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Stols-Witlox, M.J.N.; Megens, L.; Carlyle, L.

Published in:
The artist's process: technology and interpretation

Link to publication

Citation for published version (APA):
Stols-Witlox, M., Megens, L., & Carlyle, L. (2012). 'To prepare white excellent...': reconstructions investigating the influence of washing, grinding and decanting of stack-process lead white on pigment composition and particle size. In S. Eyb-Green, J. H. Townsend, M. Clarke, J. Nadolny, & S. Kroustallis (Eds.), The artist's process: technology and interpretation (pp. 112-129). London: Archetype.

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Download date: 21 Feb 2019

‘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 ($PbCO_3$) and hydrocerussite ($2PbCO_3.Pb(OH)_2$); other lead salts such as plumbonacrite ($Pb5O(OH)_2(CO_3)_3$) may

ATSR 4 15 Stols.indd 112 17/01/2012 10:18

also be found on occasion.[1] 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).[2] 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.

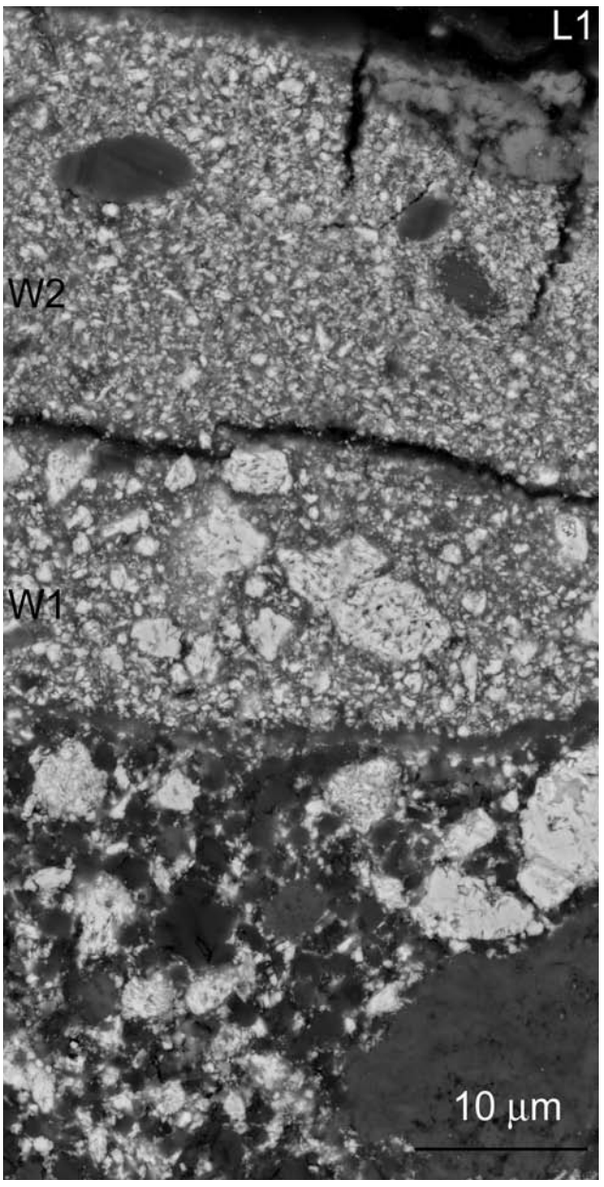

Figure 1 SEM BSE image of a cross-section from the tile floor in Vermeer's *The Art of Painting*, *c.*1666–68, 120 × 100 cm., oil on canvas. Kunsthistorisches Museum, Vienna. Bottom layer: ground with lead white particles of diverse size, calcium carbonate and earth pigments. Second layer: paint layer consisting of lead white of different size. Third layer: paint layer containing very fine lead white and larger bone black particles. Fourth layer: organic palmierite-containing deposit.

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.[3] 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,[4] thoroughly washed with distilled water.[5] Vinegar for grinding with the lead white was prepared from organic, sulphite-free red wine and vinegar culture (bacteria).[6] Lead white pigments, further

ATSR 4 15 Stols.indd 113 17/01/2012 10:18

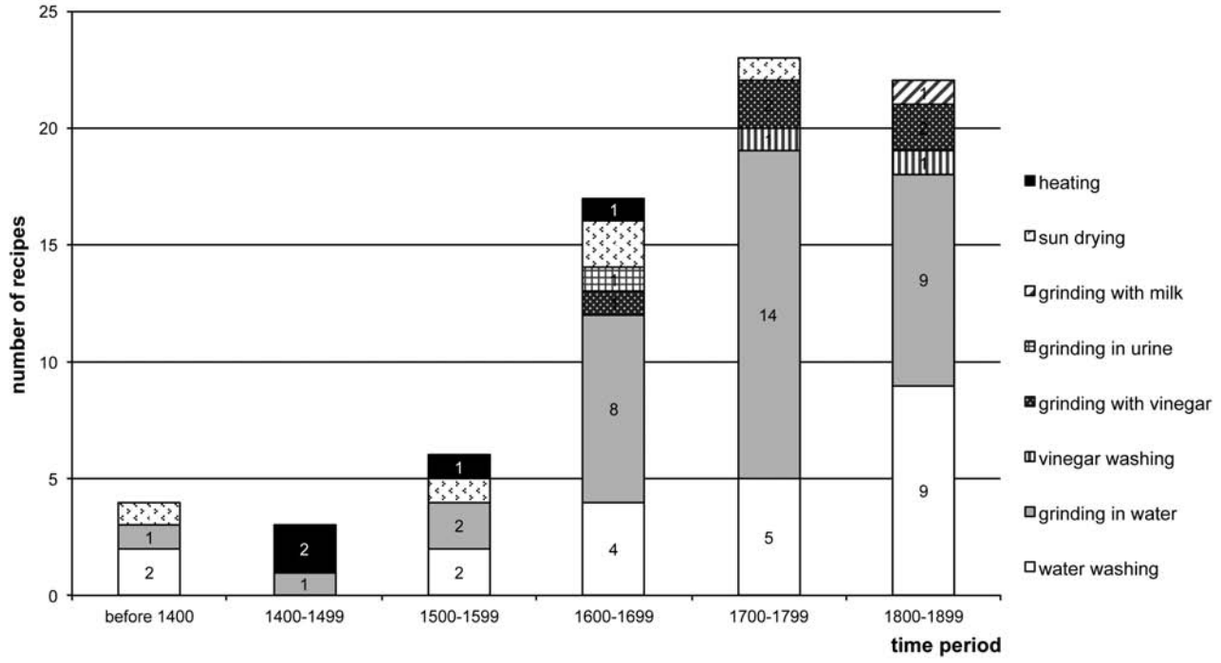

Figure 2 Occurrence over time of recipes specifying different methods for washing and grinding lead white.

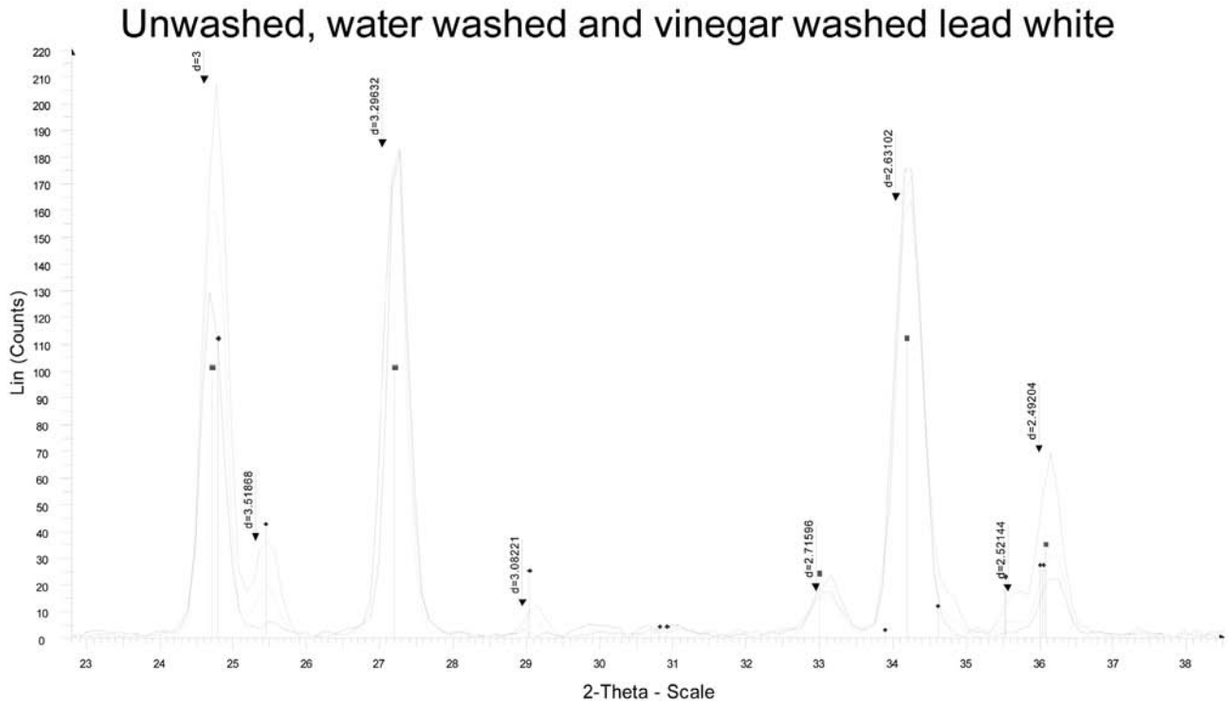

Figure 3 Diffractogram with unwashed, water-washed and vinegar-washed lead white. The position of cerussite peaks is indicated with blue vertical lines, the position of hydrocerussite peaks is indicated with red vertical lines. An increase in cerussite proportion is clearly visible in, for instance, the 25,4 position. (See Plate 40 in the colour plate section.)

