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Citation

Bjørnland, L. H., Degryse, P., Schibille, N., Eremin, K., Walton, M., Gratuze, B.,
... Shortland, A. J. (2024). The production of smalt and other cobalt compounds
at the Blaafarveværket, Modum, Norway. *Forensic Archaeology, Anthropology
And Ecology*. doi:10.1558/faae.26631

Version: Publisher's Version

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Downloaded from: <https://hdl.handle.net/1887/4175239>

Note: To cite this publication please use the final published version (if applicable).

The Production of Smalt, and other Cobalt Compounds at the Blaafarveværket, Modum, Norway

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Forensic analysis of fine art and objet d'art uses accurately determined pigment compositions to confirm or deny provenance, dating and attribution. Some of the most important pigments in this respect are those associated with cobalt. The Blaafarveværket, or Blue Colour Works, based near Modum in Norway, was a producer of cobalt products (especially smalt and zaffre) during the eighteenth and nineteenth, exploiting local cobalt ores and other raw materials locally sourced and brought in from more distant areas. century. The Blue Colour Works is now a museum and study of its archive has detailed type and quantity of its production, showing that it was perhaps the most important producer of cobalt products in the early nineteenth century, and the largest mining operation in Norway. The archive gives details of the production processes for some of the products and shows that these processes changed through time. The museum preserves samples of different parts of the intermediate and end products, showing that the output was a smalt very low in manganese and zinc, with some nickel, significant iron and arsenic and relatively raised bismuth and uranium contents. REE element analysis combined with lead isotopes shows that different sources of silica were probably used, since both are quite variable, supporting archive suggestions that external sources of silica and plant ash were employed on occasions.

Keywords: cobalt, smalt, zaffre, mining, production, Norway

Submitted January 27, 2023; Accepted July 17, 2023

Introduction

Detailed forensic analysis is increasingly being used in the art world to verify the attribution of paintings and objects. One of the key techniques employed involves identifying pigments in the object in question and comparing their characteristics to a known database of objects or pigments that have good provenance and well determined date. Particularly, useful in this respect is to be able to characterise the products of dated factory sources, where the confidence about the association between a pigment produced, its factory and therefore its date is strongest.

Blue pigments are especially useful as the colour blue has been a very important colour in art since its very inception in the form of the pigment “Egyptian blue,” in the early third millennium BC (Hatton, Shortland and Tite, 2008, 1591–1604). As such, it was the first artificially generated pigment following on from natural compounds used for white, black, yellow and red. Egyptian blue was used alongside rare naturally blue minerals, but from the 16th century BC there was another important way of producing a blue: glass coloured using compounds of copper, or especially cobalt (Shortland 2012). Of these, cobalt is the more interesting, and different sources of cobalt from factories of different dates have been used to generate similar colorants for local use or more distant trade and in the production of a range of materials such as ceramics, glass, paper, textiles and even laundrying as a whitening agent (Delmare and Rouchaleau 2013, 87–94). However, usable cobalt sources are relatively rare and because cobalt ores almost always exist in the presence of ores of multiple other elements, creating a pure colorant was difficult (Gratuze, Soulier, Blet and Vallauri 1996, 77–94). Not only that, but the unwanted secondary elements varied in different cobalt sources and almost always persist to a limited extent into the finished product. Thus cobalt colorants tend to exhibit a characteristic “fingerprint” of elemental compositions that can be linked to the ore body and associated factory, and are ideal in forensic investigation of objects (Gratuze, Soulier, Blet and Vallauri 1996, 77–94). Ten to twelve such fingerprints have now been identified, although some are known only in the finished product—i.e., the ore source is still debated (Gratuze, Pactat and Schibille 2018, 225, and Gratuze, Soulier, Blet and Vallauri 1996, 77–94). To the archaeologist and historian, cobalt colorants are therefore a valuable tool, since they allow a pigment to be traced from its production site right through to a finished object which might itself be traded. Long distance trade in pigments and objects can therefore be hypothesised where the particular characteristics of a cobalt ore body is accurately known.

In Europe from 1400AD, “smalt”, a dark-blue, crushed, cobalt-coloured glass, was the main way in which this cobalt pigment was traded (Delamare and Rouchaleau 2013). It was produced up until the late nineteenth century, when it was superseded by novel methods for producing blue colours and modern synthetic pigments including cobalt oxide. It was an important and expensive commodity, which meant that prospecting for cobalt was common. However, finding an exploitable source was rare, as noted above. The main European source was in Saxony, at Schneeberg and neighbouring towns, and although this waxed and waned due to political and economic crises, this remained the most important until the nineteenth century (Delamare and Rouchaleau 2013, 56–66). Other smaller and very limited attempted at

production were made in England, France and Spain, but their production volumes were very small (Delamare and Rouchaleau 2013, 67–83). One of the most important later sources was Norway, and “from 1823 to 1849... [Blaafarveværket, in Norway] was the largest producer in the world. It [...] supplied three-quarters of the world market for cobalt blues” (Delamare and Rouchaleau, 2013, 40). Despite this, it is comparatively little known and studied.

The Blaafarveværket

The Blaafarveværket, or Blue Colour Works (BCW), was an industrial and mining facility situated in Modum, about 40km west of Oslo, Norway. The purpose of the factory was to process locally sourced ore minerals to produce four cobalt bearing materials: smalt, zaffre, slig and cobalt oxides. “Smalt” was a cobalt-rich glass; “zaffre” was a relatively impure cobalt compound used in the colouring of glasses, glazes and other materials; “slig” was a crushed, washed and calcined cobalt ore, with very little refining, whereas “cobalt oxide” was a chemical compound—the most refined and pure of the materials produced. The exact differences between these compounds and their use are discussed later. The BCW was active from 1772 to 1898 (Berg (red.), Sæland, Nyland, Østensen, Nordrum and Kullerud 2016, 297), and different materials and quantities were produced at different times during this period. Broadly speaking, from the middle of the 1780s to the end of the 1840s the main products were smalt and zaffre. This changed in the late 1840s to the late 1860s when the production of cobalt oxide became more important. Later on, from 1870 to the close of mining in 1898, the only production was slig, which was shipped to the blue colour works in Saxony for further refining (Berg (red.), Sæland, Nyland, Østensen, Nordrum and Kullerud 2016, 298). To reflect this change in product, the technology of production also changed through the period of operation, with different methodologies used for extracting the cobalt from the ores.

