'To prepare white excellent...': reconstructions investigating the influence of washing, grinding and decanting of stack-process lead white on pigment composition and particle size
Stols-Witlox, M.J.N.; Megens, L.; Carlyle, L.

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‘To prepare white excellent…’: reconstructions investigating the influence of washing, grinding and decanting of stack-process lead white on pigment composition and particle size

Maartje Stols-Witlox, Luc Megens and Leslie Carlyle

ABSTRACT Historical recipes describe several refining methods to improve the quality of lead white, usually by grinding or washing with water and/or vinegar. Processing methods also include decanting (particle size separation based on gravitational sedimentation speed). This paper reports on reconstructions of such processing methods using historically accurate materials and techniques. Particle size separation through gravitational sedimentation is easy to accomplish. The size fraction thus produced bears a close resemblance to the very fine grade of lead white present in Vermeer’s The Art of Painting (Kunsthistorisches Museum, Vienna). Lead white is generally considered to be a basic lead carbonate, but it usually consists of basic and neutral lead carbonate, with small amounts of other lead salts. X-ray diffraction of the pigments produced with historically accurate techniques shows that all of the reconstructed processing methods influence the ratio of neutral to basic lead carbonate, favouring the formation of neutral lead carbonate.

Introduction

As the white pigment most commonly used for oil painting before the introduction of good quality zinc white or titanium white, lead white is discussed extensively in written sources. Prior to the 19th century, lead white was usually produced by the stack process: rolls or buckles of lead were exposed to dilute vinegar (acetic acid) inside glazed earthenware jars, which were buried in horse manure or spent tanning bark (both of which provided a source of heat and carbon dioxide through fermentation). The metallic lead was corroded by fumes from the dilute vinegar to form lead acetate, which in turn reacted with carbon dioxide to form (basic) lead carbonate. In large-scale production facilities, rows of earthenware jars, buried in the manure or tanning bark, were built up in stacks, which provides the derivation of the terms ‘stack process’ or ‘stack method’ and ultimately ‘stack lead white’. Although the 19th century saw the development of new production methods (such as the precipitated Clichy white and the German chamber process), artists continued to favour stack-process lead white for its excellent covering power and its rheology (Gettens et al. 1993).

Historical recipes not only described the pigment’s production, but dealt with its purification and adulteration, as well as its use in painting. An earlier publication on lead white production, quality and terminology, presents an overview of such recipes (Stols-Witlox 2011). To summarise briefly, historical sources ascribed quality differences to several parameters: not only was purity of the raw ingredients considered to be important, but also the reaction (corrosion) conditions as well as later processes such as washing, grinding and decanting of the pigment. While most painters were unable to influence reaction parameters personally as the pigment was generally produced by large-scale production, they could select lead white with particular characteristics and they were able to further process it themselves and thereby enhance its quality.

The fact that large numbers of surviving recipes for artists provide instructions for washing and grinding lead white suggests that such processing treatments may have been common (see Tables 1–3). This information is highly relevant because paintings provide evidence of the use of different qualities of lead white and it is currently unclear whether this represents variables in manufacture, careful selection by the artist, or processing by the artist, their assistants or colourmen – or perhaps a combination of all these factors.

Lead white is usually referred to as basic lead carbonate, but Olby (1966) showed that it is in fact a mixture of cerussite (PbCO₃) and hydrocerussite (2PbCO₃·Pb(OH)₂); other lead salts such as plumbonacrite (Pb₅O(OH)₃(CO₃)₂) may...
also be found on occasion. Cerussite and hydrocerussite are in chemical equilibrium, which means that depending on the circumstances they can be converted into each other (Godelitsas et al. 2003), therefore the presence and ratio of lead salts in a given pigment sample will depend on variables during the production process — such as the location of the ceramic container within the stack itself and the quality of the starting materials (Homburg and Vlieger 1996: 39) — or on processing methods. Some reports in the late 19th and early 20th century state that the freshly produced pigment contained some residual lead acetate (Carlyle 2006: 15).

Most studies have confirmed the presence of lead white in paintings by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDX). Although this method detects the presence of the element lead, it does not identify the formulation or quantity of lead salts present in the pigment. Therefore little information on the exact composition of lead whites in paintings is available in the modern technical literature. However, the SEM can provide additional information on lead white. Since SEM backscattered electron (BSE) images show particle morphology, they may be used to obtain an indication of whether or not lead white has been post-processed. This approach was employed successfully in a recent study by Boon and Oberthaler (2010) of The Art of Painting by Johannes Vermeer, dated to the 1660s. SEM BSE images demonstrate that the top layers of whitish passages in the painting contain a lead white with very small particles (possibly denoting a very fine grade of pigment), whereas for the ground and lower paint layers, a much coarser lead white has been employed with a considerably larger range of particle or aggregate sizes (see Fig. 1). Berrie and Matthew have reported similar differences in particle size (2011: 295).

How were different grades of lead white obtained, what was their exact composition and what are the possible consequences of their use for the visual characteristics of the final paint layer and for the way the paint ages? This paper investigates the effects of washing, grinding and decanting methods by reconstructing historical recipes, and analysing the effects of these treatments on pigment morphology and composition.

**Materials and methods**

Reconstructions were based on a large collection of recipes for the production and processing of lead white from both published and unpublished sources, dating from c.1500 to 1900. This recipe collection was established by the Historically Accurate Reconstruction Techniques (HART) project. The recipe collection was expanded further by the first author in subsequent doctoral research. Recipes analysed for the present study include those that concern both lead white processing recipes describing professional manufacturing practice and recipes intended primarily for artists (Fig. 2). If available, first editions were consulted. Recipes copied or translated from earlier sources were included in the research, since they may provide information on the popularity of certain procedures.

The majority of recipes for processing lead white, especially those describing professional manufacturing methods, mentioned washing or grinding with water. The second largest group consists of instructions for grinding or washing with vinegar, the latter being mentioned mainly in recipes aimed at artists (Stols-Witlox 2011). Because of their relative frequency, these two methods were selected for comparative reconstructions.

Reconstructions were executed with traditionally prepared stack-process lead white, thoroughly washed with distilled water. Vinegar for grinding with the lead white was prepared from organic, sulphite-free red wine and vinegar culture (bacteria). Lead white pigments, further
processed by grinding with vinegar and/or further water washing, were subsequently made into oil paint by hand grinding with poppy oil extracted from a single seed lot from a single supplier. The paints were spread on polyester film (Melinex) with a hogshair brush and with a drawdown bar. All dry pigments produced were analysed with X-ray diffraction (XRD), and the oil paints were then analysed in cross-section with SEM-EDX. Colour measurements were made on the paint films using a Konica-Minolta spectrophotometer.
### Table 1: Historical recipes for washing stack-process lead white.

