

Analysis of the Microstructure and Elemental Composition of Uranium-Containing Microcrystals Observed in the Characteristic Bronze-Green Matte Glaze of Rhodian-Type Gouda Pottery from the Early 20th Century

ABSTRACT

Bright red and orange colors are those that are typically associated with uranium glazes. Dutch Rhodian-type Gouda pottery from the Art Nouveau era was found to feature several uranium glazes that instead are green and yellow in color. In this study, a sample of the characteristic Rhodian-type bronze-green matte glaze was analyzed by optical microscopy and scanning electron microscopy-energy dispersive X-ray analysis, which showed the glaze to have a complex microstructure and composition. Willemite (Zn_2SiO_4) microcrystals were observed throughout the matte glaze, confirming it to be of the zinc-type.

Microcrystals of the calcium uranate $Ca_2U_3O_8$ and two non-stoichiometric sodium-calcium-uranium oxides were detected, the morphology and composition of which were found to correlate with green and yellow surface areas of the glaze. Care needs to be taken while handling and restoring Rhodian-type Gouda pottery, as the uranium glazes it features are significantly radioactive.

KEYWORDS

Uranium glaze · Uranium oxides · Microcrystals · Zinc-type matte glaze · SEM-EDS · Rhodian ware · Gouda pottery · Art Nouveau

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INTRODUCTION

Uranium oxides were used extensively as coloring agents in ceramic glazes during the first half of the 20th century (Lehnhäuser 1966; Parmelee 1951). Bright orange and red colors were, and still are, considered to be typical uranium-based colors, e.g. Fiesta ware, of which popularity peaked in the early 1930s (Baaij 2010). The city of Gouda was one of the main production centers of decorative art pottery in The Netherlands at the time (Tasman 2007), and

uranium-based red and orange glazes were used in significant quantities by the industry.

Surprisingly, micro X-ray fluorescence (μ -XRF) analysis confirmed the presence of uranium in the bronze-green colored elements of the decorative patterns on an earlier type of Gouda pottery, termed Rhodian ware (Figure 1). Rhodian ware was developed and marketed in 1910 by the Plateelbakkerij Zuid-Holland (PZH) pottery at



Figure 1. Vase with the type of bronze-green glaze analyzed in this study. PZH exhibition vase with Rhodian-pattern, L.J. Muller, ca. 1911 CE, H 105 cm × Diam. 44 cm. Private collection · Courtesy of J. van Bragt and H. J. Wolffensperger

Gouda (Baas et al. 2016). A novelty at the time because of the use of matte instead of high-gloss glazes, the uranium-containing, bronze-green matte glaze in particular is characteristic of Rhodian-type wares from the 1910s, as the glaze surface developed unique greenish-metallic and yellow areas, called frosting, during firing. Investigation using an optical microscope indicated that these types of frosting are the result of microcrystallization phenomena, which discovery prompted further investigation into their microstructure, distribution, and elemental composition.



Figure 2a. Rhodian-type jug with Srebo-pattern, PZH Gouda, ca. 1912 CE, H 15.5 cm × Diam. 13.2 cm. Private collection Bert-Jan Baas · Courtesy of J. van Bragt and H. J. Wolffensperger

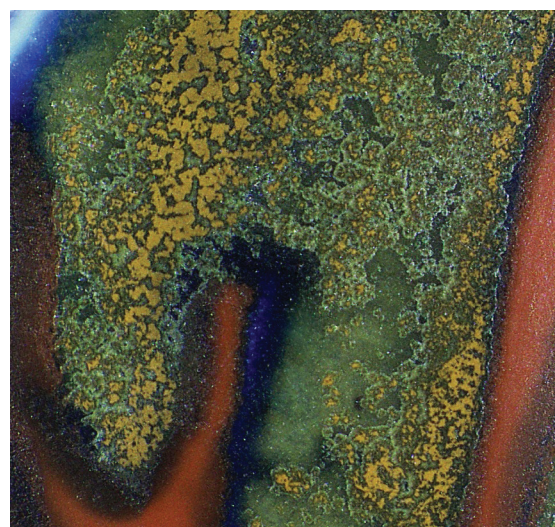


Figure 2b. Photomicrograph of a shard of Rhodian-type jug with Srebo-pattern · Courtesy of J. van Bragt and H. J. Wolffensperger

Here we describe the historical background of PZH and its Rhodian ware, together with the elemental composition analysis and microstructure of its unique bronze-green matte glaze using scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDS), based on samples obtained from shards of an early object dating from ca. 1912

(Figure 2). With this paper, we aim to generate awareness of the use of uranium oxide as a coloring agent in green colored ceramic glazes in the early 20th century. This is of particular relevance to pottery restorers, as the radioactive microcrystals at the glaze surface might be damaged during treatment, thereby generating radioactive dust that could be inhaled, ingested, or released into the environment.

