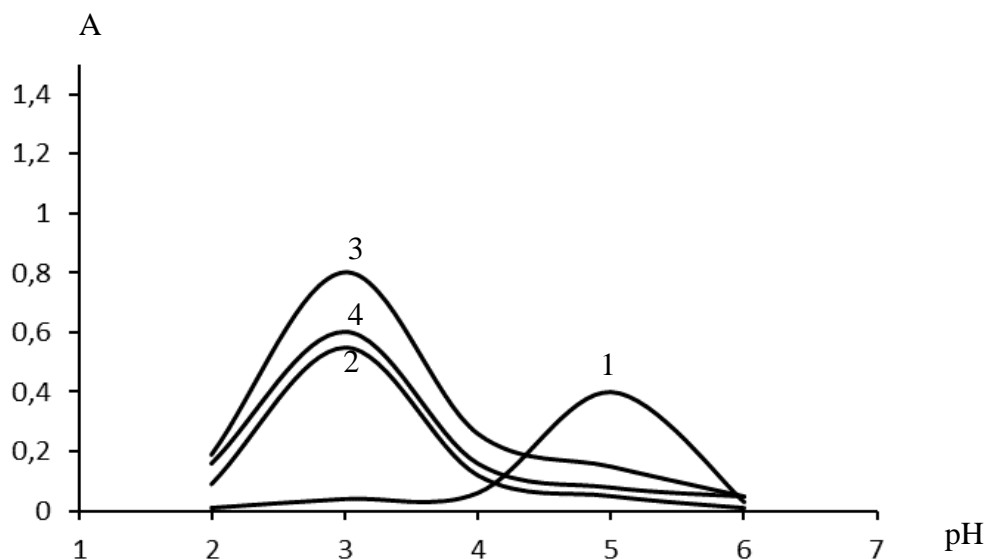
1.0-6.0. Fig. 3 shows the absorption spectra of the reagent, homogeneous-ligand and mixed-ligand complexes. The light absorption of the reagent is maximum at 408 nm (curve 1), and its homogeneous ligand complex of iron (III) at 418 nm (curve 2).

In the presence of DAM and its homologues, mixed ligand complexes of Fe (III)-R-DAM, Fe (III)-R-DAPhM and Fe (III)-R-DAOPhM are formed. As can be seen from Fig. 3, during the formation of mixed-ligand complexes, a bathochromic shift and a hyperchromic effect are observed as compared to the spectrum of the homogeneous-ligand complex. The light absorption of the complexes Fe (III)-R-DAM, Fe (III)-R-DAPhM and Fe (III)-R-DAOPhM is maximum at 458, 482 and 487 nm (curves 3, 4 and 5), respectively.

The study into dependence of optical density on the pH of the solution showed that mixed-ligand complexes of iron (III) are also formed in an acidic medium at a pH of 1.0-6.0. As can be seen from Fig. 4, the yield of the Fe (III) -R complex is maximum at pH 4.5-5.0 (curve 1) and, with the formation of mixed-ligand complexes, their maximum yield shifts to more acidic medium compared to the homogeneous-

ligand complex. The optimum pH values ( $\text{pH}_{\text{opt}}$ ) for all mixed-ligand complexes are 2.5-3.0 (curves 2, 3 and 4).

The color of the reagent and complexes depends on the pH of the medium; therefore, the absorption spectra of the complexes were studied with respect to the control experiment [R+DAM (DAPhM, DAOPhM)]. It was established that in the spectra for homogeneous-ligand and mixed-ligand complexes as compared to the control experiment, the optical density is maximum at 490 nm. The effect of reactant concentration, temperature and time on the formation of iron (III) complexes was studied. The yield of the homogeneous ligand complex Fe (III) -R is maximum at concentration of  $1.0 \cdot 10^{-4}$  M of reagent, and the mixed-ligand complexes Fe (III)-R-DAM, Fe (III)-R-DAPhM and Fe (III)-R-DAOPhM at  $8.0 \cdot 10^{-5}$  M of reagent and  $2.0 \cdot 10^{-4}$  M DAM (DAPhM, DAOPhM), respectively. The complexes are formed immediately after mixing the solutions of the components and differ in their stability. So, if the Fe (III)-R complex is stable for 2 hours and when heated to  $60^\circ \text{C}$ , the mixed-ligand complexes are stable for more than a day and when heated to  $80^\circ \text{C}$ .



