Note from the Making and Knowing Project:

This essay was written for “The Art Technological Source,” an 8-week graduate course at the University of Amsterdam offered by Dr. Marjolijn Bol in 2015 and 2016 to students enrolled in an MA program in either Technical Art History or Conservation and Restoration. Students worked on various recipes from BnF Ms. Fr. 640 on the following five topics: 1) tracing and painting on glass, 2) yellow dyes and their applications, 3) dragon’s blood, 4) painters’ varnishes, and 5) sieves.

This is an un-edited student paper that relies on an earlier state of the Making and Knowing Project’s transcription and translation of Ms. Fr. 640. For an updated version of the text, see:

> Making and Knowing Project, Pamela H. Smith, Naomi Rosenkranz, Tianna Helena Uchacz, Tillmann Taape, Clément Godbarge, Sophie Pitman, Jenny Boulboullé, Joel Klein, Donna Bilak, Marc Smith, and Terry Catapano, eds., *Secrets of Craft and Nature in Renaissance France. A Digital Critical Edition and English Translation of BnF Ms. Fr. 640* (New York: Making and Knowing Project, 2020), https://edition640.makingandknowing.org. DOI: https://doi.org/10.7916/78yt-2v41.

Chalk in yellow lake pigments

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BnF Ms. Fr. 640, fol. 63v: “Scudegrun” and fol. 10r: “Scudegrun”

Transcription and Translation

Recipe 1:

<div>
<id>p010r_2</id>
<head><m>Scudegrun</m></head>

<ab>Il se faict à <pl>Lyon</pl> avecq du <m>suc de gaulde</m> & de la <m>croye</m> incorporée<lb/>
ensemble, ou pour mieulx avecq de la <m>ceruse</m> qui est propre à<lb/>
destrempé et à <m>huile</m>.</ab>
</div>

<div>
<id>p010r_2</id>
<head><m>Scudegrun </m></head>

<ab>It is made in <pl>Lyon</pl> from the <m>juice of weld</m> mixed with <m>chalk</m> or better yet with <m>ceruse</m>, which is appropriate for <m>tempera</m> and <m>oil</m>. </ab>
</div>

Recipe 2:

<div>
<id>p063v_3</id>
<head><m>Scudegrun</m></head>

<ab>Se faict avecq de la <m>fleur de genest<m> fort bouillie<lb/>
dans de l<m>eau</m> y mettant suffisament de l<m>alum</m> Puys<lb/>
de la <m>ceruse</m></ab>
</div>

<div>
<id>p063v_3</id>
<head><m>Scudegrun</m></head>

<ab>It is made with the broom flower well boiled in water, putting in it enough alum, then some ceruse.</ab>
</div>

Introduction

Ms. Fr. 640 names two recipes for the preparation of Scudegrun on fol. 10r and 63v. They are recipes for the manufacture of yellow lake pigments. Lake pigments are the most important class of organic pigments. They are made out of a salt which functions as a substrate that gives the pigment body. This substrate is colored with vegetable or insect dyestuff.[1] Lake pigments enabled artisans to expand the limited range of mineral pigments ever since antiquity.[2]

The recipe on fol. 10r gives the option of using chalk, but preferably ceruse (lead white). The recipe on fol. 63v only names ceruse. Because of the health risks of working with lead white, chalk was used in all our reconstructions. The appropriateness of chalk for yellow lakes was confirmed by historical recipes, for instance in the popular Bolognese Manuscript (fifteenth century) that deals with pigment making.[3] Kirby, who is a modern authority in the field of lake pigment research, states that many recipes for yellow lake pigments dating from before the eighteenth century describe the use of a calcium salt like chalk.[4]

During the reconstruction of the Scudegrun recipes chalk appeared to play a large role in the manufacture of the pigment and any changes in the parameters of the experiment influenced the pigments final characteristics. Not much is known about the exact formation and final composition