RHODIAN-TYPE GOUDA POTTERY: HISTORICAL BACKGROUND AND PRODUCTION TECHNIQUE

Around the year 1900, several new art potteries were established in The Netherlands in the wake of the success of the Rozenburg Art Pottery in The Hague, which had by then acquired national and international fame with its distinctively modern, Art Nouveau-style wares (Brentjens 2018). PZH was incorporated on the 2nd of April, 1898, at the city of Gouda, located in the province of Zuid-Holland, about 30 km east of The Hague. Its founding director and co-owner, Egbert Estié (1865-1910), expressed the company's goal as producing "a new kind of art pottery in the style of the famous Delftware and Rozenburg factories" and, by marketing its products at a lower price level, PZH quickly carved out a share of the art pottery market. PZH's earliest products greatly resemble those of Rozenburg: floral patterns in an Art Nouveau style as an underglaze decoration covered with a high-gloss glaze. When, in 1900 at the Paris World's Fair, Rozenburg shocked the art world with its so-called "eggshell porcelain," PZH responded with a pottery version in imitation thereof. Although initially a financial success, PZH's business model resulted in the company being effectively banned from participation at the major exhibitions and World's Fairs from 1900 onwards, in which the director of the Rozenburg factory, the well-connected and influential J. Jurriaan Kok (1861-1919), played a major part. (Baas et al. 2018)

Combined with the Art Nouveau style losing popularity after 1904 and under the growing influence of the Hoyng family as PZH shareholders and owners of high-end retail stores of luxury goods, a new business model was adopted in which a fully new and distinctive product was to be the key to future success. Hence, in 1909, the artist-technician Daniël Harkink (1862-1953) took the

lead designing a new range of shapes, decorative patterns, and a decorating technique. The ceramic arts of historical cultures became his main source of inspiration. His shapes were inspired by Greek-Cypriot vessels from the 17th-3rd centuries BCE in the collection of Luigi Palma di Cesnola (1832-1904) at The Metropolitan Museum of Art in New York, as well as Dutch archeological pottery finds and 17th-18th century Dutch Delftware. Harkink's decorative patterns were inspired by "Persian" ceramic art of the 16th-17th centuries, mainly of the kind which was then described as "Rhodian ware," now Iznik pottery, from the collection of Charles D. Fortnum (1820-1899) at the Victoria and Albert Museum in London (Baas et al. 2018). Harkink reintroduced wheel-throwing, setting his new line apart from the slip-cast Rozenburg-style wares. He also abandoned the typical high-gloss glaze, and instead introduced the use of matte glazes at PZH, a choice which had important technical consequences.

Because of the inherent semi-opacity of matte glazes, subduing underglaze colors, Harkink developed color stains and colored matte glazes which were to be painted on top of a ground coat of unfired, white matte glaze. Decorating on a powdery surface was, however, deemed a health risk by the Dutch Labour Inspection (Hoyng 1994). The solution that was developed was to first fire the wares with the applied matte glaze ground to the relatively low heat level of Seger cone 018, approximately 710 °C. This heat treatment was insufficient to completely melt the glaze, but allowed it to become firm and sintered while retaining a degree of porosity required for the effective application of water-suspended decorative stains and matte glazes. In fact, the problem of color unevenness on the finished product due to the decorator's brushwork, which was deemed unattractive at the time, was solved to a large extent by this process as well. The sintered ground glaze reduced the absorbency of the underlying porous ceramic body just enough that a color stain or glaze applied with a brush could flow slightly, and even out, before setting. After this decorating process, a second firing to Seger cones 03a-04a, approximately 1070-1100 °C, melted the sintered ground glaze and applied decorative glazes and stains into a continuous, vitrified glaze coating with a matte finish. (Hageman archive; Kersbergen notebooks)

