

ED-XRF Analysis of a Billon Coin

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Danish skilling coins 1619-1622 exist in three types, subtypes with different mintmaster marks:

- crowned monogram, denomination and 96 in oval, fine silver 85.9%, kroneskilling, 1619, H113A-B
- crown, denomination, billon silver 31.2%, 1619, H118A-B
- crowned monogram, denomination, billon silver 31.2%, H119A (1619), H119B (1620-1623), H119C (1620-1622), H119D (1620-1621, 1623)

Anders Harck in his article in Numismatisk rapport [Harck2021] poses the question if Hede 119A (1 skilling, 1619, mint-master Johan Post), which had a measurement of silver far exceeding the prescribed silver fineness of 31.2% should be classified as belonging to the fine kroneskillinge (1 skilling Hede 113, 2 skilling Hede 112 and 4 skilling Hede 111) with a prescribed fineness of 85.9%.

In this article the results of analyzing a similar coin (Hede 119C, 1 skilling, 1620, mint-master Nicolaus Schwabe) is presented. The question is what XRF analysis says about the coin's actual fineness and what sources of error must be considered in order to make a correct classification. The coin was blanché on one side and the other side was polished to reveal the bulk of the coin.

The methodology described below can be used for analyzing other (non-mutilated) coins.

Contents

1	Coin and destructive preparation of the coin for measurement.....	2
2	Results	2
2.1	“copper” side	3
2.2	“silver” side	4
3	Measurement technique.....	5
3.1	Introducaion to ED-XRF	5
3.2	Measurement depth	6
3.3	ED-XRF instrument used	7
3.4	Analysis.....	7
4	Conclusions	7
5	Acknowledgements	8
6	Bibliography.....	8

1 Coin and destructive preparation of the coin for measurement

The coin tested is a Danish 1 Skilling from 1620 struck in Copenhagen, mint-master Nicolaus Schwabe, Hede 119 C. It has a prescribed silver content of 31.2% and a weight of 0.523g. This corresponds to a disk of 0.422 mm thickness.



Figure 1 The coin before and after preparation; left two "copper" side, right two "silver" side.

One side, the "silver" side, is used for studying the effects of blanching. The other side, the "copper" side is used to determine the composition of the body of the coin.

The reverse - the "silver" side - of the coin was subject to blanching (pickling, vitkokning, weißkochen) twice in a hot solution of 20% citric acid for 5 min each.

The obverse – the "copper" side – of the coin was polished with SiC powder on a Q tip until the (mainly) copper alloy was visible. SiC was used as an abrasive not to contaminate the alloy with other metals; like iron from a steel file.

2 Results

Details on how these results were obtained and the terminology for the element peaks (K, L, etc.) are given in 3. Concentration reported values are either Olympus Vanta proprietary (fast) calculation of concentrations or a PyMca more detailed (slow) calculation.

(in keV)	Skilling 1620, Hede 119C, Copper side	Skilling 1620, Hede 119C, Silver side	Skilling 1619, Hede 119A
Cu K 8.05	65.56 +- 0.28	29.46 +- 0.15 ratio: 0.45	24.20
Ag K 22.16	32.19 +- 0.69	51.53 +- 1.08 ratio: 1.60	73.40
Ag L 3.15	35.08 +- 0.69	88.36 +- 1.08 ratio: 2.52	-
Au L 11.44	0.032 +- 0.462	0.087 +- 0.612 ratio: 2.70	0.09
Pb L 12.61	0.73 +- 0.14	1.20 +- 0.18 ratio: 1.64	0.64
Sb K 26.36	0.11 +- 0.44	0.15 +- 0.48 ratio: 1.29	-
Zn K 8.64	0.36 +- 0.20	0.10 +- 0.18 ratio: 0.28	0.92
Bi L 13.02	0.044 +- 0.588	0.085 +- 0.797 ratio: 1.95	0.09
Fe K 6.41	0.71 +- 1.55	0.39 +- 0.97 ratio: 0.55	0.19
Ni Ka 7.48	0.058 +- 0.242	0.0078 +- 0.2042 ratio: 0.13	-
As Kb 11.73	0.063 +- 0.295	tr	-
Br K 11.92	-	0.00457 +- 0.00014	-
Co K 6.93	0.0043 +- 0.3424	tr	-
W L 9.67	0.06196 +- 0.00073	0.03056 +- 0.00061 ratio: 0.49	-
Pd K 21.18	0.0138 +- 0.0014	-	0.40
Se K 11.22	-	-	0.04