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[1] Daniel V. Thompson, *The Materials and Techniques of Medieval Painting* (New York: Dover Publications, 1956), 74-75.
[2] Jo Kirby, "Dyes, Dying and Lake Pigments – Historical Background." (Presentation, *Back to the Roots* – Workshop on the Preparation of Historical Lake Pigments. Doerner Institut, Munich, March 23-25, 2011) http://www.doernerinstitut.de/downloads/Back_to_the_Roots/Back_to_the_Roots_Kirby_I.pdf.
[3] Mary P. Merrifield, *Medieval and Renaissance treatises on the arts of painting: original text with English translations* (Mineola, NY: Dover Publications, 1999), 650.
[4] Kirby, Jo, Maarten van Bommel and Andre Verhecken, *Natural Colorants for Dyeing and Lake Pigments: Practical Recipes and their Historical Sources* (London: Archetype Publishings, 2014), 77-78.

calcium-containing lake pigment substrates. Only since a decade researchers show interest in lake pigment substrates and not much research has been carried out with a focus on calcium-containing lake pigment substrates.[5] This makes that the role and behavior of chalk in this type of substrates is not yet well understood. The observations during the reconstruction and the current state of research lead me to consider the role of chalk in the manufacture and application of yellow lake pigments.

To gain insight into the way chalk functions in the substrate of the pigment and how it influences the final pigments' properties, first the calcium-containing substrates are placed in context by comparing them to another popular type of lake pigment substrates. Hereafter a closer look is taken at the manufacture process of the pigment while focusing on the behavior of chalk. Finally, the way the chalk influences the pigments application is researched.

Hydrated alumina versus calcium-containing substrates

In his lauded book *The Materials and Techniques of Medieval Painting* (1956) Thompson subdivides the lake pigments in two groups, based on the characteristics of their substrate.[6] One group has a translucent substrate, called hydrated alumina, and the other has an opaque chalk-containing substrate. The *Montpellier Liber Diversarium Arcium*, a well-known fifteenth-century recipe book, specifically mentions a lake is a dye that is attached to a colorless substrate and describes calcium-containing substrates as a separate group of pigments.[7] De Mayerne cites painter Daniel Mytens (1590-1658) in his extensive and immensely popular seventeenth century artistic manual, who even states calcium-containing substrates place these pigments between the earth pigments.

[5] Jo Kirby, Marika Spring and Catherine Higgitt, "The Technology of Red Lake Pigment Manufacture: Study of the Dyestuff Substrate" in *National Gallery Technical Bulletin* 26 (2005), 71.
[6] Thompson, *The Materials and Techniques of Medieval Painting*, 79-80.
[7] Mark Clarke, *Mediaeval Painters' Materials and Techniques. The Montpellier Liber Diversarum Arcium* (London: Archetype Publishers, 2011), 71.

[8] Also more recent the division between lakes on hydrated alumina and calcium-containing substrates was made.[9] However, currently a lake is described as a pigment obtained by combining vegetable or animal coloring matter with some metallic oxide or earth.[10] In this annotation this current consensus is followed.

Thompson makes a distinction between the two groups of substrates based on their visual characteristics; one is translucent, the other is not. Of course this means there are also chemical differences in the formation and composition. In order to understand those differences a closer look will be taken at their formation. For the preparation of both types of substrates three components are needed: a dyestuff from plant or insect origin, alum, that functions as an acid, and an alkali. The different substrates are the result of the type of alkali reagent used.[11]

Hydrated alumina substrates are the most common substrates found in lakes. They are used for red lake pigments, which were popular due to the translucent glazes that could be made with the pigments.[12] The alkali used is potassium carbonate. If to a dyestuff-containing solution of alum potassium carbonate is added, transparent aluminum hydroxide precipitates, carbonic acid escapes as a gas and potassium sulfate is formed and stays in solution. A double decomposition has taken place: both the potassium carbonate and alum have been converted into other salts. During precipitation the aluminum hydroxide takes up the color of the dyestuff and chemically bonds with it to form a colored compound.[13] This is the final pigment.