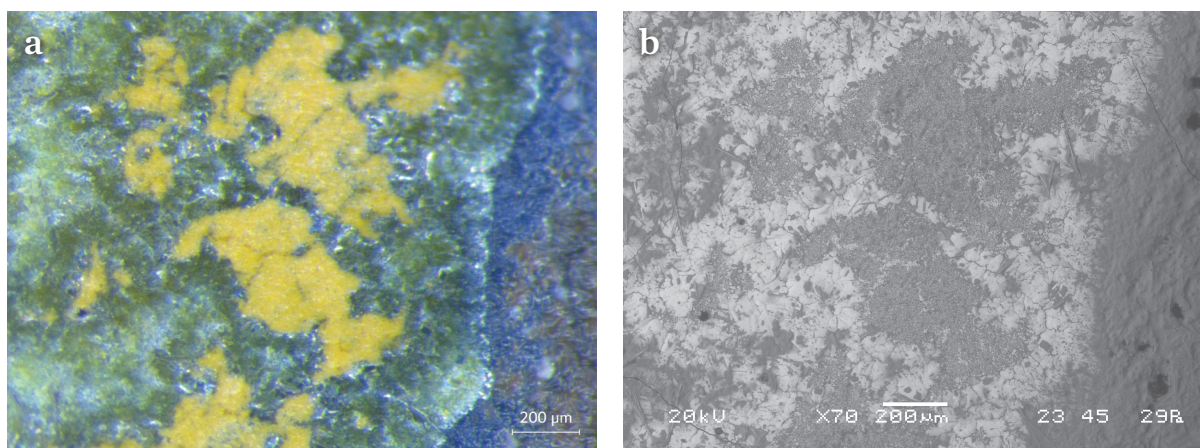


Figure 3. a) Optical micrograph at 66× magnification; b) SEM-BEI at 70× magnification of the same surface area of the glaze sample

This technique, for which the term “Rhodian” became synonymous on the factory floor, was the major technical innovation developed under Harkink’s direction. It led to a product that had the required, radically different look that set it apart from the Art Nouveau Rozenburg-style wares made prior to 1910. In the following years, Harkink perfected it, and together with other artists working at PZH, Leen J. Muller (1879-1969) chief among them (Baas et al. 2016), a plethora of new patterns were designed and marketed with great success. And although the patterns from the early 1910s went out of fashion by the late 1920s, the technique itself remained the basis for the company’s success until its closing in 1964.

EXPERIMENTAL

Samples of bronze-green matte glaze typical of Rhodian-type Gouda pottery made by PZH were obtained from shards of a jug decorated with a pattern titled “Srebo.” This object was dated to ca. 1912 based on the specific factory marks painted on the bottom of the jug, and referencing contemporary source material, including analysis by Barbara Walraven (1959-2018), José van Bragt, Henk Wolffensperger, and Bert-Jan Baas.

SEM-EDS was chosen to further investigate the greenish-metallic and yellow areas of the bronze-green matte glaze on the “Srebo” sample. The glaze surface was coated with a thin gold layer using a JEOL gold sputter coater. The sample was mounted and grounded to a sample holder with copper tape and observed in a JEOL 5910LV SEM, operating at high vacuum in Backscattered

Electron mode with an accelerating voltage of 10 kV. Semi-quantitative analysis of the crystals at the surface was performed by point analysis with a Thermo Scientific Noran System Six EDS detector. Elemental concentrations were determined applying the PROZA method and normalized to a total without carbon, which apparently was present as dirt or soot on the surface of the glaze. A cross section of the glaze was analyzed after wet-grinding one side of a shard with silicon carbide paper, 320 mesh, and polishing with alumina paste, 1 μm particle size.

RESULTS

Glaze microstructure

Figure 3a shows an optical micrograph of the surface of the glaze sample (Figure 2). Microcrystalline features are clearly visible as reflective crystals in the green areas covering the surface and as colorless needles. The microstructure of the yellow material is not discernible in visible light under low magnification. A scanning electron microscope backscattered electron image (SEM-BEI) of the same area of the glaze surface shows a distinct correlation between optical color and glaze microstructure (Figure 3b).

The crystalline nature of the observed microstructures becomes apparent at greater magnification (Figure 4). The relatively large features observed in the green areas (Figures 4a and b) have a leaf-like morphology, and are covering the glass phase of the underlying glaze layer. Additionally, they appear to contain two

microcrystalline phases that are compositionally different, but have grown together to form a composite two-phase crystalline material (Figure 4b). The yellow areas instead are characterized by the presence of smaller, irregular hexagonal single crystals of 1-5 μm in diameter, which either appear to be at the glaze surface or partially immersed in the glass phase (Figure 4c). Due to their abundance and dense distribution, these yellow areas are best described as a glaze-microcrystal composite material.

