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Dik, J.

**Publication date**  
2003

[Link to publication](#)

### **Citation for published version (APA):**

Dik, J. (2003). *Scientific analysis of historical paint and the implications for art history and art conservation. The case studies of naples yellow and discoloured smalt.*

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# NORTHERN EUROPEAN 18<sup>TH</sup> CENTURY LEAD ANTIMONATE YELLOW

### ABSTRACT

Lead antimonate yellow or Naples Yellow was the most common synthetic yellow pigment in 18th century European painting. Recent studies have shown that Italian artists made use of the pigment at much earlier dates. North European painters, however, hardly applied Naples Yellow before 1700. Instead, the traditional pigment lead tin yellow remained highly popular until about 1730, when it was replaced gradually by Naples Yellow. When the pigment was first imported from Italy, the origin of Naples Yellow remained a mystery to North European artists and craftsmen. Only during the second half of the 18th century Germans, Frenchmen and Dutchmen seemed to have learned to produce lead antimonate themselves.

In this paper the history of use and production of 18th century Northern European Naples Yellow is examined. Naples Yellow was reconstructed according to the first North European historical production recipes. Samples were taken from a number of paintings. Scientific analyses were carried out using X-ray powder diffraction (XRPD). The results show that early Northern European Naples Yellow differs in chemical composition from earlier Italian types. These findings should therefore be of interest for authentication studies. In addition, an attempt is made to explain why Naples Yellow replaced lead tin yellow.

## INTRODUCTION

Lead antimonate -or Naples Yellow- is a yellow pigment that is synthesized by calcinating a mixture of lead and antimony salts. During the Middle Ages lead antimonate usage seems to have been limited to the Islamic and Slavonian world. Through contacts with Venetian merchants the know-how on the production of lead antimonate was passed on to the Western culture during the 15th century.<sup>1</sup> The first reference to Naples Yellow as a painting pigment dates back to ca. 1600, when the author of the Mariani manuscript gave a detailed description of the production of *giallo di vasari* (potter's yellow) or lead antimonate yellow.<sup>2</sup> Mariani described three sorts of potter's yellow, including a tin or zinc containing lead antimonate phase, which have been discussed in the previous chapter.

It is generally assumed that lead antimonate was introduced in North European painting in the first quarter of the 18th century.<sup>3</sup> Lead tin yellow, the traditional yellow pigment, was slowly replaced by lead antimonate and by the 1740's lead tin yellow had fallen in complete disuse.<sup>4</sup> The first Northern documentary reference to Naples Yellow can be found in the correspondence of the Dutch painter Hendrick van Limborch. In a letter dated 18 June 1708, the Rotterdam painter Van Limborch mentions Naples Yellow, stressing its intense opaque property.<sup>5</sup>

Apparently, Northern artists were not aware of the synthetic origin of lead antimonate. A number of Italian, handwritten recipes of much earlier date are in existence, as discussed in chapter 2, but these sources, strictly intended for personal use, were not widely known and certainly not to a Northern European audience. According to a widespread 18<sup>th</sup> century rumour Naples Yellow was a volcanic mineral that was found around the Vesuvius in the Bay of Naples. Hence the name Naples Yellow was coined. The terminological aspect and the mystification of its origin will be discussed below. The production process of Naples Yellow was not published until 1758 by the Italian Passeri.<sup>6</sup> It should be stressed, however, that Passeri published the production recipe under the old Italian name of *giallolino*, which may explain why his publication remained largely unnoticed in northern Europe and was not referenced to in any of the 18<sup>th</sup> or 19<sup>th</sup> century literature on Naples Yellow. A much more authoritative article by Fougereux de Bondaroy, member of the French *Académie Royale des Sciences*, appeared only a few years later<sup>7</sup>. Fougereux has ever since been acknowledged of revealing the real origin of Naples Yellow to a wider, Northern European public. De Bondaroy carried out several experiments and mentioned a production recipe for Naples Yellow. In 1797 de Massoul stated that Naples Yellow was produced by one single supplier from Naples :

*Till the present time, the supply was drawn from Naples: formerly one person furnished the whole.*<sup>8</sup>

Like De Bondaroy, De Massoul published a production recipe. Both recipes call for similar ingredients, but both differ substantially from Italian production methods, including those described in earlier manuscripts as well the contemporary recipe given by Passeri.

Thus, literature suggests that Naples Yellow was imported from across the Alps during the first decades of the 18th century and that North European production of the pigment did not start before the middle of the eighteenth century. Therefore, the question rises whether 18<sup>th</sup> century lead antimonate yellows of Northern European origin vary chemically from earlier, notably Italian types. We therefore carried out an examination of 18th century North European Naples Yellows, involving reconstructions of historical recipes and analysis of paint samples. Our aim was to establish what types of Naples Yellow could have resulted from a new North European production method.

### THE TERMINOLOGY OF NAPLES YELLOW

The term Naples Yellow, or *Luteolum Neapolitanum*, first appeared in a treatise by Andrea Pozzo in Rome, published between 1693-1700.<sup>9</sup> Ever since the term remained in use to describe the colour of lead antimonate yellow. Soon after 1700 the first references to Naples Yellow can be found in North European literary sources. The Dutch painter Hendrick van Limborch mentioned Naples Yellow in a letter dated 18 June 1708.<sup>10</sup> The author of the *Eikelenberg* manuscript, written between 1700 and 1720, also commented on the new pigment. Eikelenberg touched on the origin of Naples Yellow and wrote:

*Naples Yellow is, as I have been told, the ash of Etna and more yellow than Schietgeel.*<sup>11</sup>

This widespread rumour of the volcanic origin is probably based on a 16<sup>th</sup> century description of *giallolino* by Cennini, who suggested that the pigment originated from stones found around volcanic craters.<sup>12</sup> The same rumour was continuously repeated in 18th century literature on Naples Yellow. In 1757 Peretny mentioned that Naples Yellow is a mineral or some sort of earth product from the area of Naples.<sup>13</sup> In his treatise of 1765, four years before De Bondaroy's publication, De Montamy gave a detailed description of the pigment, its origin and its pigment properties. Also, De Montamy stated that Naples Yellow is a volcanic product referring to contemporary geological studies of Mount Etna :

*Everybody agrees that Naples Yellow is a type of yellow rock that is extracted from the area around Naples.*<sup>14</sup>

Even well into the 19<sup>th</sup> century some authors still adhered to the notion that Naples Yellow was a natural product, although doubts were expressed about its true origin, as Robert Dossie mentioned in 1841 :

*It has been supposed to be a native earth, and is said to be found in the neighbourhood of Naples; but this is dubious, as the different parcels of it vary so much from each in specific qualities to allow the supposition of their being native earths.*<sup>15</sup>

