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IDENTIFICATION OF WHITE JEWELRY ALLOY BASED ON SILVER AND PLATINUM FOR TESTING PURPOSES

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The procedure for the identification of white jewelry alloys based on precious metals, in particular, silver, platinum and platinum group metals with different contents of alloying components, by testing on an assay stone and by the method of X-ray fluorescence analysis has been considered. The methodology for assessing the compliance of silver and platinum fineness in white jewelry alloys of different component composition with the requirements of regulatory documents and the procedure for their identification has been improved.

It has been established that the silver fineness in precious alloys of the AgCu, AgZnCu system, determined using the potassium dichromate reagent on the test stone, depends on the manifestation of the contrast of the qualitative reaction from the standard sample (assay needle).

It has been proven that for testing silver alloys containing palladium, the "Acid reagent for gold 750" is effective, which works to determine the qualitative and approximate content of silver in alloys.

It was determined that the reagent "Ferrous-cyanide potassium" is very sensitive to changes in the alloy composition of silver alloys and makes it possible to establish the silver content with an accuracy of 5 %. The presence in silver alloys of such impurities as zinc, cadmium, nickel, gold, palladium and others increases the error in determining the fineness of silver and forms a different color and shade.

It has been proven that testing of silver alloys on an assay stone with silver nitrate is effective only for the CpM system. The presence of zinc in 925 sterling silver alloys visually increases the color intensity of the sediment, which indicates a higher overestimated fineness.

It has been found that the identification of the content of precious alloys based on platinum for the presence of ligature components is carried out with a potassium iodide reagent at t=120 °C by the color and shade of the sediment.

The procedure for using potassium iodide during testing of precious platinum-based alloys has been optimized

Keywords: assay control, precious metals based on silver and platinum, assay stone

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1. Introduction

Precious white metals, in particular silver, platinum, palladium, rhodium, have long been successfully used in the manufacture of exclusive jewelry, household items for table setting, clocks, ritual, church and cult things, coins, etc. In addition, from platinum and platinum metals groups produce technical products, antineoplastic agents, dentures and the like.

The main advantages of jewelry platinum in comparison with gold are its rarity, noble white color and luster, high resistance to chemicals and air. Due to the high strength and toughness of the metal, the products do not change their properties during operation and practically do not wear out. In terms of resistance to tarnishing in air, jewelry palladium is almost as good as platinum and has a number of additional advantages: more intense white color, better pressure-processed, cheaper. Palladium jewelry alloys are the most technologically advanced with silver and nickel. Given the low strength, low resistance to chemicals and external influences, a significant part of silver jewelry is made with rhodium plating.

The demand for white jewelry has been quite stable over the past twenty years, and the market value of precious metals is systemically increasing almost every year. This predetermines the creation of substitutes-imitations of white jewelry alloys based on base metals, the identification and diagnosis of which is a complex and urgent problem for industry, trade, pawnshops, banks, assay control, and the like.

There is a wide range of chemical elements that are introduced into the composition of modern white jewelry alloys based on gold, silver, platinum and platinum group metals (PGM) to improve their technological properties. Such components also significantly affect the change in the qualitative reaction of the assay reagent and lead to significant errors in determining the fineness on the assay stones. The method of assaying on assay stones is the cheapest, most basic and widely used in assay control. However, the imperfection of the procedure for identifying white jewelry alloys, due to the constant change in their composition and the absence of assay standard needles on them, makes it impossible to correctly determine the fineness of the precious metal and affix the state hallmark.

Therefore, in solving these problems, an important role is played by the improvement of the procedure, as well as the correct choice of means and methods of identification. This will improve the test methodology for assay stones of white jewelry alloys for the needs of assay control.

2. Literature review and problem statement

The general properties of gold, the principles of alloying with Ag, Cu, Ni, Pl, Zn and heat treatment are described in [1]. The change in the technological properties of silver, depending on the characteristics of alloying and use, is described in [2]. The authors of [2] proved that the strength of silver alloys increases due to alloying, as well as heat treatment. In [1, 2], the issues of the scope of application of alloys based on gold and silver are declared, but attention is not paid to the important issue of determining the component composition and identification methods. In [3], the aim of the study is to analyze the effect of portable X-ray tubes of a complex screen of an XRF apparatus with Ag- and Rh-anodes. The paper confirms the efficiency of using Rh-anodes at the level of Ag-anodes, however, the sensitivity of determining the component composition of gold alloys of exactly white color is not indicated.

The first fundamental studies of platinum group alloys and their intermetallic compounds are described in [4]. The values of the ground state of metals and their alloys are estimated, the electronic band structures and the density of states are calculated, and the thermodynamic characteristics are analyzed. Methods for diagnostics of the component composition of platinum-based alloys were ignored by the authors.

The peculiarities of the formation of nanoparticles of the Au-Pt alloy during the decomposition of double complex salts are described in [5]. The samples were examined by double analysis of the results obtained by the methods of XRF, electron microscopy and UV-visible spectroscopy. However, the authors in their study limited themselves to the determination of the average crystal size, the study of the reliability of the white Ag-Pd-Au alloy after chlorination and sulfiding. The efficiency of using a nanometer Au film deposited on the surface of a wire of an Ag-Pd-Au alloy has been proven; however, it has not been described to identify similar alloys.

The paper [6] describes the chemical composition, optical characteristics, gives comparisons of the color of alloys based on Ag, Au, Ni, Cu. However, the results of studies are presented, in which only microscopic and sensory (visual) methods of describing the investigated alloys are used.

The fineness determination of jewelry alloys of gold by XRF is described in [7]. The work is based on a comparison of the composition of gold alloys doped with Ag, Cu, Zn, Ni, In, obtained by non-destructive XRF and destructive cupellation. However, the aim of this work is not to develop a method for identifying white alloys of unknown composition.

The authors of [8] developed a method for determining the amount of clad gold on a brass base. It is indicated that the presented method does not require dissolution of samples and provides for the use of Cu as the internal standard of the alloy. So, outside the field of vision of the authors remained questions of research of alloys of white gold.

The results of studies of antique items made of gold alloys by XRF and two-dimensional microanalysis are presented in [9]. In [10], the transmission of X-rays and γ-rays was also investigated in order to determine alloys of gold and gilding. The work describes several methods that make it possible to distinguish between items made of gold alloys and gilded items. The methods proposed by the authors provide for the use of XRF with various instruments, data analysis and the number of measurements to identify precisely gilded objects. Samples of white alloys were not investigated.

The improvement of the XRF method as an independent study is described in [11]. The aim of the study was to determine the effect of chemical composition on the intensity of X-ray radiation for gold alloys. However, the work is limited to standard alloys of known origin.

Recent studies increasingly point to the need to improve the methods for identifying alloys through the prism of various component compositions of the alloys under study.

So, in [12] neutron activation analysis (NAA) and XRF were compared for non-destructive analysis of antique coins from Ag-Cu alloys. The simultaneous determination of Ag and Au nanoparticles in water samples by total reflection X-ray fluorescence spectrometry is presented in [13]. The effect of the atomic-chemical environment in the quantitative analysis of gold by the XRF method was studied in [14]. The study of white gold alloys on assay stones was carried out in [15]. The influence of analysis methods on the accuracy of determining the gold fineness of precious alloys of various component composition is shown in [16]. However, the group of authors did not aim to compare the effect of chemical reagents on white alloys of silver and platinum.

All this allows to assert that it is expedient to conduct a study dedicated to the improvement of the method for identifying alloys based on silver and platinum by the touchstone method using sediment color standards.