Microcrystals in both morphologically different areas are enriched in a heavy element. The ubiquitous and relatively large needle-shaped microcrystals are composed of lighter elements, and correspond to the colorless needle-shaped microcrystals that were observed with optical microscopy. Figure 5 shows an elemental distribution map of a cross section of the glaze sample. The larger leaf-like features associated with the green areas have formed throughout the glaze layer as linear features, with lengths up to 200 μm , thicknesses of 0.5-1.5 μm , and random orientations and distribution (Figure 4a). The inner surface of gas bubbles trapped in the glaze is often covered with the same leaf-like microcrystals as were observed at the glaze surface. The glaze-microcrystal composite material of the yellow areas is typically associated with the glaze surface, penetrating up to 50 μm into the glaze layer (Figure 4c). The larger needle-shaped crystals present in both the green and yellow areas are also randomly distributed.

These distinct microcrystalline features clearly correlate with specific elemental distributions (Figure 5). The large needle-shaped microcrystals containing lighter elements were found to be enriched in zinc (Zn), silicon (Si), and oxygen (O), indicative of a zinc silicate (Figure 5c). X-ray diffraction analysis of the glaze sample confirmed the presence of the mineral willemite (Zn_2SiO_4 , data not shown). Intriguingly, the distinct types of microcrystals specifically associated with the green and yellow areas were each found to be enriched in the element uranium.

Compositional characterization of the uranium-containing microcrystals by SEM-EDS

A total of 119 SEM-EDS spot measurements were performed: 76 on microcrystals in the yellow

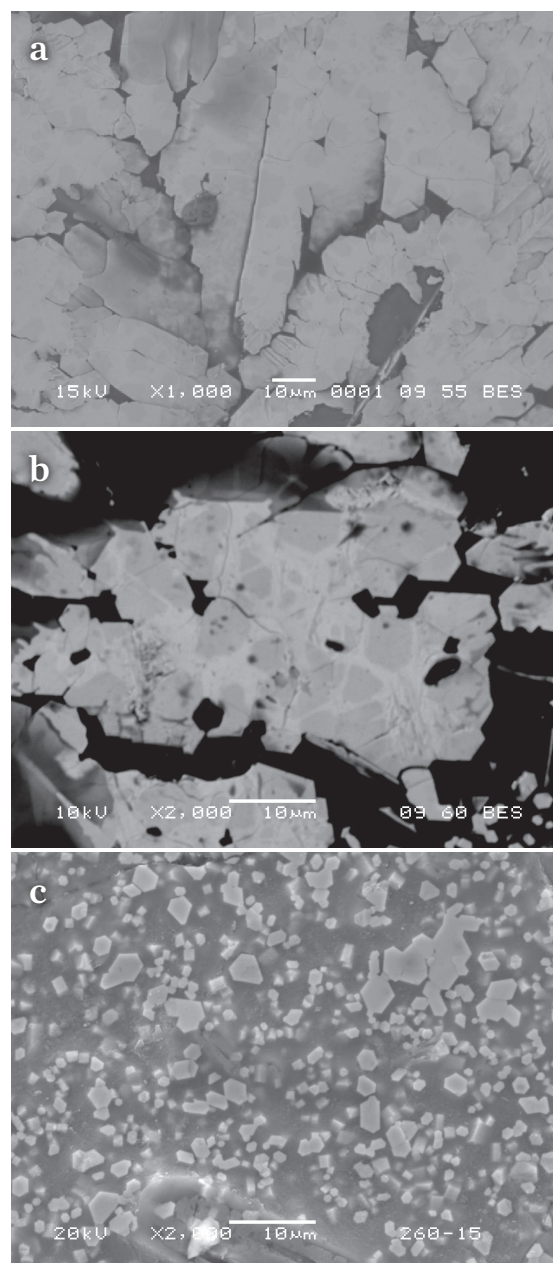


Figure 4. a) SEM-BE-TOPO at 1000 \times and b) SEM-BE-TOPO at 2000 \times magnification showing a two-phase composite microstructure in the leaf-like microcrystals on the surface of the green glaze; c) SEM-BE-TOPO of the composite microcrystal-glaze material typical of the yellow glaze surface

areas and 43 on the two observed phases of the composite microcrystals in the green areas. The combined scatter plot of the observed elements, sodium (Na), calcium (Ca), and uranium (U), against oxygen (Figure 6a), clearly reveals three distinct compositional groups (Figures 6b-d). The uranium content ranges from 22.0-25.5 atom percent (at. %). The average oxygen content of