The facility was large, at one time the largest mining operation in Norway, and produced significant volumes of cobalt compounds. This paper combines the study of a very important historic archive housed in The National Archive of Norway with the analysis of the products of the processes, preserved in the BCW, which is now a museum. It aims to describe the way cobalt compounds were extracted and the nature of their products with the purpose of allowing further work to potentially trace their trade and dissemination. It is clear that the products were exported throughout Europe, especially to England, and from there around the world, including China and Southeast Asia where they were widely used in many manufacturing areas.

The BCW museum and archive

The “Cobalt Works of Modum Foundation” (Stiftelsen Modums Blaafarveværk—Bygdemuseet Modum) was founded in 1971 and consolidated to form the BCW Museum in 2004 (See <https://www.blaa.no/om-oss/om-stiftelsen/>). This industrial museum is now made up of 60 different buildings with a total footprint of around 8000m², with land, mines, internal roads and bridges covering an area of 43 hectares or just over 100 acres (Figure 1). The Blue Colour Works private archive was collected and preserved in The National Archive of Norway under the reference code RA/PA-



Figure 1. The Blue Colour Works in 1892, showing the waterfall Haugfoss. The building to the far left is one of the calcination huts and it is connected to the giftfang arsenic catcher (long, sinuous building ending in a short smoking chimney, just left of centre). The large building just below and right of Haugfoss is the stamping mill, powered by water from the waterfall. Copyright of Buskerud Fylkesfotoarkiv.

0157-Modums Blaafarveværk. The archive consists of 53 series, 1342 portfolios and 543 boxes with a total of about 500,000 pages of written material.¹ The material is divided in 4 periods: 1776–1822 (F), 1822–1849 (G), 1849–1856 (H) and 1856–1919 (I), which are subdivided into 10 series covering the activities of the BCW including: meetings, management, legal issues and contracts, correspondence, reports on operation and production, personalia and various aspects of the local towns. The archive material was kept at different locations and under varying conditions before it was collected at The National Archives, which means that there are inconsistencies and some gaps in the record.²

In addition to the archive, there are two collections of raw materials and products of the BCW. The first is a collection of a total of 82 samples in small glass vials. These include smalts and oxides and were donated to the BCW Museum by the Norwegian University of Science and Technology (NTNU). These samples were collected in the 1920s or 1930s by the University at the BCW. Most of the 82 samples are smalts, some dated, some not, the earliest being 1782 and the latest is from 1854. There are also 2 samples of cobalt oxides that both date to 1849. In addition, there are in this collection a few examples of smalts that are clearly labelled “Schwartzfels” and another “Cöln,” indicating that they are probably German. They are probably linked to the

1. See <https://www.blaa.no/om-oss/dokumentasjonssenter-og-arkiv/>

2. See <https://www.arkivportalen.no/entity/no-a1450-0100000006338?ins=RA>

Hessen cobalt mines and blue colour works, that are contemporary with the BCW and interacted with them, along with other German producers (Delmar, 2013). A full list of the 82 is given in Supplementary Material in the Appendix. The Museum also preserves a second more informal collection of uncatalogued samples and raw materials. These include cobalt slig from the later periods (around 1860s), which were preserved in their original containers, so are certainly associated with production at BCW.

History of the BCW

The Blaafarveværk were founded in 1776 after cobalt bearing ores were discovered on the Skutterud farm (now spelt Skuterud) in the municipality of Modum in south-east Norway. Test works were started in 1773, under the leadership of staff from the Silverworks in nearby Kongsberg. In April 1776, the BCW were founded by a royal decree by the Danish Norwegian state with the aim that the cobalt extracted was to be refined into a blue pigment. To do this, properties at Fossum on the river Simoa, about a mile south from the cobalt mines at Skuterud, were bought. The power for the factory was to be derived from a waterfall on the Simoa, Haugfoss, which with a fall of 39 meters drove the BCW machinery (Lindeman 1993, 5–15).

The first colour samples were produced in 1782, but it was after the recruitment of a new director, Georg Christopher Bernstein in 1783, that BCW were able to produce higher quality smalt that was needed to make headway in European markets. Export of smalts started in the middle of the 1780s (RA, Tollregnskaper Drammen, 1786, 199b–200a). The smalt was taken to the port of Drammen and shipped to Copenhagen and Holland.

In 1813 and 1814, the Napoleonic wars caused severe problems for the BCW, when it was mortgaged to a Swedish merchant by the Danish king. In 1814 the union between Norway and Denmark ended, and years of legal disputes followed. The disputes regarded who had ownership to the works themselves, and to the remaining stock of pigments (Lindeman, 1993, 59–66). Eventually in 1822, the BCW was sold to a German banker, Wilhelm Christian Benecke (later von Gröditzberg) and his associate Benjamin Wegner. They started to rebuild the BCW by headhunting new German personnel to lead the production (Lindeman, 1993, 77–79). The German chemist Friedrich Christian Gotthilf Roscher arrived in Norway in 1822 and developed a new melting process that made it possible to use poorer cobalt ore to produce high quality smalts. This meant that more of the unmined ore could be used, something that led to a great expansion of the mining operation at Skuterud, including the introduction of underground mining which resulted in discovery of new zones of cobalt ore (Lindeman 1993, 83). In the 1830s and 1840s, the BCW were the biggest mining operation in Norway with about 1,200 employees in 1840. The BCW produced up to 180 tons of different pigments a year, a large percentage of the total European smalt and zaffre markets.

