

# Iron Containing Supplement Biofer Investigated by X-ray Fluorescence, Energy Dispersive X-Ray Spectroscopy and Mössbauer Spectroscopy

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Iron, an important microelement essential to the functioning of the body, plays a special role in the process of respiration. There are more and more new products available on the pharmaceutical market. Some of them were the subject of previous studies, especially by using the Mössbauer spectroscopy. One of the latest is Biofer. For the present tests three tablets from three different production series of Biofer were used for examination of elemental composition using the X-ray fluorescence and energy dispersive X-ray spectroscopy methods. Information about oxidation of iron contained in the tablets was obtained with by means of the Mössbauer spectroscopy.

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## 1. Introduction

Iron is a diet component essential for proper functioning of the human body. This is a component of hemoglobin contained in erythrocytes and it is active in transportation of oxygen to all tissues. Iron absorption is impaired due to either incorrect diet or diseases resulting in deficit of this element in the body [1–4]. In this case, supplementation becomes necessary. On the medical market dietary supplements containing iron can be obtained. One of these is Biofer, manufactured by Phorbio, which seems to have an interesting composition for people with iron deficiency. The active substances are the powdered hemoglobin and ferrous fumarate,  $C_4H_2FeO_4$ . In this paper three tablets of Biofer drug from three different production series were examined in order to determine if different series of the drug have a similar composition of the elements and quantity of iron.

## 2. Experiment

For the present study three series of Biofer numbered 735121 (designated as B1), 736022 (B2) and 736203 (B3) are selected. From each box of tablets, one was randomly selected. The pharmaceutical composition consisted mainly of: powdered hemoglobin, ferrous fumarate, silicon dioxide, magnesium salts of fatty acids, iron oxide and titanium dioxide (last two as colorants).

For transmission Mössbauer spectroscopy (TMS) [5–8] whole tablets, as prepared by the manufacturer, were

used which gives an effective thickness of about  $6 \text{ mg Fe/cm}^2$ . Measurements were made at room temperature in the transmission geometry with the source  $Co-57$  (50 mCi activity) in the rhodium matrix, moving at a constant acceleration. For detection of radiation the argon filled proportional counter was applied with a beryllium window of 0.3 mm thickness. The spectrum was registered with the MOSIEK-3E spectrometer in the  $2 \times 512$  channels mode with about  $10^6$  pulses per channel [5–7]. Parameters of hyperfine spectra were determined with the Recoil [5–8] in reference to  $\alpha$ -Fe.

Elemental composition of the pharmaceuticals was measured with the two X-ray based methods: X-ray fluorescence (XRF) and energy dispersive X-ray spectroscopy (EDX) [9, 10]. XRF was conducted using the Amptek experimenter kit equipped with a silicon-diode detector (the resolution of 190 eV for Fe  $K_\alpha$  line). EDX was performed in a vacuum ( $10^{-5}$  hPa) using the ISIS Link 300 analyzer from Oxford Instruments with a silicon-lithium detector (the resolution of 133 eV for the  $K_\alpha$  line of Mn). Due to the low energy of electrons (20 keV) and their small penetration depth, the tablet coatings were removed.

## 3. Results and discussion

The tested Biofer pharmaceutical composition contains two active substances supplying an organic iron in the form of hemoglobin powder (350 mg, the mass of iron is 1.2 mg) and in the form of an inorganic ferrous fumarate (25 mg, the mass of iron is 8.2 mg). The combination of these two forms of iron, according to the producer, improves absorption of iron by the body. The masses of tested tablets are respectively  $m_{B1} = 502.6 \text{ mg}$ ,  $m_{B2} = 514.4 \text{ mg}$  and  $m_{B3} = 508.2 \text{ mg}$ .

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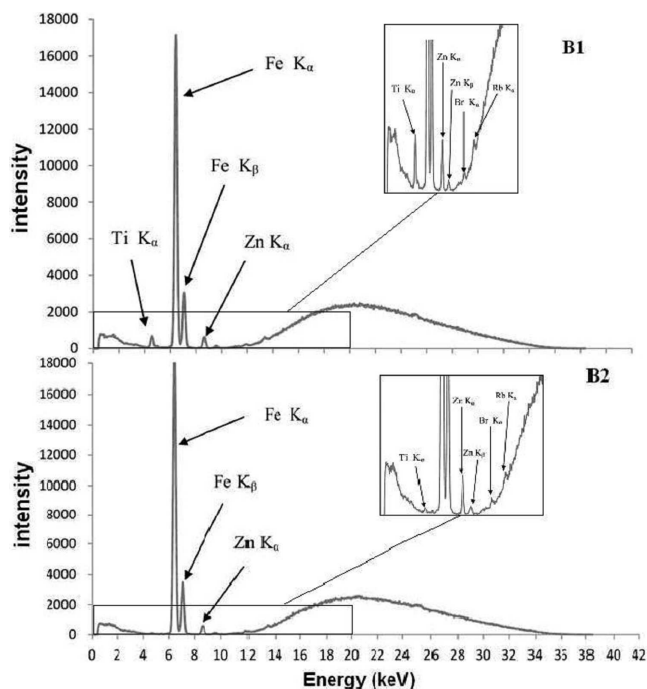


Fig. 1. XRF spectra of Biofer (samples B1 and B2).