<table>
<thead>
<tr>
<th>Author</th>
<th>Year</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pomaro, G.</td>
<td>1500–1600.</td>
<td>To purify <em>biaccia</em> very fine with water. First grind the <em>biaccia</em> very soft with water; then wash 3 or 4 times and leave to dry on the stone, then gather and keep in paper.</td>
</tr>
<tr>
<td>Andriessen</td>
<td>1552.</td>
<td>Lead white is ground on a hard stone with clean water and if you want it fair then when it is well ground leave it spread on that hard grinding stone and sprinkle on clean water and leave it to bleach in the hot sun and when it is dry humidify it again on the stone and grind it for a while and let it dry again and bleach [do this] up to four or five times. This is the most clean white that you can find if you first remove the blue lead that falls in during grinding that must always be removed or the lead white would truly stain.</td>
</tr>
<tr>
<td>Birelli</td>
<td>c.1601.</td>
<td>Then grind it, &amp; put it in a <em>pignatto</em>, &amp; for every <em>libra</em> of this flour [= lead white], add two ounces of water, &amp; put on the fire while stirring it well until the fire until smoke rises, then take it from the fire, &amp; put it in the shape you like, &amp; put it in the sun, leave until it has dried well, then remove it from the mould, &amp; put it in a straw which will make fine <em>biaccia</em>.</td>
</tr>
<tr>
<td>Veen, J.</td>
<td>van c. 1650.</td>
<td>Lead white is ground with clean water, if you want it beautiful so it can bleach on the grinding stone, strewed and sprinkled with clean water in the hot sun and when it has dried it must again be humidified a little and ground and leave it to bleach and dry up to 5 times.</td>
</tr>
<tr>
<td>Symonds, A.</td>
<td>1698.</td>
<td>To prepare white excellent. Take a good quantity of <em>Seruse</em> and mix, then as much as you want it to be thoroughly mixed with water. Then let it settle 2 or 3 hours Which done, taste the water and if it is not beautiful add two ounces of water and put on the fire while stirring it well until the water vaporise, and the ceruse being dry use of the fairest and reserve for yr use.</td>
</tr>
<tr>
<td>Veen, J.</td>
<td>van c. 1650.</td>
<td>Lead white is ground with clean water, if you want it beautiful so it can bleach on the grinding stone, strewed and sprinkled with clean water in the hot sun and when it has dried it must again be humidified a little and ground and leave it to bleach and dry up to 5 times.</td>
</tr>
<tr>
<td>King, D.</td>
<td>1668.</td>
<td>To prepare white excellent. Take a good quantity of <em>Seruse</em> and mix, then as much as you want it to be thoroughly mixed with water. Then let it settle 2 or 3 hours Which done, taste the water and if it is not beautiful add two ounces of water and put on the fire while stirring it well until the water vaporise, and the ceruse being dry use of the fairest and reserve for yr use.</td>
</tr>
</tbody>
</table>

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**Contd.**
Table 1 Historical recipes for washing stack-process lead white cont’d.

<table>
<thead>
<tr>
<th>Beurs, W. 1692. <em>De groote waereld in het kleen geschildert.</em> Amsterdam: J. and G. Jansoniis van Waesberg, 8</th>
</tr>
</thead>
<tbody>
<tr>
<td>For this choose the thickest flakes, as the best, grind them in very clean and clear water on a good grinding stone, which must not be too hard or too smooth; and when it has been ground, put it on a piece of glass, on which the water must evaporate. When dry enough, grind with the best poppy oil, which surpasses nut oil, linseed oil and other known [oils], after which put it in a clean shell or bowl, and cover with clean water, to prevent drying; and so it can be kept; to be used, when needed […] The good non-ground lead white is prepared in the same way, as flake-white, but is easier to grind.</td>
</tr>
<tr>
<td><strong>Daar toe dan kiestmen uit de dikste Schulpjen, als de beste, die men in zeer schoon en helder water vrijt op een goede keystone, die niet te hart of te glad wezen kan; en alzoo is gewreven, zet menze op een stuk van een glas, daar op het water uit droogen moet. Als ze nu droog genoeg is, vrymen die in de beste papaver oly, die de Nooij-oly, Lijnvaat-oly en andre bekenden overtreff; waar na menz en in een rein Schulpie of kommenije doet, en er schoonwater op zet, om niet te verdrogen; en dus kaimeenje goed houden; om als t voogd is, gebruikt te […] T Goede ongenoelen Looitwit bereiden op de zelbe wyze, als het Schulp-wit; dog t is gemakkelijker om vrymen;</strong></td>
</tr>
<tr>
<td>Smith, M. 1692. <em>The art of painting according to the theory and practise of the best Italian, French, and German masters.</em> London: Vendue, 71</td>
</tr>
<tr>
<td>The instruments and Materials us’d in Painting and the preparing Colours to the pallat; Lead White</td>
</tr>
<tr>
<td>White Flakes being pick’d and scratch’d must first be ground very fine in water, afterwards Temper’d on the stone with the muller with Nut-Oyle; then cover it with water in a gallipot or it will skin over. But so it must not be kept very long for the Oyle will grow Fat and make it unfit for use. For Deadcolouring you may grind white lead in Linseed Oyle.</td>
</tr>
<tr>
<td>161: If you want to grind paints then first take some lead white to clean the stone and when it has been ground add some indigo, because this white is not pure enough to use, after this take lead white and it will be pure. The white you can grind with water, and let dry and temper with nut oil or linseed oil. Lead white is ground with urine or vinegar and when dry and you want to use it temper it with oil.</td>
</tr>
<tr>
<td><strong>161: Alsom verwen vriwen wil zo noemt eerst wat lood-wit om den steen wat zayer to maken en als, t gewreven is zo doeter wat indigo bij, want dit wit dan niet zayer genoeg is om alleen te gebruiken, daer na neemt ander lood-wit en die zal zayer zijn.</strong> T wit kon gij vriwen met water, en laeten droogen en temperent met suet olij of lijn olij. Looit-wit vriwt met pis of asijn en alse gedoet is en gij die wilt gebruiken zo tempertse met olij. <strong>161: If you want to grind paints then first take some lead white to clean the stone and when it has been ground add some indigo, because this white is not pure enough to use, after this take lead white and it will be pure. The white you can grind with water, and let dry and temper with nut oil or linseed oil. Lead white is ground with urine or vinegar and when dry and you want to use it temper it with oil.</strong></td>
</tr>
<tr>
<td>The lead white may also from the start be ground with water and some think this is better, but the Venetian white must from the start be ground in oil, because one cannot temper off the water from the white.</td>
</tr>
<tr>
<td>Looidwitt vriemen met schoon water; wilmant schoon hebben zo laat het wyt gespyp genogen op den steen als t gewreven is en beprengen met schoonwater en later zoost staan bloeken in heete zon en als, t droosh is zal ment weder vochtich maken en vriwen weder een wiif tijs en weer. En bleeken tot al te hart of te hart en bleeken tot 4 of 5 maal toe dit is schoon wit en als men verwerken in oile gewreven worden, om dat men het waer winter in het oile worde, om dat men dit wit in oile gewreven worden kan.**</td>
</tr>
<tr>
<td>Then one grinds these flakes on a hard stone, like porphyry, with the muller &amp; clear water, &amp; as clean as possible to have a beautiful white; sometimes these flakes are covered with a grey or yellow material which must be grated off before grinding, which may be caused by the lead sheets that were not clean before they were enclosed in the pot. The lead white being well ground with water is left to dry, &amp; you may keep it as long as you wish.</td>
</tr>
<tr>
<td><strong>490–94: Lead white is ground with clean water; but take care, that it is beautiful so leave it spread on the stone after it has been ground and sprinkled with clean water and leave it thus to bleach in the hot sun and if it is dry it will again be humidified and reground for a while and then dried and bleached up to 4 to 5 times this is beautiful white and when you want to use it you will temper it with gum water. Note. If you grind this lead white you have to search and remove the lead which is normally in it or it would be contaminated from the start.</strong></td>
</tr>
<tr>
<td>Then one grinds these flakes on a hard stone, like porphyry, with the muller &amp; clear water, &amp; as clean as possible to have a beautiful white; sometimes these flakes are covered with a grey or yellow material which must be grated off before grinding, which may be caused by the lead sheets that were not clean before they were enclosed in the pot. The lead white being well ground with water is left to dry, &amp; you may keep it as long as you wish.</td>
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</tr>
<tr>
<td>Sprong, U. 1738. <em>Kabinet der verf-stoffen.</em> Amsterdam: Jacob Graal, 13–14</td>
</tr>
<tr>
<td>Here we will share a nice trick, on how the lead white may be purified, and made to last. Take lead white, grind it thoroughly with water on a stone; spread it in the open air; but take care, that it does not become too dry; grind it again and expose it to the air, keeping it always wet. Continue this 3 to 4 times, and it will be very white and pure.</td>
</tr>
</tbody>
</table>
| **138–9: Venetian lead or flake white must be ground with pure rain water, on a good stone, but especially with no other water, because no other water is good for the paints mentioned above. Venedis looit of schlip wit moet sterk met zayer regenwater, op een goede steen gevreyven worden, maar voor al met geen ander water, om dat geen ander water tot alle boeven gemelde verwen goet is.**
To prepare beautiful lead white.