The use of colorimetric methods for the analysis of jewelry alloys will make it possible to more accurately determine the content of a fineness of precious metals, taking into account new jewelry alloys of unknown component composition under assay control conditions.

3. The aim and objectives of research

The aim of research is to identify white jewelry precious metal based on silver and platinum of various component composition and to improve, on its basis, the standard method for establishing the fineness and type of alloy. This will allow to create standards for the color of the sediment by the interaction of a specific chemical composition of the alloy and a chemical reagent. Color standards will be used as a means of identifying white jewelry alloys of various component compositions. They will provide an accurate determination of a precious metal fineness without additional expensive X-ray fluorescence analysis.

To achieve this aim, the following objectives were solved:

– to determine the influence of the alloy composition of jewelry alloys based on silver and platinum on the accuracy of the fineness determination by the XRF method and the test method on the assay stone;

– to improve the procedure and methodology for identifying white jewelry alloys based on silver with various component compositions;

– to improve the procedure and methodology for identifying white jewelry alloys based on platinum with various component compositions.

4. Materials and methods of research

4. 1.Investigated materials and equipment used in the experiment

The objects of research were jewelry made of precious alloys of white color, received for branding by state enterprises of assay control of Ukraine, in particular, on the basis of silver, platinum, including rhodium-plated.

More than 1000 samples of jewelry based on silver, 500 samples based on platinum and platinum group metals of different component composition were studied by XRF. Some of them are shown in Fig. 1.

a *b* *c*

d *e*

Fig. 1. Some objects of research of items made of silver and platinum: *a* — earrings made of 925 sterling silver; *b* — a ring with stones made of 925 sterling silver; *c* — earrings made of 925 sterling silver; *d* — rhodium-plated silver bracelet; *e* — ring made of platinum 900

For preliminary control and correctness of determination of chemical elements in alloys, an ElvaX spectrometer (Ukraine) was used, which makes it possible to detect elements in the range from sodium (atomic number 11) to uranium (atomic number 92). All investigated products were investigated by the XRF method. The qualitative composition of the jewelry alloy was determined by the spectrum, which is reflected on the ElvaX device. The X-ray energy spectrometer is designed for:

– registration of X-ray fluorescence spectra of the samples under study;

– determination of the energy position and intensity of X-ray fluorescence lines;

– identification of the elemental composition of the samples under study;

– determination of the concentrations and relative content of elements in the sample under study.

Features of the ElvaX X-ray fluorescence spectrometer:

– fully automated measurement process – from automatic optimization of unit parameters for a specific sample to quantitative multi-element analysis;

– automatic filter changer and helium purge for the analysis of light elements, starting with magnesium;

– non-standard precision quantitative analysis of alloys;

– built-in camera for precise positioning of analysis objects.

Elvatech MCA Software is used to control the operation of the spectrometer. The program offers two main methods for use: the method of fundamental parameters, which is used for the quantitative analysis of alloys; regression algorithm using graduated dependencies previously constructed from sets of standard samples.

For the study by the XRF method, 1000 samples of jewelry made of silver alloys were selected. Based on the results of XRF, nine samples of items of 5 brands were selected, different in component composition, declared as 925 and 875 fineness of silver; as well as assay needles of CpM composition (7) (Table 1).

Table 1

Samples of jewelry and assay needles based on silver taken for research

Indicator	Sample number (1÷6, 8, 9) and assay needle (7)								
	1	2	3	4	5	6	7	8	9
Declared fineness	925	925	925	925	925	875	600 800 875 925 1000	925	925

Fourteen samples were taken from 500 samples of jewelry based on platinum of various component composition and fineness processed by the XRF method for testing on assay stones (Table 2).

Table 2

Studied jewelry samples based on platinum of various brands and fineness

No.	Sample index	Quality indicator	
		Declared fineness	Brand
1	P1	900	PtIr 900-100
2	P 2	950	PtCu 950
3	P 3	950	PtIr 950-50
4	P 4	950	PtCo 950-3,0
5	P 5	900	PtCo 900-7,0
6	P 6	950	PtPd 950-50
7	P 7	950	PtPd 950-50
8	P 8	950	PtRh 950
9	P 9	950	PtCo 950-50
10	P 10	950	PtPd 950-50
11	P 11	950	PtPd 950-50
12	P 12	900	PtPdSi 900
13	P 13	900	PtRu 900
14	P 14	950	PtRt 900-100

The investigated 14 samples of jewelry of domestic and foreign production based on platinum are represented by 11 brands: PtIr 900-100; PtCu 950; PtIr 950-50; PtCo 950-3,0; PtCo 900-7,0; PtPd 950-50; PtRh 950; PtCo 950-50; PtPdSi 900; PtRu 900, PtRh 900-100.

4. 2. Method of identification of jewelry alloys based on silver and platinum

4. 2. 1. Methods for identifying white jewelry alloys by X-ray fluorescence analysis (XRF)

The essence of XRF is the decomposition of the studied X-ray spectrum into spectral models of pure elements. By interpreting the corresponding parameters of the models, the intensity of the spectral lines is determined, making it possible to calculate the concentrations of elements. It is a non-destructive method for determining the qualitative and quantitative content of chemical elements in products.

The intensity of the secondary characteristic radiation of the atoms of the elements excited by the primary radiation of the X-ray tube directly in the sample under study is measured. The structure of X-ray spectra is determined by the energy state of electrons in the atom, and therefore the spectrum of each element is individual, that is, it is characterized by a certain set of spectral lines. The dependence of the intensity of the characteristic spectrum line on the concentration allows to make a quantitative analysis, that is, to determine the percentage of this element in the sample. For platinum alloys, the measurement error is 0.3 %. The mass fraction of platinum in jewelry alloys based on platinum was determined by calculation by the difference of 100 % and the sum of the determined components and impurities.

The selection of products for incoming inspection by the XRF method was carried out according to the following scheme: from a batch consisting of 51 to 400 products, 1 product was selected, from 401 to 1000 – at least two.

If the products corresponded to the specified fineness, then a decision was made for further research of all products from the batch by the method of testing for assay stones using the necessary assay reagents. This method is simple, fast, effective and is widely used in the work of assaying state enterprises in Ukraine.

4. 2. 2. Methods for the identification of silver-based jewelry alloys on assay stones

The assay stone test method is based on the chemical reactions of reagents with a precious metal alloy applied to the assay stone. Testing of jewelry based on silver on assay stones was carried out with the use of assay needles, reagents and without them, comparing the color of the strips of the tested alloy and the needle. Various reagents were used to test silver-based alloys. Silver nitrate, potassium dichromate – for the qualitative determination of silver alloys of at least 500 fineness; “Acidic reagent for gold of 750 fineness”, “Potassium ferricyanide” – for the qualitative and “approximate” quantitative determination of silver.

Under the influence of these reagents, a weak grayish-white bloom is formed on silver: on silver of the lowest sample relative to the reagent, the intensity of the spot increases; there is no stain on silver with the highest sample relative to the reagent. The weak effect of the reagent was enhanced by adding drops of nitric acid.

Potassium dichromate (an aqueous solution of potassium dichromate with the addition of sulfuric acid), in comparison with silver nitrate, gives a less accurate result, but is used more often. The effect of the potassium dichromate was recorded in the range from “purity” to 600 fineness, and in combination with other reagents - below 500 fineness.

With an increase in the fineness of silver items under the influence of a potassium dichromate, silver reacts with an increase in the brightness of the spot. So, silver of 600 fineness leaves a dark brownish-red sediment; for 720-820 – brown shades disappear, orange color appears; 875 – shows a red color; above 900 – “blood” color; below 600 fineness – the reaction stops.

