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ANALYTICAL INVESTIGATION OF AN ENAMELED ROYAL TRAY

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Abstract

This research aims to investigate a precious colorful royal Ottoman silver tray from the Faculty of Applied Arts' Museum, Egypt. It incorporates two sophisticated methods of enamelling: champlevé and painture enhancing a tugra central disc. The chemical composition and deterioration aspects of the enamel and metal were identified using different examination and analytical techniques such as USB digital microscope, Portable X-ray fluorescence (pXRF), scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM−EDX), and Fourier transform infrared spectroscopy (FTIR).The tray showen different signs of deterioration, such as flaking, exfoliation, scratches, pits and colour alteration of the enamel while tarnishing covered the whole metal surface. Results proved that the tray is made of partially gilded silver-copper alloy. Gold residues were found on the frame and the handles. The enamel contains basically silica, potassium, sodium and lead. The analysis and examination results in addition to the tugra central disc enabled the accurate dating of the tray to the 19th century.

***Keywords**: Enamel; Metal; Examination; Analysis; USB digital microscope; pXRF; SEM-EDX; FTIR*

Introduction

The art of enamelling metals is defined as the process of applying small granules of enamel to metal and firing at high temperatures, for the purpose of decoration [1, 2]. It has been practiced from very early times; by the Egyptian, Phoenicians, Assyrians, Greeks, Romans and Etruscans [3-5]. By the third century B.C., the enamel process had spread to Europe and the process continued to move slowly to Asia Minor and the Middle East, India, China and lately to Japan [6].

Enamel is a vitreous glaze of finely ground glass which produced from a mixture of silica, alkali compounds, which lower the melting temperature, lead oxides, salts of soda, potassium, boric oxides and various metallic oxides. Enamel can be transparent, opaque or opalescent [7, 8]. Enamel can be melted directly onto the metal surface, but more often into a depression or a cell prepared to receive it [9]. The methods of enamel application are different; they are defined as cloisonné, champlevé, painted enamel, grisaille, basse-taille and plaque-a-jour etc., [3]. A combination of some of these techniques may appear on the one and the same artefact. The metal is usually gold, silver, copper, bronze or iron [10].

Since the time of the Ottoman Emperor Sultan Selim I (r. 1512–1520) Egypt became part of the Ottoman Monarchy [11, 12, and 13]. Ottoman style objects spread all over Egypt

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together with many other objects made in Europe and Istanbul according to Turkish taste. These items included small cups called zarfs, often in gold or silver, sherbet spoon holder, flywhisks, mirrors, trays, decorative clock, pocket watches and cutlery sets [14].

Ottoman trays have been designed on four forms, a form of heart, rectangle, and circle or elliptical and mostly fitted with handles on both sides to carry.

Sometimes the tray is a piece in a collection of pieces together they constitute ship sets which reflect craftsmen skills and artistic creations, where each piece is characterized as having a unified design and a unified decoration [15].

This research aims to investigate a precious colorful enameled royal Ottoman silver tray from Egypt. It incorporates two methods of enamelling: champlevé and painture, The former is one of the oldest enamelling techniques [16], in which the enamel is laid into a depression made in the metal [8] by stamping, piercing, or etching with acids [17]. As for the painted enamel" or wet enamel, it is a style in which a picture can be built up using successive firing [18], painted enamel derive their outlines from the colour scheme only [19] after the enamel colours are mixed with lavender oil [20].

Historical back ground of the royal tray

The tray holds tugra which dates back to the Ottoman Empire 1922 (AH 1299). The most outstanding symbol of the Ottoman sultan's authority was his imperial tuğra (cipher), which was affixed to all official documents, indicating fermans, vakfiyes and correspondence; it was also carved on his seals and stamped on coins minted during his reign. Each sultan chose his personal tuğra immediately after his accession and used the same format throughout his life. It should be pointed that tugramark of the Sultan which decorated the coat-of-arms in centre of the tray was used as a silver hallmark [21, 22].

Materials and Methods

Initial examination included visual inspection, measuring dimensions and photography.

The tray was examined using USB digital microscope (1.3 Mega Pixels, Manual Focus from 10 to 500mm, and 20 X to 500X), combined with a tiny digital camera (CMOS, CRE Company, China) and is connected to a computer. The images seen through the microscope's eyepiece can show the deterioration aspects on the metal and the enamel at different magnifications.