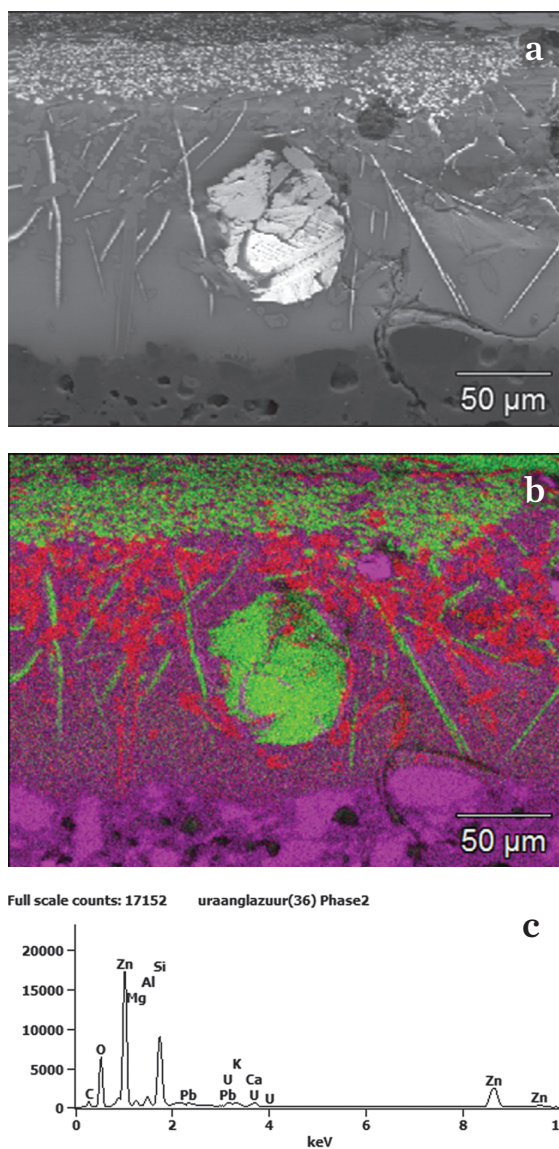


Figure 5. a) SEM-BEI and b) elemental distribution map of a cross section of the glaze sample (magenta: Si, red: Zn, green: U); c) average spectrum of the zinc-rich particles in b, a zinc silicate phase. Elements magnesium, aluminum, lead, uranium, potassium, and calcium are probably from the surrounding phases, detected because, in many cases, the particles are thinner than the depth of information of EDX.

groups I and II is approximately 2 atom percent higher as compared to group III. However, prominent differences are observed between the sodium and calcium contents of groups II and III.

The average elemental composition for each group is reported in Table 1. The microcrystals belonging to group I were identified as a calcium-uranium oxide, of which the average elemental composition is in close agreement with the stoichiometric composition $\text{Ca}_2\text{U}_3\text{O}_8$. The microcrystals

belonging to groups II and III were both found to be quaternary sodium-calcium uranium oxides. However, neither fits a concise stoichiometric composition, and are probably non-stoichiometric phases. For all three groups, uranium was calculated to be present in the oxidation state of +4 (U^{IV}).

Interestingly, a trend in distribution of microcrystals belonging to groups I-III is observed between the green and yellow areas (Table 1). The microcrystals in the green areas are enriched in groups II and III, which together form the observed composite crystals (Figure 4b). The yellow areas instead show an enrichment of microcrystals belonging to groups I and II, with a minor subset of the small single crystals in this glaze-microcrystal composite material belonging to group III.

DISCUSSION

The application of matte glazes was an international phenomenon during the art pottery revival era in the West around the year 1900 (Montgomery 1993). Due to the popularity of matte glaze wares, the international scientific ceramics community at the time turned their attention to matte glazes as well (Binns 1903). Micro- or “cryptocrystalline” matte glazes were well known at the time, including zinc silicate microcrystalline matte glazes (Riddle 1906). The detection of willemitte (Zn_2SiO_4) microcrystals in the Rhodian-type matte glaze indicates that Harkink had specific knowledge of zinc-type matte glazes. This is confirmed by former PZH employees interviewed in the 1970s-1980s, who stated that Harkink developed his matte glaze by adding zinc oxide as a matting agent to a high-gloss glaze that was commercially obtained from the ceramic glaze materials wholesaler Harrison & Son of Hanley, Stoke-on-Trent, UK (Hageman archive). The recipe of the basic Rhodian matte glaze that survives in the notebooks of PZH laboratory assistant Jan van Kersbergen confirms this method (Table 2) (Kersbergen notebooks).