Potassium dichromate allows to determine silver alloys with an accuracy of 200 fineness. The reagent “Potassium ferricyanide” allows to determine the silver content in jewelry alloys with an accuracy of up to five fineness (more accurate diagnostic signs).

4. 2. 3. Method of identification of platinum-based jewelry alloys on assay stones

To determine the fineness of platinum alloys, the following reagents were used:

1) “Acidic for alloys of gold 958” (for alloys of platinum with copper, palladium, cobalt).

2) “Acidic for alloys of gold 958” with the addition of hydrochloric acid in a ratio of 1:2 (1:3) for testing alloys of platinum with copper, palladium, cobalt, rhodium, ruthenium, iridium.

3) potassium iodide (10 %) at room temperature.

4) potassium iodide (10 %) when heated (t=70–90 °C).

5) potassium iodide (10 %) when heated (t=120 °C).

The reaction of “acidic reagent for alloys of gold 958 when testing alloys of platinum with copper, palladium, cobalt is very slow.

In this case, the reagent interacts with the alloy with the formation of characteristic dark gray deposits. The stronger the effect of the reagent, the darker the sediment, the lower the alloy fineness. The rate of the reaction and the intensity of the formed sedimentation, in addition to the sensitivity of the stone, the temperature of the reaction and the duration of heating, depends on the fineness of the platinum alloy and its ligature composition. Strips were applied to the test stone by rubbing with products made of platinum alloys, and by a test needle of the corresponding alloy composition. The strips were wetted across with “acidic reagent for alloys of gold 958, after which the stone was placed on an asbestos pad and gently heated (to about 50–60 °C). With increasing temperature, the reagent began to act on the strips. Heating was stopped immediately as soon as the effect of the reagent on the alloy strips became noticeable (about 2–4 minutes). Then the stone was removed from the grating, the excess of the reagent was removed with filter paper, and the result of the action of the reagent on the strips of the platinum alloy was compared.

Tests of alloys of platinum with copper, palladium, cobalt were carried out in the same way as with the “Acidic reagent for alloys of gold 958”, while the reaction took place more quickly and the action of the reagent was more expressive. The reagent interacts with the alloy with the formation of characteristic dark gray deposits. The stronger the effect of the reagent, the darker the sediment, the lower the alloy sample.

Tests of alloys of platinum with rhodium, ruthenium, iridium were carried out in the same way as for alloys of platinum with copper, palladium, and cobalt. However, the strips were wetted across with “Acidic reagent for alloys of gold 958” with the addition of hydrochloric acid in a ratio of 1:2 or 1:3.

The determination of the ligature composition of platinum alloys was carried out using potassium iodide (10 %) in two ways. In the first, it was slowly heated to about a temperature of *t*=70–90 °C to prevent boiling of the reagent, in the second, it was strongly heated to about *t*=100–120 °C with the boiling of the reagent.

5. Results of the study of the influence of the ligature composition of white jewelry composition on the accuracy of fineness determination

5. 1. Results of identification of jewelry alloys based on silver and platinum by X-ray fluorescence analysis

For preliminary analysis of the composition of silver alloys, selection of assay needles and reagents, all products were tested for XRF. To control the qualitative and quantitative composition of the silver alloys selected for testing on assay stones, 8 samples of various grades (C1–C6, C8–C9) and the corresponding assay needles (C7) were taken. The results of the XRF study of samples of silver-based alloys are shown in Table 3 and Fig. 2, 3.

The results of XRF analysis of the chemical composition of silver-based jewelry indicate that the samples meet the requirements for the content of the main precious metal (silver) and alloying components, and provide proper consumer properties.

The results of the study of 14 samples of jewelry based on platinum by the XRF method are shown in Table 4 (1–6).

The spectrum of a jewelry alloy based on PtCo 950-50 (sample P9) is shown in Fig. 4.

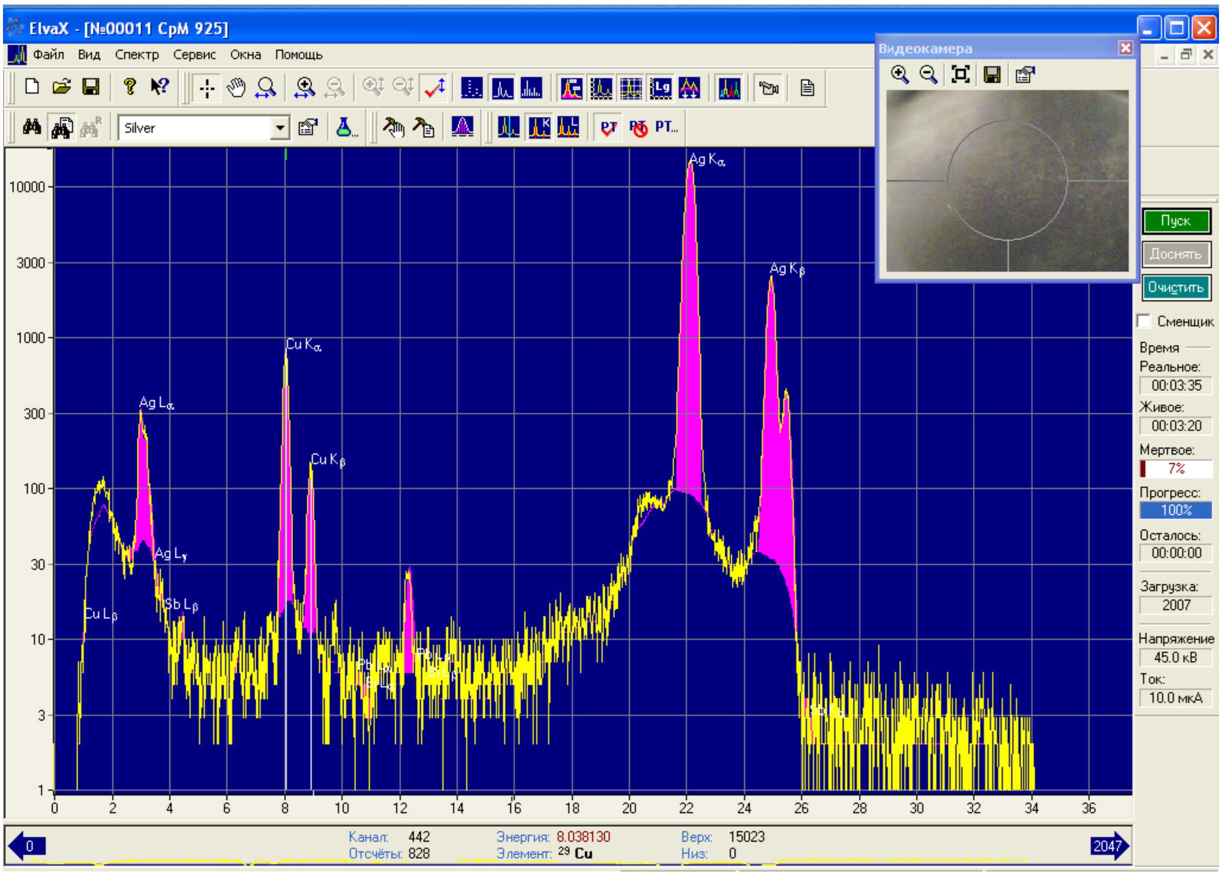
The results of the study of the content of jewelry alloys based on platinum by chemical composition indicate their compliance with the requirements. All platinum-based alloys are two-component and three-component, the main alloying components are rhodium, palladium, iridium, ruthenium, cobalt and copper. In an alloy based on platinum grade PtIr 900-100, iron impurities are present – 0.2 %.