Portable X-ray fluorescence XRF (NITON/ XLt 8138, Thermo Scientific, 592GkV, USA, with niton software version 4.2E) was used to micro analyse the metal and the enamel and to identify the weight ratios of the elements (Wt. %).

Samples of enamel and soldering material of the tray were analyzed with SEM-EDX (Quanta 250 FEG attached to EDX Unit, FEI Company, Netherlands) with accelerating voltage 30V. The samples were examined without coating at low vacuum.

The central enamelled convex disc of the tray, decorated with tugra has been made separately then it was attached to the tray base. Visual inspection revealed that there are layers of a non-metallic material between the disc and the tray, this might be a mixture of an adhesive and a filling material used to assemble the tugra disc with the tray base. Samples of the non-metallic filling material were analysed using FTIR spectrometer (4600 Jasco, maximum resolution: 0.7cm^{-1} S/N ratio: 25,000:1, in the frequency range of 4000 - 400cm^{-1}). The samples were ground and mixed with KBr powder. Spectra were obtained in the transmission mode. This analysis was undertaken at the National Research Centre (NRC) in Cairo, Egypt.

Results and Discussion

Identification and documentation

Examination enabled the full description of the tray, which is given in table 1. Face and back appearances are photographed and shown in Figures 1 and 2.

Table 1. Description of case study

Items	Description
Object number	93/5, New Number: 183
Location	Faculty of Applied Arts' Museum, Cairo, Egypt
Dimenations	31 X 32.5 cm^2.
Date	The Ottoman Empire 1299-1922 [23].
Description	The tray consists of a double layer metal sheets; the back sheet is plane while the upper is champlevé enamelled. The two sheets are assembled together by an outlining metal frame fastened all around by pins. The tray back has only two legs remaining, while the other two legs are lost. Their positions are indicated by residues of soldering material. Tugra central disc is 13.2 cm diameter and decorated with Coat of arms of the Ottoman Empire using painted enamel with different colours (Fig. 3). Green, turquoise orange-red, white, yellow, pink and blue enamel colours are used with gold foil lining, some parts of tugra.

Fig. 1. Front of the tray

Fig. 2. Back of the tray.

Fig. 3. Ottoman tugra on the central disc of the tray.

Digital microscope examination

The results of microscopic examination shown many aspects of deterioration of both metal and enamel. Cracking running throughout the enamel layer and flaking of the enamel causing loss in some parts were observed. Exfoliation of enamel exposed the metal surface underneath to the direct contact with the atmosphere resulting in corrosion and accumulation of dust in the exposed areas. However, it was thought that the metal corrosion and formation of corrosion products would probably be the responsible for the occasional loss of the enamel from the metal surface [24]. Metal corrosion also occurs when metal comes into contact with glass, the latter often shows visible signs of glass corrosion: hazing, wet surfaces or efflorescence, cracking, roughening, pitting [25].

Microscopic examination also revealed scratches, pits and colour alteration (Fig. 4). Physical damage resulting from use, abrasion and improper handling may force the enamel to fall away [26], shatter easily if it is knocked or dropped. Loss of enamel could also allow moisture to enter causing corrosion of metal and forcing more enamel to fall off [27]. Turquoise and green Enamels were the most affected compared with other enamel colours. They exhibited colour change, damage in the form of husks and exfoliation. Painted enamel white back ground around the corners exhibited larger cracks.

Tarnish covered the whole metal surface of the tray, exhibiting scratched dull, darkened and blackish appearance (Fig. 5).

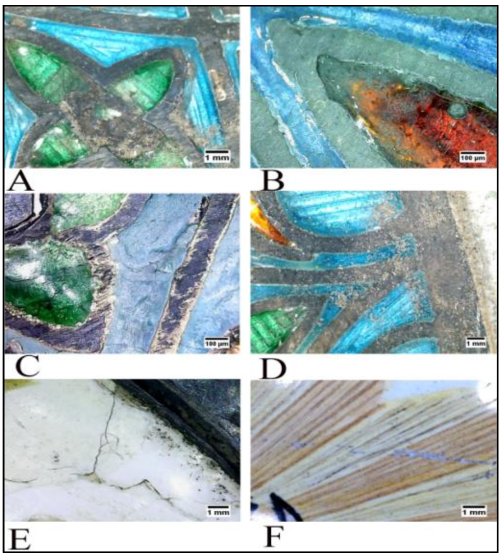


Fig. 4. Photomicrographs of the enamel:
A. loss of enamel, B. pits, C. husks, D. colour change, E. cracks, and F. scratches.