However, during the last half of the 1840s the competition from manufacturers of the new synthetic ultramarine resulted in falling prices and fewer sales. The BCW tried to respond by developing new types of oxides, but in 1849 the BCW was once more in financial difficulties and in the summer of that year was sold to its biggest

customer, the British company Goodhall and Reeves (formerly Smith, Goodhall and Reeves) (Lindeman 1993, 89). The new owners downscaled the staff and continued the production up until 1855, when it was again sold, this time to its main competitor, the Saxon company Sächsischer Blaufarbenwerks-Verein, who were closing down rivals and merging different blue colour works within Saxony (Drammen Blad, 1855). Production of smalts were stopped and only slig, oxides and some zaffre were produced. From 1870, the BCW produced only slig which was sent to the colour works in Saxony for further smelting and refining (RA, Østlandske bergdistrikt 1867–1879). In the 1890s, the BCW were converted into a wood processing works and finally closed down in the autumn of 1898, but the Saxon company continued the wood processing works until 1919 (Lindeman, 1993, 94)

Raw materials of Production

Geologically, the Skuterud Cobalt Mines are situated in the Modum Complex, which is part of the Kongsberg sector of the Precambrian supracrustal rocks of southeast Norway (Bjerkgård, Dahlgren, Raanes, Sandstad and Heldal, 2020, 28–29). The country rocks of the Modum Complex consist mainly of steeply dipping, isoclinally folded metasediments containing elongate outcrops of metagabbro-amphibolite bodies of varying size. Within the metasedimentary rocks are sulphide-rich lenses known as “fahlbands”



(Gammon 1966b) and the Skuterud Mines exploit the most westerly fahlband, where the mineralized zone was richest in cobalt (Figures 2 and 3). The cobalt mineralization consists of cobaltite (CoAsS), Skutterudite (CoAs_3), and cobalt bearing minerals of arsenic (Berg (red.), Sæland, Nyland, Østensen, Nordrum and Kullerud 2016, 298). The Norwegian Geological Survey also lists glaucodot ($(\text{Co}, \text{Fe})\text{AsS}$) and safflorite ($(\text{Co}, \text{Fe})\text{As}_2$)

Figure 2. Skuterud mine: earlier open cast mining of the cobalt bearing ore. Image from the late nineteenth century, copyright of BCW.

(Bjerkgård, Dahlgren, Raanes, Sandstad and Heldal 2020, 28) The genesis of the cobalt mineralization at Skuterud is not well understood and little research has been done on the subject.

The archive gives details of the type and source of the other raw materials used in production, specifically, quartz and potash. The quartz was local, coming from a quartz mine owned by the BCW. Some of the potash was produced by the BCW, but mainly the potash came from Norwegian, German and American producers (Lindeman 1993, 39–40). In 1783, the BCW director complained that the Norwegian potash produced “too much gal in the glass melt” (Lindeman 1993, 38) and was therefore not suitable for smalt production (Lindeman 1993, 26). Thus, the source of potash changed through time.



Figure 3. Skuterud mine: miners working underground in some of the later shafts. Images from the late nineteenth century, copyright of BCW.

Production of Smalt—1830

The method of production remained relatively unchanged from the 1780s to the 1820s, but little is known of the precise production methodology used for this period. This might still be in the archive, but to date, it has not surfaced. However, the archive does give a vivid idea of how smalts and cobalt oxides were produced later, since they were described in 1830 in a text by the head chemist (“Hytteinspektør”) Friedrich Christian Gotthilf Roscher (RA, Modums Blaafarveværk, 1830). It is likely that his account reflects production in the last decades of the eighteenth and first decades of the nineteenth centuries, but it is not clear how long before 1830 the processes had been in action. He first describes the preparation of the raw materials. The quartz was crushed with water in a stamping mill, where the water allowed the separation of the quartz grains from associated mica and iron oxides. The more or less pure quartz sand was then dried. When the potash arrived at the BCW, it was heated to free it from moisture and “sphere like substances” (RA, Modums Blaafarveværk, 1830). After this it was also crushed and sifted. The richest cobalt ore with the lowest

percentage of iron was used to produce the finest smalts with a dark colour, while the poorer cobalt ore with large quantities of iron was originally used to produce ordinary smalt, lighter in colour. The cobalt ore was first crushed and sorted by hand to pick out the richest cobalt minerals. This was then crushed again, washed and heated to produce “slig.” The slig contained a combination of different cobalt, arsenic, iron and sulphur phases. To rid the slig of arsenic and sulphur, it was strongly heated. The arsenic was given off as a smoke, which was collected in a 150-metre-long pipe construction called a “giftfang” (in English, “poison catcher”). The arsenic was refined and sold as a by-product and was used, amongst other things, to rub on the skins of cattle to deter insects and to colour wallpaper. During the heating, the cobalt and iron turned to oxides.

Roscher invented a new melting process to enrich the cobalt content in the end-product whilst using poorer ore. The method consisted of reducing the inferior ore to cobalt metal (RA, Modums Blaafarveværk, 1830) using charcoal and crushed green glass as a flux (Müller 1843, 48–49). The metal was then roasted to form an oxide. By applying this reduction method, most of the iron in the ore was separated out as slag. Using this method, previously inferior cobalt ore could produce an oxide with as high a percentage as 30–40 % cobalt (Müller 1843, 49–50).

The smalts themselves were produced by combining quartz, potash and cobalt oxide (RA, Modums Blaafarveværk, 1830). The BCW had their own smalt recipes and were very strict about quality (RA, Modums Blaafarveværk, 1830), so much attention was given to the quality of the ingredients. The components were melted in a glass type furnace to form a glass. When the liquid glass reached sufficiently low viscosity, it was poured out into a vat of cold water, solidifying and shattering it. The blue glass cullet was taken to stamping mills, crushed and then milled. During the milling the crushed glass was mixed with water and then poured into sedimentation tanks where the smalt would be separated based on its grain size: the larger, heavy grains sinking faster, forming a fining up sequence on the bottom. The finest grainsize glass was kept and the courser milled again.

The smalt was graded according to its perceived quality – generally, the finer the grainsize and the deeper the colour, the higher the quality. The coarsest grainsize was labelled “Writers sand”, the middle grainsize was labelled C for “Couleure”, and the finest milled smalts were labelled E for “Eschel” (Lindeman 1993, 45–46). The texture of each label was further subdivided. The coarser end of the C label was given an additional B (CB), the middle-labelled f (Cf), and the finest milled C were given no extra letter (Lindeman, 1993, 46). E were subdivided in two categories: the course “Fasseschel” and the fine “Sumpfeschel” (given an S after the E) (Lindeman 1993, 46).