According to the regulatory document, iron impurities should not exceed 0.04 %, this leads to a deterioration in the technological properties of the platinum alloy. The reason for the appearance of iron in the alloy may be a poorly cleaned surface of the product or contamination of the casting mold.

Table 3

Results of identification of silver-based jewelry alloys (XRF method)

Sample index	Results of the study of the chemical composition			Chemical composition according to ND	Fineness	Brand	Identification method
	atomic number	chemical element	content, %				
S1	29	Cu	7.138	7.5–7	925	AgCu 925	GOST 30649-99
	47	Ag	92.862	92.5–93			
S2	29	Cu	7.279	7.3–5.8	925	AgZnCu 925-0,6	TU U 27.4-00201514-010:2005
	30	Zn	0.204	0.2–1.2			
	47	Ag	92.518	92.5–93			
S3	29	Cu	7.281	7.5–7	925	AgCu 925	GOST 30649-99
	47	Ag	92.719	92.5–93			
S4	29	Cu	5.880	7.3–5.8	925	AgSiCu 925-0,2	TU U 27.4-00201514-010:2005
	14	Si	0.289	0.1–0.3			
	47	Ag	93.679	92.5–93			
S5	29	Cu	6.248	7.3–5.8	925	AgZnSiCu 925-1,5-0,12-0,1	TU U 27.4-00201514-010:2005
	30	Zn	0.460	0.2–1.2			
	14	Si	0.04	0.04–0.16			
	47	Ag	92.892	92.5–93			
S6	29	Cu	12.020	12.5–12	875	AgCu 875	GOST 30649-99
	47	Ag	87.980	87.5–88			
S7	Standard assay needles					AgCu 600 AgCu 800 AgCu 875 AgCu 925 AgCu 1000	–
S8	29	Cu	5.880	–	925	AgPdCu 925	–
	47	Ag	93.679				
	46	Pd	0.289				
S9	29	Cu	4.138	7.5–7	925	AgCu 925	GOST 30649-99
	47	Ag	95.862	92.5–93			



Silver

☐ Сменщик

Время

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Прогресс: 100%

Осталось: 00:00:00

Загрузка: 2007

Напряжение 45.0 кВ

Ток: 10.0 мкА

Канал: 442

Отсчёты: 828

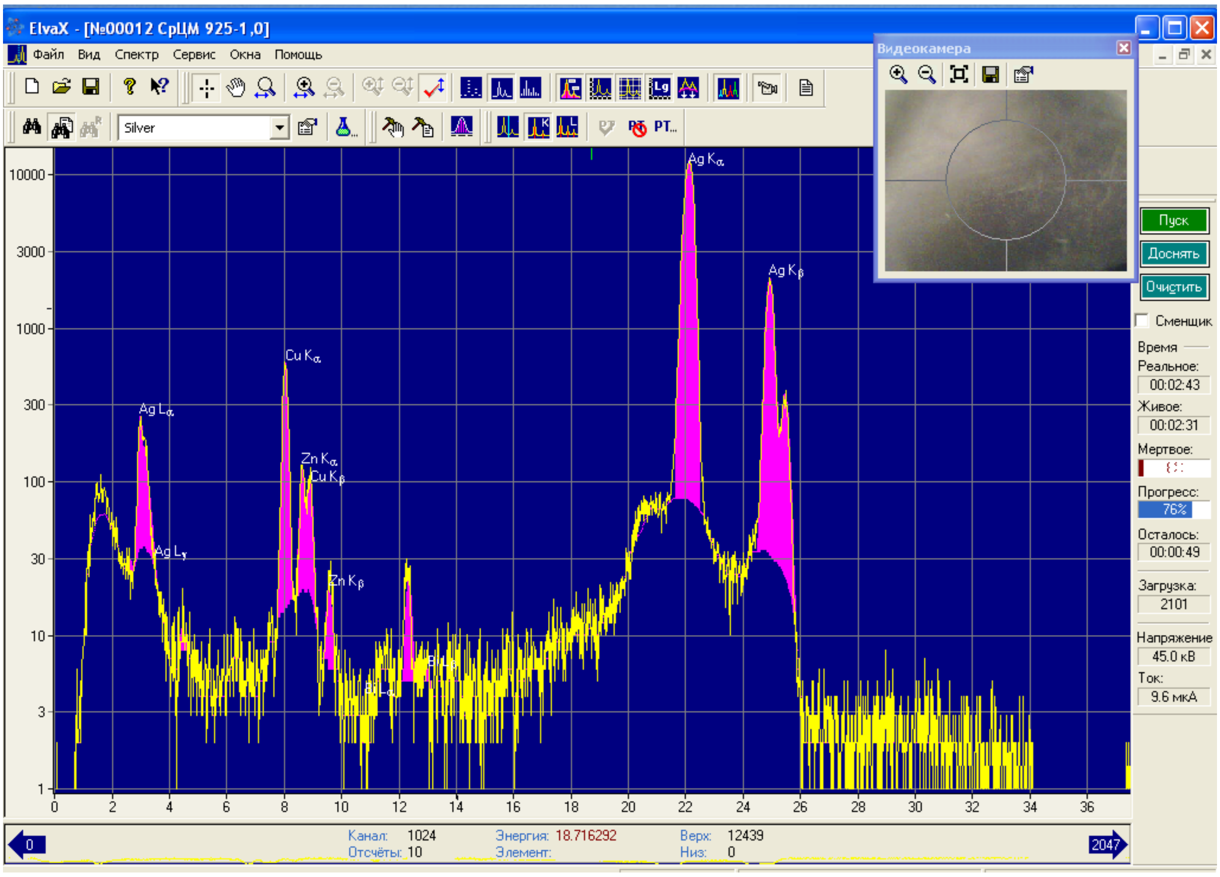
Энергия: 8.038130

Элемент: 29 Cu

Верх: 15023

Низ: 0

Fig. 2. Spectrum of jewelry alloy based on silver AgCu 925 (sample S1)