Table 1 Measurement results. The first column is the element symbol, the group and the energy of the dominant peak, the other columns are the measured concentration values in %, the second and third is for the two sides of the coin being studied (spectrum analyzed using PyMca, see 3.4) and the fourth is taken from the Harck article. The third column also gives the ratio of the measurement for the silver side to the measurement of the copper side. A value of "tr" is used when an element is reliably detected, but cannot be quantified accurately. "-" is used when the element is not reliably detected. Increased measurement time may, by reducing the statistical error, improve the

measurement and an “-“ may become “tr” or accurately quantified or “tr” may become accurately quantified.

The concentrations reported by the Olympus Vanta software is:

element	Skilling 1620, Hede 119C, Silver side	Skilling 1620, Hede 119C, Copper side
Cu	35.15855+-0.00864	64.10488+-0.00704
Ag	62.41573+-0.00920	34.01994+-0.00630
Pb	1.20843+-0.00196	0.63455+-0.00144
Sb	0.14495+-0.00305	0.08420+-0.00181
Zn	0.15987+-0.00143	0.27595+-0.00153
Bi	0.13064+-0.00088	-
Fe	0.41912+-0.00230	0.61067+-0.00180
Ni	-	0.05243+-0.00071
Co	-	-
W	0.25392+-0.00194	0.07846+-0.00169
Pd	-	-
Sr	0.00120+-0.00011	-
Zr	0.00210+-0.00013	-
Mo	0.00189+-0.00016	0.00104+-0.00014
Sn	0.10360+-0.00525	0.13789+-0.00318
Mn	-	-
Nb	-	-

The measurement values for the copper side agree well between PyMca (see 3.4) and reported by the Vanta instrument(see 3.3). For the silver side – highly in-homogenous material - the values reported by the Vanta instrument (proprietary algorithms) are generally higher; the sum of all elements except Cu and Ag is 2.42% compared to 1.91%. This is probably due to the fact that the Vanta instrument values are normalized to sum to 100%.

2.1 “copper” side

The silver content measured (Ag K) agrees well with the prescribed value; Ag L is slightly higher indicating that the polishing did not go deep enough in some small areas. They measure different depths (see 3.2) into the coin.

Apart from Cu and Ag that make up 98% of the coin there are additional elements that can indicate the origin of the metal used for making the coin. This is, however, greatly obfuscated by the coin being a mixture of silver metal and copper metal; most likely coming from different sources. One large source of Danish state income at the time were the Sound Tolls paid by ships for the use of Øresund. According to Wilcke [WilckeI, p. 234] the income was on average for the period 1621-1625: Sound Tolls 136,071, rentekammeret (the Treasury) 319,868 daler per year. Thus the Sound Toll is 30% of the sum; for 1619 it is 48% and 1620 29%. According to Venge [Venge, p. 231] the Sound Tolls were 34% of the state income in 1602. According to Jespersen [Jespersen, p. 299] the Sound Tolls were 34.5% of the state income in 1602 abd 32.9% in 1608. It was paid in a large number of different kinds of coin (and salt) – see Table 2 -; many – e.g. ducats and thalers – minted in many different locations.

Payment in	sum 1619	sum 1620
Rosenobel	8941.5	9187

GuldGylden	754.5	368
Rhinsk Gylden	1	0
Doppelte Dukater	0	11.5
Daler	173640	154589
Ort	1132.5	1022.5
Mark	8996.5	7599
Slet Mark	0	56.5
Skilling	49695	42679.5
Tønder (barrels) salt	1542	1656

Table 2 Sound Toll payments 1619 and 1620. Data from [STRO]