The quality of the colour was divided into three groups: O, M and F (Lindeman 1993, 44–45). O stood for “Ordinær” and was the poorest quality of colour. M stood for “Middels”, and F stood for “Fin”—the best quality (Lindeman, 1993, 45). F were subdivided further based on the amounts of Fs given, from F to FFFFF—the more Fs the finer the colour. Thus FFFFF (or 5F) was the finest smalt and was also called “Royalblau” (“RB”). Up until the 1830s, sometimes H for “hoch” was used to express quality, giving HHHFFF (Lindeman, 1993, 45). From 1789–1819, Lindeman, gives the quanti-

ties of materials required to produce 100 centner (4980kg; 1 centner = 100 pounds = 48.9Kg,) of FFFF smalt. This required:

- 11 centner and 25 pounds rich-middle quality slig,
- 57 pounds crushed slig,
- 16 centner and 15 pounds FFFF-Eschel,
- 4 centner and 30 pounds FF- and FFF-Eschel,
- 58 pounds cobalt oxide,
- 55 centner and 34 pounds quartz,
- 25 centner and 69 pounds potash (Lindeman, 1993, 41).

This is one of several different recipes of this type ranging through the life of the BCW, all similar, but with slightly different components or proportions. What is interesting to note is that some of the finished products (labelled Eschel) are included in the batch. In other words, the methodology recycles some of the final smalt back into the production process. It is suggested in the archive that this is because some of the Eschel smalt, being the finest grained, did not have the depth of colour required, and therefore was difficult to sell. Reworking it into the recipe bulked out the new pigment for little, or an acceptable, loss of quality. Zaffre was produced by heating the cobalt slig and mixing it with crushed quartz. It was also labelled based on its quality with varying numbers of Fs (or M or O) (Lindeman 1993, 46).

Production of Cobalt oxide—1840s onwards

Crookes and Roehrig (Crookes and Roehrig 1868, 578–579) discuss the processing of ores at Modum. They define two ores that were extracted: skutterudite (CoAs_3 , “speiss”) and cobaltite (CoAsS , “glance”). The final products of the ores are listed as cobalt oxide and “zaffre”. The first step of the process was to sort (“dress”) the ores and then they were roasted to form a finer ground mass (“schlich”, a Germanisation of “slig”). This was mixed with sulphuric acid to form sulphates and boiled dry and then roasted again. The resulting material was boiled and the liquid was then “tapped off”, leaving heavier unwanted earthy minerals behind. and moved to other vessels, in which it was heated again. It is then mixed with sodium carbonate. It is specifically stated that just enough sodium carbonate is added, to precipitate the iron and copper sulphates along with “a small quantity of cobalt”, called here Precipitate 1. Following precipitation and removal of these carbonates, the final liquid was again separated, filtered and heated got a third and last time. It was then mixed with more sodium carbonate to precipitate all the cobalt in solution as cobalt carbonate, called here Precipitate 2. This Precipitate 2 was “filtered, washed, pressed, and strongly heated in clay crucibles” resulting in cobalt oxide, the desired product in this later period. Precipitate 1, on the other hand, was smelted, producing a “speiss containing about 30 percent cobalt”, which was “then submitted to a further treatment” and was “used for the production of zaffre”. The “further treatment” is not specified. There are details of the requirements in terms of raw materials. Converted into kilos, to create one kilo of Precipitate 2 requires 6 kg of sulphuric acid, 3.5kg of sodium carbonate and 0.45 cubic metres (around 300kg) of wood.

Volume of production

Unfortunately, the archive is not complete enough to give the total production of the BCW between 1776–1898. However, the Norges Geologiske Undersøkelse (Geological Survey of Norway) has estimated, based its archives, that the total amount of extracted ore was about 1 million tons, with an average of 0.2 % cobalt (Bjerkgård, Dahlgren, Raanes, Sandsted and Haldal, 2020, 28). Of course, this estimate says little about how much finished smalts, zaffre, slig and oxides were produced from the ore. Based on accounts by Thorvald Lindeman, the total production in 1791 and 1792 averaged 125 tonnes per year. In 1793, the mill burnt down stopping production. The next figures available are for 1813–1819 which averaged 52 tonnes, with 1819 the greatest with 109 tonnes. Between 1830 and 1848, Amund Lammers stated that the BCW produced nearly 2,000 tonnes of smalts and 816 tonnes of zaffre (or 102 and 42 tonnes per year, respectively) (Lindeman, 1993, 81). However, it is important to note that the numbers vary in the sources. Between 1850–1855 there are detailed records concerning what was exported, and because almost all of the production was exported, this gives an indication of the total production. The average export over this period was 45 tonnes of “Blue colour”, while the export of “cobalt blue, refined cobalt and cobalt oxide” fell remarkably over the period from 1 tonne in 1850 to nothing, zaffre exports also fell from 22 tonnes to 2 tonnes, and “smalt blue” averaging 5 tonnes per year in the first three years, is not mentioned at all in the last three. From 1856–1894 it is estimated that 257 tons of cobalt ore were extracted (Ließmann, 1994, 32). For the year 1866, 3.6 tonnes of “pure cobalt oxide” were produced (RA, Østlandske bergdistrikt, Voigt, 1867). Following this, production continued to reduce considerably, and then finished completely (Statistisk sentralbyrå, 1874).

Methodology

To investigate the composition of the products with the aim of characterising the BCW production, samples were taken both from a selection of the 82 glass vial samples and from the other preserved samples of the production process. Small amounts from a selection were analysed. The powders were analysed by ICP-OES, whereas those preserved as uncrushed glass/smalt were analysed by LA-ICPMS. A further subset was analysed by lead isotope analysis.