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.[7] 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),[8] and the oil paints were then analysed in cross-section with SEM-EDX.[9] Colour measurements were made on the paint films using a Konica-Minolta spectrophotometer.[10]

ATSR 4 15 Stols.indd 114 17/01/2012 10:18

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

Pomaro, G. 1500–1600. *I ricettari del fondo palatino della bibliotheca nazionale centrale di Firenze*. Milan: Giunta Regionale Toscana Editrice Bibliografica, 98 (Pal. 796: c. 5v)

To purify *biacca* very fine with water. First grind the *biacca* very soft with water; then wash 3 or 4 times and leave to dry on the stone, then gather and keep in paper.
Ad conciare la biaccha sottilissima con aq(u)a. Prima macinerai la biaccha suttilisima con aqua; poi la lava 3 o 4 volte et lassala secchare in su la pietra, et poi la tolle et serba in una carta.

Andriessen 1552. *Viervoudig tractaetboeck*. Kampen: Steven Joessen, 55

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.
Lootwit wryvtmen op enen harden steen met schoon water ende wilment schoon hebben so alst wel ghewreven is laetmen uit ghespreyt ligghen op die harde wrijfsteen en besprengent met schoon water ende latent soe ligghen ende bleken in die heete son ende alst droogh is salment weder vochtich maecken op den steen ende wrijvent een poes ende laetent wederom drogen ende bleken tot vier oft vijf reysen toe. Dat is tschoonste wit datmen kan ghevijnden behouden dat ghi eerst wel uit gelesen hebt tblauwe loot datter in valt int wrijven dat moetmen altijt wel waer nemen ende uit doen oft dat Lootwit souder werckelijck af smetten.

Birelli, *c.*1601. *Opere di Giovanbatista Birelli* (no. of vols unknown), vol. 1. Florence: Giorgio Marescotti, 363

Then pound it, & put it in a *pignatto*, & for every *libra* of this flour [= lead white], add two ounces of water, & put on the fire while stirring it well together, leave on the fire until smoke rises, then take it from the fire, & put it in the shape you like, & put it in the sun, leave until it has dried well, then remove it from the mould, & put it in straw which will make fine *biacca*.
doppo lo pestate, & lo mettete in un pignatto, & per ogni libra di questo fiore [= lead white], mettete oncie due d'acqua ,& ponetelo al fuoco stemperandolo bene insieme, lo farere star tanto al fuoco che levi il fumo, poi cavatelo dal fuoco, & mettetela nella forma che volete, & ponetelo poi al sole, lassandovelo fin che sia ben'asciutto, Finalmente lo traete dalla forma, & mettetela nella paglia che farà Biacca fina.

Symonds 1984 [1649–51]. 'Secrete intorno la pittura', in M. Beale (ed.), *A Study of Richard Symonds: His Italian Notebooks and their Relevance to Seventeenth Century Painting Techniques*. London/New York: Garland, 225

Biacca ceruse, white.
When S. G. A. was to finish the quadro of Cleopat. A. M. ground Biacca that is whyting wth water very well, then to take out the water ground it in Olio di lino washing it often, pouring on water constantly. This was to purify the Biacca & take out all the lead of wch tis made.

Veen, J. van c. 1650. 'De wetenschap ende manieren om alderhande couleuren van say of sayetten te verwen'. The Hague: Royal Library, Ms. 135K44, 119

Lead white is ground with clean water, if you want it beautiful so leave it to bleach on the grinding stone, strewn 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;
Loodwit wrijftmen met schoon waeter, wilt gij't schoon hebben soo laedt het op een wrijfsteen bleijcken, uijt gestroijt ende bespronght met schoon waeter inde heete sonneschijn ende aens ,t droog is salmen ,t wederom een weijnig natten ende wrijven en laeter ,t wederom bleijcken en droogen tot ,t op 5 mael toe;

King, D. 1653–57. 'Secrets in the noble arte of miniatura or the art of limning'. London: British Library, Ms. Additional 12461, 38–9

To prepare white excellent
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 and 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 and when you perceive that the water taste fresh as other water, then let it dry or setting the bason upon a soft fire, let the residue of the water vaporise, and the ceruse being drie use of the fairest and reserve for yr use. Probatissimum

Anon. 1668. *The excellency of the pen and pencil*. London: Dorman Newman, Richard Jones, 70

Of those Colours that are to be Grownd, and how to Grind them.
I Have in the last Section told you what Colours are to be Grownd, which Washed, and which to be Steeped; it resteth now, to shew you how to Grind those Colours that are to be grownd; I shall not run over every Colour particularly, but shew you how to grind one Colour, which take as an example for all the rest.
For our example in grinding of Colours, let us take the original of Colours, viz. Ceruse, or White-lead, there is little difference between them, only one is refined, the other not.
To grind it do thus; Take a quantitie thereof, or of any other Colour to be grownd, being cleansed from all manner of dirt or filth, which you must first scrape off; then lay the Colour upon your Stone, and with your Muller bruise it a little, then put thereto fair spring-water, and between your Muller and Stone grind the Colour well together with the water till the Colour be very fine, which when you find to be enough, have in a readiness a great Chalk-stone, in which make certain troughs or furrows, into which pour your Colour thus grownd, and there let it rest till it be thorough drie, then take it out ans reserve it in papers, and those papers in boxes, for your use.

cont'd.

ATSR 4 15 Stols.indd 115 17/01/2012 10:18

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

Beurs, W. 1692. *De groote waereld in het kleen geschildert*. Amsterdam: J. and G. Jansonius van Waesberg, 8
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. *Daar toe dan kiestmen uit de dikste Schulpen, als de beste, die men in zeer schoon en helder water vrijft op een goede keysteen, die niet te hart of te glad wezen kan; en alsze is gewreeven, zet menze op een stuk van een glas, daar op het water uit droogen moet. Als ze nu droog genoeg is, vryftmen die in de beste papaver oly, die de Noot-oly, Lijnsaat-oly en andre bekenden overtreft; waar na menze in een rein Schulpie of kommetje doet, en er schoonwater op zet, om niet te verdroogen; en dus kanmenze goed houden; om als ,t noodig is, gebruikt te [.....] ,T Goede ongemalen Lootwit bereidmen op de zelve wyze, als het Schulp-wit; dog ,t is gemakkelijker om vryven;*
Smith, M. 1692. *The art of painting according to the theory and practise of the best Italian, French, and Germane masters*. London: Vendüe, 71
The instruments and Materials us'd in Painting and the preparing Colours to the pallat: Lead White White Flakes being pick'd and scrap'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 Lynseed Oyle.
Eikelenberg, S. 1679–1738. 'Aantekeningen'. Alkmaar: Municipal Archive, Mss. Eikelenberg
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. *161: Alsmen verwen wrijven wil zo neemt eerst wat loot-wit om den steen wat zuyver te maken en als ,t gewreven is zo doeter wat indigo bij, want dit wit dan niet zuyver genoeg is om alleen te gebruyken, daer na neemt ander loot-wit en die zal zuyver zijn.T wit kont gij wrijven met water, en laeten droogen en temperent met nuet olij of lijn olij. Loot-wit wrijft met pis of asijn en alse gedroogt is en gij die wilt gebruyken zo tempertse met olij.* 390–94: Lead white is ground with clean water; if you want it beautiful so leave it spread on the stone after it has been ground and sprinkled with clean water and leave it thus to bleech 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. *Loodwidt wrijftmen met schoon water; wilment schoon hebben zo laet het uyt gespreyt leggen op den steen als t gewreven is en besprengent met schoon water en latent zoo staan bleeken in heete zon en als ,t drooch is zal ment weder vochtich maken en wrijvent weder een wijl tijts en wederom droogen en bleeken tot 4 of 5 maal toe dit is schoon wit en als ment verwerken wil zalment met gom-water temperen. Nota. Alsmen dit lootwit wrijft zo moet men het loot datter gemeenlijk in is daar uyt zoeken of het zouder zonderlinge af besmet worden.*
Wiltschut, A. 1701. 'Teycken bouk'. Paris: Netherlands Institute, Coll. Frits Lugt, Ms. 1997-A.11 86, 31
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 it. *Het loodt-wit magh oock wel van versten af aen met water gewreven worden dat vinden sommige beter, maer het venets wit moet van versten af in olie gewreven worden, om dat men het waeter daer niet uijt temperen kan*
Hyre, P. de la 1730. 'Traité de la pratique de peinture', in *Mémoires de l'Academie Royale des Sciences. Depuis 1666 jusqu'à 1699* 9. Paris: Compagnie des libraires, 665
Then one grinds these flakes on a hard stone, like porphyry, with the muller & clear water, & 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, & you may keep it as long as you wish. *Ensuite on broïe ces écailles sur une pierre dure, comme porphyre, avec la molette & de l'eau claire, & le plus proprement qu'il est possible pour avoir de beau blanc; quelques fois ces écailles sont couvertes d'une matiere grise ou jaune qu'il faut ratisser avant que de les broïer, ce qui peut venir des lames de plomp qui n'étoient pas bien nettes par dessus quand on les a enfermées dans le pot. Le blanc de plomp étant bien broïé à l'eau on le laisse bien secher, & on le peut garder tant qu'on veut.*
Sprong, U. 1738. *Kabinet der verf-stoffen*. Amsterdam: Jacob Graal, 13–14
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. *Wy zullen hier te plaatse noch een fray hantgreepje ter neer stellen, hoe dat ,t loodwit kan gezuivert, en bestendig gemaakt worden. Neemt Loodwit, vryft ,t ter dege met een water op een steen: Zet ,t dus uitgestrekt leggende, in de open lught; dogh draagt wel zorg, dat ,t niet droogh en wordt; vryft , t dan andermaal en stelt ,t weer in de loght, ,t zelve gedurig nat houdende. Vervolgt dit tot drie a vier toghten toe, zoo zalt zeer wit en zuiver zyn.*
Pictorius, J. 1747. *Den geheimen illumineer-kunst*. Leiden: Wed. en Zoon van Jan vander Deyster
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. *Venediis loot of schilp wit moet sterk met zuyver regenwater, op een goede steen gevreven worden, maar voor al met geen ander water, om dat geen ander water tot alle boven gemelde verwen goet is.*