Place it [the lead white] together in a mortar, and pour on a little water, and pound the paint for as much as half an hour, (it is better with water on a mill, such as potters use for their glazes) until it becomes as pulp, then put the paint with a spoon in one or two pots, and put it in the sun, and let it become hard, then it is ready.

Schoon Looot Wit to maken.

Doet die te zaamen in een steene vysel, en giet een wynig Water daarop, en stampt de verwe wel een half uur, (het is beter met water op een mole, als de potte bakkers tot haar verglaas gebruiken) tot het wert als pap, doet dan de verwe met een lepel in een of twee potten, en zet het in de zon, en laat het hart worden, dan is het gemaakt.

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Lindenbergh, J.F. 1753. Nieuwe verlijger. Amsterdam: Steven van Esveldt, 2

Flake white. Preparation.

According to some there are two types, of which one is called Venetian, the other inland; but I have never seen more than one kind, which is one of the hardest materials used in paints, it is ground vigorously with pure rain water on a stone, a long time.

Schelpwit. Bereiding.

Hier van zyn volgens vele twee soorten, wier eene Venetsch, de andere Inlandsch genaamt word; dog ik hebbe nooit meer dan een soort gezien, 't selve is een der hardste stoffen die in de vernen gebruikt werden, men wryft die sterk met schoon regenwater op een steen, een lange tyd.

---


There is no previous preparation necessary, in the case of white lead, to its use; except washing over where it is intended for more delicate purposes; but then indeed it is always best to substitute the flake white.

Buys, E. 1774. Nieuwen en Volkomen Woordenboek (10 vols), vol. 6: K-L. Amsterdam: S.J. Baalde, 754

This lead white is ground on a porphyry stone with a little water, and made into a dough, which is made in moulds to small pointed breads, which are dried for transport;

Men wryft dit loodwit op den porphyry steen met een weinig water, en maakt er een deeg van, dat men in vormen tot kleine spitsvrye brooden maakt, die men drogen laat om te vervoren;

---


About the preparation, use and mixing of white paints.

Flake white. Preparation. It is ground vigorously with clean rain water on a stone, a long time.


men wryft het sterk met schoon regenwater op eenen steen, eeven langen tyd

---


White is made with lead white, or better, with flake white. It is first ground in water, then when it is very dry, with varnish, as thick, that it can be made into balls, make little bullets from it. This way it is used in small quantities. For large quantities it is ground with linseed oil, cold water is poured onto the greasy mixture and ground so long until the water separates out. Because it becomes more clean and manageable with water, while considering the above, also a little of the greasiness is dissolved during grinding.


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After the lead has been transformed into lead white through the vinegar vapors, it is ground on large grinding stones, which in large factories may be most economically driven by waterforce like machines, as tenderly as possible and humidified with water, so a thick pulp results. Then the lead white is cast in moulds, which are normally pyramid shaped, and dried in summer in the open air, in winter however in a moderately warm oven.

Siebenter Abschnitt. Von der weifen Farbe. 1 Weisses Bleiweiß.

Nachdem das Blei durch die Dämpfe des Eßigs in Bleiweiß zernaget ist, so wird es auf grofen Reibestenen, die als Maschinen in einer großen Fabrik am vornthliefenstften vom Wasser getrieben werden können, auf das zarteste gerieben und dabei mit Wasser angefeuchtet, daß ein dicker Brei daraus wird. Sodann wird das Bleiweiß in Formen gebildet, die gemeinnützisch pyramidenförmig sind, und im Sommer an der Luft, im Winter aber in einer mäßig warmen Stube getrocknet werden.

151: The factory grinds the lead white unmixed; however, it most commonly receives on the mill an addition of chalk. The pure flake white is poured onto the millstone, humidified with water, and this is repeated more often, while it is ground. It flows through the channel of the top stone to the other stones, and is ground by all 4 stones. From the last grinding stone it is moved through a channel into a wooden vat, and from this scooped into triangular earthenware moulds which are approximately 4 to 5 zoll high, and has the same width on top. Here it stays as long, until it shrinks, and then it is placed on wooden boards and air-dried.


cont'd.
Historical recipes for washing stack-process lead white

<table>
<thead>
<tr>
<th>Date</th>
<th>Author</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>17/01/2012</td>
<td>Maar</td>
<td>Ist nun die Feuchtigkeit genug abgeleitet, so setzt man die kleinen Bleiweishütte in die Luft, um sie vollständig auszutrocknen. Now the lead white is ready for transport. The more water runs out, is closed with paper; the pots are lined with bright blue paper, and then the mass is put in. When the moisture is removed, then the little lead white huts are placed on a wooden board in the air, to dry them completely.</td>
</tr>
<tr>
<td>11 October</td>
<td>Brodhagen, P. 1802. Anleitung zur Technologie (3 vols.), vol. 1, Hamburg; Bachmann und Gundermann, 378--9</td>
<td>The lead is grinded through the mill, and the mass is washed with water, the whiter the lead white becomes, and with when adding the finest, whitest and cleanest chalk it also results in the most beautiful lead white.</td>
</tr>
</tbody>
</table>
An unforgettable white lead colour is made from lead white, which selected and all is ground on a porphyry; until it becomes blackish. Then take a pot filled with water, wash the lead white clean, and let it settle; and casting off the water, stir it again with vinegar; repeat this another one or two times, and you will have an excellently beautiful white, suited both for water- and oilpaints...

Een onvergelijkelijk witte loodkleur wordt gemaakt van lood-wit, dat uitgezocht en wel op een' porphyriersten gewreven is, tot dat het zwartachtig wordt. Neem dan een pot vol water, wasch het lood-wit daar wel zuiver in af, en laat het bezinken; en het water er afgegooid hebbende, roer het dan weder door met azijn; herhaal dit nog een of twee maal, en zij gult een uitsluitend fraai wit, beide voor water- en olieverf geschikt, bekomt.


The biacca in the trade to purify, first pulverise, and then pour on more than once boiling water; and it is very necessary to do this with Kremnitz [white], because it contains animal glue, from which it obtains its stiffness.

La biacca di commercio per depurarla, si usas prima polverizzarla, e poi sopra gettarvi più d’una volta dell’acqua bollente; ed à molto necessaria fare questa operazione a quella di Kremnitz, perché contiene uno coll’ animale, donde ripete la sua tenacità.


One clears away the manure, takes out the jars, scrapes off the lead white adheres to the metal *), after one has moistened it to prevent dusting, grinds it with some water to a thick pulp, which one leaves to stand for some days in a warm place, through which the remaining acids fully oxidise the metallic parts **), cleans it by washing or slaking (**), if this is necessary, and has it ground ++. Often it is finally slaked.

* In Newastle the lead passes with water through rollers, which remove the lead white. This prevents any dusting. The water is removed, after the lead white has settled, with pumps. The lead is reused or melted. If thin plates are used, they are normally completely corroded.

**) This however appears not to happen in all factories. Often also lead sugar crystals are found on the plates, like a white froth on the water, which contains much sugar of lead. In order not to lose this, one adds some potash, which decomposes it and creates a deposit of carboxylic lead (= lead white).

***) Slaking is operated as usual. One has a large, square tray, which has 7 to 9 compartments, which have the same size, but different heights (or the same amount of vessels of varying heights). The water stirred with lead white floats from one into another and it deposits in the later ones ever finer lead white. Good slaking substantially adds to the quality of lead white.

†) In Holland grinding is performed on three mills standing besides each other and operated with horses. The first grinds coarsely, the second finely, the third very finely. In Berlin the mills are placed on top of each other, so the ground [material] passes straight from one into the other. It would maybe be better to grind in tons, which spin around their axis, with the aid of iron rails.