Published silver and copper coin analyses in 18 articles (see 6) show the following concentrations for the additional elements for coins minted 1500-1800:

metal	silver coins					copper coins				
	average concentration %	sigma	min	max	nr coins	average concentration %	sigma	min	max	nr coins
Au	0.12098	0.16446	-0.003416	1.036	463	0.08967	0.07872	0.03000	0.35000	30
Pb	0.36008	0.44207	0.00000	2.490	368	0.50758	0.37559	0.01	3.170	784
Sb	0.03869	0.01918	0.0004	0.23500	379	0.24630	0.19822	0.01	1.050	744
Zn	0.04680	0.17846	-0.01478	2.230	342	0.24625	0.23719	0.04000	1.000	40
Bi	0.06921	0.11045	0.00000	0.72520	304	0.05970	0.04069	0.02000	0.21000	66
Fe	0.40697	1.147	0.003300	14.428	283	0.11307	0.17146	0.02000	1.660	613
Ni	0.02032	0.03297	-0.005072	0.32294	203	0.12286	0.09711	0.03000	0.74000	567
As	0.02983	0.04481	0.00000	0.22900	223					
Br	0.38502	1.661	0.00000	9.984	47	0.01475	0.01035	0.00000	0.08000	461
Co	0.00092	0.00627	0.00000	0.04301	47	0.02000	0.01414	0.01	0.04000	4
W	0.00347	0.01359	0.00000	0.06117	47					
Pd	0.00390	0.01239	-0.007279	0.05435	217					
Se					0					

2.2 “silver” side

The silver side consists of an outermost thin layer of almost pure silver with very small amounts of elements that are mostly unaffected by the citric acid; e.g. Au and Sb. The measurement depth (see 3.2) is, however, larger than the thickness of the outermost layer so the measured concentration is influenced by the concentration of the bulk of the coin. As the measurement depth varies with photon energy the ratio of the silver side measured concentration to the copper side varies from element to element and is lower with increased measurement depth (see Table 3).

element, group, energy	Measurement depth in Ag	measurement depth in Cu	ratio
Au L 11.44	11.5 μm	7.4 μm	2.10
Sb K 26.36	18.7 μm	71.4 μm	1.26
Cu K 8.05	4.5 μm	21.6 μm	0.44
Zn K 8.64	5.4 μm	26.2 μm	0.22

Table 3 Measurement depth and ratio silver side to copper side for selected elements.

Other elements, notably Cu and Zn, are much depleted in the outermost layer. They are, however, slightly larger due to measuring into the bulk of the coin.

Br is most likely remnants of a corrosion product.

The ratio of the measurements of Ag K and L, with vastly different measurement depths, is a clear indication of surface enrichment.

The prepared coin can be modeled as a thin “silver” layer and a thicker layer with the composition given by the measurement of the “copper” side. Adjusting the thickness of the layers and comparing a Monte Carlo simulation¹ generated spectrum to the actual “silver” spectrum gives the best fit for a thickness of 8.2 μm for the thin “silver” layer. Further analysis gives the estimated composition of the “silver” layer in Table 4.

element	estimated concentration %
Au	0.0611 +- 0.4100
Pb	0.5750 +- 0.0928
Sb	0.1198 +- 0.3240
Zn	0.0510 +- 0.1676
Bi	0.0398 +- 0.4058
As	0.0101 +- 0.2260
Br	0.0050 +-0.00006
Se	0.0024 +- 0.2594

Table 4 Estimate of the composition of the thin “silver” layer.

3 Measurement technique

3.1 Introductaion to ED-XRF

There are numerous introductory texts to XRF, for example [BRUKER] and [GETTYALE]. Only a very brief summary is given here.

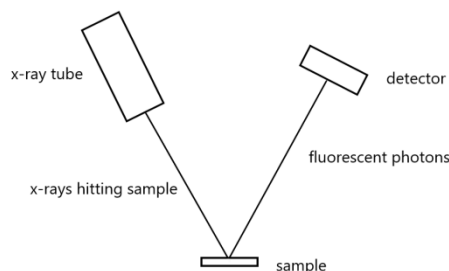


Figure 2 Simplified sketch of an XRF instrument.

In XRF a sample to be measured is hit by x-rays from an x-ray tube. These x-rays can be either absorbed, scattered, or transmitted by the sample material. When absorbed by an atom in the sample an electron is pushed to a more unstable higher orbital and when it moves back to a lower orbital the excess energy is emitted as a fluorescent photon. The energy is characteristic of the element and transition; each transition has a specific intensity. The transitions between shells are named and in the Niels Bohr’s model the shells are labeled K, L, M... Each shell has a number of sub-shells. The energy of the transition photons are detected and the corresponding group peaks in the detected spectrum are labeled

¹ A type of mathematical algorithms that are based on random numbers to solve certain problems that are difficult to solve using deterministic methods.

K, Ka, Kb, L, L1, L2, L3, ... A “peak” may consist of multiple smaller peaks at slightly different energies. Sometimes e.g. the K “peak” can be used for the measurement; in other cases the Ka or Kb gives better results as it may be further away from peaks of other elements.