Van Kersbergen’s recipe notebooks do not contain the recipe for the bronze-green glaze. However, this particular glaze is one of at least four similar uranium-glazes, including the ochre-yellow referred to as “color 961” (Table 2). The bronze-green glaze was likely achieved using an

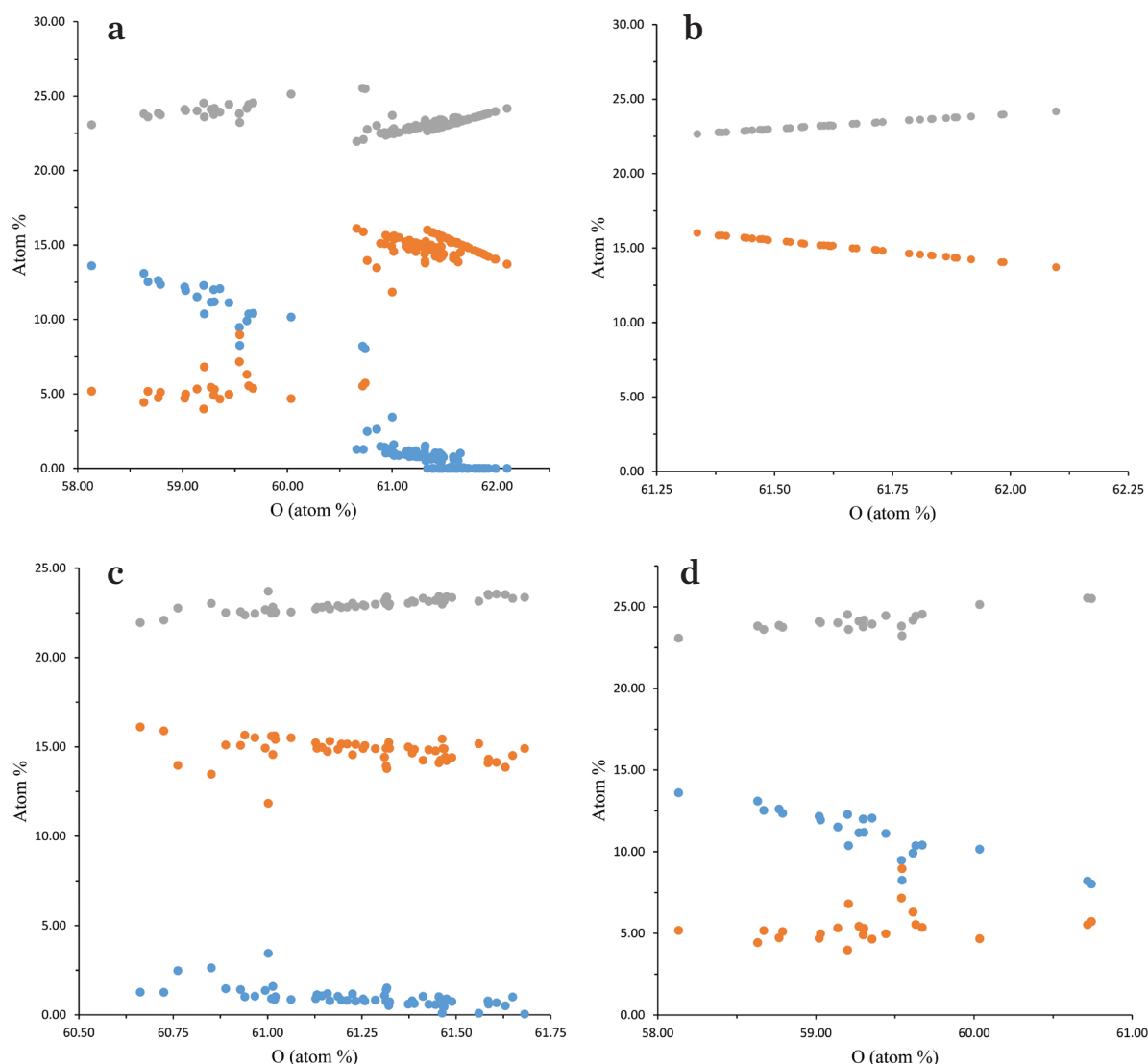


Figure 6. a) Combined scatter plot of the atom percent of sodium, calcium, and uranium versus oxygen, showing three distinct compositional groups; b-d) scatter plots of compositional groups I, II, and III (blue: Na, orange: Ca, grey: U)