#### FOUGEROUX DE BONDARROY

The French *Académie Royal des Sciences* was founded in 1666 under the ministry of M. Colbert, who had been appointed by Louis XIV.<sup>16</sup> Under Colbert's leadership, France was to strengthen its own economy, mainly by raising the level of self-support of French industries and limiting the amount of imports. This policy of autarky that later became known as *Colbertism*, notably concerned the arts and crafts and was strongly supported by the *ancien régime* until the French Revolution.<sup>17</sup> The foundation of the French *manufactures*, such as the porcelain factories of Sèvres, for example, was aimed at giving a boost to French economical and cultural development at the cost of foreign products. Amongst others, the role of the *Académie Royal des Sciences* was to provide applied scientific support to these developments. At the end of the 17<sup>th</sup> century the *Académie* envisaged a large publication on the technical description, entitled *Description des Arts et Métiers*, which was not issued until 1761 under the auspices of De Bondaroy's uncle, Henry-Louis Duhamel de Monceau.<sup>18</sup>

As has been shown elsewhere, Naples Yellow played a major role in the 18<sup>th</sup> century as the dominant synthetic yellow pigment in painting and the colouring of glass and ceramics.<sup>19</sup> As mentioned above, French artists and craftsmen were dependant on the import of this pigment from Italy, which may have been considered a restraint for raising autarky of French art production. In view of these political considerations, a strong interest of the *Académie* in Naples Yellow and its real origin is not surprising.

A production recipe for Naples Yellow was finally revealed by Fougieroux de Bondaroy (1732-1789), a member of *Académie*. De Bondaroy studied botanics and sciences and was admitted as a member in 1758. His main areas of interests were mineralogy and the technology of arts and crafts. Like many aristocratic young men, De Bondaroy made a lengthy journey to Italy, visiting all major Italian cities in the summers of 1763 and 1764. De Bondaroy kept a diary of five volumes in total, four of which have been conserved, spread over three different libraries in Italy and France. The fifth volume is considered lost. The initial plan of publishing his diary was never carried out, although many of his later publications seem to have been based on experiences during his travel. The handwritten

journal and other documents, including various drawings by De Bondaroy were studied recently, giving a rather precise account of his main interests and the itinerary of his travel.

De Bondaroy had a large interest in the technology of arts and crafts, such as the textile and glass industries and the production of artificial gemstones and pigments. He visited the main production sites of these crafts such as the jewel workshops of Genes and the glass production island of Murano in Venice. De Bondaroy noted that all craftsmen working in these industries were highly secretive about their handicraft:

*just like in France, everyone makes a mystery of his own working method.*<sup>20</sup>

In addition, De Bondaroy paid a visit to the area of Naples, where he carried out mineralogical research of volcanic compounds of the Vesuvius. De Bondaroy must have read Passeri's work on Naples Yellow during his stay in Italy, though he doesn't mention this source in his own work. Passeri's recipe for Naples Yellow, which he calls *giallolino*, is almost identical to some of the recipes mentioned by Piccolpasso and Mariani in their 16<sup>th</sup> century manuscripts :

*Giallolino, or the colour of gold, is made of a pound of antimony, half a pound of lead, an ounce of allume di fecia and another ounce of common salt.*<sup>21</sup>

In short, his travel to Italy, sponsored by the French *Académie*, gave De Bondaroy insight into the real origin of the pigment known as Naples Yellow. Two years after his return to France, De Bondaroy published the production recipe for Naples Yellow, after the pigment had been declared a mineral for more than five decades.

## RECIPES

Based on his experiences in Italy and several experiments carried out by himself, de Bondaroy published the following recipe for Naples Yellow:

*I put together 12 ounces of ceruse, 1 ounce of alum, 1 ounce of sal ammoniac and 3 ounces of diaphoretic antimony. I mixed this as well as possible and put it in a flat plate, unvarnished and covered; I exposed it to a moderate fire for seven or eight hours (e). The plate that I had used, I retrieved it from the fire, I obtained a piece of stone, more or less bound, which became soft when crushed up, greasy to touch and of the same colour as Naples Yellow, and it had all its properties. I also made it with blanc de plomb, diaphoretic antimony, sal ammoniac and alum, and I've had a beautiful yellow and heavier.*

(e) A less violent fire, but continued longer, gives a better result; the fire of the reverberatory<sup>22</sup> of the potters, the plate being placed in the upper part of the oven is preferable to all others.<sup>23</sup>

A similar recipe was published in 1797 by De Massoul, which seems to be a copy of the recipe by De Bondaroy with a minor adjustment concerning the reaction time :

*It has been found that Naples Yellow is produced by art, and is a metallic calx. It is now brought to equal perfection with that of Naples, by incorporating with Spanish White, calcined Alum, Salt Ammoniac and Diaphoretic Antimony, all pure. Having well pounded this mixture put in a flat earthen pan, which must not be varnished; cover it, and let it remain upon moderate fire, during seven or eight hours. It would succeed better, were it placed for twelve hours, at the top of an oven, upon a reverberated potter's fire.*<sup>24</sup>

## INTERPRETATION OF SOURCES

As earlier research of technical sources has shown, the historical terminology in source literature is not always clear.<sup>25</sup> This is also the case in the recipes on lead antimonate yellows studied. De Bondaroy's *sel ammoniac* and De Massoul's *Salt Ammoniac* most likely correspond to sal ammoniac, the mineral name for ammonium chloride. However, the meaning of other ingredients, including *Spanish white*, *Diaphoretic Antimony* and *calcined Alum* is not precisely clear. Both authors made detailed remarks on the reaction time required, but only vague indications are given concerning the reaction temperature.

### *Blanc de Plomb, Céruse, Spanish White*

At first sight, all three terms (Blanc de Plomb, Céruse, Spanish White) seem to be synonyms for lead white, or basic lead carbonate  $[2\text{Pb}(\text{CO}_3)\cdot\text{Pb}(\text{OH})_2]$ . Spanish white is a rarely used synonym for lead white.<sup>26</sup> However, De Bondaroy makes a clear distinction between *blanc de plomb* and *céruse*. We suspect that different qualities are indicated with these terms, as is the case with the Dutch terms for lead white *schulpwit* and *loodwit*. *Schulpwit* was used for a pure form of lead white, while *loodwit* indicated a mixture of lead white and chalk  $[\text{CaCO}_3]$ .<sup>27</sup> In his recipe De Bondaroy provides a further clue on the difference between *ceruse* and *blanc de plomb*, as he states that the use of *blanc de plomb* results in a yellow that is heavier, or in French *plus pesant*. While the term *heavy* (in English) can be used metaphorically, thus referring to the shade of yellow, *pesant* also may relate directly to the weight of the sample. This difference in weight may have been caused by the use of two types of lead components:

one type consisting of pure lead white and another type of lead white adulterated with chalk and thus having a lower specific weight.

It should be mentioned that the use of lead white was a major change in the production technique of Naples Yellow. Earlier Italian sources steadily call for the use of *piombo abrugiato*, or burnt lead, meaning lead monoxide, as has been shown in chapter 2.