An example of the XRF measurements, performed in air and in backscattering geometry, is presented in Fig. 1. Due to high absorption of X-rays with the energy less than 3 keV in air, the characteristic radiation originating from light elements was not observed. Optimal parameters of X-ray tube (40 kV and 15  $\mu$ A) ensures

TABLE I

Relative percentage of elements in Biofer pills B1, B2, B3 obtained from XRF spectra.

Element	B1	B2	B3
Ti	2.75 $\pm$ 0.14	0.03 $\pm$ 0.02	3.91 $\pm$ 0.17
Fe	91.83 $\pm$ 0.67	92.21 $\pm$ 0.63	88.44 $\pm$ 0.63
Zn	5.10 $\pm$ 0.18	7.28 $\pm$ 0.26	7.30 $\pm$ 0.25
Br	0.31 $\pm$ 0.03	0.48 $\pm$ 0.04	0.36 $\pm$ 0.03

TABLE II

Relative percentage of elements in Biofer B1, B2, B3 obtained from EDX measurements.

Element	B1	B2	B3
C	56.52	57.44	57.15
O	38.92	37.55	38.05
Na	0.34	0.58	0.35
Si	0.69	0.64	0.64
P	0.28	0.26	0.29
S	0.36	0.37	0.36
Cl	0.58	0.6	0.59
K	0.88	1.02	1.01
Fe	1.44	1.53	1.58

minimum background count is in the energy range from 4 to 10 keV. In this range spectrometer is very sensitive. In the recorded spectra the characteristic radiation peaks come from Ti, Fe, Zn, Br. The relative percentages were determined with the XRS-FP package and presented in Table I. The trace amount of Rb was also observed in each sample. In sample B2 only a trace amount of Ti was recorded, compared to about 3% in B1 and about 4% in B2 samples.

EDX measurements were taken in the vacuum  $10^{-5}$  hPa, so the low-energy characteristic radiation coming from light elements could be registered. Due to the low energy of electrons (20 keV) and related small thickness of penetration for testing EDX, the pills coating was removed. Therefore Ti (component coating) does not occur in the spectra. The program used for analysis automatically detects elements and their contents in the sample. The results of measurements are shown in Table II. In each of the pills, the same elements with a similar percentage were detected.

TABLE III

Mössbauer parameters of iron compounds in Biofer pills B1, B2, B3.

Compound	IS	QS	Iron state	Area [%]
	[mm/s]			
B1				
ferrous fumarate	1.21	2.24	Fe 2+	87.3 $\pm$ 1.7
Fe 3+	0.35	0.92	Fe 3+	9.1 $\pm$ 1.3
Oxyhemoglobin	0.16	2.08	Fe 2+	3.5 $\pm$ 1.2
B2				
ferrous fumarate	1.21	2.23	Fe 2+	89.6 $\pm$ 1.3
Fe 3+	0.41	0.82	Fe 3+	5.8 $\pm$ 1.0
Oxyhemoglobin	0.17	1.94	Fe 2+	4.6 $\pm$ 1.0
B3				
ferrous fumarate	1.21	2.24	Fe 2+	89.1 $\pm$ 1.9
Fe 3+	0.34	0.88	Fe 3+	5.7 $\pm$ 1.4
Oxyhemoglobin	0.21	1.97	Fe 2+	5.1 $\pm$ 1.5

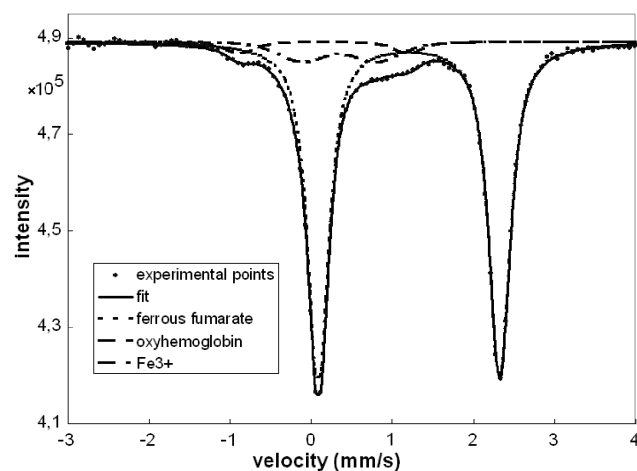


Fig. 2. Mössbauer spectra of Biofer (B1 sample).

The Mössbauer spectroscopy measurements were taken using the MOSIEK-3E [4–8] analyzer. As an example, the resulting spectrum for B1 sample is shown in Fig. 2. The spectra reveal three doublets. The most intensive one comes from ferrous fumarate and two smaller ones from oxyhemoglobin and  $\text{Fe}^{3+}$  iron compound, which is probably pollution of ferrous fumarate [2]. All TMS parameters of iron compounds are presented in Table III. As a result of the measurements obtained by MS in the tested tablets, the hemoglobin was found only in the oxygenated state.

#### 4. Conclusions

The measurements confirmed the presence of iron in the forms declared by the manufacturer, hemoglobin iron fumarate. The iron content in each sample is at a similar level. The absence of titanium in the sample B2 shows that the manufacturer does not follow the production standards. In all samples there were detected a few percents of the  $\text{Fe}^{3+}$  content, which probably was the pollution of iron fumarate.

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