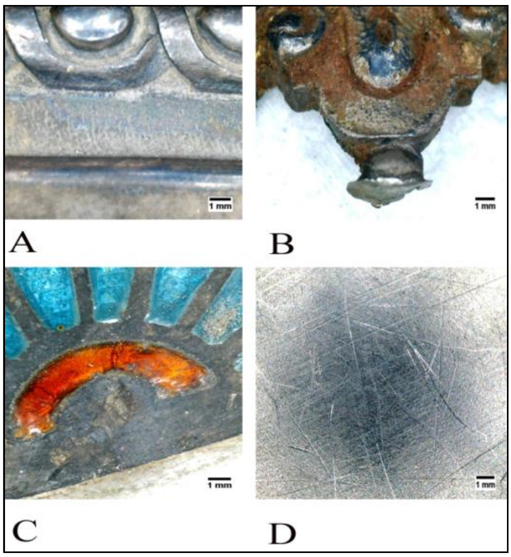


Fig. 5. Photomicrograph of the metal:
A. Black layer, B. corrosion products, C. cutting, and D. scratches.

Portable X-ray fluorescence (pXRF)

pXRF analysis of different metal areas showed differences in composition. Table 2 presents the results; the main components of the tray back, handle, frame and the legs, are silver and copper. However, the tray back is almost silver (95.38 wt. %) with a little amount of copper (3.27wt. %). The handles and the frame composition were approximately identical. The presence of gold (12.84% -10.42 %) may due to gilding of these parts; this layer was obscured by the black tarnish.

Analysis results proved that the tray legs are made of silver copper alloy (93.98% Ag and 3.88 % Cu). Pure silver is too soft to be used alone. So the addition of copper considerably increases the strength, hardness and wear-resistance of silver without leaving a deleterious effect on its ductility and formability [28]. Gold residues on the handles and the frame of the tray indicated selective gilded. This is suggested to be applied by fire gilding technique to give the impression of sound gold parts or for creating a fascinating aspect by using the decorative effect of the different noble metals in combination with other materials [29].

Table 2. Analysis results of the metal parts of the tray using pXRF

Elements	Composition (wt.%)			
	Back	Handle	Frame	Legs
Ag	**95.38**	**81.08**	**83.24**	**93.98**
Au	**0.05**	**12.84**	**10.42**	**nd**
Cu	**3.27**	**2.71**	**2.36**	**3.88**
Sn	0.22	1.23	0.44	nd
Sb	0.58	0.16	0.28	0.34
In	0.01	0.14	0.08	0.03
Cd	0.05	0.02	0.03	0.06
Pd	0.01	nd	nd	0.01
Pb	0.04	0.24	2.37	0.21
Se	nd	nd	nd	0.01
Pt	0.03	nd	nd	nd
Zn	0.09	0.24 5	0.40	0.91
Fe	0.16	1.27	0.23	0.53
Mn	0.01	0.03	0.05	0.01
Cr	0.07	0.01	nd	0.01
V	0.01	0.01	0.01	0.01
Ti	0.01	0.01	0.07	0.01

Notes: nd - not detected.

SEM- EDX analysis

Analysis of the solder

Result of EDX analysis of the solder material that was used to assemble the legs are presented in table 3 and shown in figure 6. The results indicate the presence of lead-tin soft solder. Soft soldering is a term describes the process in which pieces of metals are joined together at a low temperature: considerably below red heat. The solder generally employed for this purpose is an alloy of lead and tin. This non-corrosive eutectic is usually used for joining silver alloy artefacts [30]. Some well-preserved soft-soldered artifacts were discovered since the time of King Tut, which dates back to 1350 BC. The pieces were of crude construction, suggesting that soft soldering was not as well developed as hard soldering techniques in the Mediterranean cultures. Historians now believe that soft soldering was also developed in Northern and Central Europe by Celts and Gauls in ca. 1900 BC [31].

The flux can be zinc chloride [32, 33], whilst this flux does not remove oxides; it prevents film formation and then burn off [34].