#### *Diaphoretic Antimony, antimoine diaphorétique*

The term diaphoretic is derived from the greek word διάφορητικός. *Diaphoretic* is a term from medicine, which means to revoke sweating. In 18th century medicine various chemicals were said to have such a purgative sweating effect on the human body.<sup>28</sup> A more chemical description of diaphoretic antimony has been given in the 1694 treatise *Histoire generale des drogues* by Pierre Pomet. In chapter VII Pomet wrote:

*The Diaphoretic of Antimony, or chalk of antimony, is the antimony from Poitou and nitric acid incorporated together and by the means of fire and warm water one makes a powder of it, which -when almost dry- is formed into little trunks and then dried properly and one keeps it like this.*<sup>29</sup>

The area of Poitou, around the city of Poitiers in France, indeed proves to be rich in antimony minerals. Antimony is found to the west of Poitiers close to Saint Priest, Limoges, and at Estagnat and Villechaise, to the south of Confolens.<sup>30</sup> Thus, in view of the descriptions given by Pomet, the most likely translation of *Antimoine diaphoretique* seems to be antimony trioxide ( $Sb_2O_3$ ) that is formed by oxidizing metallic antimony with the help of nitric acid.

#### *Alum*

Alum, or *alun* in French, refers to aluminium potassium sulfate dodecahydrate [ $KAl(SO_4)_2 \cdot 12H_2O$ ], as is also suggested in contemporary French dictionaries. The recipe is the first to call for the use of this material in the production of Naples Yellow. Alum was a well-known material in pigment technology since the Antiquity. It was used as a substrate for organic colourants, such as madder and other red lakes. The use of alum in the production of lead antimonate, however, should be considered a novelty, but may have a simple explanation (see discussion).



## MATERIALS AND TECHNIQUES

For the reconstruction experiments use was made of lead white,  $2\text{Pb}(\text{CO}_3)\cdot\text{Pb}(\text{OH})_2$  [PDF 13-131], antimony trioxide,  $\text{Sb}_2\text{O}_3$  [PDF 5-534], *alum* or aluminium potassium sulfate dodecahydrate,  $\text{KAl}(\text{SO}_4)_2\cdot 12\text{H}_2\text{O}$  [PDF 7-17], *sal ammoniac* or ammonium chloride,  $\text{NH}_4\text{Cl}$  [PDF 2-887]. X-Ray powder diffraction (XRPD) was used to ascertain the identification of these materials. A mixture of the ingredients was made according to the weight percentage mentioned in the recipe: 12 parts lead white, 1 part alum, 1 part sal ammoniac and 3 parts antimony trioxide. The ingredients were mixed in a mortar and ground to a fine powder. The mixtures were then put in high temperature crucibles (crucible type 131-01, *Haldenwanger*, Berlin, Germany) and heated at various temperatures during a period of 8h in a preheated oven (type LF1, *Vecstar*, United Kingdom). After cooling the reaction products were ground again and measured using XRPD. Diffraction data were collected using a Phillips *X'Pert Pro* diffractometer (*Philips*, Eindhoven, the Netherlands).

Samples of Naples Yellow were taken from various historical paintings, mostly from the yellow paint of gold jewels or embroidery of 18th century portrait paintings. Most portraits were signed and dated and in some cases the exact geographical origin was known. In all instances samples were taken only after receiving a written consent of the owner of the pictures. The structure and composition of these paint samples was determined by XRPD. Use was made of an *Enraf-Nonius* FR 552 Guinier Johansson camera (*Enraf-Nonius*, Delft, The Netherlands) equipped with a Johansson monochromator using  $\text{CuK}\alpha_1$  radiation ( $\lambda=1.54060 \text{ \AA}$ ). The samples were prepared by pressing the powder to a thin layer onto mylar foil. In order to improve particle statistics the sample holder was rotated in the specimen plane. For indexing of the pattern the accurate positions of as many lines as possible were collected by reading out the Guinier photographs with an optical instrument. Special software was used for peak analysis as well as multiple phase identification, including *Profit* version 1.0b, *X'Pert Plus* version 1.0 (*Philips*, Eindhoven, the Netherlands) and *PCPDFWIN* version 2.1.<sup>31</sup>

table 1 phase composition of reconstructions of De Bondaroy recipe

temp in °C	650–700–750–800	900–1000–1100
Reaction Products	$\text{Pb}_2\text{Sb}_2\text{O}_7$ (<75%) $\text{K}_2\text{Pb}(\text{SO}_4)_2$ $\text{Pb}_3\text{O}_4$ $\text{Sb}_6\text{O}_{13}$	$\text{Pb}_2\text{Sb}_2\text{O}_7$ (>75%) $\text{K}_2\text{Pb}(\text{SO}_4)_2$ + minor other phases

## RESULTS

The reaction products of the reconstruction experiments are summarized in table 1. The formation of lead and antimony oxides was observed at lower temperatures ( $T<900^\circ\text{C}$ ). Presence of well-crystallized cubic  $\text{Pb}_2\text{Sb}_2\text{O}_7$  [PDF 74-1354] started at  $900^\circ\text{C}$ . At lower tempera-

tures  $\text{Pb}_2\text{Sb}_2\text{O}_7$  showed broader peaks. All other materials present gave well-defined diffraction maxima at all temperatures. Interestingly, at all reaction temperatures investigated minor amounts of  $\text{K}_2\text{Pb}(\text{SO}_4)_2$  [PDF 29-1015] proved to be present in the product.

Most paint samples examined turned out to consist of a Naples Yellow phase as well as varying amounts of lead white. The admixture of lead white to yellow paint was a common practice of artists and its presence in the paint samples is not surprising. The Naples Yellow part of certain samples (table 2) showed a multiphase composition similar to the De Bondaroy reconstruction sample synthesized at  $T > 900^\circ\text{C}$ . In these samples  $\text{Pb}_2\text{Sb}_2\text{O}_7$  and  $\text{K}_2\text{Pb}(\text{SO}_4)_2$  were identified as main and minor phase respectively. The diffractograms and peak lists of the De Bondaroy  $900^\circ\text{C}$  sample as well as sample D86 that is representative for this group of paint samples are shown in Appendix I, figures I.1 and I.2 and tables I.1 and I.2.