Silver

☐ Сменщик

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Прогресс: 76%

Осталось: 00:00:49

Загрузка: 2101

Напряжение 45.0 кВ

Ток: 9.6 мкА

Канал: 1024

Отсчёты: 10

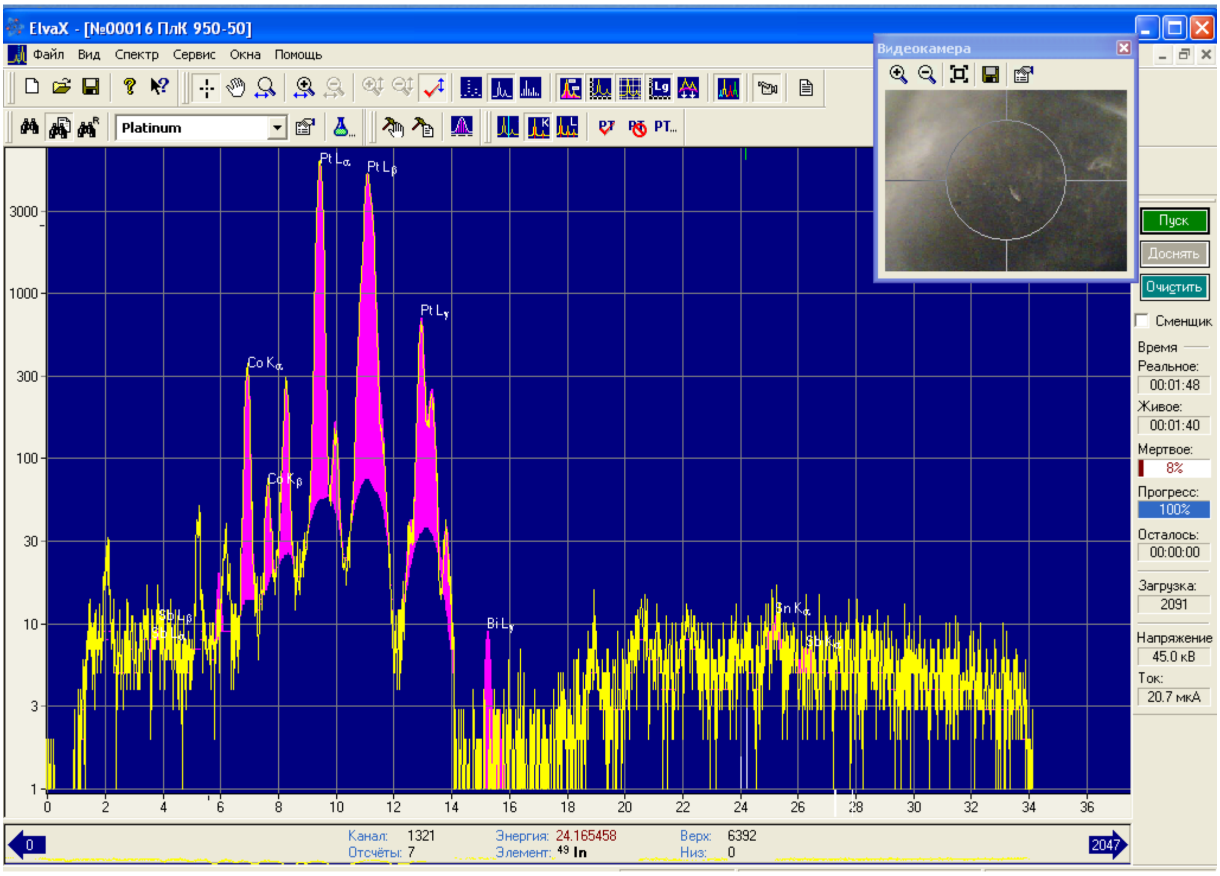
Энергия: 18.716292

Элемент:

Верх: 12439

Низ: 0

Fig. 3. Spectrum of jewelry alloy based on silver AgZnCu 925-0.6 (sample S2)



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Загрузка: 2091
Напряжение: 45.0 кВ
Ток: 20.7 мкА

Канал: 1321
Отсчёты: 7
Энергия: 24.165458
Элемент: 49 In
Верх: 6392
Низ: 0

Fig. 4. Spectrum of a jewelry alloy based on platinum grade PtCo 950-50 (sample 9)

Table 4

Results of identification of jewelry samples based on platinum by XRF and assay stone methods

Sample number	Results of the study of the chemical composition			Fineness	Brand
	atomic number	chemical element	content, %		
1	2	3	4	5	6
P 1	26	Fe	0.2	900	PtIr 900-100
	77	Ir	8.2		
	78	Pt	91.7		
	82	Pb	0.01		
P 2	29	Cu	4.92	950	PtCu 950
	78	Pt	95.10		
P 3	77	Ir	4.9	950	PtIr 950-50
	78	Pt	95.2		
P 4	27	Co	3.6	950	PtCo 950-3,0
	78	Pt	95.2		
P 5	27	Co	7.4	900	PtCo 900-7,0
	78	Pt	91.5		
P 6	26	Fe	0.04	950	PtPd 950-50
	46	Pd	4.44		
	78	Pt	95.44		
P 7	26	Fe	0.04	950	PtPd 950-50
	46	Pd	4.74		
	78	Pt	95.15		
P 8	45	Rh	4.1	950	PtRh 950
	78	Pt	95.8		
P 9	27	Co	4.80	950	PtCo 950-50
	78	Pt	95.20		

Continuation of Table 1

1	2	3	4	5	6
P 10	29	Cu	2.32	950	PtPd 950-50
	46	Pd	2.64		
	78	Pt	95.14		
P 11	29	Cu	2.0	950	PtPd 950-50
	46	Pd	2.68		
	78	Pt	95.32		
P 12	14	Si	3.92	900	PtPdSi 900
	46	Pd	5.04		
	48	Pt	91.01		
P 13	44	Ru	3.92	900	PtRu 900
	46	Pd	4.94		
	48	Pt	91.01		
P 14	45	Rh	7.20	900	PtRh 900-100
	78	Pt	92.8		

5. 2. Results of identification of silver jewelry alloys on assay stones

Testing of assay needles made on the basis of silver alloys of various fineness of the CpM brand (samples S7) (Table 3) on assay stones with a standard reagent – potassium dichromate – produces a red color of the sediment of varying intensity. The technique is described in clause 4. 2. 2. Studies have established that the saturation of the red color of the sediment with increasing fineness becomes lighter – from dark red to red (Fig. 5).

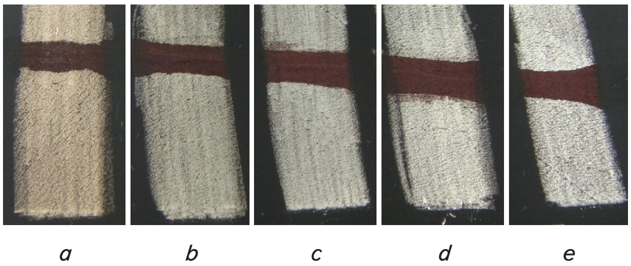

Fig. 5. Results of testing assay needles based on silver alloys of various fineness (samples S7) of potassium dichromate: *a* – silver alloy of 600 fineness; *b* – silver alloy of 800 fineness; *c* – silver alloy of 875 fineness; *d* – silver alloy of 925 fineness; *e* – silver alloy of 1000 fineness

The results indicate that as the silver content of the alloy increases, the color of the sodium sediment becomes lighter. In addition, other alloying components do not significantly affect the color of the sediment. Tests have shown that the determination of a sample of standard silver alloys (AgCu 925) (Ukraine) on test stones can be carried out with test needles and without the use of reagents, comparing the rub color of the alloy under test and the color of the test needle.

Compliance with the declared sample and the results of XRF was checked on all eight samples of jewelry based on silver of various brands. Rubs were applied to the test stone with the samples, on both sides of the reference assay needle of 925 fineness (in the center), and rubbed over the rub with a chromium peak solution. The color of the sediment on rubbers from the products is lighter than from the sediment on the reference assay needle, and this indicates the compliance of the tested alloy with the declared fineness – 925 and XRF results (Fig. 6).

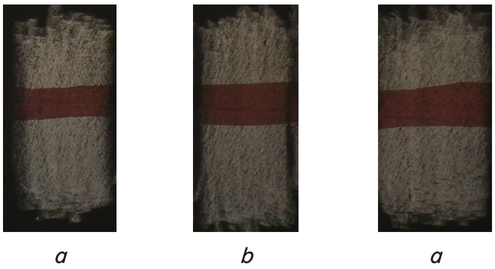

Fig. 6. Typical dark red sediment of 925 sterling silver alloy (as an example, sample S1) under the action of a potassium dichromate: *a* – a line of sample S1; *b* – a line from a AgCu 925 assay needle; *c* – line of sample S1

Alloy of silver brand AgCu 925 with rhodium plating, like S9 is tested (bracelet, Italy). Upon examination, a digital fingerprint was found on one link of the bracelet – 925. There is no state assay control in Italy, so the manufacturer applies the fineness. To determine the silver alloy, a reagent based on potassium dichromate (chromic peak) was used. The reagent was applied to the surface of the product; no reaction occurred. This indicates the presence of a coating on the surface. To determine the depth of the coating and establish a sample of the alloy, the product is scraped with a file and a chrome peak is applied to a small cleaned surface. The appearance of a spot with a bright red color indicates a high-grade silver content in the alloy, which corresponds to 925 fineness.