ICP-OES

The major and minor element composition of a selection of smalt samples was determined at the geochemical laboratories of KU Leuven (Belgium) by inductively coupled plasma optical emission spectroscopy (ICP-OES) after lithium metaborate fusion using a Varian 720-ES instrument (simultaneous ICP-OES with axially viewed plasma). The instrumental setup is described in Brems *et al.* 2012 (2012, 2897–2907). Smalt powders were weighted and approximately 10 times the amount of LiBO_2 was added. The samples were then fused at 1000° C for 10 min and dissolved in 3% HNO_3 + 1.5% HCl . A series of HIQU single element solutions (CHEM-LAB, Belgium) was used to prepare calibration solutions. Standard reference materials were used to control the procedure (NIST 610, BCR-1G, MRG-1, WMS-1). Precision was better than 5% and accuracy better than 10% for the values reported.

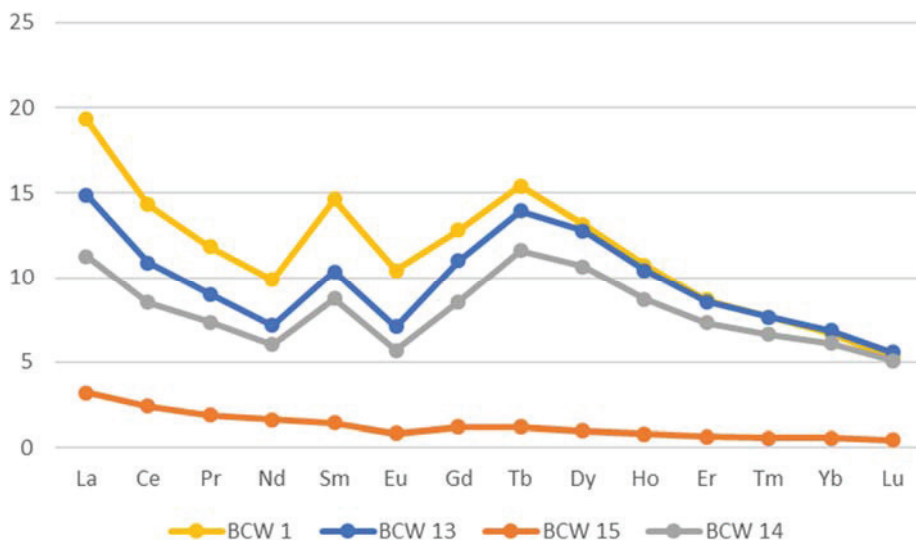


Figure 4. LA-ICPMS data showing the chondrite normalized REE data for the four BCW smalts analysed.

LA-ICP-MS

Four glass cullet samples, known to be smalt before it was crushed, were repeatedly analysed (n=6) by LA-ICP-MS at IRAMAT-CEB (Orléans, France). The analyses were performed using a Thermo Fisher Scientific ELEMENT XR mass spectrometer with a linear dynamic range of twelve orders of magnitude thanks to the combination of a Faraday detector and a dual-mode SEM detector. The laser was a Resonetics M50E

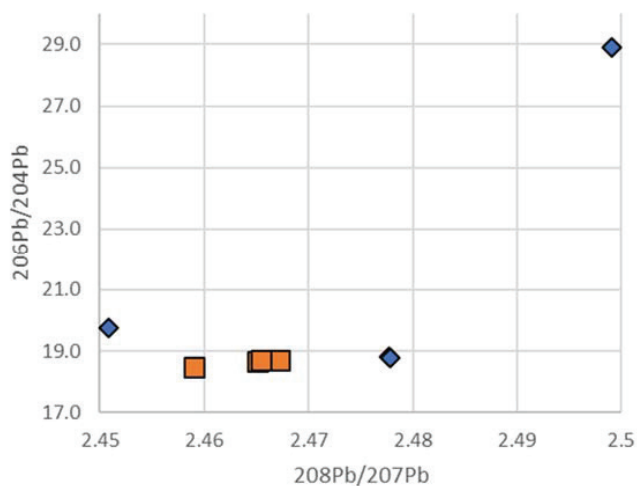


Figure 5. LIA analysis of eight examples of smalts, four from BCW (blue diamonds) and four with German labels (orange squares).

eximer laser operating at a wavelength of 193 nm, 5mJ and a repetition rate of 10 Hz. The analytical protocol followed is the one typically employed for the analysis of glass and followed the published methodology of Gratuze (Gratuze 2016, 137–139).

MC-ICP-MS

Eight separate samples (ca. 0.05 g) of smalt were dissolved for lead isotopic analysis at the geochemical laboratories of KU Leuven (Belgium), after which dissolved samples were taken to a

class-10 clean lab facility at Ghent University (Belgium) for chromatographic lead isolation relying on the use of Pb-SPEC resin (Eichrom Technologies). Full details of the sample preparation and laboratory procedures are provided by Rademakers *et al.* 2017 (Rademakers, De Putter and Degryse 2017, 1251–1270). Single-collector ICP-MS was used to determine the Pb concentration to evaluate target element recovery and allow adequate dilution, such that the Pb concentration (150 µg/kg) in all sample and standard solutions measured matched within ±12%. Lead isotope ratios were determined using a Thermo Scientific Neptune Multi-Collector –Inductively Coupled Plasma–Mass Spectrometer (MC-ICP-MS). The uncertainty values measured fall within the currently accepted range for lead isotope analysis (errors <0.005% for all ratios).

Results

The results of the OES analyses are shown in Table 1. The first column gives the assigned laboratory number (with the number related to the 82 vials, see Appendix), the second is the historic description of the sample that was written on label on the vial. The third column is an interpretation from both the label and the result as to what part the sample plays in the production process. This first four analyses, D-11, D-12, D-13 and D-14 are of concentrated, processed bulk ore samples, or “slig”, so represent the very beginning of the process after the ore has been sorted, crushed and perhaps a preliminary roasting. D-11 is lower in cobalt, arsenic, nickel and iron, and higher in silicon and aluminium and other elements than the other three. D-13 and D-14 have a high concentration of cobalt at around 20–22% Co, with similar amounts of iron and about half that in arsenic, so lower ratios than in the slig. The titanium concentration is very different between the two samples, with D-13 four times that of D-14. The concentration of the Ni is also increasing, perhaps connected with the rising concentration of the cobalt. Interestingly, the copper content is very variable, from only 0.5%–7.3% Cu.