A second group of paint samples proved to consist of pure cubic  $\text{Pb}_2\text{Sb}_2\text{O}_7$ . In these samples no by-products could be detected (table 3). The presence of lead tin yellow type II,  $\text{Pb}_2\text{SnO}_4$  [PDF 75-1849], mixed with varying, minor amounts of  $\text{SnO}_2$  [PDF 41-1445] was established for a third group of painting samples (table 4). The use of lead oxide chloride, or  $\alpha\text{-Pb}_7\text{O}_6\text{Cl}_2$  [PDF 6-393], also known as *Turner's Yellow*, was identified in two cases (table 5).

table 2  $\text{Pb}_2\text{Sb}_2\text{O}_7$  with small amounts of  $\text{K}_2\text{Pb}(\text{SO}_4)_2$  identified in 18<sup>th</sup> century yellow paint

artist	Painting	date	geographical origin	collection	Inv. nr.	Sample ID.
Hubert Robert	Artist in his studio	after 1770	Paris (?), France	Museum Boymans-v.B	2586	D85
Roland Delaporte	Still life with fruit and bread	after 1760	Paris, France	Museum Boymans-v.B	1121	D86
François Boucher	Chinoiserie	1740-1770	Paris, France	Museum Boymans-v.B	2573	D87
Circle of August Querfurt	Hunting scene	1750-1760	Germany	Private collection	n.a.	D95
Follower of Francesco Fontebasso	Death of Sophonisba	2 <sup>nd</sup> half 18 <sup>th</sup> c.	Italy	Private collection	n.a.	D96
Spanish School	Saint Francis Xavier	2 <sup>nd</sup> half 18 <sup>th</sup> c.	Spain	Private Collection	n.a.	D72
Pietro Bardellino	Moses and Aaron	18 <sup>th</sup> c.	Italy	Private Collection	n.a.	D73

table 3  $\text{Pb}_2\text{Sb}_2\text{O}_7$  without by-products identified in 18<sup>th</sup> century yellow paint

Artist	Painting	date	geographical origin	collection	Inv. nr.	Sample ID.
Nicolas Lancret	The tease	1730-1740	France	Huis Doorn	HuD 1718	D90
Willeboirts Bosschaert	Madonna with Child		Flanders	Rijksmuseum	n.a.	AW 98/3
Circle of Giovanni Paolo Spadino	Still life with fruits	early 18 <sup>th</sup> c.	Italy	Private Collection	n.a.	D97

Jan Toorenvliet	Scene in a backyard	early 18 <sup>th</sup> c.	Italy ? Holland ?	Private Collection	n.a.	D57
Follower of Charles Lebrun	King Louis XIV as Roman Emperor	17 <sup>th</sup> c.	France	Private Collection	n.a.	D58

table 4 **Pb<sub>2</sub>SnO<sub>4</sub> with small amounts of SnO<sub>2</sub>** identified in 16<sup>th</sup> to early 18<sup>th</sup> century yellow paint

Artist	painting	date	geographical origin	collection	Inv. nr.	Sample ID.
A. Houbraken	Eternity seated by a Fire	early 18 <sup>th</sup> c.	Holland	Private Collection	n.a.	D7
Follower of Frans Pourbus	Portrait of a Noblewoman	16 <sup>th</sup> c.	Netherlandish	Private Collection	n.a.	D6
Jan Mijtens	Portrait of a family	17 <sup>th</sup> c.	Holland	Private Collection	n.a.	D12
Follower of Jan Breughel I	The sense of smell	17 <sup>th</sup> c.	Holland	Private Collection	n.a.	D9
P. Moreelse	Portrait of a Man	early 17 <sup>th</sup> c.	Netherlandish	Private Collection	n.a.	D8

table 5  **$\alpha$ -Pb<sub>7</sub>O<sub>6</sub>Cl<sub>2</sub>** identified in 18<sup>th</sup> to early 19<sup>th</sup> century yellow paint

Artist	painting	date	geographical origin	collection	Inv. nr.	Sample ID.
Follower of Jacob Ferdinand Voet	Portrait of Wilhelm Justern von Stein zu Rechtenstein	ca. 1690	Southern Germany	Private Collection	n.a.	D23
Tobias Stranover	Still-life with Fruits	1686-1724	Hungarian School	Private Collection	n.a.	D35

## DISCUSSION

The examination of 18<sup>th</sup> century yellow paints showed the presence of four groups of synthetic yellow pigments. First of all, the occurrence of lead tin yellow is in line with common observations on this pigment. The pigment was found on paintings of the early 18<sup>th</sup> century and of older date.

Secondly, a number of early 18<sup>th</sup> century samples was found to contain fairly pure amounts of lead antimonate yellow, suggesting a production method that followed earlier Italian methods as discussed before in Chapter 2. Though based on a limited number of paint samples, it seems as if this pure form of lead antimonate yellow is present only on paintings of the early 18<sup>th</sup> century and younger date. It would be interesting to establish a possible terminal date of use of this particular type of Naples Yellow on the basis of a more representative amount of 18<sup>th</sup> century pigment samples. No elemental analysis was performed on our samples, so that the presence of tertiary metals such as Sn or Zn, as described in Chapter 2, can not be excluded completely. The complete absence of any by-products, however, seems to be a strong indication for a lead antimonate without other tertiary metals.

The third group of lead antimonate pigments seems to be associated with the production method as reconstructed above.  $K_2Pb(SO_4)_2$  was identified as minor phase component associated with  $Pb_2Sb_2O_7$  as main phase. It should be mentioned that a few minor peaks at different positions in both the paint samples and reconstruction diffractograms remained unattributed for. In addition, paint samples seem to consist of approximately twice the amount of  $K_2Pb(SO_4)_2$  compared to the reconstruction sample, suggesting the use of a larger amount of alum than mentioned in the original production method. Thus, the phase composition of the reconstruction sample and the painting samples are very similar, but not identical.

The production process by De Bondaroy differs strongly from earlier, Italian production recipes for the pigment. Notably the introduction of alum, or  $KAl(SO_4)_2 \cdot 12H_2O$  as ingredient must be considered a major novelty in the manufacturing of lead antimonate, or Naples Yellow. The formation of minor, but detectable amounts of  $K_2Pb(SO_4)_2$ , a colorless and probably unwanted by-product, may be explained by the addition of  $KAl(SO_4)_2 \cdot 12H_2O$  as initial ingredient. After hydrolysis at high temperatures Pb seems to have replaced Al in the sulphate compound. Small amounts of Al may still be present in the  $K_2Pb(SO_4)_2$  phase, since a separate Al-containing phase could not be detected. It would be interesting to examine individual phases further for the Al-contents. Interestingly,  $K_2Pb(SO_4)_2$  was also found in various Naples Yellow paint samples of French, German, Italian and Spanish 18<sup>th</sup> century origin. As yet, this phase composition has not been found on 17<sup>th</sup> century or earlier types of Naples Yellow. It should be noted that *alum* is not mentioned in any of the known 16<sup>th</sup> and 17<sup>th</sup> century Italian production recipes, including the Mariani, Piccolpasso and Darduin manuscripts. Therefore, the use of alum seems to be an 18<sup>th</sup> century novelty in the production process that may explain the presence of  $K_2Pb(SO_4)_2$  in 18<sup>th</sup> century samples of lead antimonate yellow. However, pending further research of earlier samples of Naples Yellow a *terminus post quem* for the occurrence of lead antimonate mixed with minor amounts of  $K_2Pb(SO_4)_2$  can not be established. At present it cannot be excluded that De Bondaroy was not the first to use alum in the production of Naples Yellow. The widespread finding of  $K_2Pb(SO_4)_2$  in Italian, French and German mid-18<sup>th</sup> century lead antimonates suggests that the pigment was produced using alum as ingredient already before De Bondaroy published his recipe in 1769. As mentioned, De Bondaroy's publication does not refer to earlier or contemporary publications on the synthesis of Naples Yellow. One would have expected a reference to the 1758 publication by Passeri, to which De Bondaroy must have had access during his trip to Italy in 1762. Possibly, De Bondaroy reproduced the knowledge on the production of Naples Yellow, including the use of alum, from an earlier, unknown source.