ATSR 4 15 Stols.indd 116 17/01/2012 10:18

411–12: To make 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 Loot Wit te 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.

Lindenbergh, J.F. 1753. *Nieuwe verligter*. 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 veele twee soorten, wier eene Veneetsch, de andere Inlandsch genaamt word; dog ik hebbe nooit meer dan een soort gezien, ,t selve is een der hardste stoffen die in de verwen gebruikt worden, men wryft die sterk met schoon regenwater op een steen, een lange tyd.

Dossie, R. 1764. *The Handmaid to the Arts* (2 vols), vol. 2. London: J. Nurse, 123

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. *Nieuw 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 into small pointed breads, which are dried for transport;
Men wryfd dit loodwit op den porphyr steen met een weinig water, en maakt ,er een deeg van, dat men in vormen tot kleine spitswyze brooden maakt, die men drogen laat om te vervoeren;

Anon. 1777. *Nieuwen verlichter* (2 vols), vol. 1. Gendt: Philippe Gimblet en gebroeders, 21

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.
Van de bereyding, gebruyk en vermenging der witte verwen. Schelpwit. Bereydinge.
men vryft het sterk met schoon regenwater op eenen steen, eenen langen tyd

Wiegleb, J.C. 1777. *Anleitung zur Chemie für Künstler und Fabrikanten.* Gotha, Langensalza: Carl Wilhelm Ettinger, 183

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.
Weiß wird mit Bleijweiß, oder besser, mit Schieferweiß, gemacht. Man reibet es zuerst mit Wasser, dann, wann es recht trocken, mit Firniß ab, so dicke, daß man es ballen kann, formiret Küglein davon. So wirds im kleinen gebraucht. Im großen wird es mit Leinöl gerieben, kalt Wasser auf das fettigte Mengsel gegossen und wieder so lange bis das Wasser sich abgesondert, gerieben. Denn es wird mit dem Wasser schöner und gefüger, weil dieses oben gedachtermaßen, auch von den Fettigkeiten etwas weniges im Reiben auflöset.

Anon. 1794. *Wiener Farbenkabinet.* Vienna/Prague: Verlag der v. Schönfeldschen Handlung

148–9: Seventh section. About white paints. 1. White lead white.
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 weißen Farbe. 1 Weißes Bleiweiß.
Nachdem das Blei durch die Dämpfe des Eßigs in Bleiweiß zernaget ist, so wird es auf großen Reibesteinen, die als Maschinen in einer großen Fabrik am vortheilhaftesten vom Wasser getrieben werden könnnen, auf das zarteste gerieben und dabei mit Wasser angefeuchtet, daß ein dicker Brei daraus wird. Sodenn wird das Bleiweiß in Formen gebildet, die gemeiniglich 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 top 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 three 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.
Die Fabrik reibt das Bleiweiß zwar auch unvermischt; allein, größtentheils erhält es auf der Mühle einen Zusatz von Kreide. Das reine Schieferweiß schüttet man blos auf den obersten Mühlstein, benetzt es mit Wasser, und wiederholt dieses zum öftern, während daß es gemahlen wird. Es läuft durch gedachte Rinnen vom dem obersten Steine zu den drei übrigen, und wird von allen 4 Steinen völlig zermalmet. Von dem letztern Reibsteine läuft es durch eine Rinne in ein hölzernes Gefäß, und aus diesen wird es in kleine dreikantige Formen von Thon, welche ohngefähr 4 bis 5 Zoll hoch, und oben eben so weit sind, geschöpft. In diesen steht es so lange, bis es zusammenfällt, und alsdenn wird es auf den Brettern eines hölzernen Gestells in der Luft getrocknet.

cont'd.

ATSR 4 15 Stols.indd 117 17/01/2012 10:18

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

153: If you want the flake white really beautiful, you have to grind it with pure water four times on the grinding stone, and as fast as possible. The more one grinds, the more white it becomes. Some grind it first with vinegar, and wash it afterwards with water, because they believe, that the vinegar, through which it was first made, makes it more white; but it is better, to use water right away. If you wish to preserve it after grinding, let it dry in small pieces or grains in a clean place, without dust, and then it can be preserved very well. However if it is intended for oil painting, then mix it, after it has been well ground for four times, with very white poppy oil, by beating the white pulp in order to remove the water and replace it with oil. Then it is ground again very fine, and only a little at a time, preserve it in some glazed jar, and pour a finger high of water on top, in order to preserve the paint, and to prevent a thick skin on top. The preparation with water renders the flake white more clear and white, compared to, when it is ground straight away with oil. This beautiful white is used for glazes on ordinary lead white.
Will man das Schieferweiß recht schön haben, so muß man es viermal auf dem Reibstein mit reinem Wasser, und so hurtig als möglich abreiben. Je mehr man reibt, desto weißer wird es. Einige reiben es sogleich mit Essig ab, und waschen es nachher im Wasser, weil sie glauben, der Essig, wodurch es erst entstanden, mache es weißer; es ist aber besser, gleich Wasser zu nehmen. Will man es nach dem Abreiben aufheben, so läßt man es in kleinen Stücken oder Körnern an einem reinlichen Orte, wo es nicht staubt, trocknen, und kann es alsdenn sehr wohl aufheben. Ist es hingegen zur Oelmalerei bestimmt, so vermischt man es, nachdem es zum viertenmal gut abgerieben worden, mit sehr weißen Mohnöl, indem man das Weiße immer schlägt, um das Wasser heraus, und das Oel an dessen Stelle zu bringen. Man reibt es alsdenn von neuem sehr klar, und nur wenig auf einmal, hebt es in einem irdenen glasurten Gefäß auf, und gießt einen Finger hoch Wasser darüber, damit die Farbe besser sich halte, und sich keine dicke Haut darüber anlege. Die Zubereitung mit Wasser macht das Schieferweiß reiner und weißer, als wenn man es sogleich mit Oel abreibet. Dieses schöne Weiß wird mit zum Glasuren auf gemeines Bleiweiß gebraucht.

Oeconomische Courant, 15 October 1800. No. 167, 82

Economical knowledge. About LEAD WHITE, its fabrication, and trade.
The lead, chalked by the vinegar into lead white, is ground very fine on large grinding stones, which in a factory are most economically turned by water, or crushed, and with water mixed into a thick pulp. Then the lead white is shaped in moulds, which ordinarly have the shape of a blunt sugar loaf; these loaves are dried in summer in the open air, and in winter in a moderately heated room.
Oeconomische kundigheden. Over het LOODWIT, het fabriceeren van, en den Handel met hetzelve.
Het Lood, door den Azyndamp tot Loodwit verkalkt zynde, wordt op grootte wrijfsteenen, die by eene Fabriek het voordeeligst werktuiglyk door het water gedreven worden, zeer fyn gewreven of gemalen, en met water tot een dikke brei aangemengd. Vervolgens vormt men het Loodwit in vormen, die doorgaands de gedaante hebben van een stompsuikerbroodjen; wordende deze broodjes by den zomer in de opene lucht, en ,s winters in een taamlyk warm vertrek gedroogd.

Leen, *c.*1800. *Over teken- en schilderkunst & raad aan kunstverzamelaars.* [Delfshaven?], 4

To render white, whatever sort, unequally beautiful it is ground clean in water, dried and then crushed and ground with fresh poppy oil.
Om het wit, welke soort ook, ongemeen schoon te hebben wrijft men hetzelve schoon in water, laat het drogen en dan fijn stoten en met blanke verse papaver of heulolie wrijven.