Nine analyses represent finely powdered smalts, the final product of the process. In terms of the elements associated with the ore, these products range in concentrations from 0.8–4.0% Co, 0.6–4.4% Fe and 0.3–4.0% As. Overall, the cobalt, iron and arsenic contents of the smalts are about a tenth of the zaffre. The three smalts that are marked as having the best colour (D-3, D-6 and D7) have the highest cobalt contents and that marked as the best colour example, marked FFFFC, (D-6) has the highest arsenic. Nickel contents range from below detection limits to 3,000ppm Ni, so are quite variable although the overall nickel content is lower than the average zaffre. The use of the wood ash as a glass flux is evident especially in the potassium content of the smalts, with concentrations averaging 10.5% K, but with very little magnesium and calcium (the highest is 8,280ppm Ca, but most of <3,000ppm). Some bismuth is detected in three of the smalts, the others are below detection limits. A variable amount of copper is also present, with contents ranging from 240–4,000ppm Cu. The analysis of four samples of larger fragments of smalt by LA-ICPMS (Table 2) showed a similar pattern. Cobalt contents range from 6591ppm to over 3.7% and three have arsenic compositions of >8% As (significantly higher than the OES

Table 2. LAICPMS results.

	Element
BCW 13	ME glass 1827
BCW 15	R III glass from 1832
BCW 14	R I glass from 1834
BCW 1	SFFE glass from 1839
BCW 13	ME glass 1827
BCW 15	R III glass from 1832
BCW 14	R I glass from 1834
BCW 1	SFFE glass from 1839

	Li	B	Na	Mg	Al	Si	P	Cl	K	Ca	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	As	Rb	Sr	Y	Zr	Nb	Mo	
BCW 13	77	77	3616	545	4170	313382	670	4249	129573	3278	290	10.08	6.48	171	10731	9365	1409	396	248	2.35	80956	149	7.41	28.55	36.98	1.36	111.97	
BCW 15	75	56	1036	648	2642	340480	220	1465	131176	5029	71	3.12	2.76	109	3607	37731	3362	340	180	2.49	23668	351	7.50	2.44	7.09	0.30	9.34	
BCW 14	54	43	716	776	3118	325209	270	1535	111428	3524	317	7.74	13.20	236	14060	6591	3898	385	331	2.07	83912	186	9.30	17.49	38.66	1.35	135.49	
BCW 1	57	102	418	418	3194	283286	739	1310	125582	2745	292	16.34	3.83	166	22549	35460	1751	425	287	2.56	104894	153	5.82	28.58	40.35	1.72	354.14	
	Ag	Cd	In	Sn	Sb	Cs	Ba	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	W	Pb	Bi	Th	U
BCW 13	12.86	1.31	0.29	11.23	32.68	2.44	121.69	5.47	10.44	1.23	5.08	2.40	0.62	3.37	0.81	4.87	0.89	2.13	0.28	1.70	0.21	0.96	0.17	35.13	252	8472	1.73	294
BCW 15	9.54	0.51	0.09	2.61	24.89	1.41	26.41	1.16	2.28	0.25	1.14	0.32	0.07	0.36	0.07	0.37	0.06	0.15	0.02	0.13	0.02	0.17	0.03	2.24	72	6479	0.39	9
BCW 14	123.54	1.78	0.39	22.42	87.46	1.71	419.98	4.14	8.17	1.00	4.28	2.02	0.49	2.62	0.67	4.07	0.74	1.82	0.24	1.51	0.19	1.00	0.10	4.73	397	5543	1.37	202
BCW 1	3.06	0.88	0.34	14.13	51.65	1.32	36.26	7.08	13.70	1.62	7.02	3.38	0.91	3.93	0.89	5.01	0.91	2.16	0.28	1.67	0.20	0.99	0.43	53.21	298	6458	2.02	624

results for other smalts). All have significant bismuth (0.5–0.8% Bi) and three are high in uranium, with over 200ppm U. One of the smalts (BCW 15, R III glass from 1832) is distinctive in terms of rare earth element composition from the other three (see Table 3) and has the highest cobalt and the lowest arsenic contents. Overall, this has a very different colourant and trace element composition, although similar in major elements.

The final product analysed by OES was two samples of the “cobalt oxide” chemical reagent, produced in 1849. Marked “Normal pattern” (D-2) and “New pattern” (D-10), they are significantly different. D-2 has a cobalt content of 20%, with 11.7% arsenic and 1% nickel. Very few other elements are present in significant quantities. D-10 has much higher cobalt (54% Co) and nickel (5.2% Ni), but much lower arsenic, only 920ppm As.

The lead isotope results for the four BCW smalts are very variable and, in some samples, highly radiogenic, especially BCW 19 and BCW 2, with $^{206}\text{Pb}/^{204}\text{Pb} = 19.753$ and 28.901 , $^{207}\text{Pb}/^{204}\text{Pb} = 15.695$ and 16.432 and $^{208}\text{Pb}/^{204}\text{Pb} = 38.467$ and 41.065 . The lead isotope ratios for the presumed Hessen smalt samples from Schwarzenfels and Cöln are much more homogenous. The three samples marked Schwarzenfels (BCW 49, 58 and 81) are practically identical to all the BCW smalts, with $^{206}\text{Pb}/^{204}\text{Pb} = 18.6$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.65$ and $^{208}\text{Pb}/^{204}\text{Pb} = 38.4$ – 38.6 . Slightly different is BCW 60, marked as from Cöln with $^{206}\text{Pb}/^{204}\text{Pb} = 18.480$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.639$ and $^{208}\text{Pb}/^{204}\text{Pb} = 38.455$.