A silver object, which has been joined using soft solders, poses several problems. The solder has often been applied extensively, thus disturbing the surface and the colour of the object. The solder joints are often weak in comparison to mechanical joints. The solder may also be the cause of a form of damage to the silver object known as fretting. Fretting is a term used to indicate the damage caused by the dissolution of silver by tin-lead solders, the silver being "eaten away" as it wears. Fretting appears as dark spongy spots in the silver surface [35], this may explain the break-up of two legs and one of the handles of the tray case study around solder points (Fig.7).

Table 3. EDX analysis results of the solder used in the tray

El.	Pb	Sn	Zn	Ag	Si	Ca	Fe	Cu	O	Al	Total
Wt.%	47.95	23.84	8.03	3.32	1.88	1.86	1.15	1.41	9.73	0.83	100

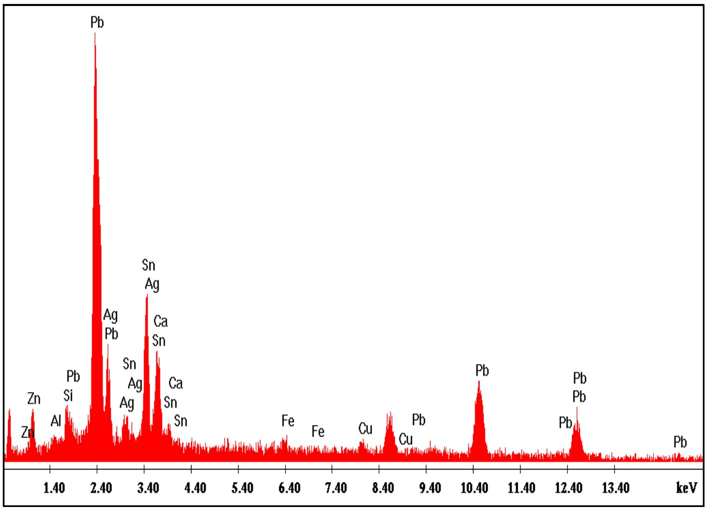


Fig. 6. EDX spectrum of the solder of the tray.

A B

Fig. 7. The areas where the solder was applied, the broken legs (A) and the broken handle (B).

Analysis of the enamel colours

Enamel colours, green, turquoise, red and white were analysed. The result of EDX analysis, shown in Table 4. Indicated that the enamels consist mainly of silica, lead oxide in addition to other components. Basically enamel is glass technically called silica to provide special properties such as lustre, fusibility, and elasticity [36], silica is basically silicon dioxide [1, 4]; Among the components of the enamel, the alkali such as Potassium and sodium in the form of oxides. The bright, polished, sparkling effect of enamels is partly due to potash in their composition, while the presence of soda renders the enamel more elastic [37].

Lead oxide is the ideal material for lowering the firing temperature, it also possess the highest refractive index and confers the greatest brilliance [4]. Softness and hardness of enamel is determined by the amount of lead oxide present. The higher the content of lead oxide the softer the produced enamel and the more liable it is to be decomposed by atmospheric influences and chemical agencies [37, 38].

On the other hand, the harder the enamel the greater the quantity of silica contained in it, and the greater the resistance to atmospheric or chemical action.

EDX analysis results presented in Table 4 and Figures 8, 9, 10 and 11 proved that all enamel colours of the tray contain significant amounts of silica and lead oxide with little amounts of the alkalies, sodium and potassium oxides in addition to traces of aluminium oxide, magnesium oxide and calcium oxide.

Metal oxides such as copper were usually used for colouring the enamel. It is responsible for the green enamel in this case study (Table 4 and Figure 8). Copper can present in different oxidation states, such as cupric oxide (CuO) which gives green colour [39].

Turquoise enamel (Table 4 and Figure 9) also contains copper oxide in addition to high percentages of potassium and sodium oxides if compared with other enamel colours. This result is in agreement with the findings of *L.F. Day* [40], in 1907 and latterly stated by *Kırmızıet al.* [41] in 2010 that the turquoise colour was achieved by the presence of copper ions together with a high alkali medium (<10–12 % K_2O + Na_2O) in addition to a high content of lead oxide PbO.

These results for green and Turquoise enamels are in agreement with the findings of *W.A. Mohamed and N.M. Mostafa* [42] in 2017 that the green and Turquoise enamel from tray backing to Hyderabad in India in the 19th century have the same results consist of copper ions.