Brodhagen, P. 1802. *Anleitung zur Technologie* (3 vols), vol. 1, Hamburg: Bachmann und Gundermann, 378–9

This mill [= mill for grinding lead white] can also be made in small, in this manner: get a large stone and have it caved out in the shape of half a ball, on the upper edge a small channel is carved, through which the mass drips into another jar placed below. Then you have another stone, the shape of half a ball, which exactly fits into the hollow of the first. In the middle of this non-hollowed stone must be a hole, in order to be able to place a funnel, through which the mass is poured in. Through some iron rings, which are secured to the surface of the stone, one sticks a bar, by which a man turns the muller. During grinding you add some water, to make the mass drain off better.
On top of the container, into which the lead white drains from the mill, you place a hair sieve, through which the mass must pass into the container, to retain the coarse materials. When the container is full, you place another one underneath and continue grinding, until all is done. In the containers, in which the lead white runs off, it settles at the bottom, and the water standing on top is removed with taps or holes. Afterwards you pour the lead white on a linen cloth, roughly stretched on a frame, where the [remaining] water drains off. Then it is further dried, either on a tile stove, which one fuels with a small fire, and then applies the mass on top, where it quickly dries; or most commonly like this: one takes planks with holes, in which conically shaped earthenware pots are placed. The hole, which these pots have at the bottom, and through which the 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. Now the lead white is ready for transport. The more 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. The remaining black deposit consists of undissolved lead, which may be reused for a fresh batch.
Diese Mühle [= mill for grinding lead white] kann man auch im Kleinen machen, und zwar so: Man läßt einen großen Stein, wie eine halbe Kugel, hohl aushauen, oben am Rande wird eine kleine Rinne eingehauen, durch welche die Masse während des Reibens in ein anderes untergestelltes Gefäß herausläuft. Man hat nun noch einene Stein, von der Form einer Halbkugel, der grade in die Höhlung des andern hineinpaßt. In der Mitte muß dieser nicht ausgeholte Stein ein Loch haben, um einen Trichter hineinstecken zu können, durch welchen die Masse hineingegossen wird. Durch ein paar eiserne Ringe, die in der Oberfläche des Steins fest stzen [typing error in original text], steckt man eine Stange, vermittelst deren ein Mann den Läufer bewegt. Während des Mahlens gießt man immer etwas Wasser nach, damit die Masse desto mehr abläuft.
Auf das Gefäß, in welches das Bleiweiß aus der Mühle läuft, setzt man ein Haarsieb, durch welches die Masse in das Gefäß laufen muß, wodurch das grobe Zeug zurück bleibt. Wenn nun ein Gefäß voll ist, setzt man ein anderes unter und fähret so fort zu mahlen, bis alles fertig ist. In den Gefässen, in welchen das Bleiweiß laufen, setzt es sich nun zu Boden, und das darüber stehende Wasser wird durch Zapfen oder Löcher abgelassen. Nach diesem thut man das Bleiweiß, auf eine grobe in Rahmen gespannte Leinwand, wo das Wasser vollends abläuft. Dann wird es weiter getrocknet, entweder auf ein Ziegelherd, wo man ein wenig Feuer darunter gemacht, und dann die Masse darauf thut, wo sie bald trocknet; oder am gewöhnlichsten so: mann nimmt Bretter mit Löcher, in die man kegelförmige irdene Gefäße steckt. Das Loch, das dies Gefäß unten haben muß, und durch welches das Wasser abläuft, wird mit Papier verstopfet; man füttert die Gefäße mit hellblauen Papiere, und thut dann die Masse hinein. Ist nun die Feuchtigkeit genug abgelaufen, so setzt man die kleinen Bleiweishütte auf ein Brett an die Luft, um sie vollends auszutrocken. Nun ist das Bleiweis zum Versenden fertig. Je mehr die Masse mit Wasser gewaschen wird, desto weisser wird das Bleiweis, und bei dem Zusatz von Kreide giebt die feinste, weißte und reinste Kreide ebenfalls das schönste Bleiweis. Der zuletzt bleibende schwarze Budensatz ist noch unaufgelößtes Blei, das man wieder zum frischen Ansatz brauchen kann.

ATSR 4 15 Stols.indd 118 17/01/2012 10:18

Fokke Simonsz., A. 1804. *De kunst van tekenen en schilderen in waterverwen, enz*. Leiden: A. and J. Honkoop, 83

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, stirr 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 onvergelijkelijke witte loodkleur wordt gemaakt van lood-wit, dat uitgezocht en wel op een' porphiersteen gewreeven 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 afgegooten hebbende, roer het dan weder door met azijn; herhaal dit nog een of twee maalen, en gij zult een uitneemend fraai wit, beide voor water- en olieverwen geschikt, bekomen.

Marcucci, L. 1816. *Saggio Analitico-chimico*. Rome: Lino Contedini [2nd corrected and augmented edition] 64–5

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, perchè contiene une coll' animale, donde ripete la sua tenacità.

Leuchs, J. 1825. *Vollständige Farben- und Färbekunde*. Nuremberg: Handlungs-Zeitung, 20–21

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 Newcastle 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 balls.
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, schabt das an dem Metall hängende Bleiweiß ab *), nachdem man es befeuchtet hat, um das Stauben zu verhüten, stößt es mit etwas Wasser zu einem diken 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 dis nöthig ist, und läßt es malen †). Oft wird es zulezt noch geschlämmt.*
**) Zu Newcastle läßt man das Blei unter Wasser durch Walzen gehen, welche das Bleiweiß abdrüken. So wird aller Staub vermieden. Das Wasser zeiht man, nachdem das Bleiweiß sich gesezt hat, mit Pumpen ab. Das Blei wird wieder gebraucht oder eingeschmolzen. Wendet man dünne Platten an, so ist es aber gewöhnlich ganz zerfressen.*
***) Dies scheint indeß nicht in allen Fabriken zu geschehen. Man findet oft auch Bleizukerkristalle an den Platten, so wie einen weißen Schaum auf dem Wasser, der viel Bleizuker enthält. Um diesen nicht zu verlieren, sezt man etwas Potasche zu, welche ihn zersezt und kohlesaures Blei niederfallen macht.*
****) Das Schlämmen geschicht wie gewöhnlich. Man hat eine große vierekige Kufe, das 7 bis 9 Abtheilungen hat, die gleich groß, aber verschieden hoch sind (oder eben so viel verschieden hohe Fässer). Das mit dem Bleiweiß abgerührte Wasser geht von einer in die andere und sezt in den spätern immer feineres Bleiweiß ab. Gutes Schlämmen trägt wesentlich zur Güte des Bleiweißes bei.*
†) In Holland geschieht das malen auf drei neben einander stehenden und durch Pferde getriebenen Mühlen. Die erste malt grob, die zweite fein, die dritte ganz fein. In Berlin heben die Mühlen übereinander, so daß das Gemalene von einer gleich in die andere übergeht. Besser wäre es vielleicht in Tonnen, die um ihre Achse würden, mit Hulfe eiserner Kugeln, zu malen.
*Das gemalene Bleiweiß läßt man einige Zeit unter Wasser, drükt es dann in irdenen oder blechene Formen, welche die Gestalt kleiner kegelförmiger Hüte haben, und läßt es zuerst in diesen, später offen an der Luft *) troknen (wobei man besonders schwefliche Dünste von demselben abhalten muß), und dann in papier einschlagen.*
**) In England auch durch Dampf geheizten Zimmern. Man läßt es in den Formen nicht ganz troken werden, da es sonst schwer herauszunehmen sein würde.*

Riffault, M.J. 1826. *Manuel théorique et pratique du peintre en batimens, du doreur et du vernisseur*, 2nd edn. Paris: Roret, 7–8

After approximately six weeks, one reveals the jars, and, unrolling the lead sheets, one finds them almost completely attacked and changed into a large quantity of lead sub-carbonate and a little quantity of acetate of the same metal (salt which results from the combination of acetic acid with the lead). One separates these two salts from the lead portion which is still in its metallic state, one grinds and washes them; all that is acetate dissolves, while all that is sub-carbonate deposits as very dense layers of one or two centimetres thickness.
Au bout d'environ six semaines, on découvre les pots, et, en déroulant les lames, on les trouve presque entièrement attaquées et converties en une grande quantité de sous-carbonate de plomb et une petite quantité d'acétate de ce métal (sel provenant de la combinaison de l'acide acétique avec le plomb). On sépare ces deux sels des portions de plomb qui sont encore à l'état métallique, on les broye et on les lave; tout ce qui est acétate se dissout, tandis que tout ce qui est sous-carbonate se dépose sous forme de couches très-denses de un à deux centimètres d'épaisseur.

cont'd.

ATSR 4 15 Stols.indd 119 17/01/2012 10:18

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

Gray, S.F. 1828. *The Operative Chemist.* London: Hurst, Chance, and Co., 646–7
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 manufactory; 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.
Mérimée, M.P.L. 1830. *De la peinture à l'huile*. Paris: Mme. Huzard, 225
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 consistence convenable: alors on en remplit des pots coniques, où elle prend la forme qu'on lui connait.*
Vergnaud, A.D. 1831. *Manuel du peintre en batimens, du fabricant de couleurs, etc.* Paris: Roret, 12
Silver white. This white is used by easel painters, and is nothing but a lead white of the first quality, which one prepares with more care, and which one releases, by a large number of washings, of every possible impurity. *blanc d'argent. Ce blanc dont se servent les peintres de tableaux, n'est qu'un blanc de plomb de première qualité, que l'on prépare avec le plus grand soin, et que l'on débarrasse, par un grand nombre de lavages, de toute espèce d'impureté.*
Anon. 1836. *The Painter's, Gilder's, and Varnisher's Manual*, new edn. London: M. Taylor/ New York: William Jackson, 18
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.
Schmidt, C.H. 1857. *Vollständiges Farben – Laboratorium*. Weimar: Voigt, 36–9
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. *Darauf wird das abgelöste Bleiweiß zwischen gußeisernen Walzen in einem hölzernen, dicht verschlossenen Kasten zermahlen, [gesiebt], sodann unter verticalen oder horizontalen Granitsteinen mit wasser fein gemahlen, [der] Brei in conische, nicht glasirte Töpfe von 5 Zoll Höhe und 3½ Zoll Durchmesser am obern weiten Theile gethan, welche auf einem Trocknengerüste stehen und täglich einige Male gerüttelt werden, um die Ablösung des Bleiweiß brodes von der Form zu bewirken. Nach 3 bis 4 Tagen wird die Form umgekehrt und das Brod im Sommer in warmer Luft, im Winter und bei feuchtem Wetter in einer mit warmer Luft geheizten Trockenkammer getrocknet. Zulezt schlägt man das Bleiweiß in blaue papier ein.*
Gentele, J.G. 1860. *Lehrbuch der Farbenfabrikation*. Braunschweig: Friedrich Vieweg und Sohn,159
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 1½-1 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.