So, if during the action on the silver alloy with chromium peak a bright red sediment forms on the surface, then there is no coating. If there is a coating, including rhodium, the reagent does not react with it. This may also indicate that non-precious metals or low-grade silver (below 500 samples) have been used. When testing coated silver items, the test should be carried out on the basis of the results of chemical analysis on test stones, since XRF gives distorted data, and showed the results of the study.

During testing of silver alloys containing palladium (example S8) jewelry workshop “X” (Ukraine), declared 925 fineness, used “Acid reagent for gold 750”. This reagent works to determine the qualitative and “rough” content of silver in alloys. A drop of this reagent formed a white syrup-like sediment with a blue tint on the strip of silver alloy, which indicates a high purity of the silver alloy and the content of palladium in it (Fig. 7).

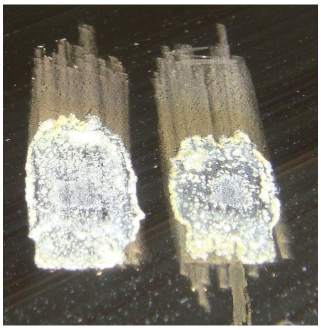

Fig. 7. The result of the action of "Acidic reagent on 750 gold" on an alloy of silver with a palladium content, 925 fineness (sample S8) with the formation of a white curd with a blue shade of sediment

The study of non-standard alloys based on silver with various components showed that when exposed to "acidic reagent on gold 750", sediments of different shades are formed, in particular with greenish or bluish-blue tints. The thicker the sediment, the higher the silver sample in the alloy. When testing a silver alloy with this reagent, the sediment did not get wet, it dried out slowly.

The study with the "Potassium ferricyanide" solution on silver assay standard needles (samples S7) was carried out to establish the reference color. On the preliminarily prepared test stone, uniform dense strips of soda were applied with silver needles of various samples (ranging from 600 to 1000), moistened with the "Potassium ferricyanide" Potassium ferricyanide and kept for 10 s. Excess reagent was removed with filter paper. Then the same procedure was performed, keeping the reagent on the strips by rubbing for 20 s. (Fig. 8).

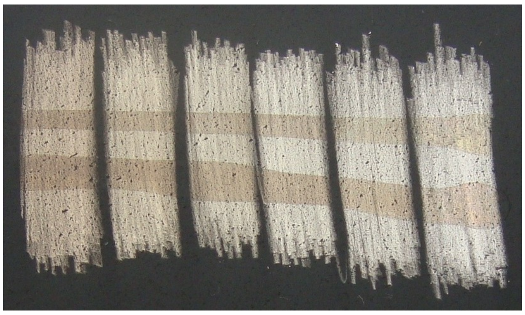

Fig. 8. The result of the action of the "Potassium ferricyanide" solution on silver alloys from 600 to 1000 fineness: upper strip — duration 10 s; lower — 20 s (samples S7)

Sedimentation coloration results indicate a visual difference between them. It was revealed that the higher the alloy sample, the lighter the sediment color.

The "Potassium ferricyanide" solution is very sensitive to changes in the alloy composition of silver alloys and allows the determination of the silver content with an accuracy of up to 5 fineness. However, the presence of impurities in silver alloys (zinc, cadmium, nickel, gold, palladium, etc.) increases the error in determining the sample of silver and forms a different color and shade. In such cases, tests were additionally carried out with "Acid reagent for 750 gold".

Under the action of the silver nitrate reagent, a weak grayish-white bloom is formed on a silver alloy containing copper (AgCu 925, sample S3): on silver of the lowest sample relative to the reagent, the intensity of the spot increases; there is no stain on silver with the highest sample relative to the reagent. The weak effect of the reagent was enhanced by adding a drop of nitric acid.

When testing silver alloys with zinc content (AgZnCu 925-0,6, sample S2) with this reagent, the color intensity of the sediment visually increases, indicating a higher sample than the XRF results (sample S2).

Consequently, the presence of zinc in silver alloys 925 visually increases the intensity of the color of the sediment, indicating a higher fineness than according to the XRF results (sample S2). But, like S3, it turned out to be more stable under the influence of assay silver nitrate (Fig. 9).

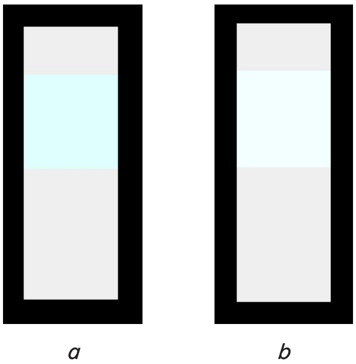

Fig. 9. Scheme of testing alloys based on silver 925 under the action of the "Silver nitrate" reagent: *a* — AgZnCu 925-0,6 (sample S2); *b* — AgCu 925 (sample S3)

The test results indicate that effective reagents for determining the qualitative and quantitative composition of silver alloys are solutions "Potassium ferricyanide", "Potassium dichromate", "Silver nitrate".

Thus, effective reagents for determining the qualitative and quantitative composition of silver alloys are solutions "Potassium ferricyanide", "Potassium dichromate", "Silver nitrate".

In case of an indistinct difference between the samples, associated with the content of alloying additives, it is possible to additionally use the solution "Acid reagent for gold 750".

5. 3. Results of identification of platinum jewelry alloys on assay stones

Tests of precious alloys based on platinum containing various alloying components (Pd, Rh, Ir, Ru, Cu, Co) were carried out on assay stones of natural origin with a fine-grained structure. Since platinum-palladium alloy has the lowest chemical resistance between pure platinum and alloys with rhodium, ruthenium and iridium, the best methods have been chosen to determine each of them. Since platinum-palladium alloy has the lowest chemical resistance between pure platinum and alloys with rhodium, ruthenium and iridium, optimal methods have been chosen to determine each of them. "Acidic reagent for 958 gold" was adopted to determine the content of palladium; slow heating with potassium iodide (10 %) – palladium, ruthenium, iridium, copper, cobalt; strong heating with boiling of the reagent – rhodium or copper impurities.

Under the action of the first reagent on rubbing of samples (samples P 6, P 7, P 10, P 11) and an assay needle 900 for platinum, an identical dark gray sediment was formed on all strips. This is an identification mark for platinum-palladium-based alloys. Two samples have a sediment slightly darker than the assay needle, indicating an increased platinum content compared to the assay needle (but within the 900 fineness norm).

To determine the alloying components of the platinum-based alloy (palladium, rhodium, ruthenium, iridium, copper, cobalt), the assay stone was slowly heated to a temperature of 120 °C with potassium iodide (10 %) applied to the strips. When heated, the rules were followed: drops of the reagent should not combine with each other.

The excess of the reagent evaporated and deposits of various shades remained on the strips, which indicates the presence of platinum group metals (palladium, rhodium, iridium, ruthenium) in the platinum alloy (Fig. 10). The chemical composition of the objects of study is given in Table 5.

To control the copper content in alloys based on platinum (samples P 2, P 10, P 11), potassium iodide was heated to boiling at a temperature of 100–120 °C. As a result, a more saturated yellow color of the sediment was obtained on the rubbing of the samples compared to the assay needle.

It was found that the more copper in platinum-copper alloys, the darker the deposit on the strip and vice versa. So, according to the intensity of the color of the sediments, their shades, it was concluded that the sample and the presence of copper in a precious alloy based on platinum.