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[8] A.M. Stoll, "Gelbe Pflanzenlacke, Schuttgelb und Safran" Maltechnik Restauro (1987), 88.

[9] For the modern source that does not characterize calcium-containing substrate lakes, see: Stoll, "Gelbe Pflanzenlacke, Schuttgelb und Safran", 88.

[10] Nicholas Eastaugh, Valentine Walsh, Tracey Chaplin, *Pigment Compendium: A Dictionary and Optical Microscopy of Historical Pigments (*Oxford: Butterworth Heinemann, 2008), 221.

[11] Thompson, *The Materials and Techniques of Medieval Painting*, 79-80.

[12] Jo Kirby, "The Technology of Red Lake Pigment Manufacture: Study of the Dyestuff Substrate", 71.

[13] Thompson, *The Materials and Techniques of Medieval Painting*, 79.

Chemical behavior during formation

Calcium-containing substrates are prepared with another alkali. The alkali can be any type of calcium salt, but was usually calcium carbonate in the form of chalk.[14] These lakes were prepared by precipitating the alumina hydroxide out of the alum with the calcium. Aluminum hydroxide precipitates as before, carbonic acid escapes as a gas [Fig. 1]. Instead of soluble potassium sulfate, now insoluble calcium sulfate is formed [Fig. 2]. Because of its insolubility this precipitates out along the alumina.[15] The composition of these substrates is not well characterized, but it is expected that they contain a relatively small amount of hydrated alumina next to calcium sulfate and a variable amount of unreacted chalk.[16]

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[14] David Saunders and Jo Kirby, "Light-induced Colour Changes in Red and Yellow Lake Pigments" in *National Gallery Technical Bulletin* 15 (1994), 83.
[15] Thompson, *The Materials and Techniques of Medieval Painting*, 80.
[16] Kirby, *Natural Colorants for Dyeing and Lake Pigments,* 77.

Figure SEQ Figure * ARABIC 1. Carbonic acid gas forms in the reaction and wants to escape. Thereby it creates foaming. This foaming immediately occurs after the first chalk is added.

Figure 2. During the gradual addition of the chalk a bright yellow and opaque precipitate forms at the bottom of the beaker. This is the calcium sulfate to which the color of the dyestuff is attached by the mordanting function of the alum

During the reconstruction of the recipe on fol. 10r in which alum is not named, it became apparent how important the role of alum is in this reaction with chalk. Mineral acids like alum readily decompose chalk.[17] A batch of pigment prepared without

[17] Ibid, 94.

the use of the acid took up none of the color [Fig. 3]. Like in textile dyeing a mordant needs to be present to chemically bind the otherwise water-soluble dyestuff to the chalk that has a very absorbent character and could be a suitable substrate.[18] The observation that the chalk will not take up any color is not in line with the statement of Saunders and Kirby that on occasion lakes were prepared with a calcium carbonate but without an acid. They note this will lead to a less strong chemical binding.[19] However, the observations during the reconstructions indicate the binding that occurs is so weak that it does not overcome the water-solubility of the dye and therefore omitting an acid will not lead to a colored pigment.

Figure 3. Oil paints made with different pigment batches. The pigments of the middle samples are made with the regular three components and show a bright yellow color. The top and bottom samples are made with pigments made without the use of alum. The color of the resulting paint is compared to the color of plain chalk mixed with the same oil. No color difference is observable

Experiments were carried out with a reversed order of adding the ingredients, as named in historical recipes, to see whether the chalk would react differently.[20] This type of reversed addition of material is more common in hydrated alumina substrates.[21] What observations during reconstruction of the Scudegrun recipe on fol. 63v in which alum was added to an alkaline dyestuff solution made clear was that there were visible differences in the two batches: the substrate created with the reversed order of addition took up more color and

[18] Jo Kirby. *A Closer Look: Techniques of Painting* (London: National Gallery Company, 2011), 32.