Besides lead and antimony, a number of tertiary compounds have been mentioned in Naples Yellow recipes, in particular *feccia di vino calcinato*, *allume di feccia*, and *feccia*. All these terms seem to refer to the same compound, potassium hydrogen tartrate. One could assume that De Bondaroy, or an unknown previous author, erroneously translated the term *allume di feccia* as regular alum or aluminium potassium sulfate dodecahydrate. Thus, the use of this

tertiary material, suggested by De Bondaroy and later on taken over by many others,<sup>32</sup> could be explained by a simple translation mistake with far-reaching consequences.

Lead oxide chloride or  $\alpha\text{-Pb}_7\text{O}_6\text{Cl}_2$  was found on two paintings of early 17<sup>th</sup> and mid 19<sup>th</sup> century respectively. The origin, production and occurrence of this yellow pigment are not well understood. The pigment has been synthesized for many centuries though on a marginal scale.<sup>33</sup>  $\alpha\text{-Pb}_7\text{O}_6\text{Cl}_2$  is prepared by heating a mixture of lead oxide with sodium chloride or sal ammoniac. Possibly, the latter ingredient inspired De Bondaroy for using it in the production of Naples Yellow. The pigment seems to be lightfast and has a fairly good hiding power.  $\alpha\text{-Pb}_7\text{O}_6\text{Cl}_2$  has been known under various aliases such *patent yellow*, *mineral yellow*, *Turner's yellow* and *Montpellier yellow*.<sup>34</sup> Historical specimens of this compound, kept in mineral collections, have long been regarded as mineralogical samples under the term "lorretoite". Like the 18<sup>th</sup> century Northern European assumption of Naples Yellow being a volcanic mineral, the anthropogenic origin of "lorretoite" remained hidden until revealed by White in 1979.<sup>35</sup> Further insight into the history of this pigment would require a separate research project similar to the present study on lead antimonate yellow.

#### THE DISCONTINUATION OF LEAD TIN YELLOW AFTER CA. 1750

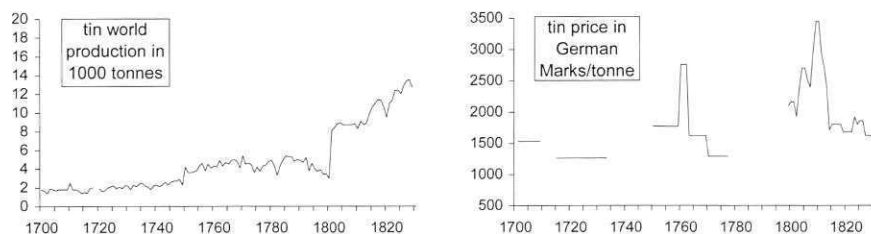
When Naples Yellow appeared in painting studios, first in Italy, later in North European painting workshops, the traditional yellow pigment was lead tin yellow. This pigment has been synthesized since Roman antiquity and remained in use during the Middle Ages. One of the production centres of this pigment must have been Flanders, since the Mariani Ms. and other Italian sources refer to it as *Giallo di Fiandri*.

For some unknown reason, the production and use of lead tin yellow came to a halt somewhere around the middle of the 18<sup>th</sup> century. An explanation for the gradual but complete disuse of this pigment has not been given so far.<sup>36</sup> From a technical point of view, the discontinuation of lead tin yellow is a complete mystery. Lead tin yellow has excellent pigment properties. Lead tin yellow is a perfectly stable, lightfast compound. It has positive drying effects on oil and gives good hiding powder with oily binding media. The pigment can be ground to a very fine powder and still retain its brilliant yellow colour. In addition, lead tin yellow can be manufactured in a variety of yellow hues, depending on temperature, similar to the production of lead antimonate. In general, lead tin yellow and lead antimonate yellow have very similar material characteristics. It must therefore be questioned whether 18<sup>th</sup> century painters were able at all to distinguish between both pigments. Thus, the positive handling characteristics, the excellent optical properties and -above all- its similarity to lead antimonate give no obvious reason for the substitution of lead tin yellow around the 1750's.

However, from an economical point of view the discontinuation of lead tin yellow is less surprising. While the production of tin remained fairly stable around the middle of the 18<sup>th</sup> century, fragmentary data on price development for tin seem to show a peak around 1760

(figure 1).<sup>37</sup> Similar data do not exist for antimony, but the price for antimony is expected to be lower than for tin.<sup>38</sup>

**figure 1** Development of global tin production and price development of tin in German Marks/tonne during the 18<sup>th</sup> century



Apart from economical reasons, also other factors could have contributed to the change from lead stannate to lead antimonate. One important element is the complete lack of written sources describing the production of lead tin yellow. Although the pigment has been mentioned in various sources -under vague and possibly different nomenclature-, the exact production recipe does not seem to have been published. Not a single manuscript, let alone a more widely available publication, on how to produce lead tin yellow has been preserved. Indeed, the availability of written sources on how to produce Naples Yellow may have been a decisive factor in the final disuse of the lead tin yellow.

The transition of lead tin yellow to lead antimonate yellow took place around the middle of the 18<sup>th</sup> century. During this period, the age of Enlightenment, the natural sciences were beginning to take shape and painting materials, among other fields, became the subject of various scientific studies. A pigment like Naples Yellow with its mysterious origin must have been an appealing topic to these early scientists, such as De Bondaroy. Since this renewed interest for Naples Yellow seems to coincide with the discontinuation of lead tin yellow around the middle of the century, a causal relation between both developments may exist.

One other factor may have played a role. Could it be possible that lead tin yellow was substituted, simply because Naples Yellow appealed much more to the imagination of painters and craftsmen than lead tin yellow? At first thought, the choice of pigments for a painting or art object seems to be a practical, down-to-earth selection of colouring materials, made by the artist without any immaterialistic considerations. Recent pigment studies, however, have shown that the selection of pigments may have had deliberate art theoretical connotations in certain cases, as has been demonstrated by McKim-Smith.<sup>39</sup> The origin of a pigment, its source, and other connotations may have been decisive factors in the choice of pigments, as shown by the following example.