When testing precious alloys based on platinum with a cobalt content on assay stones, a salad color of the sediment was found under the action of potassium iodide (samples P 5, P 6). In addition, the sediment in alloys with a lower mass fraction of platinum (sample P 5, PtCo 900-7.0) under the influence of potassium iodide at room temperature is darker.

So, if the ligature component in platinum alloys is non-precious metals (in particular cobalt), then the sediment in alloys with a lower mass fraction of platinum becomes darker under the action of potassium iodide at room temperature.

When testing jewelry alloys based on platinum with different rhodium content under the action of potassium iodide, the following was found. The sediment of a darker green color was in sample P 14 with a high rhodium content (PtRh 900-100) as compared to sample P 8 (PtRh 950-50) with a slight heating.

Table 5

Results of the study of jewelry alloys based on platinum with the "Potassium iodide" reagent

Sample number	Results of the study of the chemical composition by XRF			The color of the sediment from the action of the «Potassium iodide» reagent
	atomic number	chemical element	content, %	
P 1	26	Fe	0.2	Light brown
	77	Ir	8.2	
	78	Pt	91.7	
	82	Pb	0.01	
P 2	29	Cu	4.92	Light yellow tint
	78	Pt	95.10	
P 3	77	Ir	4.9	Light brown
	78	Pt	95.2	
P 4	27	Co	3.6	Dark salad
	78	Pt	95.2	
P 5	27	Co	7.4	Salad
	78	Pt	91.5	
P 6	26	Fe	0.04	Yellow-lemon
	46	Pd	4.44	
	78	Pt	95.44	
P 7	26	Fe	0.04	Yellow-lemon
	46	Pd	4.74	
	78	Pt	95.15	
P 8	45	Rh	4.1	Greenish
	78	Pt	95.8	
P 9	27	Co	4.80	Salad
	78	Pt	95.20	
P 10	29	Cu	2.32	Light yellow tint
	46	Pd	2.64	
	78	Pt	95.14	
P 11	29	Cu	2.0	Light yellow tint
	46	Pd	2.68	
	78	Pt	95.32	
P 12	19	K	3.92	Orange tint
	46	Pd	5.04	
	48	Pt	91.01	
P 13	44	Ru	3.92	Dark red tint
	46	Pd	4.94	
	48	Pt	91.01	
P 14	45	Rh	7.20	Greenish
	78	Pt	92.8	

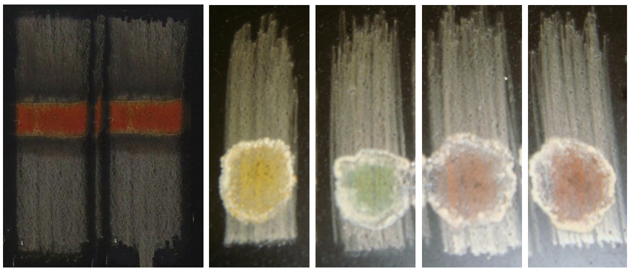

Fig. 10. The result of the action of potassium iodide on platinum alloys with various alloying components: *a* – with a palladium content (5 %) and potassium – a sediment of a hot yellow tint (sample P12); *b* – with a palladium content – a yellow-lemon shade (samples P 6, P 7, P 10, P 11); *c* – with rhodium content – greenish tint (sample P 14); *d* – with iridium content – brown tint (samples P 1, P 3); *e* – with ruthenium content – dark red tint (sample P 13)

Consequently, when testing precious platinum-based alloys on assay stones, a certain pattern was found. If the ligature component in platinum alloys is palladium, rhodium, iridium, ruthenium, then the sediment in alloys with a large mass fraction of platinum under the action of potassium iodide will be darker with slight heating.

In this case, platinum, and not the ligature component (palladium, rhodium, iridium, ruthenium), first of all enters into the reaction with potassium iodide when "heated", because they are higher in chemical resistance than platinum.

Determination of a sample of a platinum-based alloy is associated with a qualitative determination of the alloy composition, is achieved by the conditions of testing and correctly selected reagents.

The research results show that the most effective for determining the quantitative and qualitative composition of platinum alloys is the "Potassium iodide" reagent (10 %), the effect of which is manifested when heated from 70–80 °C to 100–120 °C.

Thus, the most effective reagent for determining the quantitative and qualitative composition of platinum alloys is the potassium iodide reagent (10 %), the effect of which is manifested when heated from 70–80 °C to 100–120 °C.

6. Discussion of the results of identification of white alloys based on silver and platinum

The accuracy of the results of establishing the fineness in white alloys based on silver and platinum can be explained by the different content of alloying components. And given the fact that different countries produce silver and platinum jewelry alloys with various alloying components, the result of fineness identification varies greatly and is different from the results described by the methodology in force in Ukraine.

The composition of alloys based on silver, providing it with a white color, includes: silver, copper, zinc, silicon, tin, indium, and sometimes palladium. These components can be expanded in different countries of the world. It can be considered the most convenient and cheapest method for the identification of standard silver alloys (not less than 500 fineness) by test for assay stones using the potassium dichromate reagent. The results of the study showed a bright visualization of the red sediment and a change in its saturation as a characteristic identification feature, evidenced by the qualitative and quantitative content of silver (fineness) (Fig. 6). Let's believe that the processes occurring and constitute the essence of research based on the chemical reactions of this reagent with an alloy on the test stones. And also on a visual comparison of the color intensity of the sediment formed on sodium chloride from the alloy under study with the color of the sediment on sodium hydroxide from the assay needle. Potassium dichromate reacts with the components of the silver alloy applied to the test stone, with the formation of red deposits of varying intensity from dark red to bright red, depending on the alloy fineness and the sensitivity of the test stone. The reaction proceeds according to the scheme:

$$2Ag+2H_2SO_4+K_2Cr_2O_7 \rightarrow Ag_2Cr_2O_7\downarrow+K_2SO_4+2H_2O+ +SO_2,$$

$$Cu+2H_2SO_4 \rightarrow CuSO_4+2H_2O+SO_2,$$

$$2(AgCu)+6H_2SO_{4+}K_2Cr_2O_7 \rightarrow Ag_2Cr_2O_7\downarrow+K_2SO_4+ +6H_2O+2CuSO_4+3SO_2.$$

It was found that the higher the silver fineness, the brighter the red sediment of silver dichromate ($Ag_2Cr_2O_7$) becomes, which, when sedimentd, is a convincing identification feature of silver-based alloys.

The study of silver alloys with various alloying components (Fig. 7) "Acidic reagent for gold 750" showed a more effective effect in determining their qualitative and quantitative composition. The study resulted in the formation of insoluble curdled white sediment of silver chloride. An essential requirement of the study is the application of the same density of rubbing of both the alloy and the assay needle on the assay stone. The sample determination error using the reagent did not exceed 10 fineness. Hydrochloric acid (HCl), which is part of the "Acidic reagent for gold 750", acts on silver to form an insoluble curdled white sediment of silver chloride (AgCl). It was found that the thicker the sediment, the higher the silver fineness. In addition, the sediment had a different shade of color if the composition included other alloying components, in particular zinc, cadmium, nickel, palladium and other metals. As an example, the given alloy of silver containing palladium has salad-colored sediment (Fig. 7), and the presence of impurities of zinc, cadmium and nickel provides sediments of a blue tint. For such alloys, this reagent does not give an error in establishing the sample, unlike other reagents for silver.

On rubbing of silver alloys below 500 samples, the syrup-like sediment of silver chloride (AgCl) acquires an intense blue tint, but not due to the presence of impurities in the alloys, but due to the formation of copper compounds.