[19] It goes beyond the scope of this annotation to go into the exact chemical reactions that occur in the manufacture of the pigment. However, more is known about the difference between the real complex-forming, that takes place during the double decomposition of the two salts resulting in a good chemical connection between the dyestuff and the substrate, and the adsorption of color by the unreacted chalk which in an example of a less strong bond. For more information, see: Anna Amot, et al, "Complexation of apigenin and luteolin in weld lake: a DTF/TDDFT investigation" in Physical Chemistry Chemical Physics 12 (2010), 6672-6684. Saunders, "Light-induced Colour Changes in Red and Yellow Lake Pigments", 84.

[20] For an example of such a recipe, see: "*Libre Secondo de Diversi Colori s Sise da Mettere a Oro: a Fifteenth-Century Technical Treatise on Manuscript Illumination*" quoted in Arie Wallert, The Getty Conservation Institute in Historical Painting Techniques, Materials, and Studio Practices (University of Leiden, June 26-29, 1995), 41.

[21] Kirby, "The Technology of Red Lake Pigment Manufacture: Study of the Dyestuff Substrate", 80.

resulted in a more compact dried pigment what made it more difficult to create a smooth paint [Fig. 4, fig. 5]. Research showed how in the formation of hydrated alumina substrates two different compounds form depending on the order of addition of the components. However, that the small amount of hydrated alumina that is present in the substrate brings about these different characteristics is not likely, since in her research Kirby states that the occurring differences are only detectable using analytical techniques.[22]

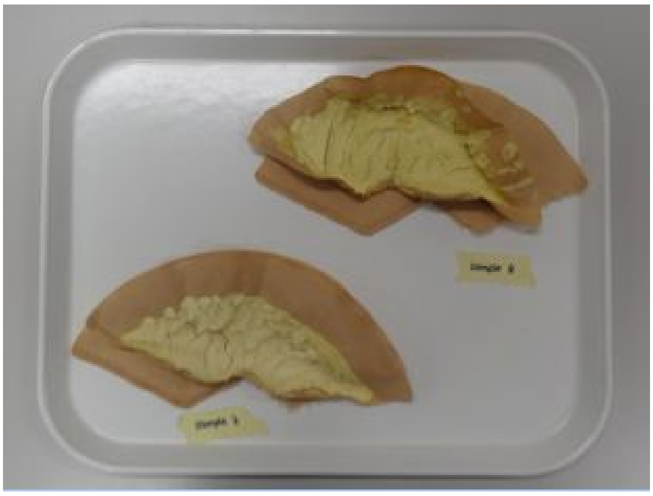

Figure 4. Comparison between sample 7, which is made by adding the components in the regular order, and sample 8, made by reversed addition. Sample 8 took up more color and is denser than the samples created in the usual order.

Figure 5. It took longer to finely grind the pigment of sample 8 (bottom) and in combination with cold pressed linseed oil the resulting paint still shows large particles, whereas sample 7 (center) was easily ground and created into a smooth paint.

Pigment properties

While reconstructing the recipe on fol. 63v it became apparent that it is very likely that calcium-containing substrates all have an amount of unreacted chalk left. When the chalk, being an alkali, neutralized the acidic environment containing the dyestuff a situation occurs which can be called saturation since the chalk can no longer go into reaction. This point can be observed when the formation of foam stops, very slow and careful addition of the chalk helps to define the exact moment [Fig. 6]. All chalk added after this point functions as an extender: it gives the pigment more body. However, since it was observed that

[22] Ibidem

unreacted chalk cannot sufficiently bind the dyestuff it might be clear that excess chalk will make the pigment lighter and less intense [Fig. 7].[23]

Figure 6. When the acidic conditions are neutralized by the addition of enough chalk, the formation of carbonic acid stops. The bubbles will become larger, the foam layer starts to get thinner and, finally, dissolves.

Figure 7. Four pigment samples made using the same amount of alum and dyestuff. The amount of chalk added to the sample in the right bottom corner was doubled as to the other three samples. It is obvious that the color of this pigment is lighter and less intens.