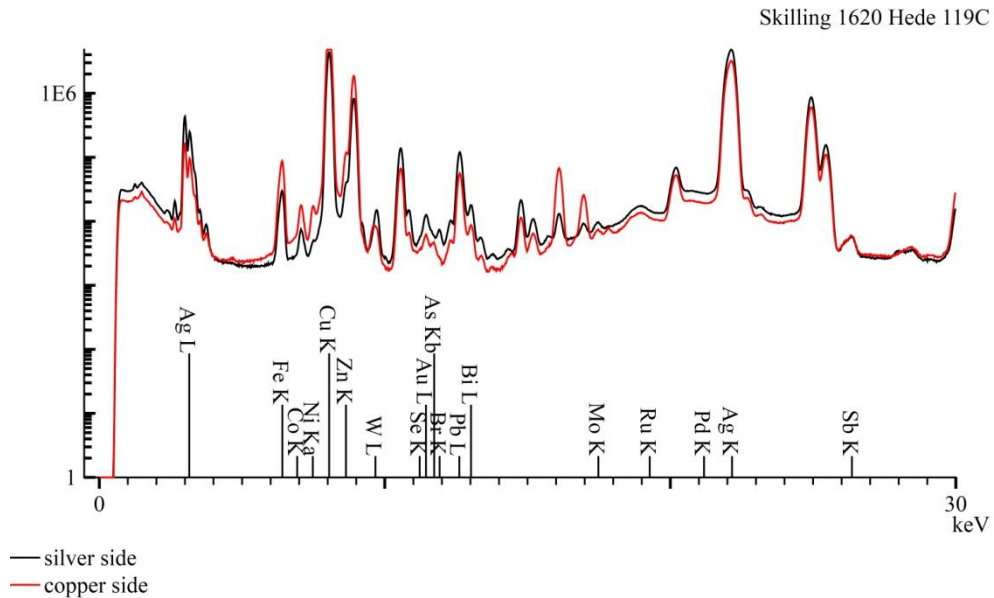


Figure 3 Measured spectra with some peaks marked.

From the size of the peaks the element concentrations are derived. Software in the instruments perform this calculation using proprietary algorithms tailored to different kinds of sample; e.g. precious metals, metal alloys, soil samples. The spectrum can, typically, be exported and analyzed using different software. In many cases the calculation is more elaborate, giving a better result, but using more computing resources.

3.2 Measurement depth

When the emitted fluorescent photons (x-rays) traverse the sample being measured some are absorbed and do not reach the detector. The absorption differs with the sample material traversed and the energy of the photon and gives a measurement depth for a particular line. For a blanded billon coin the lines have a measurement depth of 2 μm for Ag L and 70 μm for Ag K (in pure silver) and 2 μm for Ag L and 44 μm for Ag K (in pure copper).

As an example consider a sample consisting of an 8 μm layer of 85% Ag and 15% Cu (the "silver" layer) and a 414 μm layer (the "copper" layer) of 30% Ag and 70% Cu

An Ag L peak, with a measurement depth of 2 μm , measures only the top quarter of the "silver" layer and is thus an accurate measurement of the Ag in the "silver" layer.

An Ag K peak, however, with a measurement depth of 52 μm measures the full "silver" layer and 44 μm of the "copper" layer and gives a measurement of Ag closer to that of the "copper" layer.

An Au L peak with a measurement depth of 10 μm gives a measurement of Au close to the Au content of the "silver" layer (only a small component is the content of the "copper" layer).

An Sb K peak with a measurement depth of 36 μm gives a measurement of Sb closer to that of the "copper" layer.

3.3 ED-XRF instrument used

The equipment used was a Olympus Vanta VMR CCC A3 E hand-held instrument run at 40 kV, 25.6 μ A, and with a 2mm Al filter. The measurement time was 900s realtime.

3.4 Analysis

Rather than using the results given by the instrument, the measurement spectra were analyzed using PyMca [PyMca]. It uses the Fundamental Parameters method that converts the measured peaks into concentrations using atomic parameters, such as atomic binding energies, fluorescent yields and absorption values. It requires detailed instrument parameters, such as geometry, x-ray tube spectrum, and detector information; in a configuration file. Input to PyMca is the configuration file, which also includes a list of peaks for which a result concentration is desired, and the spectrum to be analyzed.