Discussion

A combination of the archive records and the analysis carried out above allows some of the processes being carried out at the BCW to be delineated for the first time. Details from the archive records show that the first stage in the preparation of the cobalt ores was crushing and then sorting to manually pick out the minerals highest in cobalt. After this stage it was crushed again and then heated again. The resulting product was called “slig” (or “schich” in some German texts). In the last period of the BCW production, slig was the only output and was sent to other factories, particularly Germany, for further processing. Four surviving samples (D-11, D-12, D-13 and D-14) are interpreted as slig, although two are marked “Concentrate”, one as “Durchschnittsprobe” (average sample) and one probably “DP”, an abbreviation of this. The names “Haugfoss” and “Skuterud” associated with two of them suggest different stamping mills on site. Three samples are dated to between 1859 and 1862. D-11 has higher amounts of silicon, aluminium, calcium, sodium, magnesium, etc. suggesting that it has a higher content of silicate minerals, and therefore “gangue” than D-12. Perhaps its description as “Number 2” suggests that it is not the best quality. Both samples have high iron, cobalt, copper and arsenic. However, D-12 has nearly 5% Zr, which seems unusual, although zircon is a very common mineral in the Modum deposits (Grorud, 1997, 31–38). All samples are low in manganese and relatively low in nickel. This slig was used as the colouring component for all products at BCW, although it required further processing to be used in finished products. Slig retained a significant amount of sulphur and arsenic which were considered undesirable. Two methods were described in the records to reduce the content of both.

The earlier version seems to be that the slig was strongly heated. This drove off the more volatile arsenic which was deliberately collected in a long “giftfang” tunnel for use in other processes. It also roasted the ore converting the sulphides to oxides. The second method seems to have been used from around 1840 onwards and consisted of using sulphuric acid to convert the sulphides to sulphates which are then dissolved in water and reacted with sodium carbonate to precipitate metal hydroxides. This has an advantage in that the cobalt sulphate tends to react first, so by splitting the batch the cobalt content could be increased. It was therefore especially useful for ore where the cobalt content was too low to use the direct roasting to oxide approach. The archive makes clear that it was this change in technology that allowed the BCW to exploit previously unusable ore and thus extended its life. Interestingly, this sulphate/carbonate reaction is exactly the one proposed for the extraction of the first cobalt pigment in Late Bronze Age Egypt, when cobalt bearing alum (also sulphates) and natron were probably mixed to produce the usable hydroxide (Shortland and Tite, 2006, 153–168).

The main product through most periods was smalt. This required the processed slig to be mixed with quartz and a wood-ash based flux. According to the archive, both of these could be sourced locally, however the wood ash also came from other sources in some (unspecified) periods. Some of these appear to be very distant from the BCW, the USA being mentioned in the records. There is therefore the potential for the wood ash to be variable in composition. There is also the possibility, although not clear from the records, that quartz might have been used from other sources too. The analysis of the smalts by OES and uncrushed smalt by LA-ICPMS showed that the composition was quite variable. All are high in silicon from the quartz (typically around 30% Si or 64% SiO₂) and potassium from the wood ash (11% K or 13.2% K₂O). They are low in sodium, magnesium, aluminium, calcium and titanium, all averaging <5,000ppm. The cobalt content of the smalts averages 2% Co overall, although there is a wide range, with one only 0.6% and one at 4%. For elements that are classically associated with cobalt such as manganese and zinc, they are very low at only 100–200 ppm, suggesting that this cobalt source does not have significant manganese or zinc. Other elements are higher, especially iron at 1–3% (but occasionally higher) and it is clear that not all the arsenic is removed by the processing, many having 0.5–2% As, but others are much higher, up to 10% As. While sometimes below the detection limit by OES, it is clear that bismuth is probably present in the smalts, sometimes up to over 8,000ppm Bi. Three of the uncrushed smalts analysed by LA-ICPMS also have hundreds of ppm uranium, again almost certainly introduced from the slig and therefore the ore. Modum deposits have been shown to contain uraninite and Bi-rich minerals (Grorud, 1997, 31–38). Nickel and copper are also present, as they were in the slig. The ratios of cobalt to both these elements are very variable, especially the copper, but average 15 (Co:Ni) and 45 (Co:Cu). There is no clear relationship between the designated quality of the smalt or its date (where known) and the composition.

REE patterns and LIA also show that there is some significant variation in the smalts, almost certainly due to variation in raw materials. The REE pattern derived from the LA-ICPMS data (Table 3) shows three of the samples (BCW 1, 2 and 14) are

Table 3. Lead isotope analysis results.

BCW 2	FEB glass from 1838
BCW 19	SFFFE
BCW 18	FFFFC
BCW 42	Modum Blaafarveværk MC
BCW 60	MES original pattern v. Cöln 25th April 55
BCW 81	SFFE Schwarzenfels
BCW 49	sFFF E Schwarzenfels [?]
BCW 58	FFFE Schwarzenfels [?]

		206Pb/ 204Pb	207Pb/ 204Pb	208Pb/ 204Pb	207Pb/ 206Pb	208Pb/ 206Pb	s 206Pb/ 204Pb	s 207Pb/ 204Pb	s 208Pb/ 204Pb	s 207Pb/ 206Pb	s 208Pb/ 206Pb
Norwegian smalt samples	BCW 2	28.901	16.432	41.065	0.56855	1.42086	0.003	0.002	0.005	0.00002	0.00005
	BCW 19	19.753	15.695	38.467	0.79456	1.94735	0.004	0.003	0.007	0.00006	0.00014
	BCW 18	18.821	15.682	38.855	0.83321	2.06448	0.004	0.004	0.010	0.00008	0.00023
	BCW 42	18.771	15.672	38.832	0.83491	2.06880	0.004	0.004	0.011	0.00005	0.00020
German smalt samples	BCW 60	18.480	15.639	38.455	0.84625	2.08092	0.002	0.001	0.003	0.00003	0.00006
	BCW 81	18.694	15.655	38.624	0.83743	2.06609	0.002	0.002	0.005	0.00003	0.00009
	BCW 49	18.653	15.651	38.580	0.83907	2.06835	0.003	0.002	0.007	0.00005	0.00012
	BCW 58	18.684	15.655	38.597	0.83790	2.06581	0.002	0.001	0.004	0.00003	0.00008

very similar with a LREE enrichment and relatively high samarium, but the fourth (BCW 15) mirrors chondrite pattern compositions very closely with no enrichment. It is unclear from the label why BCW 15 should be so different, the best solution being that it represents a different quartz raw material source from the other three, which perhaps have a local pattern. More work on the quartz source at the BCW is needed to determine if this is the case. LIA also show considerable variation. Some of the BCW samples have quite extreme values which might be expected from material made with a pre-Cambrian ore source such as the Modum Complex cobalt deposit, but the huge variation points to a variety of mineral mixtures being used. The LIA results of the BCW smalts were compared to three samples marked Schwarzenfels (BCW 49, 58 and 81) and one Cöln (BCW 60). The Schwarzenfels samples are all practically identical, while the Cöln sample is similar but slightly less radiogenic. They do not show the variation seen in the BCW samples.