oxide stain of a similar recipe, and the recipe of “color 961” gives a good impression of the types of materials that were used. These color stains interacted with the underlying pre-sintered, zinc-type Rhodian matte glaze, and melted into a coherent glaze layer during the final firing process. The observed elemental distribution in the glaze cross section is consistent with this method (Figure 5). The fusion of a uranium oxide-rich surface deposit with the underlying zinc-containing matte glaze, followed by diffusion of uranium ions into the glaze layer, could well explain the observed distribution pattern of uranium in the glaze cross section. The formation of microcrystals of the three compositional groups

that are observed, as well as their distribution at the surface and throughout the glaze layer, is likely to be a function of local variations in concentration of the elements that make up these microcrystals, caused by the decorator’s brushwork (Table 1).

The three compositional groups I-III reveal a complex chemistry. Indeed, the breadth of especially non-stoichiometric phases of ternary and polynary uranium oxides is surpassed by only a few other elements (Keller 1975). The elemental composition of microcrystal group I closely fits the stoichiometric composition $\text{Ca}_2\text{U}_3\text{O}_8$. Given the observed stoichiometric composition, the characterization of the group I compound as a calcium uranate seems justified.

GROUP	O	Na	Ca	U	RATIO O/U	FORMULA	GREEN	YELLOW
I (41) ^a	61.64 ± 0.19	–	15.08 ± 0.57	23.28 ± 0.38	2.65 ± 0.04	Ca ₂ U ₃ O ₈ or Ca _{0.65} UO _{2.65} or (CaO) _{0.65} UO ₂	9	32
II (55)	61.25 ± 0.25	0.99 ± 0.57	14.77 ± 0.69	22.99 ± 0.38	2.66 ± 0.04	Na _{0.13} Ca _{1.93} U ₃ O ₈ or Na _{0.04} Ca _{0.64} UO _{2.66} or (Na ₂ O) _{0.02} (CaO) _{0.64} UO ₂	18	37
III (23)	59.34 ± 0.60	11.08 ± 1.56	5.44 ± 1.06	24.14 ± 0.62	2.46 ± 0.05	Na _{4.13} Ca _{2.03} U ₉ O _{22.12} or Na _{0.46} Ca _{0.23} UO _{2.46} or (Na ₂ O) _{0.23} (CaO) _{0.23} UO ₂	16	7

^a The number in parentheses reports the number of measurements

Table 1. Results of elemental composition analysis by SEM-EDS characterizing groups I, II, and III. Averaged at. % are reported for each element detected. The columns Green and Yellow report the observed frequency of each compositional group in the green and yellow areas of the glaze sample.

GLAZE/STAIN	AMOUNT	MATERIAL
Rhodian glazuur N° 1	75 kg	Enamel glaze A600, Harrison & Son, Hanley, UK
	25 kg	Zinc oxide
	7 kg	Sanda
Kleur 961	200 g	Urangelb 1 ^a , Edlich & Weisse, Meissen, Germany ^b
	10 g	Copper oxide
	10 g	African Blue 183GS, Wengers Ltd., Hanley, UK
	30 g	White Engobe N° 114, Wengers Ltd., Hanley, UK

^a Sand was likely ball-milled to a fine powder prior to its use as a glaze ingredient.

^b According to H.A. Verlee, the successor of Harkink, this material was in fact “yellow uranium oxide,” or sodium diuranate Na₂U₂O₇.

Table 2. Recipes of “Rhodian glaze N° 1” and “color 961” from the notebooks of PZH laboratory assistant J. van Kersbergen

Both microcrystal groups II and III, however, were found to be non-stoichiometric phases. Group II has a small sodium content that approaches the detection limit of the EDS instrument, as is evident from the large standard deviation (Table 1). The calcium-content, however, is proportionally smaller relative to Ca₂U₃O₈. The uranium-oxygen ratio is identical, within experimental error, to that of group I. Hence, group II also likely

comprises a U₃O₈-uranate, where sodium oxide (Na₂O) exists as a substitution solid solution in Ca₂U₃O₈.