Opaque white enamel (Table 4 and Figure 10), contains basic components of enamel, described above in addition to other characteristic element that is arsenic (As) in the form of arsenic trioxide (As_2O_3). Lead arsenate, an opacifier, which was introduced around 1750 and became the most commonly used opacifying agent in the 19th century, causing the As_2O_3 content of 11th century opaque enamels [43]. So, the presence of this oxide refers to the use of lead arsenate pacifier.

Red enamel (Table 4 and Figure 11) contains the essential components of the enamel without colouring oxides. The examination results obtained by USB digital microscope confirmed the existence of a gold layer beneath the enamel (Fig. 12). This is an old practice of decoration with enamel in which, a layer of silver or gold foil is applied beneath the enamel layer. When transparent enamel is fused over the foil it will give greater brilliancy and protection [17], these results are in agreement with the findings of *W.A. Mohamed*, *2011* [44] that the enamel was applied on gold of about 20 carats in objects from painted enamel Qajar pendant.

Table 4. EDX analysis results of the enamel colours

Enamel colour	Elements	Na_2O	SiO_2	MgO	As_2O_3	Al_2O_3	K_2O	CaO	CuO	PbO
Green	**Wt. %**	3.39	52.85	nd	nd	1.30	8.47	0.89	3.67	29.43
Turquoise	**Wt. %**	3.30	56.88	1.95	nd	nd	11.00	nd	3.57	23.31
Opaque white	**Wt. %**	2.51	49.51	nd	4.40	1.21	7.86	nd	nd	34.50
Red	**Wt. %**	3.68	51.54	nd	nd	nd	6.25	nd	nd	38.53

Notes: nd = not detected.

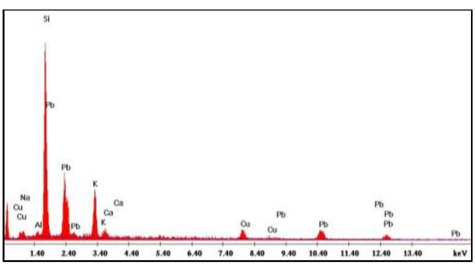


Fig. 8. EDAX spectrum of the green enamel

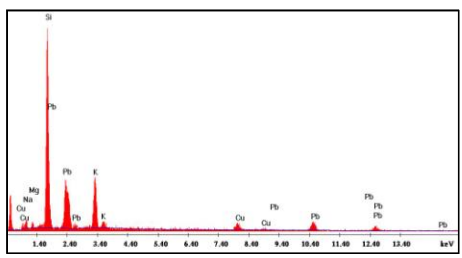


Fig. 9. EDAX spectrum of the turquoise enamel

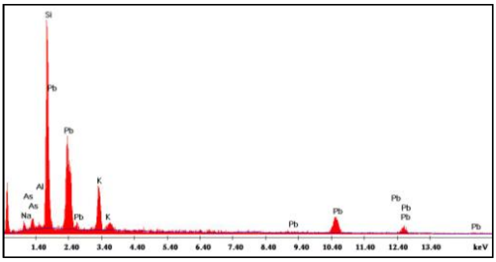


Fig. 10. EDX spectrum of the white enamel

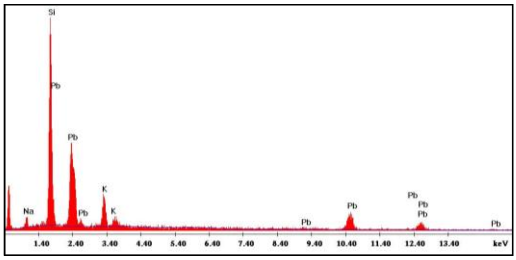


Fig. 11. EDX spectrum of the red enamel.

Fig. 12. Digital microscope photomicrograph of the gilding layer beneath the enamel layer.

A relatively high lead concentration (20%) was found in the transparent enamel. this result is in agreement with the assumption that lead potash glass was used for the painted enamel [44]. This can indisputably be assigned to the 19th century production [43]. The use of arsenic in the form of oxide to obtain opaque white enamel has also started and commonly used during the 19th century, So, the presence of high lead and arsenic indicated the date of the tray, which is of Sultan Abdul Hamid II second 1876-1909 dHr (AH 1293-1327).