The ground lead white is left under water for some time, then pressed into earthenware or sheet metal moulds, which have the shape of small conical hats, and left to dry first in these, then in the open air *(†) (during which very sulphurous vapours must be kept away) and then wrapped in paper.

*) In England also in rooms heated by vapour. One does not let them dry completely inside the moulds, because they would be difficult to remove.

Man räumt dann den Mist weg, nimmt die Töpfe heraus, schüttet das an dem Metall hängende Bleiweiß ab *), nachdem man es befeuchtet hat, um das Stauben zu verhüten, stößt es mit etwas Wasser zu einem dicken Brei, den man einige Tage an einem warmen Orte ruhig stehen läßt, damit die anhängende Säure die metallischen Theile noch vollends oxidirt **), reinigt es dann durch Abwaschen oder Schlämmen ***) wenn dies nöthig ist, und läßt es malen †). Oft wird es zuletzt noch geschlämt.


††) In Holland geschieht das malen auf drei nebeneinander stehenden und durch Füße getriebenen Mühlen. Die erste malt grob, die zweite fein, die dritte ganz fein. In Berlin heben die Mühlen übereinander, so daß das Gemalte von einer gleich in die andere übergeht. Besser wäre es vielleicht in Tonnen, die um ihre Achse würden, mit Hülfe eiserner Kugeln, zu malen.


*) In England auch durch dampfgeheizten zimmern. Man läßt es zuerst in diesen, später offen an der Luft *) troknen (wobei man besonders schweflige Dünste von demselben abhalten muß), und dann in papier einschlagen.

**) Diese vorzüglichen farben von dem zimmern. Man löst es unter wasser, wasch die löse innerhalb von einigen tagen, und lassen sie sich dann in einer zimmerei, die einige malen, und dann in einem warmen ort trocknen. Man läßt es dann in eine zimmerei, die einige malen, und dann in einen warmen ort trocknen.

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**) Man löst es unter wasser, wasch die löse innerhalb von einigen tagen, und lassen sie sich dann in einer zimmerei, die einige malen, und dann in einen warmen ort trocknen. Man läßt es dann in eine zimmerei, die einige malen, und dann in einen warmen ort trocknen.

††) In Holland geschieht das malen auf drei nebeneinander stehenden und durch pferde getriebenen Mühlen. Die erste malt grob, die zweite

***) Einen unvergleichlichen witte loodkleur wordt gemaakt van lood-wit, dat uitgezocht en wel op een' porphyriersten gewreven is, tot dat het zwartachtig wordt. Neem dan een pot vol water, wasch het lood-wit daar wel zuiver in af, en laat het bezinken; en het water er afgegooid hebbende, roer het dan weder door met azijn; herhaal dit nog een of twee maal, en zij gult een uitsluitend fraai wit, beide voor water- en olieverf geschikt, bekomt.


TO PREPARE WHITE EXCELLENT...
Historical recipes for washing stack-process lead white

In about a fortnight the corrosion is finished, and the sheets of white lead are found near 1/4 inch thick, and covered in some places with crystals of sugar of lead. As much as can be got off by a moderate degree of force, is very carefully washed. This washing is esteemed the most delicate part of the whole manufacture; during the progress of it, a white scum appears which is taken off, and a little pearlash being added to it, it is changed into white lead, of a beautiful whiteness, and is sold for choice purposes: the remainder is mixed with a pure sulphate of barytes, brought from the Tyrol, in different proportions, according to the market for which it is designed.

Part of the sediment left in the cistern is well washed and produces a dull milk-white lead, with several portions of fresh water. Generally the washing is not continued to such exactness, because buyers prefer white lead that has a slight bluish tinge; now the copper contained in the litharge produces the colour, provided the settling is not washed too much. A grey tinge is sometimes preferred; which is produced by adding a small quantity of common ivory black, which must, however, be well mixed with the white lead.

The flat sheets which cover them [= the jars] provide the flakes, which are kept aside to spread in the trade without any extra preparation. The spirals are unrolled, the smallest and most delicate flakes are removed, and ground with water on horizontal stones. One then washes this ceruse; one lets it deposit and drain, until it has reached a convenient consistency: then one fills conical pots, where it takes the shape that we all know. Les lames plates qui les recouvrent fournissent les écailles, que l'on met à part, pour les repandre dans le commerce sans autre préparation. Les spirales sont déroulées, on en retire des écailles plus petites et plus friables, que l'on broie à l'eau sous les meules horizontales. On lave ensuite cette céruse; on la laisse déposer et égoutter, jusqu'à ce qu'elle soit en bouillie de consistance convenable: alors on en remplit des pots coniques, où elle prend la forme qu'on lui connaît.

When cakes of white lead are purchased ready prepared, small particles of lead in the metallic state are not unfrequently found, owing to the preparation having been imperfectly executed; and in grinding the colour, this metallic part, becoming divided by the motion of the muller, gives a greyish tint to it. To avoid this inconvenience, if you do not prepare your white lead yourself, be careful to ascertain, as well as you can, in purchasing it, whether it is pure, and select the thinnest cakes. In grinding it, your slab and muller should be perfectly clean; because there is often a little acid moisture in white lead, which renders it very apt to attract any parts that remain of colours previously ground. To obtain white lead of a very fine quality, it is often necessary to grind it several times.

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Then the removed lead white is ground in cast iron rollers in a wooden, well closed box, [sieved], then ground fine with water in vertical or horizontal granite stones, the pulp put into the upper wide end of conical, non glazed jars of height 5 Zoll and diameter 3½ Zoll in, which are placed on a drying rack and are shaken a number of times each day, to promote the loosening of the lead white bread from the mould. After 3 to 4 days the mould is turned and the bread is dried in summer in the warm air, in winter and in moist weather in a drying room heated with warm air. Finally the lead white is wrapped in blue paper.


Grinding can, as said before, be executed in two manners, depending on whether hard or soft lead white shall be made. If hard [lead white] is made, the leadchalk dough or the dry lead white powder stirred with water is placed on the mill, while the stones are typically in motion, making 1½-2 turn per second. The pulp is scooped into the little “Sarge” [meaning unclear, probably hole in the centre of the stone] of the running stone with a copper spoon with a long handle and so much is added, that the “Sarge” is filled. It takes some time, before its contents have sunken down, when it is again filled, until bit by bit a batch, for instance 10 Centner has gone through. A worker can operate 4 to 5 stones, standing besides each other. The material that collects in the “Sarge” of the lower stone is moved forward to the outlet with a shovel more often, where it falls into a smaller vessel. During first grinding it is often necessary to raise the upper stone a little, because the mass is very thick and therefore does not easily flow between and through both stones. When everything has been ground and the ground mass is a thick pulp, the stone is ground clean by rinsing it with water, the hole of the base stone is washed, for which a coarse brush or a scrubber is used, and then the same pulp is ground more finely several times in the same manner with more closely placed stones. It is better, however, and this is most often done, that the raw lead chalk is passed through one stone and that the fine grinding is done in some other set of stones, which are turned simultaneously, so what comes off the first stone is scooped onto the second, from there onto the third and fourth stone and the grinding takes place evenly and uninterrupted. If soft lead white is made, then one can act the same way both with pure lead white and with mixed types until the finishing; but to prevent it from becoming hard, careful washing is inevitable. For rinsing one needs a number of large vessels, the larger, the better. These vessels are filled with water, the ground lead white is added in and shaken very well, which is very difficult, because it does not dissolve easily. Then it is left to settle, until the liquid is clear and can be drained off. This stirring with fresh water, settling and draining is repeated several times. Then the very thick lead white pulp is placed in a filter, then in presses, to remove the water and is then in irregular chunks left to dry in air or in drying stoves, depending on the equipment available.
Grinding to soft lead white can however also be executed in another manner, which facilitates rinsing and dividing. Because the lead chalk is ground with a lot of water, the ground material does not move like thick porridge, but as an easily movable liquid through the stones and leaks out by itself from the openings in the centres of the base stone. In this case the grinding stones can be easily set up in the following manner: they are placed around the vertical shaft like steps, so that whatever runs off the first stone, directly enters the second through a channel and then flows into the third and fourth and from there to the water removal vessel, in which the lead white, as explained before, is rinsed. This is the usual set-up of the German mills. During rinsing in large factories a substantial amount of lead is lost yearly, which is washed out and drains off with the washing water and lead acetate, and is too dilute to be further usable. This loss can amount to some percents. To prevent this, one can do two things. One adds to the first water, with a yellowish or white lead wash, another ‘Maß’ of a crystal soda solution, which results in a precipitation, in which the dissolved lead settles as carboxylic lead oxide. Or – which is the best method – one directs all the drainage wash water into a slake, to which an excess of chalk or carboxylic chalk has been added, where the lead then settles bit by bit as lead white and calcium carbonate dissolves in return. After a long time one examines the contents of the slake and it is possible that it contains the most easily pure white lead has accumulated inside, and then feeds it again.