ATSR 4 15 Stols.indd 120 17/01/2012 10:18

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 which the lead white is stirred, one "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 draining 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 empties it when a somewhat useable, however not very pure lead white has accumulated inside, and then feeds it again.

Das Vermahlen kann nun, 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 ½ - 1 Umdrehung machen. Man schöpft den Brei mit Hülfe 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 niedergesunken ist, worauf man wieder nachfüllt, bis nach und nach eine Partie, z. B. 10 Centner durchgegangen sind. Ein arbeiter kann 4 bis 5 Steine, die neben einander stehen, bedienen. Was in der der Sarge des Bodensteins sich von Gemahlenen ansammelt, wird durch ein Schäufelchen öfters nach vorn in den Ablauf geschafft, wo es in einen kleineren Ständer fällt. Bei dem ersten Mahlen ist es häufig nöthig, den Stein etwas höher zu stellen, denn weil die Masse sehr dick wird, läuft sie nich gern unter und durch die beiden Steine. Wenn Alles durchgemahlen ist und nun das Gemahlene einen zähen Brei darstellt, so wird der Stein durch Nachspülen von Wasser rein gemahlen, die Bodensteinsarge 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 gestellten Steinen hindurch- und feingemahlen. Besser ist es jedoch, und so wird es auch meistens gehalten, dass man den rohen Bleikalk stets einen Stein passiren lässt und das Feinmahlen auf einigen andern Gängen, die zu gleicher Zeit in Bewegung sind, ausgeführt wird, dann wird dasjenige, was vom ersten Steine kommt, auf den zweiten, von da etwa auf den dritten und vierten Stein geschöpft und das Mahlen geht dann regelmässig und ununterbrochen 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 nich hart werde, ist ein sorgfältiges Auswaschen unumgänglich erforderlich. Zum Auswaschen hat man dann eine Anzahl grosser Ständer nöthig, die um so besser, je grösser sie sind. Diese Ständer füllt 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 Aufrühren mit frischem Wasser, Absitzenlassen und Abziehen wiederholt man einige Male. Alsdann bringt man den sich sehr dick absetzenden 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 Vermahlen zu weichem Bleiweiss kan aber auch von vorn herein auf eine Weise erfolgen, wodurch das Auswaschen und Zertheilen leichter vor sich geht. Der Bleikalk wird nämlich mit vielem Wasser vermahlen, so dass das Gemahlene nicht als dicker Brei, sondern als eine leichter bewegliche Flüssigkeit durch die Steine und von selbst aus den Oeffnungen der Sargen um die Bodensteine herausläuft. In diesem Fall ist leicht eine solche Anordnung der Mahlgänge möglich, dass sie um das vertical stehende Triebrad herum stufen- oder treppenweise aufgestellt sind, so dass dasjenige, was vom ersten Steine abläuft, direct durch eine Rinne zu dem andern Stein tritt und so zum dritten und vierten und von da in die Abwässerungsstanden, worin das Bleiweiss, wie vorhin schon erwähnt, abgewässert wird. Dies ist die gewöhnliche Aufstellung der deutschen Mühle.

Bei diesem Abwässern geht in größeren Fabriken eine nicht unbedeutende Menge Blei jährig verloren, welche ausgewaschen wird und mit dem Waschwasser als essigsaures Blei davon läuft, und zwar in einer Verdünning, wo es kaum benutzbar ist. Dieser Verluft kann bis einige Procente ausmachen. Um ihm zu entgehen, kann man auf zweierlei Weise verfahren. Man setst dem ersten Wasser, womit das Bleiweiß aufgerührt worden war, einige Maß einer Lösung von krystallisirter Soda hinzu, welche einen Niederschlag erzeugt, indem sie das gelöste Blei fällt, und gleichfalls als kohlensaures Bleioxyd. Oder man läßt, was jedenfalls das beste ist, alle die fallenden Waschwasser in einen Sumpf laufen, worin ein Ueberschuß von Kreide oder kohlensaurem Kalk gethan worden ist, wo dann das Blei sich nach und nach als Bleiweiß ausgescheidet und kohlensauren Kalk dafür in Lösung geht. Nach längerer Zeit untersucht man den Inhalt des Sumpfes und entleert und beschickt ihn von neuen, wenn sich ein einigermaßen brauchbares, wenn auch nicht ganz reines Bleiweiß darin angesammelt hat.

Riffault, 1874. *A Practical Treatise on the Manufacture of Colors for Painting*. London: Sampson Low, Marston, Low & Searle.

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 collected at 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.

IV. The white lead is mixed with water in troughs, so as to form a soft paste which passes successively through several horizontal mill-stones before it is thoroughly comminuted. This wet grinding is absolutely without danger, since the men do not touch the white lead with their hands, but carry it in scoops or ladles.

V. In all the works which we have visited, the soft paste of white lead is poured into conical earthenware pots, which are dried in a stove room. The greater part of the water is expelled, and the blocks becoming contracted, are easily removed from the pots. Their thorough drying is finished in another, or the same, stove room. […]

VI. The greater part of the white lead in lumps requires to be ground and sifted again before it is ready for sale. This second grinding, in the majority of works, is still done with vertical stones rolling upon a stone bed. The ground stuff is shovelled into the hopper of a cylindrical sieve, enclosed in a wooden box, where the fine white lead falls. That which has not passed through the meshes of the sieve is collected in another box, and ground anew. The sifted white lead is removed from its box, after the dust has subsided, and packed in barrels either by shaking, or by a slight ramming.

cont'd.

ATSR 4 15 Stols.indd 121 17/01/2012 10:18

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

78–85: The French or Clichy Process, by Thénard
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.

Mierzinski, S. 1881. *Erd-, Mineral- und Lackfarben.* Weimar: Voigt, 53

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.
Das durchgewaschene Bleiweiss wird dann zwischen horizontalen Mühlsteinen von 80 bis 100 cm Durchmesser (ganz nach Art von Getreidemühlen), unter beständigem Zufliessen eines Wasserstrahles, gemahlen. Als Mülhsteine werden nur die sogenannten französischen benutzt. Man hat eine Anzahl von Mühlen für die beste Sorte und eine andere für das zu lange in der Kammer Gebliebene, welches nicht ganz so weiss ausfällt. Nach beendigung des Mahlens schlämmt ein Wasserstrahl das Bleiweiss, mit Ausnahme der gröbsten Theile, von den Steinen ab, worauf es in Absatztröge fliesst. Nach einiger Zeit wird das Wasser aus diesen ausgepumpt und der Bleiweisskuchen ausgestochen.

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 and 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.[11] 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

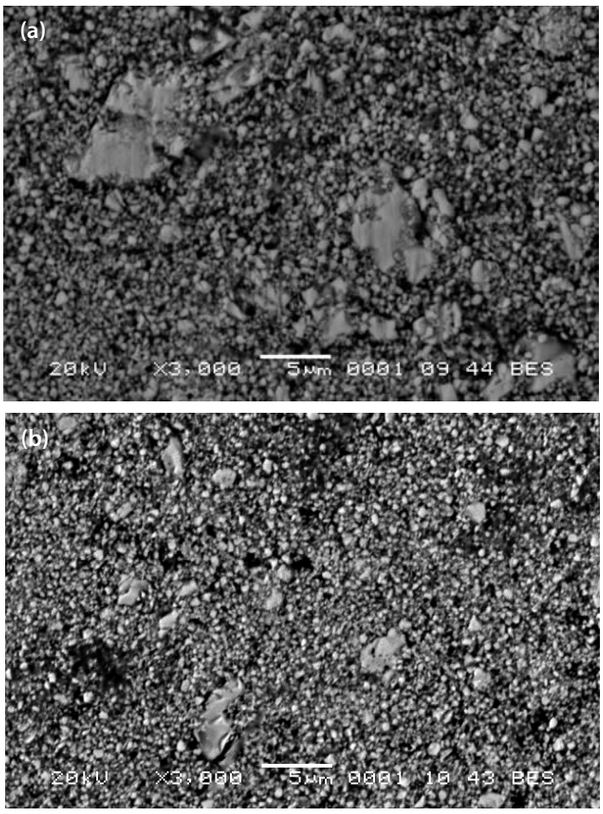

Figure 4 SEM BSE image of stack-process lead white (a) ground in water, paint prepared with poppy oil, (b) ground in vinegar, paint prepared with poppy oil.

