1895

White lead

Evans Walker Buskett

THESIS

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FOR THE DEGREE OF

BACHELOR OF SCIENCE

IN

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White lead is a hydrated carbonate of lead. Of the many formulae given $2PbCO_3.Pb(HO)_2$ is probably the most acceptable, but the amounts of $PbCO_3$ and $Pb(HO)_2$ vary very much in the products of different factories. Good white lead is an amorphous white pigment possessing great opacity, density, and covering power, and the property of drying quickly when mixed with linseed, or similar oil, and exposed to air. The hydroxide combines with the oil forming a white elastic coating or paint; but lead hydroxide posesses but little opacity which is given by the carbonate. Thus it will be seen that both are essential when a good paint is desired. White lead is used to a great extent with other pigments, to serve as their base, and to give them body and enable them to dry quickly.

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White lead was known to the ancients under its Greek name psimithium or its Roman name cerussa.

The earliest account of the process of manufacture is by Theophrastus in his "History of Stones" written about 300, B.C. He describes the process as follows: "Lead is placed in earthen vessels over sharp vinegar, and after it has acquired some thickness of a sort of rust, which it commonly does in about ten days, they open the vessels and scrape it off as it were, in a sort of foulness; they then place the lead over the vinegar again, repeating over and over again the same method of scraping it till it has wholly dissolved. What has been scraped off they then beat to powder and boil for a long time, and what at last subsides to the bottom of the vessel is cerusse." Vitruvius, a Roman architect of the first century B.C., says: "It will be proper to explain in what manner white

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lead is made. The Rhodians place in the bottom
of large vessels a layer of vine twigs, over which they
pour vinegar, and on the twigs they lay masses
of lead. The vessels are covered to prevent evaporation,
and when, after a certain time, they are
opened the masses are found changed into
white lead." Pliny, one hundred years later,
says, "Psimithium, which is also known as
cerussa, is another production of the lead works,
and the most esteemed comes from Rhodes."
Pliny describes the process as follows: "It is
made from very fine shavings of lead placed
over a vessel filled with the strongest vinegar,
by which means the shavings become dissolved;
that which falls into the vinegar
is first dried and then pounded and sifted,
after which it is again mixed with
vinegar and is then divided into tablets and
dried in the sun during summer. . . . .

---- It is also made in another way. The lead is thrown into jars filled with vinegar, which are kept closed for ten days; the sort of mould which forms upon the surface is then scraped off, and the lead is again put into the vinegar until the whole is consumed."

Dioscorides, who wrote, in the first or second century, a work on Materia Medica and Botany, says; "Ceruse is made in the following manner; having poured vinegar, as sharp as possible, into a broad mouthed pitcher, or an earthen jar, fasten firmly a mass of lead near the top of the jar upon a mat of reeds, previously stretched beneath, and throw over the jar a cover, that the vinegar may not evaporate until the lead, dissolved and dripping down like rain,

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has disappeared; then having strained off
the clear water, which remains upon the surface,
pour into a vessel that which is viscid;
this must be dried in the sun, presently
pulverized in a hand mill, or in some other
manner, and sifted; afterwards what remains
hard or solid must be reduced to fine
particles and likewise sifted; the same process
must be repeated in turn three or four
times. That is best which first passes through
the sieve, and this must also be employed for the
relief of the eyes; that which is next sifted out
holds the second place, and in succession
the others in their order. Others, having
suspended a stick of wood about the middle
of the jar, place the mat of twigs before mentioned
upon it, in such a manner that
it may not touch the vinegar,
and throw in the lead,

putting on a cover and sealing it tightly. After ten days, removing the cover, they look in, and when the material has been dissolved they complete the other operation as we have described." Dioscorides says that this may be done in winter, "If you place the jar over braziers, cauldrons, or furnaces; for heat applied to it shows the same effect as the sun."

These methods, however, are not acceptable in modern, as they do not provide for the presence of carbon dioxide, and Dioscorides above mentions the need of heat. It is clear that, following out these methods to a letter, lead acetate would be produced. Lead acetate was used in medicines while white lead is mentioned by Pliny as a pigment, for which purpose lead acetate is not adapted. Both however were known

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as cerussa. The ancients clearly understood the necessity of supporting the lead out of contact with the vinegar.