Das Vernahlen kann man, wie schon gesagt, auf zweierlei Weise vor sich gehen, je nachdem hartes oder weiches Bleiweiß dargestellt werden soll. Wird hartes dargestellt, so kommt der Teig des Bleikalks oder das mit Wasser angerührte trockene Pulver des Bleiweißes auf die Mühle, während sich die Steine gewöhnlich so bewegen, daß sie in der Secunde 1 1/2 - 1 Umdrehung machen. Man schöpft den Brei mit Hilfe eines langgestielten kupfernen Löffels in die kleine Sarge des Läufers und gibt so viel auf, bis die Sarge voll ist. Es dauert dann einige Zeit, bis deren Inhalt niedergedunkelt ist, worauf man wieder nachfüllt, bis nach und nach eine Partie, z. B. 10 Centner durchgerinnt ist. Ein arbeiter kann 4 bis 5 Steine, die neben einander stehen, betreiben. Was in der der Sarge des Bodensteins sich von Gemahlenem ansammelt, wird durch ein Schaufeleichen ofters nach vorn in den Ablauf geschafft, wo es in einen kleineren Ständer fällt. Bei dem ersten Mahlen ist es häufig nötig, den Stein etwas höher zu stellen, denn weil die Masse sehr dick wird, läßt sie sich nicht gut unter und durch die beiden Steine. Wann Alles durchgemahlen ist und wenn das Gemahlenem einen zären Brei darstellt, so wird der Stein durch Nachspülen von Wasser rein gemahlen, die Bodensteinmarge auch ausgewaschen, wozu man sich eines groben Haarpinsels oder einer Bürste bedient, und nun wird derselbe Brei noch einige Male auf eben dieselbe Weise bei enger gesteilet Steinen hindurch- und feingemahlen. Besser ist es jedoch, und so wird es auch meistens gehalten, daß man den rohen Bleikalk stets einen Stein passieren lässt und das Feinmahlen auf einigen anderen Gängen, die zu gleicher Zeit in Bewegung sind, ausgeführt wird, dann wird dasjenige, was vom ersten Steine kommt, auf den zweiten, von da auf den dritten und vierten Stein geschöpft und das Mahlen geht dann regelmäßigen und unterbrochen fort.

Soll weiches Bleiweiß dargestellt werden, so kann man zwar sowohl bei reinem Bleiweiß, als den gemischten Sorten bis zum Fertigmahlen eben so verfahren; aber damit es sich nicht hart werde, ist ein sorgfältiges Auswaschen unumgänglich erforderlich. Zum Auswaschen hat man dann eine Anzahl grosser Ständer nötig, die um so besser, je grösse sie sind. Diese Ständer fällt man mit Wasser, bringt das gemahlene Bleiweiß hinein und rührt es wohl auf, was aber schwer ist, weil es sich nicht gut zertheilt. Dann lässt man es absitzen, bis die Flüssigkeit klar ist, welche man darauf ablaufen lässt. Dieses Aufspüren mit frischem Wasser, Absitzenlassen und Abziehen wiederholt man einige Male. Als dann bringt man den sich sehr dick ansammelnden Bleiweißbrei in Filter, von da auf Pressen, um das Wasser zu entfernen und dann in unregelmäßigen Stücken zum Trocknen an der Luft oder in Trockenstuben, je nachdem dazu die Einrichtungen vorhanden sind.

Das Vernahlen zu weichem Bleiweiß kann aber auch von vorn herein auf eine Weise erfolgen, wodurch das Auswaschen und Zertheilen leichter vor sich geht. Der Bleikalk wird nämlich mit viel Wasser vermahlen, so dass das Gemahlene nicht als dicker Brei, sondern als eine leicht bewegliche Flüssigkeit durch die Steine und von selbst aus den Öffnungen der Sarge um die Bodensteinen herausläuft. In diesem Fall ist leicht eine solche Anordnung der Mahlgänge möglich, so daß sie um das vertikal stehende Triebrad herum stufen- oder treppenweise aufgestellt sind, so daß dasjenige, was vom ersten Steine abläuft, direkt durch eine Rinne zu dem andern Stein tritt und zu dem dritten und vierten von da in die Abwässerungsstanden, worin das Bleiweiß, wie vorhin schon erwähnt, abgewaschen wird. Dies ist die gewöhnliche Anstellung der deutschen Mühlen. bei diesem Abwässern geht in grösseren Fabriken eine nicht unbedeutende Menge Blei jährlich verloren, welche ausgewaschen wird und mit dem Waschwasser als essigsaures Blei davon läuft, und zwar in einer Verdünnung, wo es kaum benutzt werden kann. Dieser Verlust kann bis einige Procente ausmachen. Um ihm zu entgehen, kann man auf zweierlei Weise verfahren. Man setzt dem ersten Wasser, womit das Bleiweiß aufgerührt worden war, einige Maß von einer Lösung von krystallisirter Soda hinzu, welche einen Niederschlag erzeugt, indem sie das gelöste Blei fällt, und gleichfalls als kohlensaures Blei oxydirt ist. Oder man läßt, was jedenfalls das beste ist, alle die fallenden Waschwassere in einem Sumpf laufen, worin ein Liebeswick von Kreide oder kohlensaures Kalk gebildet worden ist, wo dann das Blei sich nach und nach als Bleiweiß ausgeschieden und kohlensaures Kalk dafür in Lösung geht. Nach längerer Zeit untersucht man den Inhalt des Sumpfes und entleert und beschickt ihn von neuen, wenn sich ein eingerieven brauchbarer, wenn auch nicht ganz rein Bleiweiß darin angesehemmt hat. 


63–72 The next dry grinding is, in the majority of cases still effected under vertical zones, rolling upon a horizontal bed. The ground lead is then shovelled into a cylindrical metallic sieve with fine holes, and enclosed in a wooden box. The powdered white lead is then fed into the bottom of the box, and the small flattened particles of metallic lead, fall from the lower end of the sieve into a special receiver. The sifted white lead is mixed with water, and thoroughly ground under mill-stones.

[English factories: The white lead is also deprived, by washing, of certain soluble salts which may injure its purity; moreover the subsequent operation is always effected with the aid of water.}
The settled carbonate of lead is first washed with a small proportion of water, which is added to the decanted solution of acetate. The washing is then continued with larger quantities of water, which are thrown away, since they are too poor in acetate. The paste of white lead is put into pots, and dried in the stove room...

... After a certain length of time, the settling tank is sufficiently filled with white lead, that is, when this material reaches the level of the overflow. The solution is then made to pass into other vessels and the white lead is washed in washing tanks, which are provided with wooden horizontal stirrers having a rotary motion. The settled white lead is covered with twice its volume of pure water and stirred. Three washings take place, and at each, the material is allowed to deposit, and the water above is decanted. The white lead is then conducted into large basins built of porous stones, which absorb part of its dampness. After a few days, the material is divided into blocks which are still quite wet, and which are pounded by wooden vertical stamps falling into a wooden trough inclined from the front backwards.

Table 1 Historical recipes for washing stack-process lead white cont'd.