Of course it could also be a well-considered choice to lighten and de-intensify the pigment using chalk.[24] Thompson even states that yellows tend to be obvious and conspicuous and therefore easily disturb the balance of a painting.[25] Since many recipes were not very detailed in the amount of chalk to add, like the recipe for Scudegrun on fol. 10r, it might have been the case that artisans were familiar with this type of process and were aware of what an excess of chalk would do with the resulting pigment. This idea is strengthened by the fact that there are recipes, in which both the actual compound formation and unreacted chalk are addressed, for example recipe no. 105 in the Bolognese Manuscript.[26] Moreover, the use of extenders was common and different materials were known that only gave body to the pigment without lightening it, like starch.[27]

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[23] Kirby, *Natural Colorants for Dyeing and Lake Pigments,* 94.
[24] The use of chalk to lighten a lake pigment with a calcium-containing substrate makes sense since it is known chalk was also used as a pigment on its own to create slightly translucent whites. Eastaugh, *Pigment Compendium, 98.*
[25] Thompson, *The Materials and Techniques of Medieval Painting,* 175.
[26] Stoll, "Gelbe Pflanzenlacke, Schuttgelb und Safran", 88.
[27] For the use of chalk as an extender, see: Nicholas Eastaugh, *Pigment Compendium,* 98 and 406. For different materials used as extenders, see: Kirby, "The Technology of Red Lake Pigment Manufacture: Study of the Dyestuff Substrate", 83.

The ratio of chalk and gypsum also influences the size and roughness of the pigment particles and their transparency.[28] The particle size of chalk is not controlled because is it a natural material.[29] Chalk is composed of 'nanofossil' remains of unicellar algae called coccoliths. These coccoliths are oval, discoidal, spherical and button-shaped, have uneven surfaces and are about 3 to 8 micrometers in diameter [Fig. 8].[30] The calcium sulfate crystals have a slightly greater volume than the chalk particles and are crystals.[31] Their surfaces are even [Fig 9].[32] This means that they scatter light less and are more translucent than the chalk particles.[33] Therefore, the more unreacted chalk is present, the less translucent and finer the final pigment will be, whereas when no unreacted chalk is present the pigment will be coarser but more translucent.[34]

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[28] "*Libre Secondo de Diversi Colori s Sise da Mettere a Oro: a Fifteenth-Century Technical Treatise on Manuscript Illumination*", Wallert, 41.
[29] Rutherford J. Gettens, Elisabeth West Fitzhugh and Robert L. Feller, "Calcium Carbonate Whites" *Studies in Conservation* vol. 19 no 3 (1974), 159.
[30] Rutherford, "Calcium Carbonate Whites", 158.
[31] Ibid, 166.
[32] Nicholas Eastaugh, Valentine Walsh, Tracey Chaplin, *Pigment Compendium: Optical Microscopy of Historical Pigments (*Oxford: Butterworth Heinemann, 2008), 290-295.
[33] Eastaugh, *Pigment Compendium,* XXIII.
[34] Ibid*, XVII.*

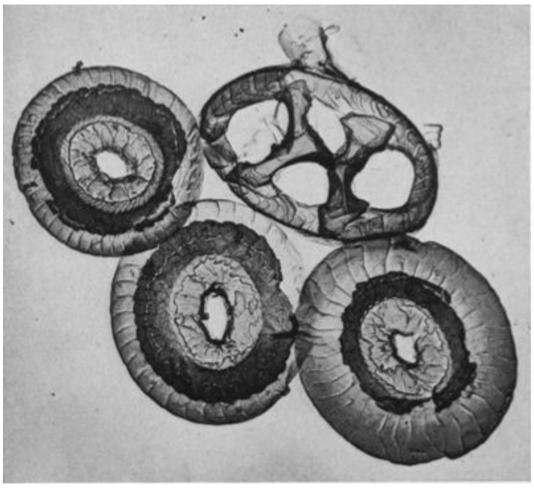

Figure 8. The variety of nanofossils present in chalk enlarged 5600x. It is clear how rough the surface of those small fossils is.