It is, however, known that the carbon dioxide came from the decomposition of the vinegar, as at Klagenfurth, in Carinthia, where white lead is made in chambers, the acetic acid and carbon dioxide being simultaneously produced by fermentation of extract of dried grapes or raisins, or, the residue of grapes after pressing.

For a number of centuries the white lead demand was met by the priests in monasteries, all over Europe.

The first place where white lead was made a commercial article, was Venice, which for a long time supplied the greater part of the demand. Their methods were afterwards used in

Holland and England and became known as the "Dutch method."

There are a great many methods employed in the manufacture of white lead, but only the most important of these will be described in detail. These are three in number 1st "The Dutch"; 2nd "The English"; 3rd "The French"; taken in the order named.

The Dutch process comprises the following operation.

1st. Casting the lead in required form
2nd. Building the beds.
3rd. Taking the beds apart.
4th. Picking up.
5th. Grinding &c.

The fifth operation will be described in detail for all the methods. The picking up is also dispensed with in modern

works.

I Casting the lead.— Lead is melted in ket
tles covered with hoods to protect the work-
men from fumes, which are danger
ous, especially when old leads are being
melted. When lead is melted it is cast on
a large ironplate and when enough has
solidified the lead on top is run of, leaving
a thin sheet about sixty centimetres long,
ten centimetres wide, and a few millime-
tres thick, which weighs about one kilo-
gram. These sheets are cut in two and
rolled into spirels, for the pots. These ope-
rations present very little danger to the
workmen.

II Building the beds.— Beds are built of
stone, or brick. They are about one metre
deep in the ground. Their dimensions
are five metres long, four wide, and six high.

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Stable manure, or spent tan are used in
the beds. Where stable manure is used
operation requires only forty days, while with
spent tan, it requires sixty to ninety days.
A layer, thirty centimetres wide and forty high,
is built around the bed with old manure,
the span is then leveled up with fresh manure
over which the pots rest. Each layer contains
over 1200 pots each filled, with about a fourth of a
litre of vinegar, up to the two knobs on which
the spirel of lead rests. This is covered with
lead sheets and then with scantlings.
Room is left between these scantlings for
draft, which are then covered with boards.
On these boards another, similar, layer
is built, until bed is filled.
An average bed requires --

1. 8 two-horse loads of stable manure;
2. 800 litres of vinegar per layer or 2400 per bed;

3. 1200 to 1500 kilograms of lead per layer, or from 10,000 to 12,000 kilograms per bed.
Four men can build a bed in four days: that is, a bed requires sixteen days work.
There is no danger in building these beds.
III Taking the beds apart. — The beds are taken apart in the following manner: The manure, boards, scantlings, of the top layer removed. The corroded lead is put into a box where the longest pieces of noncorroded metal are removed. The workmen say that there is little danger in this work; especially if they are allowed to smoke. In some works the noncorroded metal is smelted but in others it is used to cover the pots.
12,000 Kgr. lead give – carbonated lead 10000 Kgr. noncorroded lead 400 Kgr. The increase in weight is there for about 25%. Gloves may be used in taking beds apart, but are very unhandy.

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The English method differs from the Dutch in using spent tan instead of manure, thereby lessening the liability of discoloration by sulphureted hydrogen; but the use of tan lengthens the time necessary for production of white lead. Pyroligneous acid is sometimes used instead of vinegar.

The lead should be of the purest and softest kind. Manufacturers sometimes purify the lead themselves. Copper and antimony produce a white lead which is greyish in color. A red or pink coloration is attributed to the presence of a suboxide of lead. Lead is cast into thin plates [grates?], so as to expose as large a surface as possible to the action of the vapors of acetic acid, carbon dioxide, and water, by pouring the molten metal on a sloping grooved iron plate, from which they are easily

detached. These are sometimes made by
machinery. The machine consists of an
endless chain of iron plates, each
plate being a mold, into which the lead
is poured as the molds pass in succession
beneath the spout of the lead kettle.
The castings are delivered onto a leather
belt covered with sheet iron. This machine
makes from fifty to sixty castings in
one minute. The corrosion is effected in
stacks, which are built in brick work chambers.
They are usually built side by side along
the side of a shed. The dimensions of
these chambers are variable, being from
twelve by twenty feet, to sixteen feet square,
and about twenty five feet in height.
The front wall has an opening four feet
wide running from top to bottom,
and is used in building up the stack