78–85: The French or Clichy Process, by Thénard
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The washed lead white is then ground between horizontal mill stones of 80 to 100 cm diameter (exactly in the manner of the flour mill), under continuous flow of water. Only so called French stones are used as mill stones. There are a number of mills for the best quality and some others for the [lead white] which has remained in the chamber too long, which does not turn out so white. After finishing the grinding, a water jet removes the lead white, except the coarsest parts, from the stones, after which it flows into the settling troughs. After some time the water is pumped off and the lead white cakes are removed.


Water washing

A particularly detailed description of how to wash lead white is provided in a recipe entitled ‘To prepare white excellent’ recorded by Daniel King (1653–57: 38–9):

Take a good quantity of Seruse, and being grosly braised put it into a fine earthen bason or great China dish then put it to a good quantity of running water wherein wash the Ceruse till it be thoroughly mixed with water. Then let it settle 2 or 3 hours which done, tast the water you shall find it tast harsh and unpleasant. Observe well the tast and poure the water off and cast it away. You shall find this water to carry away with it a deale of grease rising on the top. Then add to as much more water, and then stirre the ceruse as before and tasting it poure off the cleare water reserving the bottome. This work you must reiterate some dozen times or more till by continuall washing you find your water hath washt away all the salt out of the Ceruse.

The salt mentioned by King most probably consisted of lead acetate. During water washing of lead white carried out by the HART project, lead acetate levels were monitored; after numerous water changes lead acetate was no longer detectable. An overview of water-washing recipes within the period under investigation (Table 1) shows that the main methods used were either washing with large amounts of clean water, repeated grinding with water on the slab, or a combination of both treatments. Comparison of XRD analyses of unwashed and water-washed lead white confirmed the findings of the

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**Figure 4** SEM BSE image of stack-process lead white (a) ground in water, paint prepared with poppy oil, (b) ground in vinegar, paint prepared with poppy oil.

**Figure 5** Lead white and chalk with a drop of wine vinegar: gas bubbles have formed.
To render *biaccia* extraordinarily more white. Take lead white in flakes, choose the most beautiful, and grind well on the marble with vinegar, and [it] will become black; then take an earthenware beaker full of water, and wash your white well, then let [it] settle well at the bottom, and pour off the water by inclination. Return to grinding with vinegar and wash; and having done this approximately 3 or 4 times, you will have a *biaccia* that will be perfectly beautiful for miniature painting, as well as oil painting.

To rendre la *biaccia* plus blanche extraordinai rament. Prendete *biaccia* di piombo in scaglie, elegete la più bella, e macinatela bene sul marmo con aceto, e diventar nera, allora prendete un vaso di terra piena d’acqua, e lavate il vostro bianco bene, poi lasciatelo bene dur in fondo, e verrà l’acqua per inclusione. Tornatela a macinare con aceto et a lavare; e fatta questa operazione medi.- 3 o 4 volte, che haverà una *biaccia* che sarà *perfettamente* bella tanto per miniare, quanto per dipingere a olio.

Ahomen verwen wrijven wil zo neenst eerst wat loot-wit om den steen want zuyver te maken en als .grewven is zo dooeter wat indigo bij, want dit wit dan niet suyver genoeg is om alleen te gebruiken, maar na neenst ander loot-wit en die zal zuyver zijn. Til ket gij wrijven met water, en laeten droogen en temperen met net olj of lijn olj. Loot-wit wrijften met pis of asijn et alse gedroogen is en gij die wilt gebruiken zo tempertse met olj.

Prenez du blanc de plomb en écailles, choisissez le plus beau, et broyez bien ces écailles sur la pierre, avec du vinaigre, et il deviendra noir; alors prenez une terrine pleine d’eau, et laissez bien votre blanc, puis le laissez bien rassoir, et versez l’eau par inclinaison; broyez-le encore avec du vinaigre et le relavez, faisant cela trois ou quatre fois, et vous aurez un blanc qui sera parfaitement beau tant pour l’emulsion que pour la peinture à hâle.

Table 2 Historical recipes for processing lead white with vinegar.

<table>
<thead>
<tr>
<th>Author</th>
<th>Date</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Barrow, J.</td>
<td>1735</td>
<td>Traité des couleurs matérielles, Paris: Sauigrain &amp; Lamy [repr. 1737, Geneva, Minkoff], 5–6</td>
</tr>
<tr>
<td>Buc’hoz, M.</td>
<td>1786</td>
<td>Recueil de secrets sur et expérimentés a l’usage des artistes (no. of vols not known), vol. 3. Paris: by the author, 293–2</td>
</tr>
</tbody>
</table>

To prepare white excellent...

...Some say it is better, if some rectified spirits of wine be pour’d on it, which will clear it from any dross that may be in it; this (as it is very probably) must be pour’d off, when the spirit of wine has done its work.

To rendre lead white extraordinarily fine. Take lead white in flakes, choose the most beautiful, & grind these flakes well on the stone, with vinegar, & it will become black; then take an earthenware ter rine full of water, & wash your white well, then let settle well, & pour off the water by inclination; grind again with vinegar & earthenware again, doing this three or four times, & you will have a white that will be perfectly beautiful both for miniature painting and for oil painting.


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As noted above, besides water washing, artists were often advised to grind or wash lead white with vinegar before use. Recipes generally specified thorough water washing afterwards to remove all traces of this acid. Some recipes described how the lead white would discolor and become black during vinegar grinding. This blackening would disappear after washing with water. Several recipes mentioned heating lead white with vinegar as a refining method. Barrow (1735) stated that rectified vinegar caused 'fermentation' of the lead white (presumably the formation of gas bubbles). Table 2 provides an overview of recipes for washing with vinegar that were published within the period under investigation. Although the number of recipes prescribing vinegar washing is lower than those for water washing, it is evident that vinegar is mentioned frequently throughout the time period under investigation.

### Table 2: Historical recipes for processing lead white with vinegar. contr’d.

<table>
<thead>
<tr>
<th>Author</th>
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<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anon.</td>
<td>1794</td>
<td><em>Wiener Farbenkabinett. Vienna/Prague: Verlage der von Schönfeldschen Handlung</em></td>
</tr>
</tbody>
</table>

### Bouvier, M.P.L. 1827.


One of my friends has told me of a procedure to obtain a very pure white. I have not had occasion to test it myself, but I have every confidence in its truth and its faithfulness; besides this procedure, which is practised by others, carries with it everything that is required to convince of its effectiveness, being founded on principles that reason can only approve of.

Take, for instance, a litre, more or less, of good Krems white, subject it, in small batches, to a first grinding with water, without too much concern that it becomes very fine: you take it off the stone in the thickness of a strong cream, and you take care that the first batches do not dry, but that they remain a little liquid. To this end you put every batch of colour in a new well glazed pot, or in a faience bowl, or of pipe clay. When all your white is ground and in a rather thick pulp, pour on top of this pulp the amount of a drinking glass of very good distilled white vinegar; you stir and stir it every hour, and this during one day; you take, to stir it, a new pipe, which the acid of the vinegar does not attack. If your vinegar is good and well distilled, it will remove all little strange particles that may be present in the white, in a manner that nothing remains that can damage your colour. This colour thus prepared provides a very perfect white; but you have to take care to remove all the vinegar by repeated washings, and until the water that floats on top of the white (when you have allowed it to settle) has no more acid taste taking it on the tongue.

The white ground again in pure water, at least three times, has a superior quality; used also with gum water to paint in gouache, it is light, very subtle, and does not turn at all on paper, like happens to Kremnitz white that has not been subjected to this operation.