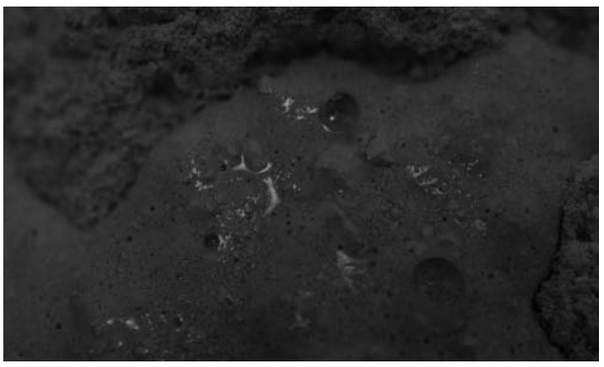

Figure 5 Lead white and chalk with a drop of wine vinegar: gas bubbles have formed.

ATSR 4 15 Stols.indd 122 17/01/2012 10:18

Table 2 Historical recipes for processing lead white with vinegar.

Fortunato of Rovigo 1967 [1659–1711]. 'Raccolta di Secreti', in M.P. Merrifield, *Medieval and Renaissance Treatises on the Arts of Painting.* Mineola: Dover [repr. 1980], cli
To render *biacca* 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 *biacca* that will be perfectly beautiful for miniature painting, as well as oil painting. *Per rendere la biacca piu bianca straordinariamente. Prendete biacca di piombo in scaglie, elegete la più bella, e macinatela bene sul marmo con aceto, e diventar nera, allora prendete un vaso di terra plena d' acqua, e lavata il vostro bianco bene, poi lasciatelo bene dur in fondo, e verrate l'acqua per inclinazione. Tornatela a macinare con aceto et a lavare; e fatta questa operatione med.- 3 o 4 volte, che havera una biacca che sara perfettam" bella tanto per miniare, quanto per dipingere a olio.*
Eikelenberg, S. 1679–1738. 'Aantekeningen'. Alkmaar: Municipal Archive, Mss. Eikelenberg, 390–94, 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. *Alsmen verwen wrijven wil zo neemt eerst wat loot-wit om den steen wat zuyver te maken en als ,t gewreven is zo doeter wat indigo bij, want dit wit dan niet zuyver genoeg is om alleen te gebruyken, daer na neemt ander loot-wit en die zal zuyver zijn.T wit kont gij wrijven met water, en laeten droogen en temperent met nuet olij of lijn olij. Loot-wit wrijft met pis of asijn en alse gedroogt is en gij die wilt gebruyken zo tempertse met olij.*
d'Emery 1709. *Nouveau recueil de secrets et curiositez.* Amsterdam: Estienne Roger, 134–5
To render 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 terrine full of water, & wash your white well, then let settle well, & pour off the water by inclination; grind again with vinegar & wash 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. *Pour rendre le blanc de plomp fin extraordinairement.* *Prenez du blanc de plomp en écaille, choisissez le plus beau, & broyez bien ces écailles sur la pierre, avec du vinaigre, & il deviendra noir; alors prenez une terrine pleine d'eau, & lavez bien vôtre blanc, puis le laissez bien rasseoir, & versez l'eau par inclination; broyez-le encore avec du vinaigre & le relavez, faisant cela trois ou quatre fois, & vous aurez un blanc qui sera parfaitement beau tant pour l'enluminure que pour la peinture à huile.*
Barrow, J. 1735. *Dictionarium polygraphicum* (2 vols), vol. 2. London: C. Hitch, C. Davis, S. Austen (n.p.)
But if you use white lead, first rectify it with white wine vinegar, this will cause a fermentation, and the white will soon settle; then pour off the vinegar, and wash it with common water. The method of washing it is thus: Put the powder into a glass of water, stir it about, and presently pour off the water, while it is white, into some other clean glass or vessell let it settle, and then pour off the water from it, and it will be excellently fine. … 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 probable) must be pour'd off, when the spirit of wine has done its work.
Le Pileur d'Apligny 1779. *Traité des couleurs matérielles*, Paris : Saugrain & Lamy [repr. 1973, Geneva, Minkoff], 5–6
When you wish to use ceruse in painting, you have to purify it with white vinegar: so it is ground on a porphyry, while wetting it with vinegar, until it is an impalpable powder: then one places it for washing in a vase filled with water, which one shakes for some time: one lets settle the largest particles, then one transfers the white water to another vessel: one lets precipitate the white, & then one decants the water that floats on top. One obtains by this method a very beautiful and very fine white, cleansed of anything that might change its whiteness: the reason is that this white is not subject to darkening because of the badly dissolved lead particles that it contains, or at least very disposed to altering itself by the least contact with the air; the vinegar achieves the complete dissolution of these particles, much better than a mineral acid, because it does not contain *phlogistique*, at least also developed: it is good for this reason only to purify the amount that you need. *Lorsqu'on veut employer la céruse en peinture, il faut la purifier avec du vinaigre blanc: on la broie donc sur un porphyre, en l'arrosant de ce vinaigre, jusqu'à ce qu'elle soit en poudre impalpable: on la met ensuite pour la laver dans un vaisseau plein d'eau, qu'on agite quelque temps: on laisse rasseoir les parties les plus grossieres, puis on transverse l'eau blanche dans un autre vaisseau: on laisse précipiter le blanc, & alors on décante l'eau qui surnage. On obtient par ce moyen un blanc très-beau & très-fin, dépouillé de tout ce qui pourroit altérer sa blancheur; la raison en est que ce blanc n'est suject à noircir qu'à cause des particules de plomb mal dissoutes qu'il contient, ou du moins très-disposées à se revivisier par le moindre contact de l'air; le vinaigre acheve l'entiere dissolution de ces particules, beaucoup mieux que ne seroit un acide minéral, parce qu'il ne contient pas de phlogistique, du moins aussi développé: il est bon pour cette raison de n'en purifier qu'à mesure qu'on en a besoin,*
Buc'hoz, M. 1786. *Receuil de secrets sur et expérimentés a l'usage des artistes* (no. of vols not known), vol. 3. Paris: by the author, 291–2
Or choose the most beautiful lead white in flakes, very white & very soft: grind it on a stone with vinegar, it will become black; but when you have washed it in very clear water, let it settle well; pour off the water by inclination, then grind again with vinegar, & wash again; repeat the same four to five times; it will become very beautiful & very bright: it is very good for illumination & for painting in oil. *Ou bien choisissez du beau blanc de plomb en écailles, bien blanc & bien tendre: broyez-le sur la pierre avec du vinaigre, il deviendra noir; mais quand vous l'aurez lavé dans une eau bien claire, laissez-le bien rasseoir; versez ensuite l'eau par inclination, puis broyez de nouveau avec du vinaigre, & relevez; réitérez de même quatre à cinq fois; il deviendra très beau & très-éclatant: il est très-propre à l'enluminature & à peindre en huile.*

cont'd.

ATSR 4 15 Stols.indd 123 17/01/2012 10:18

Table 2 Historical recipes for processing lead white with vinegar. cont'd.

Anon. 1794. *Wiener Farbenkabinet*. Vienna/Prague: Verlage der von Schönfeldschen Handlung
152: When one uses lead white for painting, it must be cleaned with white wine vinegar. For this purpose one grinds it on a porphyry, after having moistened it with this wine vinegar, until it has become an impalpable powder. Then it is poured into a vessel filled with water for washing, which is shaken for a while, then the coarsest particles are left to settle at the bottom; after that the still whitish water is poured into another vessel, and the white, which is still in the water, is left to settle at the bottom, after which one decants the supernatant water. In this manner one obtains a very beautiful and fine white, which above all has been cleaned of anything, which may harm its whiteness and may change it. Because this white only tends towards grey or black, because the lead particles which remain inside either have not been dissolved completely, or have the tendency to change back into lead upon the least contact with air. The wine vinegar however helps to dissolve these lead particles completely, and even better than any mineral acid would, because this does not have an inflammable nature, at least not openly. *Wenn man das Bleiweiß zum Malen gebrauchen will, so muß es mit weißem Weineßig gereiniget werden. Man reibet es demnach auf einem Porphyrstein, nachdem man es mit diesem Weineßig angefeuchtet hat, so lange, bis es zu einem unfühlbaren Pulver geworden ist. Hierauf wird es zum Abwaschen in ein Gefäß voll Wasser geschüttet, welches man eine Zeitlang schüttelt, läßt es eine kurze Zeit die größsten Theilchen zu Boden fallen, und giesset das noch weißgefärbte Wasser in ein anderes Gefäß, und lässet alles Weiße, das noch in dem Wasser ist, zu Boden setzen, worauf man das darüberstehende Wasser abgiesset. Auf diese Art erhält man ein sehr schönes und feines Weiß, welches von allem entlediget worden, was seiner Weiße Schaden thun und solche verändern könnte. Denn dieses Weiß fällt nur deswegen gern in das graulichte oder schwärtliche, weil die Bleitheilchen, die darinnen sind, entweder nicht genugsam aufgelöset worden, oder doch zum wenigsten bei der geringsten Berührung der Luft sehr geneigt sind, sich wieder in Blei zu verwandeln. Der Weineßig aber hülft dazu, daß diese Bleitheilchen völlig aufgelöset werden, und zwar weit besser, als es eine mineralische Säure thun würde, weil derselbe kein brennliches Wesen, wenigsten nicht offenbar mit sich führt.*
153: see Table 1.
Fokke Simonsz., A. 1804. *De kunst van tekenen en schilderen in waterverwen, enz.* Leiden: A. and J. Honkoop, 83
See Table 1.
Bouvier, M.P.L. 1827. *Manuel des jeunes artistes et amateurs en peinture*. Paris: Levrault, 4–5
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 faithfullness; 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 *livre*, 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. *Un de mes amis m'a indiqué un procédé pour obtenir du blanc de toute pureté. Je n'ai pas eu occasion d'en faire l'essai moi-même, mais j'ai toute confiance dans sa véracité et sa bonne foi; d'ailleurs ce procédé, qui est pratiqué par d'autres, porte avec soi tout ce qu'il faut pour convaincre de son efficacité, étant fondé sur des principes que le raisonnement ne peut qu'approuver.* *Prenez, je suppose, une livre, plus ou moins, de beau blanc de Crems, faites-lui subir, en petites parties, une première broyée à l'eau, sans vous attacher à ce qu'il soit très-fin; vous le relèverez de dessus la pierre en bouillie de l'épaisseur d'une forte crème, et vous aurez soin que les premières broyées ne sèchent point, mais qu'elles se conservent un peu liquides. Pour cela vous mettrez chaque relevée de couleur dans un pot neuf bien vernissé, ou dans un bol de faïence, ou de terre de pipe. Quand tout votre blanc est broyé et qu'il est en bouillie assez épaisse, versez par-dessus cette bouillie la valeur d'un gobelet à boire de très-bon vinaigre blanc distillé; vous remuerez et brouillerez le tout toutes les heures, et cela durant une journée: vous prendrez, pour le remuer, un tuyau de pipe neuf, que l'acide du vinaigre n'attaquera pas. Si votre vinaigre est bon et bien distillé, il nettoiera toutes les petits particules étrangères qui peuvent se trouver dans le blanc, en sorte qu'il ne restera plus rien qui puisse ternir votre couleur. Cette couleur ainsi préparée donne un blanc très-parfait; mais il faut avoir soin d'en extraire tout le vinaigre par des lavages récidivés, et jusqu'à ce que l'eau qui surnage sur le blanc (quand on l'a laissée reposer) n'ait plus aucune saveur d'acidité en la portant sur la langue. Ce blanc ainsi rebroyé à l'eau pure, au moins trois fois, est d'une qualité supérieure; employé aussi à l'eau de gomme pour peindre la gouache, il est léger, très-subtil, et ne reluit point sur le papier, comme il arrive au blanc de Cremnitz qui n'a pas subi cette opération.*