**Figure 4.** Influence of pH on absorbance of Fe(III) complexes at  $\lambda_{\text{opt}}$  on the background of the control experiment.

1. Fe(III)-R, 2. Fe(III)-R-DAM, 3. Fe(III)-R-DAPhM, 4. Fe(III)-R-DAOPhM.

$C_{\text{Fe}} = 4.0 \cdot 10^{-5}$  M,  $C_{\text{R}} = 1.0 \cdot 10^{-4}$  M,  $C_{\text{DAM(DAPhM, DAOPhM)}} = 2.0 \cdot 10^{-4}$  M, KFK-2,  $l = 1$  cm.

The ratios of components in the composition of the formed colored complexes were established by method of isomolar series, the Starik-Barbanel's relative yield method and the equilibrium shift [14]. All methods showed that ratios of components in the homogeneous ligand complex are 1:2, and in the mixed ligand complexes - 1:2:2.

The calibration graph is linear in the range

of iron (III) concentration of 0.22-11.20 mcg/ml for the complex Fe (III)-R, 0.17-6.72 mcg/ml for the Fe (III)-R-DAM and Fe (III)-R-DAOPhM complexes and 0.11-11.2 mcg/ml for the Fe (III)-R-DAPhM complex. Molar absorption coefficients, as well as other chemical and analytical characteristics of the complexes are shown in Table 2.

**Table 2.** Chemical-analytical characteristics of iron (III) complexes with 3-(4'-fluorophenylimine) – 1-phenyl-butanone-1 in the presence of diantipyrylmethane and its homologues.

Reagent	pH <sub>opt</sub>	$\lambda_{\max}$ , nm	$\Delta\lambda$ , nm	Stoichiometric ratios	$\varepsilon \cdot 10^3$ (at $\lambda_{\max}$ )	Linearity range of calibration curve, mcg/ml
Fe(III)-R	4.5-5.0	418	10	1:2	10.00±0.12	0.22-11.20
Fe(III)-R-DAM	2.5-3.0	458	50	1:2:2	12.75±0.20	0.17-6.72
Fe(III)-R-DAPhM	2.5-3.0	482	74	1:2:2	20.00±0.24	0.11-11.20
Fe(III)-R-DAOPhM	2.5-3.0	487	79	1:2:2	15.25±0.19	0.17-6.72

The study into the influence of foreign ions on the determination of iron (III) showed that in the presence of diantipyrylmethane and its homologues, the selectivity of the reaction significantly increased. The method of iron (III) determination in a mixed-ligand complex Fe (III)-R-DAPhM has the highest selectivity. For example, the determination of iron (III) does not interfere with more than 5,000-fold amounts of alkali, alkaline earth and rare earth metals; 1500-fold - Ni (II), Co (II), Cd (II), Mn (II), Mg (II); 1000 – fold - Cu (II), Zn (II), Pb (II), Cr (III),  $\text{HPO}_4^{2-}$ ; 350-fold - Al (III), Ga (III), In (III), Th (IV), U (VI), F<sup>-</sup>,  $\text{C}_2\text{O}_4^{2-}$ ; 100-fold - V (V), Sb (V), Sn (IV) Ti (IV).

The developed technique was applied to determine trace amounts of iron in red and Hovsani onions.

**Analysis technique.** A sample of onion weighing 300 g after drying is placed in a graphite cup, burned in a muffle furnace at 550-750° C until complete decomposition of organic substances. The resulting ash is dissolved in a mixture of 15 ml of HCl and 5 ml of  $\text{HNO}_3$  in a glassy carbon dish and treated three times with 4-5 ml of HCl at 60-70° C until the nitrogen oxides are completely distilled off. Next, the mixture is dissolved in distilled water, filtered in a flask with a capacity of 100 ml and diluted to the mark. An aliquot of the solution is transferred to a 25 ml volumetric flask, 2.0 ml of a  $1.0 \cdot 10^{-3}$  M reagent solution, 0.5 ml of a  $1.0 \cdot 10^{-2}$  M solution of DAPhM added and the volume reached the mark with ammonium acetate buffer solution pH=3.0.