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Take, for instance, a litre, more or less, of good Krems white, subject it, in small batches, to a first grinding with water, without too much concern that it becomes very fine: you take it off the stone in the thickness of a strong cream, and you take care that the first batches do not dry, but that they remain a little liquid. To this end you put every batch of colour in a new well glazed pot, or in a faience bowl, or of pipe clay. When all your white is ground and in a rather thick pulp, pour on top of this pulp the amount of a drinking glass of very good distilled white vinegar; you stir and stir it every hour, and this during one day; you take, to stir it, a new pipe, which the acid of the vinegar does not attack. If your vinegar is good and well distilled, it will remove all little strange particles that may be present in the white, in a manner that nothing remains that can damage your colour. This colour thus prepared provides a very perfect white; but you have to take care to remove all the vinegar by repeated washings, and until the water that floats on top of the white (when you have allowed it to settle) has no more acid taste taking it on the tongue.

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Table 3: Historical descriptions for pigment decanting or other methods of particle size election.

<table>
<thead>
<tr>
<th>Author</th>
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</tr>
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<tbody>
<tr>
<td>Mayerne, T.T.</td>
<td>De Picturae &amp;qve subalternarum artium</td>
<td>Lead white ground first with water, then washed &amp; allowed to settle, decanting the turbid water, makes a deposit that is very beautiful, &amp; dies less than the deposit.</td>
</tr>
<tr>
<td>King, D.</td>
<td>Secrets in the noble arte of miniatura or the art of limning</td>
<td>Le blanc de plomb broye premièrement avec eau, puis lavé &amp; laissé rassee, en decantant l'eau trouble, fait une résidence qui est tres belle, &amp; meurt moins que le fonds.</td>
</tr>
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</table>

Mr. Hilliards manner of preparing Ceruse.

Having ground your ceruse in water without gum & put it into a vial glass with a good quantity of faire water, and being well shaken together let it stand a while, and before it be settled pour off the third part of the water and let it settle. Then pour yet likewise one other third part out of the vial and reserve the last part in the vial still. This water third divided into three parts let stand still till all be settled and the water cleared, then dry it or make the colour being now settled in the bottome dry by evaporating. Then in tempering it with gumme use it at your pleasure.

Anon. | 1668. | The excellency of the pen and pencil. |

As in the grinding of colours I gave you an instance but in one for the rest, like I shall do for those colours which are to be washed; I will make my instance in red-lead, which you are to Wash in this manner.

Put a quantity thereof into a clean earthen dish, and pour thereto fair water, stirring the colour and water together with your hand or otherwise, then let it stand a while, and you shall see a filthy greasi scum arise and lie above the water, with other filth; pour this water quite away, and put other clean water to the colour, and stir it about again, pouring away the water (if foul) the second or third time; then add more water, and stir the colour about again till the water be thick and troubled; but yet free from filth, then gently pour this troubled water into a second earthen dish, leaving in the first dish all the drags. Into the second bason put more fair water, and with your hand stir about the colour as before; do thus two or three times, and take (if your colour be very foul) a third earthen dish, and add more water, and keep stirring, till at last the water become clear, and the colour remain fine at the bottom of the dish; pour away your water gently, and you will find some colour remaining and flicking to the edges of the dish, which when it is dry, you may (with a feather) strike away like flower, which reserve as the choicest and purest of all; if in a pound of this red-lead you have an ounce of good indeed, prize it, for it is troublesome to procure. The other, which is not altogether so pure, may be serviceable for some uses, though not for all. What hath been said of this colour is to be understood of all other that are to be washed; therefore for washing of colours let this suffice.

Anon. (A.P.S.) | 1770. | Nauwkeurige beschryving van het schilderen der zwarte konstprinten. |

One crushes the flake white in a marble mortar with an iron pestle into a coarse powder. On top of this one pours water, until it becomes a pulp: then grind it further with a stone or glass pestle for some time, until one notices, that it has largely been ground fine. Then a larger amount of water is added, and, after having stirred everything well, let it stand 15 to 20 counts, after which one pours out the liquid with the fine particles that are still retained inside it into a sugar or bowl water. The settled more coarse part is ground again and treated in the same manner. And this grinding, pouring on and off is repeated until some blackish, or brown coarse particles remain. The decanted water is left until all the materials have settled; upon which one pours off the superfluous water, and lets the remaining drip through a funnel of blotting paper: finally one dries the remaining pulp between papers. The longer one lets the material settle in the beginning, the more fine the particles will be: but this time is long enough. Anyone who is somewhat used to this work can grind as much as a pound of this white very fine in 2 hours. One may without any fear use this manner of fine-making for all earthy paint materials.

Men stoot het schelpwit in een maarmere mortier met een eyere zyere stamper tot een grof poeyer. Hier op giet men water, tot het een pap werde: dan wryst men verders met een steene of glaaze stamper eenigen tyd lang, tot men bemerkt, dat er een goed deel fyn gewreven is. Hier op giet men er by een ruime hoeveellheid waater, en, naa alles wel omgeroerd te hebben, laat men t 15 à 20 tellen staan, waarna men t vegt met de daar noch in opgheboden wordende fyne deeltjes in een zuiker glas of kom overgiet. Het gezakte grovere deel wort wederom gewreeven, en op dezelfde wyze behandeld. En dit wrywen, op-, en afijten herhaald men zo long tot er eenige zwartagtie, of bruine grooje deeltjes te rug blyven. Het afgegostene water laat men staan tot alle stoffe geraakt zy; wanneer men theroverlievde water er afijtet, en t overige door een tregter van lek papier er laat uitdriepen: eindelyk droogt men de overgeblevene pap tuschen papieren. Hoe langer men in den beginne de stof laat zakken, dies te fynder zullen de deeltjes zyn: doch deze bepaalde tyd is lang genoeg. Die eigensins aan deze behandeling gewend is kan in 2 waren tyds wel een pond van dit wit fyn wryven. Men mag zonder eenige vreyde wyze van fynmaaking by alle aardagtie verwijste niet in, t werk stellen.

Le Pileur d'Apligny 1779. | Traité des couleurs matérielles. |

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Le Pileur d'Apligny 1779. | Traité des couleurs matérielles. |

Anon. | 1794. | Wiener Farbenkabinet. |

Method to wash raw colours.

The traders or manufacturers of pigments wash them in large cases or wooden jars, in which there may enter hundreds. They use two taps, at different heights, to first let run off the first waters, on which float all the light materials; then another, lower down, to separate the good colour from the sediment, which is filled with stones and other heavy materials.

Manière de laver les Couleurs brutes.

Les marchands ou fabricans de couleurs les lavent dans de grandes caisses ou jarres de bois, où il en peut entrer des quintaux. Ils pratiquent deux robinets, à différentes hauteurs, pour laisser d'abord écouler les premières eaux, sur lesquelles surnagent tous les corps légers; puis un autre, plus bas, pour séparer la bonne couleur davec le sédiment, qui est rempli de pierres et autres corps pesans. cont'd.
While the lead white formed in this operation [= of Krems white] has not been allowed to obtain the hardness of that obtained by the Dutch method [= stack process], it is not necessary to grind; one obtains it very well divided by levigation, by means of a very simple installation. It is composed of a large case that contains nine cases or compartments, of decreasing height. One puts in the most raised case the flakes of lead white, separated first from the metal that has not been attacked. One then enters water from a higher reservoir, and one stirs strongly with a rake: the water overflows quickly, runs into the second case, then into the third, and arrives like this successively in the ninth.

One understands that the lead white molecules, taken along by the water, are the more thin the more far they have been carried. Which is found in the lower case is thus the first quality white. One empties the cases into large vats, where the deposit is formed in more or little time: one then collects it, and when it has drained sufficiently, one puts it in earthenware vases, where it dries and takes the shape of square loaves.

**Table 3** Historical descriptions for pigment decanting or other methods of particle size election cont'd.

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**Figure 6** SEM BSE image of paint made with lead white and chalk, water washed, paint prepared with poppy oil. False colour image with all lead counts in red, and all calcium counts in green: (a) first ground with water, and (b) first ground with vinegar (See Plate 41 in the colour plate section.)
To prepare the test materials do not reveal apparent changes in their visual characteristics (Fig. 6a and b).