Although microcrystal group III was found to be a quaternary uranium oxide composed of the same elements as group II, the relative composition is very different. The lower O/U-ratio of group III is inconsistent with a U₃O₈-uranate. A near-

stoichiometric overall formula can be calculated, which is in close, but not exact, agreement with sodium calcium uranate $\text{Na}_4\text{Ca}_2\text{U}_9\text{O}_{22}$. However, when the composition is normalized to uranium, it can also be expressed as a solid solution of Na_2O and calcium oxide (CaO) in uranium dioxide (UO_2), where Na_2O and CaO are present in a 1:1 ratio.

Solid solutions of Na_2O and CaO in UO_2 with a wide range of compositions have been extensively researched (Keller 1975; Sali, Sampath, and Venugopal 1996; Kim and Kim 2014). However, a full characterization of the atomic structure will be required to determine whether the microcrystals of groups I-III are either ternary or quaternary uranates, solid solutions of Na_2O and CaO in UO_2 , or a combination of both. Unfortunately, to the authors' knowledge, no literature description of any of the observed compounds exists. This gap in the literature is not surprising, given that modern research into uranium oxides is dominated by the field of nuclear reactor science, where the extreme conditions, such as temperatures exceeding 1800 °C, are very different from those in the peat-fired pottery kilns operated at PZH.

At approximately 23 atom percent, a significant amount of uranium is present in the microcrystalline material of the bronze-green matte glaze. As PZH's Rhodian-type pottery predates the invention of nuclear fission, non-depleted uranium is present, which raises questions about radiation levels and safety. A survey performed in 2007 shows that radiation levels produced by uranium-containing glazes, to which β -radiation is the most significant contributor, can be of legitimate concern (Luthjens and Van Doorn 2008). The survey mostly focused on orange and red uranium glazes from the 1930s, and contact radiation levels, as well as the radiation dose at a 30 cm distance for exposure of the naked eye, were found to often exceed acceptable levels. These levels are, however, calculated to a 24 hour per day exposure, which in practice never occurs. As the bronze-green glaze never covers an object in its entirety, the total radiation dose is less compared to the Art Deco era pottery. The survey by Luthjens and Van Doorn showed that the plexiglass of display cabinets effectively reduces the emitted radiation to background levels, thereby shielding the observer: curator, collector, museum visitor,

etc. The greatest concern is the potential release of radioactive microcrystals or glaze dust while handling the items or working on them during restoration. Radioactive dust entering the body through inhalation or ingestion poses a serious health risk. Environmental issues too are a concern, as dust is readily released into the environment. (Luthjens and Van Doorn 2008)

Various types of Gouda pottery are ubiquitous in collections of Dutch art pottery that focus on the first half of the 20th century. At least a dozen museums in The Netherlands own significant collections of Gouda pottery, whereas the community of private collectors exceeds 400 individuals in The Netherlands alone. Internationally, Gouda pottery is present in museum and private collections as well. Being actively exhibited, traded, and collected, Gouda pottery is often handled by museum curators, antiques dealers, collectors, and restorers. With this paper, we hope to contribute to the awareness of the potential health and safety risks associated with uranium glazes, in particular those found on Rhodian-type Gouda pottery that, due to their green colors, do not fit the paradigm of their kind.

CONCLUSION

Recent archival research and analysis of surviving Rhodian-type Gouda pottery yielded new insights into the history of this type of Dutch art pottery from the Art Nouveau era. The microstructure of the bronze-green glaze typical of Rhodian-type ware was analyzed by optical microscopy and SEM-EDS. The presence of needle-shaped willemite (Zn_2SiO_4) microcrystals confirmed the basic matte glaze to be of the zinc-type. Elemental composition analysis by SEM-EDS showed that three compositionally distinct groups of uranium-containing microcrystals are present, and morphological differences based on their shape, size, and distribution were found to correlate with the green- and yellow-colored surface areas of the glaze. The elemental composition analysis allowed these microcrystals to be tentatively characterized as the calcium uranate $\text{Ca}_2\text{U}_3\text{O}_8$ and two non-stoichiometric phases of sodium-calcium-uranium oxides. Care needs to be taken while handling and restoring Rhodian-type Gouda pottery, as its characteristic bronze-green glaze with surface microcrystals contains non-depleted uranium, a significant radiation source.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support of Gouda pottery historian Barbara Walraven (1959-2018), without whose relentless archival research much of the history of PZH would still be obscure, and José van Bragt and Henk Wolffensperger for sharing their invaluable knowledge and research based on their signature collection of Rhodian-type Gouda pottery. The authors would also like to thank Kate van Lookeren Campagne for her diligent advice on the manuscript content.

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