**Table 3.** Results of determination of iron (III) in food products (n=5, P=0.95)

Analyzed object	Found Fe, % weight.	
	R+DAPhM	AAS
Red onion	$(3.92 \pm 0.05) \cdot 10^{-2}$	$(3.89 \pm 0.05) \cdot 10^{-2}$
Hovsani onion	$(1.3 \pm 0.04) \cdot 10^{-1}$	$(1.5 \pm 0.10) \cdot 10^{-1}$



The results are presented in Table. 3 as compared to the analysis of the atomic absorption method (AAS). The results of the proposed method and AAS are in good agreement with each other. Thus, the proposed

method for the determination of iron (III) with 3-(4'-fluorophenylimine) – 1-phenylbutanone-1 in the presence of DAPhM is simple, rapid to provide reliable results.

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### 3- (4'-FLÜORFENİLİMİN) -1-FENİL BUTANON-1 REAKTİVİNİN KRİSTAL QURULUŞUNUN VƏ ANALİTİK XÜSUSİYYƏTLƏRİNİN TƏDQIQI

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Benzoilaseton əsasında yeni üzvi reagent 3-(4'-flüorfenilimin)-1-fenilbutanon-1 sintez edilmişdir və rentgen quruluş analizi ilə onun kristal quruluşu öyrənilmişdir. Reaktiv kristalları monoklinikdir:  $a = 6.4520(6) \text{ \AA}$ ,  $b = 28.685(2) \text{ \AA}$ ,  $c = 7.2003(6) \text{ \AA}$ ,  $\alpha = 90^\circ$ ,  $\beta = 91.163(2)^\circ$ ,  $\gamma = 90^\circ$ ,  $V = 1332.3(2) \text{ \AA}^3$ , boşluq qrupu  $P2_1/n$ ,  $Z = 4$ . Rentgen quruluş analizinin nəticələri reagent molekulunun trans konfiqurasiyasına malik olduğunu göstərdi. Potensiometrik titrləmə metodu ilə reagentin dissosiasiya sabiti ( $pK = 9.89 \pm 0.05$ ) və bir sıra metallarla komplekslərinin davamlılıq sabiti müəyyən edilmişdir. Diantipirilmetan və onun homoloqları, diantipirilfenilmetan və diantipiril-o-hidroksifenilmetan iştirakı ilə reagentin dəmirlə (III) əmələ gətirdiyi kompleks birləşmələri öyrənmək üçün spektrofotometrik metoddan istifadə edilmişdir. Qırmızı və Hövsan soğanlarında dəmirin(III) mikromiqdarının birbaşa müəyyənəşdirilməsi üçün yüksək seçici üsul hazırlanmışdır.

**Açar sözlər:** sintez, rentgen quruluş analizi, kompleksəmələgəlmə, dissosiasiya sabiti, davamlılıq sabiti, dəmir (III), diantipirilmetan, diantipirilfenilmetan, diantipiril-o-hidroksifenilmetan

### ИССЛЕДОВАНИЕ КРИСТАЛЛИЧЕСКОЙ СТРУКТУРЫ И АНАЛИТИЧЕСКИХ ВОЗМОЖНОСТЕЙ 3-(4'-ФТОРФЕНИЛИМИН)-1-ФЕНИЛБУТАНОНА-1

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Синтезирован новый органический реагент на основе бензоилацетона-3-(4'-фторфенилимин)-1-фенилбутанона-1 и методами рентгеноструктурного анализа установлена его кристаллическая структура. Кристаллы реагента моноклинные:  $a = 6.4520(6) \text{ \AA}$ ,  $b = 28.685(2) \text{ \AA}$ ,  $c = 7.2003(6) \text{ \AA}$ ,  $\alpha = 90^\circ$ ,  $\beta = 91.163(2)^\circ$ ,  $\gamma = 90^\circ$ ,  $V = 1332.3(2) \text{ \AA}^3$ , пространственная группа  $P2_1/n$ ,  $Z = 4$ . Результаты РСА показали, что молекула реагента в кристалле имеет транс-конфигурацию. Определены константа диссоциации реагента ( $pK = 9.89 \pm 0.05$ ) и константы устойчивости его комплексов с рядом металлов методом потенциометрического титрования. Спектрофотометрическим методом исследовано комплексообразование реагента с железом(III) в присутствии диантипирилметана и его гомологов - диантипирилфенилметана и диантипирил-o-оксифенилметана. Разработана высокоселективная методика непосредственного определения микроколичеств железа в красном и Говсанском луке.

**Ключевые слова:** синтез, рентгеноструктурный анализ, комплексообразование, константа диссоциации, константа стабильности, железо (III), диантипирилметан, диантипирилфенилметан, диантипирил-o-оксифенилметан.