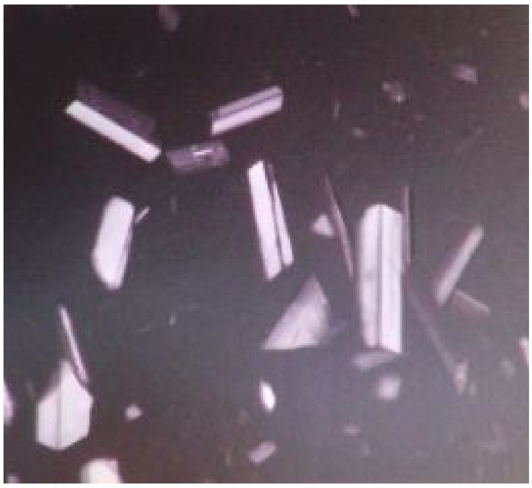

Figure 9. The smooth crystal surface of calcium sulfate particles enlarged 100x. This example shows gypsum particles, one of the several closely linked forms of calcium sulfate.

Application

As the author-practitioner indicates in the Scudegrun recipe on fol. 63v, lake pigments with a calcium-containing substrate were made into water-based and oil paints.[35] To describe the transparency of materials, their refractive index is used; the lower the refractive index, the more light-scattering takes place and the more opaque a material is.[36] The closer the refractive indices of the pigment and medium lay together, the less light scattering takes place.[37] The refractive index of calcium sulfate (1.520-1.53) is somewhat higher than that of chalk (1.486-1.64).[38] In the reconstruction cold pressed linseed oil (1.48-1.49) was used in accordance with Kirby.[39] The refractive index of drying oils is usually closer to the one of the of pigments closer water-based media: the refractive

35 Kirby, *Natural Colorants for Dyeing and Lake Pigments,* 104.
36 Eastaugh, *Pigment Compendium, XVI-XXVIII.*
37 White, "Some Observations on the Binder and Dyestuff Composition of Glaze Paints in Early European Panel Paintings", 219.
38 "Cameo Materials Database: Alumina Trihydrate", Museum of Fine Arts Boston, accessed March 24, 2016. http://cameo.mfa.org/wiki/Alumia_Trihydrate. "Cameo Materials Database: Gypsum", Museum of Fine Arts Boston, accessed March 24, 2016. http://cameo.mfa.org/wiki/Gypsum. "Cameo Materials Database: Chalk", Museum of Fine Arts Boston, accessed March 24, 2016. http://cameo.mfa.org/wiki/Chalk.
39 "Cameo Materials Database: Linseed Oil", Museum of Fine Arts Boston, accessed March 24, 2016. http://cameo.mfa.org/wiki/Linseed_oil. Kirby, *Natural Colorants for Dyeing and Lake Pigments,* 90.

index of water is 1,333.[40] As a result, oil paints are always more translucent and less bright (more saturated) than water-based paints [Fig. 10].[41]

Figure 10. A piece of wood covered with the different paint samples. The yellow paint at the center of the top edge is mixed with egg white. This results in a bright and opaque yellow paint. The samples applied to the right top corner and right bottom corner are made with the same pigment as the pigment in egg white, but are here mixed with cold-pressed linseed oil. This results in a less bright, darker (more saturated) and more translucent paint as expected.

The lake pigments with alumina substrates were largely valued for the translucent glazes they produced in an oily medium.[42] This raises the question why yellows were made using calcium salts that lowered their translucency when used to make paint. Thompson states medieval color-makers considered yellow lakes most beautiful when they were somewhat opaque, light and brilliant.[43] Moreover, yellow pigments were mainly used to imitate gold, modify

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40 Ward, Gerald W.R. *The Grove Encyclopedia of Materials and Techniques in Art* (Oxford: Oxford University Press, 2008), 253.
"Cameo Materials Database: Water", Museum of Fine Arts Boston, accessed March 24, 2016. http://cameo.mfa.org/wiki/Water.