Fourier Transform Infrared Spectroscopy (FTIR)

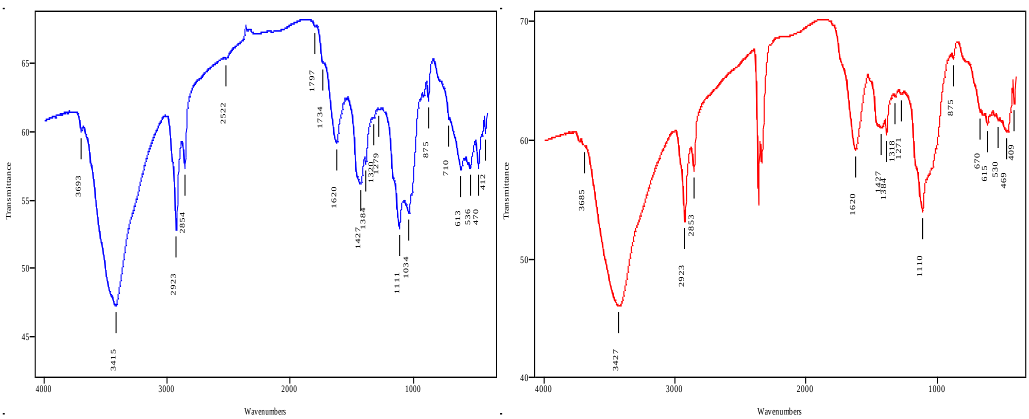
The unknown filling material underneath tugra central disc (Fig. 13) was identified by FTIR.

Fig. 13. The non-metallic filling material underneath tugra disc**.**

Spectra shown in (Fig. 14) indicated the relative intensities of intermolecular hydrogen bonding OH stretching at 3415 – 3427cm^{-1} is higher, C-H group (stretching due to aromatic and symmetric stretching) also appeared at 2924 –2852cm^{-1} and appeared (stretching vibration) C=O at 1620, 1111, 1034cm^{-1}, and the C-H group (bending vibration) appeared at 876, 779, 777cm^{-1}.

The region between 1800 and 1100cm^{-1} comprises bands assigned to cellulose [45]. Cellulose shows an O–H stretching band near 3330cm^{-1}, C–H stretching bands in the 3000–2900cm^{-1} region, C–H bending bands in the range 1500–1300cm^{-1}, and C–O ether stretching band at 1030cm^{-1} [46], Band assignments according to the literature are listed in tables 5 [47, 48 and 49].

The data obtained from FTIR stated that the filling material composed of paper [50], the purpose of which is to raise, support and protect the tugra disc. This makes tugra disc more distinguished than the tray ground.



(a) (b)

Fig. 14. FTIR Transmittance spectra of samples (a) and (b) of the filling material at 400 to 4000 cm^{-1}.

Table 5. FTIR analysis results of the filling material

Wavenumber cm-1 sample (a)	Wavenumber cm^{-1} sample (b)	Functional group bands	Assignment
3415	3427	OH stretching	Cellulose, Lignin and hemicellulose
2924, 2854	2924 - 2852	C-H stretching	Cellulose, Lignin and hemicellulose
1620	1620	Conjugated C=O stretching	Due to oxidation of cellulose
1427	-	CH_2 bending	Cellulose + lignin
1385	1385	CO_3 stretching + CH_2 bending	Cellulose + hemicellulose
1111, 1034	1111	C-O stretching	Cellulose and hemicellulose
876	876	C-H aromatic bending out of plan	Lignin + cellulose
777	779	C-H bending vibration	Lignin

Conclusions

Examination of the royal 19th century Ottoman silver tray shown many aspects of deterioration of both metal and enamel (e.g. cracking running throughout the enamel layer, flaking, exfoliation, scratches, pits) and colour alteration of enamels (e.g. tarnishing covering the whole metal surface of the tray, exhibiting scratched dull, darkened and blackish appearance).

pXRF analysis proved that the main components of the tray are made of Ag-Cu alloy with some residues of gilding layers on the frame and handles.

EDX analysis of the solder material that was used to assemble the legs indicated the presence of lead - tin soft solder. And the results of the enamel analysis by EDX confirmed that all of enamels consist of silica, potassium, sodium and lead In addition to some of the distinctive elements of each colour. And this type of analysis has proved that the tray dates back to the 19th.

The extraordinary filling material, which was found underneath tugra central disc, was identified by using FTIR analysis. Cellulose, hemicellulose and Lignin proved that it composed solely of paper.

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