Hieronymus Bosch, painter of the famous triptych *Garden of Earthly Delights*, depicted the Expulsion from Paradise on the inner left wing.<sup>40</sup> The scene takes place under the shadow of a giant dragon tree, or *Draco Draconis L.* This dragon tree is the natural source for an organic red colourant, known as dragonblood that was mostly used during the Middle Ages. The resin of the dragon tree is coloured to a deep red by a fungal organism on the bark of the tree. According to a widespread rumour, however, the dragonblood tree grew from the blood of dragons. This mysterious origin of the colourant must have made a deep impression on Middle Age contemporaries. The pigment has a very poor lightfastness and at the same time must have been extremely expensive, since it had to be imported from the Canary Islands, natural home of the dragon tree. Yet, the pigment enjoyed great popularity among Middle Age painters and craftsmen. The use of *dragonblood* can only be explained by its mysterious, fantastic reputation, which must have had an enormous appeal to painters.

This type of considerations may also help to explain why lead tin yellow was replaced by Naples Yellow. When Naples Yellow entered Northern European painter's workshops Italy was still considered to be the artistic epicentre of painting. Italian Renaissance masters were regarded as unrivalled artists, whose works were copied and imitated. Among these role model artists were Raphael, Michelangelo, Corregio and Titian. Sir Joshua Reynolds (1723-1792), first president of Britain's Royal Academy of Art, exemplified this view in his famous *Discourses*.<sup>41</sup> Many young Northern European artists spent one or more years in Italy before establishing a painter's workshop in their native country, as the mid-18<sup>th</sup> century Dutch painters' biographer Johan van Gool mentioned.<sup>42</sup> In this context it should be noted that early 18<sup>th</sup> century Northern European use of lead antimonate yellow seems to occur with artists that had some sort of relation with Italy, as pointed out by Groen and De Keijzer.<sup>43</sup> More in general, the formal education of young academics was concluded by making a *Grand Tour* through Italy. It can therefore be argued that Naples Yellow, a painting material supposedly used in Italian painting, exercised a similarly strong appeal to Northern European painters. How could an artist imitate -let alone surpass- Italian painting without the appropriate painting materials? Such considerations would certainly have made an appealing marketing slogan for the pigment dealer offering Naples Yellow for sale.

In conclusion, three factors -separately or in combination- may explain why lead antimonate yellow replaced lead tin yellow. First of all, antimony may have been a cheaper alternative for the production of the pigment. Secondly, the lack of written information on lead tin yellow and a simultaneous scientific interest for Naples Yellow seem to have contributed to the oblivion of lead tin yellow. In third place, as pigment of supposed Italian origin, Naples Yellow must have been an appealing pigment to artists during a period in which Italian artworks were still regarded as the summit of the art of painting.

## CONCLUSION

This study has shown that 18<sup>th</sup> century recipes for the production of Naples Yellow differ from earlier Italian descriptions of the pigment. The French scientist Fougeroux de Bondaroy published the first Northern European production recipe in 1769. His method of synthesis includes *alum* as ingredient, which is a significant process innovation. The use of alum causes the formation of minor amounts of  $K_2Pb(SO_4)_2$  as by-product of the dominant yellow phase  $Pb_2Sb_2O_7$ . This characteristic phase composition was found in paint samples of various 18<sup>th</sup> century paintings. In addition, pigment reconstructions made following the details given by De Bondaroy proved to consist of  $K_2Pb(SO_4)_2$  and  $Pb_2Sb_2O_7$  as minor and major phases respectively. Although no clear terminal date for the occurrence of this type of Naples Yellow could be established, the *terminus post quem* seems to be in the mid or early 18<sup>th</sup> century. This finding should be of interest for authenticity studies.

Naples Yellow replaced lead tin yellow during the first decades of the 18<sup>th</sup> century as dominant yellow pigment. After 1750 the pigment lead tin yellow is discontinued completely. From a technical point of view the replacement of lead tin yellow cannot be explained, as pigment properties of both materials are almost identical. However, economical and art theoretical factors can be invoked to explain the replacement of lead tin yellow. In addition, the lack of written sources on the production of lead tin yellow may have played a role.

## ACKNOWLEDGEMENTS

This project was enabled through support of auction house Christie's Amsterdam. The Old Master Picture Department (drs Marina Aarts, drs Hanneke Verschuur and drs Hagar Cappers) provided support in the collection of sample material from paintings. The owners of the pictures examined are acknowledged for allowing sampling of their works of art. Drs Friso Lammertse and Mrs. Annetje Boersma are thanked for the sampling of paintings from Museum Boymans-van Beuningen. Drs D. Verroen is thanked for the sampling of paintings from Museum Huis Doorn. Prof. Roger Burt of Exeter University is acknowledged for helpful comments on European mining history. Ed Sonneveld and drs Kees Goubitz from the Laboratory for Crystallography, Universiteit van Amsterdam are thanked for their help in collecting and interpreting XRPD data. Jephtha Dullaert, graduate student at the Institute for Art History, University of Amsterdam, is acknowledged for bibliographical support.



## APPENDIX I

figure I.1 Diffractogram of De Bondaroy reconstruction at 900°C, measured with Philips *X'Pert Plus* at 50mA, 40kV with a stepsize of 0.01° and a holding time of 6s

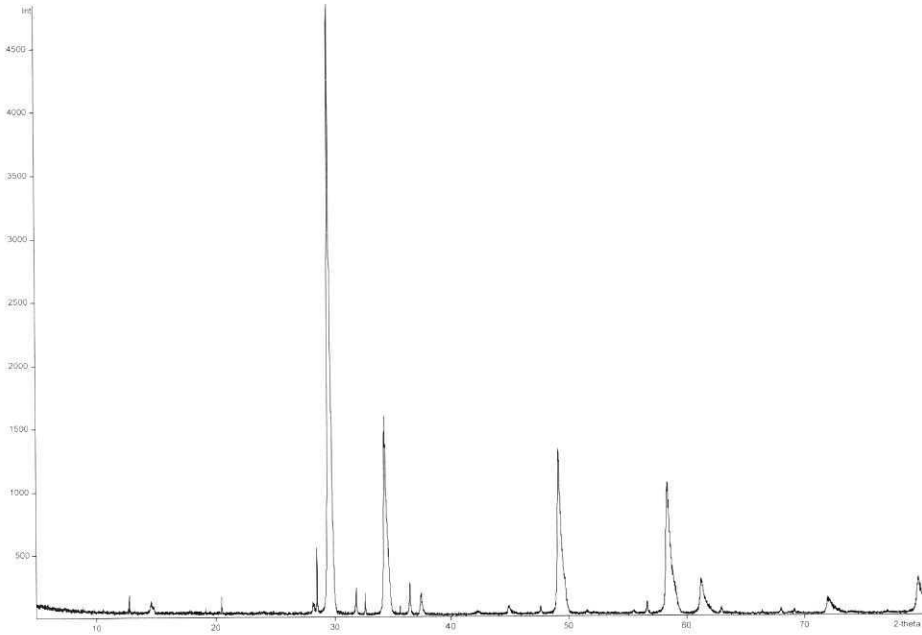


table I.1

peak list of De Bondaroy Naples Yellow, 900°C. In order to compare all phases at the same scale the reflection, intensities of PDF 29-1015 have been multiplied by 10. PDF peaks with  $I/I_0 \leq 10$  are not listed, unless when matching with an observed reflection