HART project: lead acetate was indeed removed from lead white by repeated washing (Carlyle 2006: 25–6). Furthermore, our reconstructions showed a shift in the balance between cerussite and hydrocerussite, with the washed lead white containing less hydrocerussite.

Vinegar grinding

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 discolour 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.

A series of grinding experiments following the instructions of Le Pileur d'Apligny was executed with unprocessed

ATSR 4 15 Stols.indd 124 17/01/2012 10:18

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

Mayerne, T.T. de 1620–44. 'Pictoria Sculptoria & quae subalternarum artium'. London: British Library, Ms. Sloane 5052, 96v

(Paulus van Somer) Lead white ground first with water, then washed & allowed to settle, decanting the turbid water, makes a deposit that is very beautiful, & dies less than the deposit.
Le blanc de plomb broye premierement avecque eau, puis lavé & laissé rasseoir, en decantant l'eau trouble, faict une residence qui est tres belle, & meurt moings que le fonds.

King, D. 1653–57. 'Secrets in the noble arte of miniatura or the art of limning'. London: British Library, Ms. Additional 12461, 39–40

Mr. Hilliards manner of preparing Ceruse.
Having ground your ceruse in water without gum & put it into a viall glasse with a good quantity of faire water, and being well shaken together let it stand a while, and before it be settled poure off the third part of the water and let it settle. Then poure yet likewise one other third part out of the viall and reserve the last part in the viall 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 setled 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*. London: Dorman Newman, Richard Jones, 71–2

As in the grinding of colours I gave you an instance but in one for all 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 greasie 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 dregs. 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*. Groningen: Broekema, 27

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 poures out the liquid with the fine particles that are still retained inside it into a sugar glass or bowl. 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 schulpwit in een maarmere mortier met eene yzere stamper tot een grof poeyer. Hier op giet men water, tot het een pap werde: dan wryft 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 hoeveelheid waater, en, naa alles wel omgeroerd te hebben, laat men ,t 15 à 20 tellen staan, waarna men ,t vogt met de daar noch in opgehouden wordende fyne deeltjes in een zuiker glas of kom overgiet. Het gezakte grovere deel wort wederom gewreeven, en op dezelfde wyze behandeld. En dit wryven, op- en afgieten herhaald men zo lang tot er eenige zwartagtige, of bruine groove deeltjes te rug blyven. Het afgegootene water laat men staan tot alle stoffe geraakt zy; wanneer men ,t overvloedige water er afgiet, en ,t overige door een tregter van lek papier er laat uitdruipen: eindelyk droogt men de overgeblevene pap tusschen papieren. Hoe langer men in den beginne de stof laat zakken, dies te fynder zyllen de deeltjes zyn: doch deze bepaalde tyd is lang genoeg. Die enigszins aan deeze behandeling gewend is kan in 2 uuren tyds wel een pond van dit wit fyn vrywen. Men mag zonder eenige vreeze deeze wyze van fynmaaking by alle aardagtige verfstoffen in ,t werk stellen.

Le Pileur d'Apligny 1779. *Traité des couleurs matérielles*. Paris : Saugrain & Lamy [repr. 1973, Geneva, Minkoff], 5–6

See Table 2.

Anon. 1794. *Wiener Farbenkabinet*. Vienna/Prague: Verlag der von Schönfeldschen Handlung, 152

See Table 2.

Bouvier, M.P.L. 1827. *Manuel des jeunes artistes et amateurs en peinture*. Paris: Levrault, 114

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 d'avec le sédiment, qui est rempli de pierres et autres corps pesans.

cont'd.

ATSR 4 15 Stols.indd 125 17/01/2012 10:18

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

Mérimée, M.P.L. 1830. *De la peinture à l'huile*. Paris: Mme. Huzard, 228–9
While the lead white formed in this operation [= 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 stirrs 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. *Comme le blanc de plomb formé dans cette opération [= blanc de Krems] n'a pu acquérir la dureté de celui qu'on obtient par la méthode hollandaise [= stack process], il n'est pas nécessaire de broyer; on l'obtient très divisé par la lévigation, au moyen d'un appareil extrèmement simple. Il est composé d'une grande caisse contenant neuf cases ou compartimens, de hauteur décroissante. On met dans la case la plus élevée les écailles de blanc de plomb, séparées préalablement du métal qui n'a point été attaqué. On y fait arriver de l'eau d'un réservoir supérieur, et l'on remue fortement avec un rable: l'eau déborde bientot, s'écoule dans la seconde case, puis dans la troisième, et arrive ainsi successivement dans la neuvième.* *On concoit que les molécules du blanc de plomb, entrainées par l'eau, sont d'autant plus ténues qu'elles ont été charriées plus loin. Ce qui se trouve dans les cases inférieures est donc le blanc de première qualité. On vide les cases dans de grands cuviers, où le dépot se fait en plus ou moins de temps: on le recueille ensuite, et lorsqu' il est suffisament égoutté, on le met dans des vases de terre poreux, où il sèche et prend la forme de pains carrés.*
Schmidt, C.H. 1857. *Vollständiges Farben – Laboratorium*. Weimar: Voigt, 44–5
Washing of carboxylic lead is very [easy] and is very similar to most processes in this category. One typically uses a large wooden vat, normally of a square shape and divided into several sections, whose number differs between 7–9. These sections have the same size, but not the same height, so that the fluid always drains from one into another, from the first, for instance, into the second and so on. The water, which is poured into the first section of the case, moves bit by bit through the others and everywhere it is stirred a little. The water deposits in proportion the lead white it carries with it, and the precipitation of lead white in the last section is the finest. If one has washed lead white in this manner, one puts it into large vessels, where one washes it again, always leaving it in the water. *Das Waschen des kohlensauren Bleies ist sehr [einfach] und hat mit den meisten Verfahrungsarten [dieser] Kategorie große [Aehnlichkeit]. Man bedient sich gewöhnlich einer großen hölzernen Kufe, gemeiniglich von viereckiger Gestalt und in mehre Fächer abgetheilt, [der] Zahl von 7-9 verschieden ist. Diese Fächer sind von gleicher Geräumigkeit, aber von ungleicher Höhe, so daß [die] Flüssigkeit des einen immer in diejenige des andern über läuft, von der ersten, z. B., in die zweite und so [fort].* *Das Wasser, welches man in die erste Abtheilung der Kufe schüttet, gelangt nach und nach in alle [andren] und überall wird es ein Wenig umgerührt. Das Wasser sezt verhältnißmäßig das Bleiweiß ab, welches es [mit] sich führt, und der Niederschlag von Bleiweiß in der [letzten] Abtheilung ist am Feinsten. Hat man auf diese [Weise] das Bleiweiß gewaschen, so bringt man es in große [Tuber], wo man es nochmals wäscht und immer im Wässer läßt.*

stack-process lead white and vinegar (Le Pileur d'Apligny 1779: 5–6, table 2). Dry, unwashed pigment was transferred to the granite grinding slab and wine vinegar was added dropwise until the consistency of double cream was reached. The paint was ground into a smooth paste, transferred to a glass jar and then washed eight times with distilled water to remove all traces of vinegar. No pigment discoloration as mentioned by Fortunato and others was noted, however vinegar grinding did result in compositional changes. These became evident when the differently treated pigments were analysed with XRD. Comparison of XRD results of untreated lead white, lead white ground with water and that ground with vinegar showed that water washing raised the proportion of cerussite by removing the other forms of lead salts, while washing with vinegar resulted in an even higher concentration of cerussite (Fig. 3). Although no strong difference in the resulting particle sizes or shapes could be noted between both washing methods, grinding in vinegar did seem to result in a slightly raised proportion of small particles as compared to clumps of aggregated particles (Fig. 4a and b).