The final product analysed is the cobalt oxide (BCW 78, “normal pattern” and BCW 79, “new pattern”). According to the archive this required careful preparation to make sure that the cobalt was as pure as possible. This seems to have been largely successful, as the contents of almost all elements analysed by OES (apart from cobalt) is each less than 1%, often much less. There are two exceptions. The first is nickel which is 1% Ni in BCW 78 and 5% in BCW 79, very approximately correlating with the cobalt content. The second is the arsenic content, which is very different in the two, at 11%, BCW is much higher. If this represents a change in the production, then the “new pattern” succeeds in eliminating almost all the arsenic from the product,

but continues to concentrate the nickel at a roughly similar Co:Ni ratio. Interestingly, although the cobalt and nickel contents are relatively high compared to the smalts, the bismuth content is still below detection. There also is significant zinc in the cobalt oxide samples, the only category here to have any real zinc (the LA-ICPMS suggesting that the zinc content of the smalts is only 150–300ppm Zn).

The lack of zinc in the slig suggests that this may be coming in from another component or raw material. However, since the records of how the cobalt oxide was made have not yet been found in the archive, then what this might be is uncertain.

Conclusions

The production at the BCW at Modum lasted for just over a hundred years, eventually ending 1890s. It was a major producer of smalts for some of this period and the volume of the production can be partly traced in the archive. At least two production methods are described in the archive for the manufacture of smalt, the first involving roasting of the sulphide ores to oxides and the second a conversion to sulphate and then hydroxide using sulphuric acid and sodium carbonate. Another methodology was used to create cobalt oxides, especially later in the production period. Analysis of preserved smalts now kept in the BCW Museum showed that the output was a smalt with 65% silica and 13% potash. The Norwegian cobalt ores produce smalt that is very low in manganese and zinc, with some nickel, and significant iron and arsenic. The smalts also seem to have relatively raised bismuth and uranium contents. These elements act as a potential signature to provenance Norwegian smalts potentially found or used elsewhere. However, it should be noted that the Saxon ores were exploited for bismuth and later uranium, so it is likely that smalt made from those deposits, will also contain significant amounts of these elements (Veselovsky, Ondrus and Kominek, 1997, 127–132). Trace elemental (especially REE) and LIA analyses showed that the BCW production is more complex than a simple use of local materials. Backed up by references in the archive, it seems probably that some plant-ash and /or quartz is being imported to the facility to use with or instead of local equivalents. This makes the final compositions of the smalts quite variable, perhaps in contrast to at least the lead isotope compositions of the German ores (although more analyses are certainly required). However, there is enough consistency to potentially enable the BCW smalts to be traced when they are exported from the factory and used in other industries in Europe and further afield. This will be the aim of further work in this area.

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Supplementary Material, Annex A

<https://doi.org/10.17862/cranfield.rd.21587499.v1>

82 samples in small glass vials, retained by the Blue Colour Works Museum (Stiftelsen Modums Blaafarveværk – Bygdemuseet Modum), but originally in the collection of the Norwegian University of Science and Technology. The label on each vial is recorded.

[...] indicates a note or text missing or untranslatable.

1. SFFE glass from 1839
2. FEB glass from 1838
3. ME glass from 1827
4. MEBS glass from 1851
5. OEst glass from 1848
6. RC III
7. FFC
8. FC
9. OES from ? Blaafarveværket 1852
10. MEBSG from ? Blaafarveværket 20th of May 1852 576 [...]
11. PKO new normal pattern from 15th of January 1849
12. Not labelled
13. ROO glass from 1854
14. R I glass from 1834
15. R III glass from 1832
16. FFF glass from 1851
17. MEBS glass from 1834
18. FFFFC
19. SFFFE
20. REOSg
21. FEB
22. OESg 1853

23. EE
24. OES
25. Modum Blaafarveværk RO + glass from 1855
26. FFF glass
27. FFFE testglass
28. Modum Blaafarveværk glass MEB 1842
29. 5FC 2
30. FFFFC
31. FFFC
32. FFFC
33. FEC
34. SFC 1
35. FC
36. FCB
37. Nr 141
38. Nr 121
39. Nr 111
40. FCB
41. MC
42. Modum Blaafarveværk MC
43. MCB
44. MCB
45. OC
46. EFFF
47. FFFFE 5 [“5” might be “S”]
48. S FFF E
49. s FFF E Schwanzenfels [best transliteration possible]
50. Nordic FFFFE
51. RE III S g
52. Nr 131
53. MSB [...] 30/₃₁₈₅₄
54. Writers sand from 1811
55. FFE
56. FE

- 22 Bjørnland, Degryse, Schibille, Eremin, Walton, Gratuze, Braekmans and Shortland
57. R E 3 S g
 58. FFFE Schwarzenfels [best transliteration possible]
 59. RE III
 60. MES original pattern v. Köln 25th April 55
 61. OE
 62. MEB
 63. ME
 64. MEB
 65. ME
 66. FEB
 67. PO normal pattern
 68. 600 pund FKO 19th November 56 70. 0 Co 4,7 ni
 69. [Just labelled with a question mark]
 70. ME2
 71. FFESg1
 72. FESg2
 73. OEg2
 74. OE II
 75. OE 3
 76. HPB
 77. MEBSt
 78. AKOSt normal pattern from 15th January 1849
 79. FKO normal pattern 15th [possibly May or June] 1849
 80. RKO new normal pattern 15/1 1849
 81. SFFE Schwarzenfels
 82. November 1782 “authentic” [probably] MC

There are also two samples of nickel labelled “Geröstet Ni-speise” and “Nickle powder – Nickle sample v. Klefva”.

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