41 White, "Some Observations on the Binder and Dyestuff Composition of Glaze Paints in Early European Panel Paintings", 216.

42 White, "Some Observations on the Binder and Dyestuff Composition of Glaze Paints in Early European Panel Paintings", 216.

43 Thompson, *The Materials and Techniques of Medieval Painting,* 188.

greens and give sharpness to other yellows, ochres for example, instead of representing yellow things.[44] A possible explanation for the use of calcium carbonates in yellow lake pigment substrates could be that the tinting-strength of yellows on an opaque substrate might be too low to gain good results in these uses.[45]

In 1994, Saunders and Kirby found that the use of calcium-containing substrates was not a way to improve the light fastness off the fugitive yellow colorants. From their experiments is became apparent that when calcium salts are added to the extracted dyestuff, the resulting lake pigment is more fugitive than when the same dyestuff is precipitated on an alumina substrate. When chalk is added in excess, and functions as an extender, the stability of the lake is reduced even further.[46]

As described above, yellow lake pigments were often used combined with other pigments to alter yellows and greens, and their substrates usually still contain unreacted chalk. This makes this type of lake pigments not compatible with alkali-sensitive colors, like Prussian blue, because the unreacted chalk can discolor these pigments.[47]

Conclusion

During the reconstruction of the two Scudegrun recipes in Ms. Fr. 640 chalk appeared to play a large role. It is due to its water-insoluble and opaque nature that chalk can indeed play a large role. When chalk reacts with the alum insoluble opaque calcium sulfate precipitates, as opposed to the potassium sulfate that is formed in the manufacture of transparent hydrated alumina substrates. The calcium sulfate becomes part of the substrate as a third component next to the hydrate alumina and dyestuff. Excess chalk can be present as the forth component.

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[44] Ibid, 174. Kirby, *Natural Colorants for Dyeing and Lake Pigments,* 77.
[45] Nicholas Eastaugh, *Pigment Compendium,* 406.
[46] Saunders, “Light-induced Colour Changes in Red and Yellow Lake Pigments”, 88-89.
[47] Clarke, *Mediaeval Painters' Materials and Techniques. The Montpellier Liber Diversarum Arcium,* 214.

The opacity and insolubility of this third and possible fourth component of the substrate make recipes that use chalk more sensitive than recipes using potassium salts: any excess chalk will directly alter the visual appearance as well as the working properties of the pigment. Although experiments showed that the effect of the use of chalk in the lake substrates is large, they also emphasized the equal importance of all three components (dyestuff, alum and chalk) to create a workable pigment.

Since not much research is done into the role of chalk in lake pigment substrates many questions still need to be answered. A step towards a better understanding of the composition of calcium-containing substrates and factors influencing the composition of the final substrate using analytical techniques would be helpful in characterizing chalk as part of the substrate. Historical research into the reasons for using chalk as the basis for a substrate would also be a big step towards a more complete understanding of the role of chalk in such substrates.

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List of illustrations

Figure 5. *Comparison of working properties of pigments created by normal and reversed addition of acid and alkali*. Photo taken by L. Wolfkamp, lab partner during reconstructions.

Figure 6. *Stoppage of foaming*. Photo taken by L. Wolfkamp, lab partner during reconstructions.

Figure 7. *Comparison of pigment samples confirming the lightening properties of excess chalk.* Photo taken by L. Wolfkamp, lab partner during reconstructions.

Figure 8. *Nanofossils in chalk enlarged 5600x.* Photo published in Gettens, Rutherford J., Elisabeth West Fitzhugh and Robert L. Feller, "Calcium Carbonate Whites" *Studies in Conservation* vol. 19 no 3 (1974), 168.

Figure 9. *Calcium sulfate crystals enlarged 100x.* Photo published in Eastaugh, Nicholas, Valentine Walsh, Tracey Chaplin. *Pigment Compendium: Optical Microscopy of Historical Pigments (*Oxford: Butterworth-Heinemann, 2008), 294.

Figure 10. *Comparison of pigments used in oil and water-based media.* Photo taken by author.