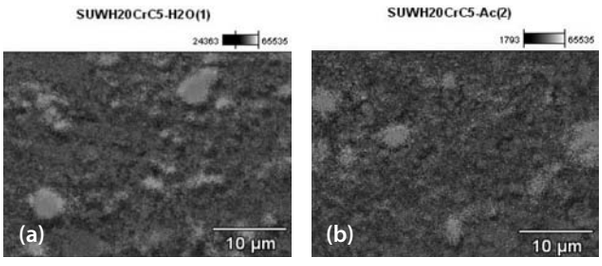

Figure 6 SEM BSE image of paint made with lead white and chalk, water washed, paint prepared with poppy oil. False colour image with all lead counts in red, and all calcium counts in green: (a) first ground with water, and (b) first ground with vinegar. (See Plate 41 in the colour plate section.)

Effect of washing on lead whites in mixture with calcium carbonate

Before the 19th century when barium sulphate became the main extender or adulterant for lead white, calcium carbonate was often mixed with lead white to produce cheaper grades of the pigment (Stols-Witlox 2011). Reconstructions show that when vinegar is brought into contact with lead white mixed with calcium carbonate, gas bubbles appear, as the calcium carbonate reacts with the acetic acid in the vinegar (Fig. 5).

Given this reaction, it was postulated that in some cases grinding with vinegar would have served to lower the calcium carbonate content in adulterated lead whites. To test this hypothesis, a mixture of lead white and calcium carbonate was ground into a fine, smooth slurry with either distilled water or with vinegar then subsequently water-washed to remove any trace of vinegar. Contrary to expectations, XRD did not indicate a significant shift in calcium carbonate content and

ATSR 4 15 Stols.indd 126 17/01/2012 10:18

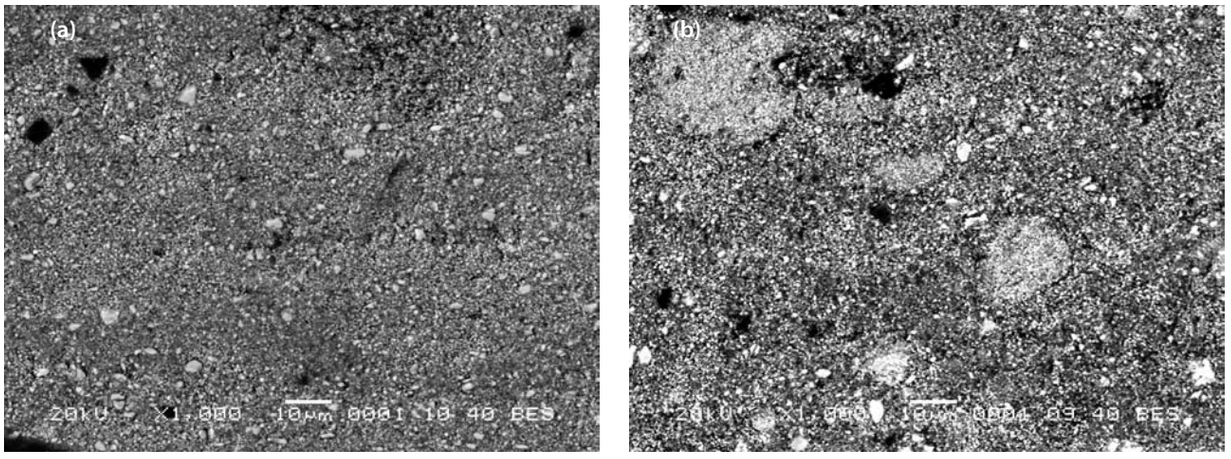

Figure 7 SEM backscattered electron image of a cross-section from paint made with (a) the most coarse part of the lead white: the deposit after decanting, and (b) decanted lead white (first decanting). Paint prepared with poppy oil.

the SEM BSE images made from the paints prepared from 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.[12] 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.[13] 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 all treatments to influence the balance between cerussite and hydrocerussite, all resulting in a higher proportion of cerussite.

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

ATSR 4 15 Stols.indd 127 17/01/2012 10:18

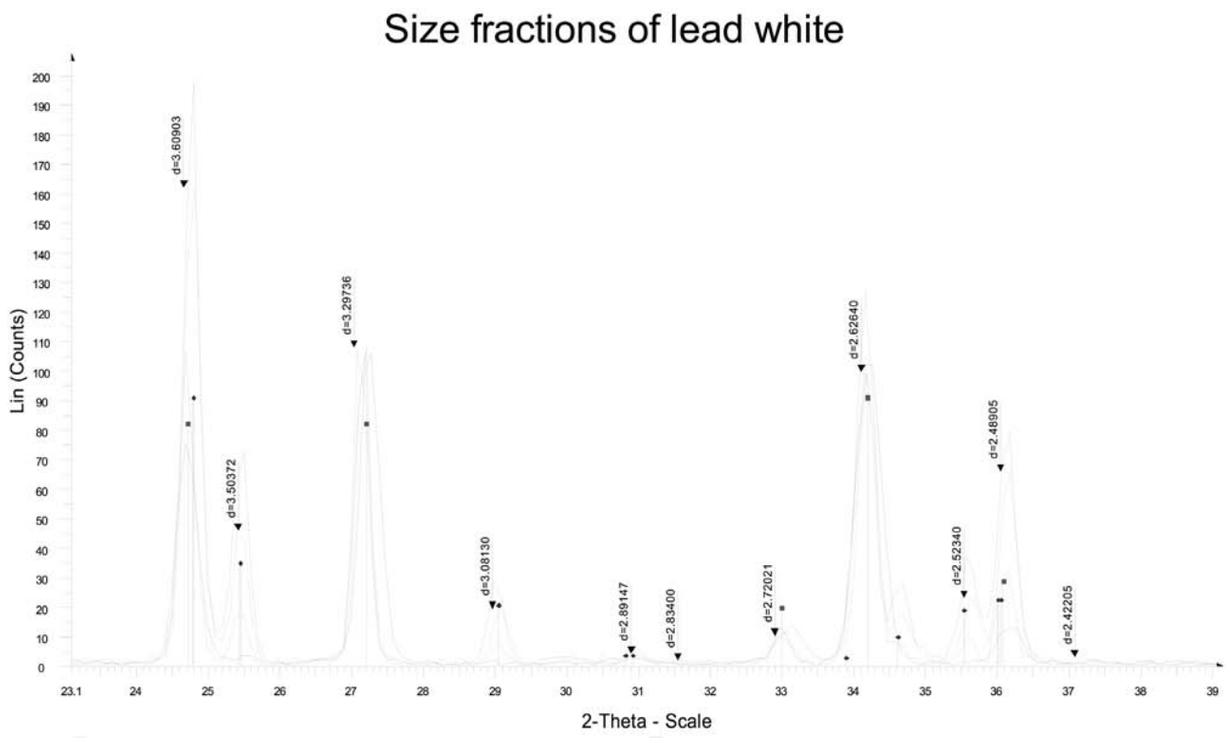

Figure 8 Diffractogram demonstrating the shift in balance between cerussite and hydrocerussite caused by decanting. Untreated lead white (blue), the deposit after decanting (green), the first decanted pigment (yellow), the pigment decanted last (red). Cerussite peaks are indicated with blue vertical lines, hydrocerussite peaks with red vertical lines. An increase in cerussite concentration is clear at the 25,4 position. (See Plate 42 in the colour plate section.)

lead whites. A higher proportion of cerussite therefore does not necessarily create a whiter paint.[14] 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.[15] 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. Argon 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.
3. HART project (2002–2006), led by Leslie Carlyle, within the de Mayerne Programme, sponsored by the Dutch Organisation for Scientific Research (NWO). Doctoral research by Stols-Witlox on the subject of historical recipes for preparatory layers for oil paintings in northwest Europe, 1400–1900, University of Amsterdam.
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 (Brouwland 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. Poppy 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

ATSR 4 15 Stols.indd 128 17/01/2012 10:18

cedar oil on a Silicon zero diffraction plate. Diffractograms were acquired in reflection mode with CuKα 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 scientific energy-dispersive X-ray spectrometry system with Noran System Six software.
10. Konica Minolta CM-2600d spectrophotometer, white calibration 7004450.
11. Carlyle (2006). Analysis performed by Katrien Keune.
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.

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Authors' addresses

- Maartje Stols-Witlox, University of Amsterdam, Ateliergebouw, Hobbemastraat 22, 1071 CZ Amsterdam, The Netherlands. (m.j.n.stols-witlox@uva.nl)
- Luc Megens, Cultural Heritage Agency of the Netherlands, Ateliergebouw, Hobbemastraat 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)

ATSR 4 15 Stols.indd 129 17/01/2012 10:18