De Bondaroy 900°C		$Pb_2Sb_2O_7$ PDF 74-1354		$K_2Pb(SO_4)_2$ PDF 29-1015							
I/I <sub>0</sub>	d value	I/I <sub>0</sub>	d value	I/I <sub>0</sub>	d value						
25	6.92			350	6.948	13	2.5540		350	2.557	
17	6.06	52	6.004						130	2.526	
16	4.64			250	4.641	50	2.4744				
26	4.328			450	4.333	24	2.4053	49	2.3859		
11	3.167			180	3.515						
11	3.1602								250	2.166	
101	3.1309	25	3.1357	999	3.138	3	2.1584		300	2.156	
999	3.0259	999	3.0022			5	2.1394	2	2.1228		
37	2.8138					3	2.0657		350	2.068	
30	2.7506					5	2.0229				
308	2.6215			700	2.749	13	2.0160	20	2.0014		
		352	2.6000			5	2.0063				
						10	1.9109			250	1.910
						237	1.8545	362	1.8384		
						5	1.7729	14	1.7579	200	1.772
						19	1.6244	3	1.5859	200	1.651
						149	1.5815	323	1.5678	110	1.586
						30	1.5687				

36	1.5142	80	1.5011			22	1.3112	41	1.3000		
22	1.5113					7	1.3009				
8	1.4762	6	1.4562			26	1.2023	101	1.1929		
8	1.3779										
7	1.3583	5	1.3539	140	1.361						

figure I.2 peak list of sample D68, measured with Guinier camera diffraction at 20mA, 40kV for 2h

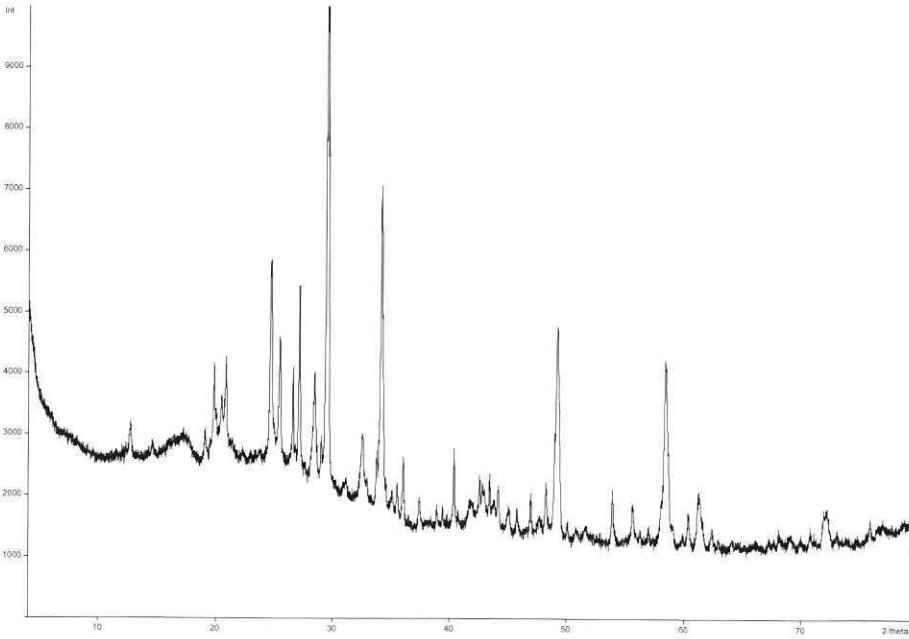


table I.2

peak list of sample D86. In order to compare all phases at the same scale the reflection intensities of PDF 29-1015 and PDF 13-131 have been multiplied by 10. PDF peaks with  $I/I_0 \leq 10$  are not listed, unless when matching with an observed reflection

SAMPLE D86		$Pb_2Sb_2O_7$ PDF 74-1354	$K_2Pb(SO_4)_2$ PDF 29-1015	$2 PbCO_3 \cdot Pb(OH)_2$ PDF 13-131			
I/I <sub>0</sub>	d value	I/I <sub>0</sub>	d value	I/I <sub>0</sub>	d value	I/I <sub>0</sub>	d value
61	6.95			350	6.948		
34	6.07	52	6.004				
54	4.64			250	4.641		
164	4.465					600	4.470
92	4.335			450	4.333		
170	4.2530					600	4.2500
391	3.6001					900	3.6100
240	3.4970			180	3.515		

199	3.3483				
372	3.2829			900	3.290
199	3.1384	25	3.1357	999	3.138
999	3.0223	999	3.0022		
34	2.8724				
131	2.7469			700	2.749
35	2.7153				200 2.715
80	2.6512				
605	2.6249	352	2.6000		999 2.623
41	2.5932				
35	2.5559			350	2.557
60	2.5253			130	2.526
129	2.4893				300 2.491
59	2.4031	49	2.3859		
31	2.3133			100	2.318
38	2.2845				
154	2.2318				500 2.231
29	2.1642			250	2.166
34	2.1565			300	2.156
70	2.1229	2	2.1228		300 2.120
54	2.1111				
46	2.1026				200 2.099
78	2.0837				
36	2.0647			350	2.068
76	2.0492				300 2.046
46	2.0103	20	2.0014		
47	1.9820				
76	1.9337				
26	1.9050			250	1.910
82	1.8868				200 1.884
399	1.8504	362	1.8384		300 1.856
31	1.8199	14	1.7579		
25	1.7965			100	1.792
26	1.7701			200	1.772
103	1.7002				400 1.696
71	1.6502			200	1.651
29	1.6156	3	1.5859		200 1.649
347	1.5794	323	1.5678	110	1.586
52	1.5320				200 1.584
35	1.5299				300 1.530
109	1.5121	80	1.5011		
40	1.4872				300 1.513
36	1.3759				200 1.485
25	1.3591	5	1.3539	140	1.361
28	1.3302				
66	1.3069	41	1.3000		
					300 1.309
					300 1.292
38	1.2531				300 1.251
27	1.2380			130	1.234