Decanting

Pigment washing can be used for particle size selection through gravitational sedimentation: when suspended in a liquid, heavier particles settle more quickly. Empirical knowledge of this process was evident in several 17th-century recipes (Tables 2 and 3). The methods described were simple: the pigment was suspended in water by stirring and the top part of the liquid was decanted before all particles had settled. Decanting was advised for lead white and earth pigments, among others. Decanting or sedimentation systems were developed further in an industrial setting during the 19th century (Table 3). There, pigment was suspended in water and flowed through interconnected vessels of different heights. The heaviest particles settled first and the lighter particles travelled farthest.

Our experiments showed that sedimentation is very successful in separating smaller lead white particles from larger pigment particles or aggregates. A small amount of ground lead white was suspended in a large volume of distilled water. The liquid was stirred and the pigment was allowed to settle for two minutes. The top half of the liquid was then poured into a second beaker. There, the pigment was allowed to settle further. After two hours, half the water was transported into a third beaker, using a pipette in order not to disturb the lead white that had settled near the bottom. After drying, all lead whites were ground with poppy oil and applied to Melinex.

A distinct difference in so-called particle size is immediately evident in the SEM BSE images (Fig. 7a and b). The decanted lead white bears a resemblance to the fine lead white used in the paint layers of Vermeer’s *The Art of Painting*, which strongly suggests that such fine particles were selected through decanting. XRD showed that the fine lead white contains a higher proportion of cerussite than the deposit (Fig. 8). This demonstrates that decanting not only separates larger and smaller particles, but that the smaller particles are of a different composition from larger, lead white particles or aggregates. This difference may in fact explain the tendency of traditionally prepared lead white to aggregate.

If the XRD results for the dry pigment samples can be used as an indication of the composition of the lead white in paint, they appear to contradict the results of Welcomme et al. (2007), who found a higher ratio of hydrocerussite to cerussite in the paint layers as compared to the ground layers of paintings (dated between 1512 and 1516) by Matthias Grünewald.

Investigation of the change of composition of lead white in paint is ongoing. Yet unpublished results from the HART project appear to show that the ratio of hydrocerussite to cerussite is lower in lead white paint tempered with linseed oil than in the dry pigment powder from which it was prepared. The different observations on the ratio of hydrocerussite to cerussite might also be linked to the equilibrium between the two in water, which can easily shift to either side under ambient conditions (Godelitsas et al. 2003).

Conclusion and future research

Our reconstructions of water washing, vinegar grinding and decanting have provided more insight into the effects of these processing methods on visual characteristics of particles within paint cross-sections, as well as on their composition. The question of whether oil paints containing only very fine lead white were created by extensive grinding or by decanting can now be decided in favour of decanting. Our reconstructions have shown that the ratio of cerussite to hydrocerussite is lower in lead white paints tempered with linseed oil than in the dry pigment powder from which it was prepared.

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The present research may only be considered as a first step, since it has not yet addressed the consequences of these processing methods for paint handling and for long-term paint stability.

Are there any benefits to using lead white with a high proportion of cerussite? Colour measurements indicate that after two months of natural ageing, vinegar-treated lead whites have yellowed considerably more than water-washed
lead whites. A higher proportion of cerussite therefore does not necessarily create a whiter paint. Research by de Behault (2010) showed that small-sized lead white particles may have been selected by artists wishing to optimise blue-hued scattering effects, as seen for instance in the cool grey skin on blue grapes.

Yet another reason to post-process lead white may have been to enhance pigment stability. During the 19th century, authors considered hydrocerussite more reactive than neutral lead carbonate. Continued monitoring of the oil paints produced during the present research is required to compare the long-term stability of neutral cerussite and hydrocerussite within an oil binding medium. Different ageing characteristics are to be expected: the white top layer in the marble floor tiles of Vermeer’s The Art of Painting, created with very fine lead white, demonstrated severe delamination, which – for a large part – was ascribed to incompatibility of this layer with the more flexible lower layers created with pigments of a more diverse particle size (Boon and Oberthaler 2010: 236).

Acknowledgements

The authors thank the Dutch Organisation for Scientific Research (NWO) for sponsoring the HART project; Jef Seynaeve for providing stack-process lead white; Katrien Keune for analysis of the HART samples; Andreas Bilo for assistance with colour measurements; Jaap Boon, Elke Oberthaler and the Reproductions Department of the Kunsthistorisches Museum in Vienna for permission to use the image of the Vermeer cross-section; and Sigrid Eyb-Green and Emilie Froment for checking translations of the recipes.

Notes

1. Plumbonacrite is reported to transform easily to hydrocerussite and is not always detected in lead white samples (Olby 1966). Its presence was analysed with XRD by Hallebeek of the (then) Netherlands Institute for Cultural Heritage in lead white samples analysed for the HART project (Carlyle 2006: 50).

2. Aragon ion polishing of lead white oil paint cross-sections (Boon and van der Horst 2008), followed by ultra high magnification with SEM demonstrates that so-called large ‘particles’ of lead white are not solid material but are composed of aggregates of small particles.


4. Produced by Jef Seynaeve, Belgium, for the HART project.

5. Distilled water was added to previously crushed lead white. Pigment and water were stirred for two minutes. After settling of the pigment, supernatant water was poured off and replaced with fresh distilled water 8–10 times. This method closely follows the one used by the HART project (Carlyle 2006: 25–6).

6. 50 ml of vinegar culture (Brouvland of Everlo, Belgium, purchased 18/9/2010) was added to 500 ml sulphite-free organic red wine (Stellar Organics, Shiraz, South Africa, imported from Coenecoop Wine Traders B.V., 2742 RC Waddinxveen). The liquid was allowed to acidify during four weeks in a cool, dark place. The resulting vinegar had a slightly pink colour and a pH of 3–4 (Macherey-Nagel pH-Fix 0–14).

7. Poppies oil produced by the HART project; see Carlyle (2006) for details.

8. The crystalline phases were analysed by XRD using a Discover D8 microdiffractometer with a general area detection diffraction system (GADDS) two dimensional detector (Bruker AXS, Karlsruhe, Germany). Powdered samples were applied in a little
cedar oil on a Silicon zero diffraction plate. Diffractograms were acquired in reflection mode with CuKa radiation (40 kV, 30 mA). The GADDS software was used for integration and the Bruker AXS Eva software for phase identification using the PDF database.

9. JEOL 5910LV with thermo scientifique energy-dispersive X-ray spectrometry system with Noran System Six software.

10. Konica Minolta CM-2600d spectrophotometer, white calibration 7004450.


12. The settling speed of particles depends not only on their mass and density, but also on their shape.

13. Earlier experiments, repeated during the present research, ruled out the possibility that very fine grades of lead white could be produced by extensive grinding on the slab (Carlyle 2006).

14. Procedure according to ASTM 2001 standard to determine the Yellowness Index (YI) E313:73; illuminant D65 (daylight, colour temperature 65°), 10° observer (CIE 1964), three automatically averaged measurements in three different locations. Vinegar-treated lead whites had a ΔYI roughly twice as large as the water-treated lead whites.

15. Mulder 1865: 302–3; Carlyle 2001: 260. Experiments undertaken by Rhodes and Van Wirt (1923) showed that slightly basic lead carbonate retarded the initial oxidation of linseed oil and did not promote final oxidation as much as a more basic lead white.

References


Authors’ addresses

• Maartje Stols-Witlox, University of Amsterdam, Ateliergebouw, Hobbeimastraat 22, 1071 CZ Amsterdam, The Netherlands. (m.j.n.stols-witlox@uva.nl)

• Luc Megens, Cultural Heritage Agency of the Netherlands, Ateliergebouw, Hobbeimastraat 22, 1071 CZ Amsterdam, The Netherlands. (l.megens@cultureelerfgoed.nl)

• Leslie Carlyle, Department of Conservation, Faculty of Sciences and Technology, New University of Lisbon, Caparica Campus, Caparica 2829-516, Portugal. (L.Carlyle@btinternet.com)