In order to verify the results and to provide calibration an extensive set of Certified Reference Materials (CRM: a sample with a precisely known composition) were measured. Example scatter plots of the PyMca result versus the known CRM content is shown in Figure 4. For each element of interest and the group permitting the most accurate result a linear least squares fit is performed of measured values and the corresponding certified concentration yielding a calibration curve. Note that for Ag both the K and L groups are fitted separately as these have much different measurement depth and gives an indication of surface enrichment.

The measured values calibration methodology of [CHARM1] and [CHARM2] was used.

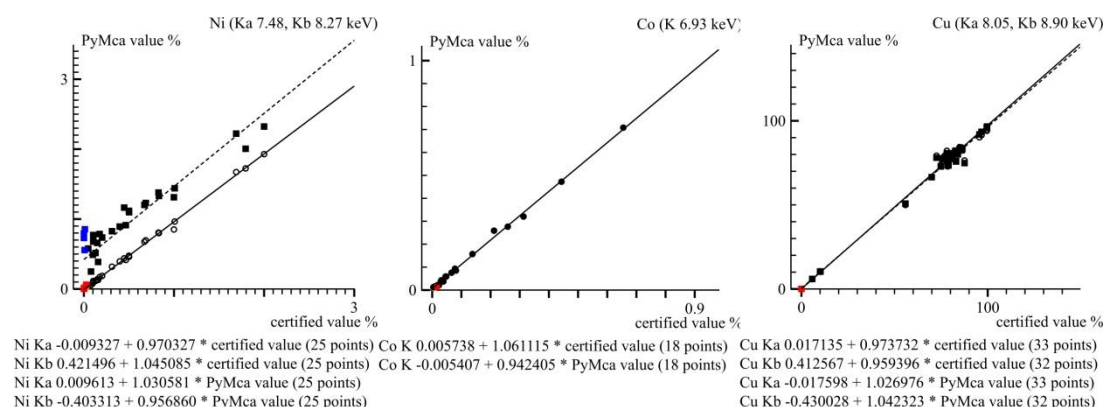


Figure 4 Sample calibration curves. For Ni the calibration for Ka (open circles) and Kb (filled squares) are shown; clearly Ka gives the more accurate results. The Co calibration curve shows that the measurement is accurate to very low concentrations.

The estimate of the thickness of the thin “silver” layer was achieved by Monte Carlo simulation of generating spectra consisting of two layers; one consisting of Ag 80% Cu 10% and a thicker having the measured composition of the “copper” side. The XMI-MSIM tool in PyMca was used to generate these spectra for various thicknesses of the thin layer and the best fit gives a good estimate of the actual thickness. The corresponding spectrum was subtracted from the measured spectrum of the “silver” side and a PyMca fit was performed giving an estimate of the composition of the thin layer.

4 Conclusions

The analysis of billon coins (and any other inhomogenous material) requires great care and access to multiple element groups; typically not available in the instrument software. Ag K and L being an important one for “silver” coins. The measured values for the “silver”

side is, according to the measuring instrument software, Ag 62.42%, while the analysis of the spectrum is Ag L 88.36% and Ag K 51.53% (i.e. the measuring instrument value is one-third of the way between Ag K and Ag L). The "correct" value (for the coin's bulk) is 32.19%; much lower than the value from the measuring instrument and Ag K. The "only" thing one can say with certainty about the Ag content of the bulk, based on the measurement of the "silver" side, is that it is lower than both the measuring instrument value and the Ag K value.

The measurement of Hede 119A was made with an instrument manufactured by Bruker (S1 Turbo SD) and with its software the Ag content is 73.40%. The difference between 62.42% and 73.40% can be explained by the use of different, proprietary, algorithms that give different results for highly inhomogeneous materials. It is clear that the skilling 1619, Hede 119A is most unlikely a kroneskilling, but rather a billon coin, since the Ag measured concentration of 73.4% is much lower than the prescribed 85.9%.

5 Acknowledgements

Joosje van Bennekom, Senior Conservator, Rijksmuseum, Amsterdam very kindly measured the coin and provided the spectra of the coin for analysis and of numerous CRMs for comparison. Anders Harck provided detailed comments; especially for 3 to make the content understandable for people new to XRF. Inger Bolt Jørgensen and Sven Aagaard for references for the Sound Toll fraction of the Danish state income.

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