## NOTES

- <sup>1</sup> see Chapter 2
- <sup>2</sup> *ibid.*
- <sup>3</sup> H. Kühn, "Lead-Tin Yellow", in : *Studies in Conservation*, vol. 12, 1968, p.7-33
- <sup>4</sup> *ibid.*
- <sup>5</sup> Library of the University of Amsterdam, Afdeling Zeldzame Werken, standnr.I C 23, letter dated 18 juni 1708
- <sup>6</sup> G. Passeri, *Nuova raccolta d'opuscoli scientifici*, Rome, 1758, vol. 6
- <sup>7</sup> M. Fougeroux de Bondaroy, "Mémoire sur le Giallolino ou jaune de Naples", in: *Mémoires de l'Académie Royale*, 1769, p.303-314
- <sup>8</sup> C. de Massoul, *A Treatise on the Art of Painting*, London, 1797, p.137
- <sup>9</sup> M. P. Merrifield, *Original Treatises on the Arts of Painting*, vol. 2, London, 1849, p. 196
- <sup>10</sup> Library of the University of Amsterdam, Dept. 'Zeldzame Kostbare Werken', inv. nr.I C 23, letter dated 18 juni 1708
- <sup>11</sup> Eikelenberg MS, photomechanic reprint at the ICN, Amsterdam, folia 70r :  
*Napels geel is, zoo mij gezegt is, de As van Etna, en geler als Schietgeel*
- <sup>12</sup> V. Thompson, *The Craftsman's Handbook : The Italian "Il Libro dell'Arte"*, New York, 1961, p.28
- <sup>13</sup> A. J. Peretny, *Dictionnaire portatif de peinture, sculpture et gravure*, Paris, 1757
- <sup>14</sup> M. de Montamy, *Traité des couleurs pour la peinture en émail et sur la porcelaine*, Paris, 1765, p. 260-263.
- <sup>15</sup> R. Dossie, *The Handmaid of the Arts*, London, 1841
- <sup>16</sup> A. Richardt, *Colbert et le Colbertisme*, Paris, 1997, p.267
- <sup>17</sup> L. Gossman, *French Society and Culture*, London, 1972, Chapter 2
- <sup>18</sup> M. F. Perez, M. Pinault, "Le voyage en Italie de Fougeroux de Bondaroy", in : *Diz-huitième siècle : revue annuelle*, Société Française d'Étude du XVIIIe Siècle [ed.], Paris, 1969, p.95-105
- <sup>19</sup> I.Wainwright et al., "Lead Antimonate Yellow", in : R. L. Feller, *Artists' Pigments : A Handbook of Their History and Characteristics*, Volume 1, Washington, 1986, p.219-254
- <sup>20</sup> from diary of De Bondaroy, copied from *ibid.*, p.98
- <sup>21</sup> see note 5

<sup>22</sup> A special type of oven, in which the the walls are shaped to reflect the heat and gas stream from the heating elements to the centre of the chamber. Therefore, the position in the chamber is highly temperature-dependant. A position in the *upper part of the oven*, as De Bondaroy mentions, will therefore yield a much lower temperature.

<sup>23</sup> Fougeroux de Bondaroy 1769, p.303-314, p.312 :

*Je joignis à 12 onces de céruse, 1 once d'alun, 1 once de sel ammoniac & 3 onces d'antimoine diaphorétique; je les mêlai le mieux q'il fut possible & les mis dans une terrine plate, non vernissée & couverte; je les exposai à un feu modéré pendant sept à huit heures (e). La terrine que j'avais mise, étant retirée du feu, j'ai obtenu une pierre plus ou moins liée, qui broyée devient douce, grasse au toucher, de la même couleur que le jaune Naples, & qui en a toutes les propriétés. Je l'ai fait aussi avec le blanc de plomb, l'antimoine diaphorétique, le sel ammoniac & l'alun, & j'ai eu un beau jaune & plus pesant.*

*(e) Un feu peu violent, mais long-temps continué, réussit mieux; le feu de réverbère des Potiers de terre, la terrine étant placée à la partie supérieure du fourneau, est préférable à tout autre*

<sup>24</sup> C. de Massoul, *A Treatise on the Art of Painting*, London, 1797, p.138

<sup>25</sup> see earlier recipe terminology in chapter 2

<sup>26</sup> Furetière mentions *Blanc d'Espagne* as a synonym for *blanc de plomb*, see note below

<sup>27</sup> T. Goedings and K. Groen, "Dutch pigment terminology I : A seventeenth century explanation of the word 'schulpwit'", in : *Bulletin of the Hamilton Kerr Institute*, No.2, 1994, p.84-86

<sup>28</sup> A. Furetière, *Dictionnaire Universel*, The Hague/Rotterdam, 1690, and Hatzfeld and A. Darmesteter, *Dictionnaire Général de la Langue Française*, Paris, 1832, p.737

<sup>29</sup> P. Pomet, *Histoire générale des drogues, traitant des plantes, des animaux, des minéraux* (...), Parijs, 1694, vol. III, p.59

<sup>30</sup> A. Lacroix, *Minéralogie de la France et de ses colonies : description physique et chimique des minéraux, étude des conditions géologiques de leurs gisements*, Paris, 1893-1910, vol. 2, p.453-454

<sup>31</sup> © JCPDS-ICDD, June 2000

<sup>32</sup> such as De Massoul, see his recipe

<sup>33</sup> H. Kühn, "Farbmaterialien : Pigmente und Bindemittel", in : H. Kühn et al., *Reclams Handbuch der künstlerischen Techniken*, Band 1, Stuttgart, 1988, p.28

<sup>34</sup> R. D. Harley, *Artists' Pigments c. 1600-1835*, London, 1970

<sup>35</sup> J. S. White, "Lorettoite discredited and chubutite reviewed", in : *American Mineralogist*, vol. 64, 1979, p. 1303-1305

<sup>36</sup> H. Kühn, "Lead-Tin Yellow", in : A. Roy (ed.), *Artists' Pigments : A Handbook of their History and Characteristics*, vol. 2, Washington, D.C., 1993, p.83-112

<sup>37</sup> data taken from C. J. Schmitz, *World Non-Ferrous Metal Production and Prices, 1700-1976*, London, 1979

<sup>38</sup> written communication with professor Roger Burt, University of Exeter, expert on European mining history, 17.6.2002

<sup>39</sup> G. McKim-Smith et al., *Examining Velazquez*, New York, 1988

<sup>40</sup> A. Wallert, M. van Bommel and J. van der Horst, 'Sanguis Draconis: dragons blood', *Dyes in History and Archaeology*, forthcoming volume (2003), 10pp.

<sup>41</sup> E. Waterhouse, *Reynolds*, London, 1973

<sup>42</sup> J. van Gool, *De Nieuwe Schouburg der Nederlantsche Kunstschilders en schilderessen*, 's-Gravenhage, 1751, see also: L. de Vries, *Diamante gedenkzuilen en leerzaeme voorbeelden*, Groningen, Groningen, 1990

<sup>43</sup> K. Groen and M. de Keijzer, "Examination of the Painting Technique of Nine Dutch Pictures of the First Half of the 18th Century", in : *ICOM Committee for Conservation, Preprints for the Conference*, Edinbrugh, 1996, p. 360-366