When testing silver alloys with the "Potassium ferricyanide" reagent, clear identification signs were established in the form of colored sediments (Fig. 8), which are formed in the form of light orange silver ferrocyanide potassium ferricyanide, dirty green copper ferrocyanide $Cu[Fe(CN)_6]$ and other ferrocyanides. It was found that the lower the fineness of silver alloys, the darker the sediments, the higher the fineness of silver alloys, the lighter the sediments.

The use of the classical "Silver nitrate" reagent for the diagnosis of silver alloys showed an effective effect only for standard alloys with copper with the formation of a white sediment (above 875 fineness) according to the following scheme (Fig. 9):

$$(AgCu)+2AgNO_3 \rightarrow 3Ag\downarrow+Cu(NO_3)_2.$$

With a decrease in the fineness, the color of the sediment gradually changed to gray-ash. For silver alloys containing zinc, cadmium, nickel, palladium, the tests provided a systematic error in the fineness determination (Fig. 9).

When identifying platinum alloys on assay stones with the "Potassium iodide" reagent at t=120 °C, the presence of various alloy components of the alloy was established (Fig. 10). Thus, palladium with potassium settled in the form of an orange-colored sediment of potassium chloropaladate (Fig. 10, *a*), with palladium – a yellow-lemon shade (Fig. 10, *b*). Rhodium formed a greenish sediment by sedimentation of rhodium dioxide hydrate ($Rh(OH)_4$) (Fig. 10, *c*). Iridium formed a brown sediment (Fig. 10, *d*) and ruthenium, which appeared as dark red sediment (Fig. 10, *e*). Copper provided a yellow tint to the sediment.

It was found that the use of the "Potassium iodide" reagent solves three essential identification problems, not solved in the existing method. At a temperature of about 120 °C, the ligature component of a platinum-based jewelry alloy is determined by its color shade. At room temperature, the presence of platinum group metals in the alloy (except for palladium) is confirmed, which do not form a colored tint, unlike copper, cobalt or palladium. At a temperature of about 70 °C, the reagent makes it possible to establish alloy fineness.

"Aqua regia", which is a part of the reagent, dissolves platinum, forming platinum-hydrochloric acid (H_2PtCl_6), which with potassium ions forms yellow-colored potassium chloroplatinate (K_2PtCl_6), practically insoluble in high concentrations of potassium ions. Conclusions on the platinum sample are made based on the results of a comparative assessment of the shades of the sediment on the alloy strips, tested, and strips from the assay needle. Thus, the presence of palladium (Pd) in the platinum-based alloy provides an orange-colored sediment (Fig. 10, *a*), since potassium chloropaladate (K_2PdCl_6) formed in this process has a red color. The more palladium in the alloy, the more intense the orange color of the sediment. Visually, there is a significant difference in the color of the deposits of platinum alloys with 5 % and 20 % palladium, on the basis of which it is possible to judge the percentage of palladium in the alloy (Fig. 10, *b*). In the case of determination of the alloy of platinum with iridium (Ir), a colored sediment with a brown tint is formed (Fig. 10, *d*). In the presence of rhodium (Rh) in platinum alloy, dirty green sediments are formed on the strips of soda by the release of rhodium dioxide hydrate (Fig. 10, *c*).

The studies carried out to determine the fineness of white jewelry alloys based on silver and platinum with various reagents on assay stones showed their effectiveness in assay control, pawnshops, expert institutions, customs, and found a number of certain limitations. It was found that the fineness determination, the qualitative and quantitative composition, should be started with weak reagents. This is due to the fact that almost every batch arriving for assay control has different ligatures of jewelry alloy, for which assay needles are absent. Before testing an unknown alloy, it is necessary to carry out XRF, which increases the cost of the assay services. The process of testing various platinum alloys requires mandatory control of the temperature of the reagent, the presence of colored sediment standards (identifiers of color and color intensity).

Therefore, the creation of an identification base of sediment color standards for white jewelry alloys of different component composition is a very important step in improving the technique and developing its procedure.

And given the rapid development of jewelry and fashion for them, the content and color varieties of jewelry alloys are changing, white non-precious alloys are more often used. This indicates the relevance and prospects for further research of a wide range of platinum, silver and other white alloys to prevent their falsification and to clearly define the sample. However, this is due to the duration of collection of the studied samples and their high cost.

7. Conclusions

1. It has been established that accurate fineness determination, with the appearance of a characteristic colored sediment, depending on the composition of silver precious alloys on test stones, is effective through the use of a number of reagents. The reagents of silver precious alloys, on the basis of which identification color samples have been created, include: "Silver nitrate", "Potassium dichromate", "Acid reagent for gold 750", "Potassium ferricyanide". The "Silver nitrate" reagent was determined to be effective only for the standard composition of silver alloys with the formation of a white sediment of varying amounts and intensity. The zinc content of 925 silver alloys visually increases the color intensity and the amount of sediment, acquiring a blue tint. This indicates a higher sample compared to the color intensity of the sediment of the standard alloy and the results of XRF. The use of the reagent "Potassium dichromate" for a silver alloy forms a brighter and lighter red sediment in higher silver fineness. In addition, it has been found that all other alloying components do not change the color of the sediment, that is, they do not affect the accuracy of the sample determination. "Acidic reagent for gold 750" is the most effective for the determination of a fineness of non-standard alloys based on silver containing zinc, palladium, gold, cobalt, silicon, etc. The fineness determination of an alloy based on silver is aimed at assessing the shade and concentration of insoluble curdled sediment of silver chloride, which increases with increasing fineness. Jewelry alloy based on silver, which includes palladium, forms syrup-like sediment of a salad shade, the presence of impurities of zinc, gold, cadmium and nickel forms a sediment of a blue tint. The "Potassium ferricyanide" reagent provides clear identification features of silver-based alloys in the form of color shades of sediments, in particular, light orange silver ferrocyanide, dirty green copper ferrocyanide and other ferrocyanides. It has been found that the lower the fineness of silver alloys, the darker the sediments, the higher the fineness of silver alloys, the lighter the sediments. The results of the study have been established a clear correlation between the sample indices of precious alloys based on silver, determined by X-ray fluorescence analysis and testing on assay stones.

2. It has been established that the exact fineness determination, the qualitative and quantitative composition of the precious metal based on platinum on the assay stones is effective with the "Potassium iodide" reagent with the formation of sediments of varying intensity, color and shade, which serve as identifiers. It has been found that under the action of the reagent "Potassium iodide" reagent on the scratches of alloys based on platinum at a temperature of about 70 °C by the amount (intensity) of the sediment – the platinum fineness is determined. At a temperature of 120 °C, an orange color of the sediment forms on the test stone, in-

dicating the content of palladium. The greenish tint of the sediment color indicates the rhodium content. The brownish tint of the sediment is evidenced by the iridium content of the platinum alloy. The dark red color indicates that the platinum contains ruthenium, the yellow color indicates the copper content. Tests at room temperature allow to detect the platinum group metals (except palladium) in the alloy, which do not form a colored tint, unlike copper, cobalt or palladium. The sample values of precious platinum-based alloys were determined by X-ray fluorescence analysis and a full correlation was established by test on assay stones.

3. The procedure and method for identifying white jewelry alloys based on precious metals with different component compositions has been improved by creating color identifiers that can establish the fineness and composition of the jewelry alloy. When testing precious alloys based on platinum "potassium iodide", it is important to control the following indicators:

1) the rub width – min. 6 mm and the distance between them – from 5 mm (to prevent mixing of reagent drops when moving the test stone to the asbestos pad for heating);

2) the quality and nature of the origin of the assay stone, which must be natural and suitable in sensitivity for working with platinum when heated;

3) the composition of the investigated alloy – according to the content of the assay needle of the corresponding composition or by the XRF method.

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