The opening being closed with boards as the stack rises in height. In building a stack the floor is first covered with ashes, on this is spread a layer of fermenting tan, to a height of about three feet, which is tamped down and surface leveled. The pots, which are partially filled with dilute acetic acid, about two to three percent, are now placed on the tan. They are placed close together leaving about six inches between the last row of pots and the walls. Two sizes of pots are used; a large size with a projecting ring on the inside, upon which a thin disc of lead rests; which serve to support the boards covering the middle pots. On the top of the pots are piled lead crates to a depth of five inches; between the lead and the covering of flooring boards a space of six or eight inches is left. The space between the walls and

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pots is filled with tan. Another bed is built on this in the same way except that the tan is only about one inch in thickness. The stacks are built up about twenty feet and then covered with tan. In a stack twelve by twenty feet a bed contains twelve tons of lead in one thousand pots, five and one half inches in diameter, containing two hundred gallons of dilute acetic acid. The opening at the top is left unclosed for examination. In large stack ventilation is required which is accomplished by placing wooden spouts in the corners of the stack. Stack is now left about three months. The temperature sometimes rising to 42.5 ^{0}C, at which temperature acetic acid, water, and also carbon dioxide are evolved, which gradually attack the lead transforming it into white lead. At the end of three months

the stack is unloaded. The top tan is removed and that which is not mouldy is put with the fresh tan, to be used again. The crates are loaded into trays and carried to the mill in which the white lead is separated from the non corroded metal. The crates retain their shape, but are greatly increased in bulk, and have a white opaque appearance. The best corrosions are those resembling porcelain in appearance. Sometimes the lead is discolored which discoloration may be due to tany matter present in the acid, or by the dripping of water from the tan. Before removal each layer, as it is exposed, is thoroughly moistened with water. White lead taken direct from stack is not uniform in composition. It consists mainly of

$2\ PbCO_3 \cdot Pb(HO)_2$. All corrosions contain

lead acetate which is washed out. The pots are usually found empty and dry. They are washed, dried, and used again Pieces of white lead which fall on the tan are picked up, and tan is raked to secure any further quantity of white lead that they contain. The corrosions are next separated from the non corroded metal and ground. The methods of grinding and washing vary in different works. The crates are usually sunk in a cistern of water and pushed with a rake between corrugated rollers running under a stream of water. This method obviates dust which is very poisonous. The white lead is then crushed, by passing through a pair of smooth gun metal rollers, and raked about over a perforated

plate under a constant spray of water. The coarse white lead is passed on to a grinding stone which is fed by hand or by an endless belt. After passing through these stones the white lead has the appearance of a thick white mud, and is reground until it is so fine that it may be carried along by a stream of water to the settling tanks; the lead which settles in the troughs leading to the settling tanks is reground. The grinding and washing are conducted in such a way that the same water may be used over again. This water generally contains lead salts in solution which are precipitated by sodium carbonate. In the tanks the white lead gradually settles to the bottom and the water is

siphoned off. When the mud is thick
enough it is ladled out into drying dish-
es. These dishes are of clay twelve inch-
es in diameter and three inches
deep. The dishes are then stacked on
iron shelves in drying stoves which
are heated by pipes passing around
the bottom, connecting with a grate
outside of the stove. The mud con-
tains 25 to 30 per cent water this is
dried the temp never exceeding 68°C.
The white lead when dry is taken
from the stoves and packed in casks
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or indigo to modify the yellow
tint due to stains from the tan.

Another English method is as follows;--
Lead is melted in a large iron pot and made
to flow on to the hearth of a reverberatory
furnace, so as to convert the lead into oxide.
This is obtained in a finely divided state by the
following arrangement of furnace. The
hearth of the furnace has a gutter into which
the molten mass flows, the sides of the gutter
being perforated, so that the molten
litharge flows out, while the lead sinks
to the bottom. The litharge is mixed with
1/100 part of its weight of a solution of lead
acetate, and placed in a series of closed troughs,
communicating with each other and
admitting of the passage of pure carbon
dioxide, obtained from burning coak in
a furnace provided with a blast.

The mixture is stirred by machinery to accelerate the absorption of the gas. The white lead made by this method covers well and is prefered to that made by the wet or "French Method," which is as follows:

If a solution of basic lead acetate be treated with carbon dioxide, part of the oxide of lead in the salt is converted into carbonate, and the remainder becomes neutral acetate. By adding a new proportion of litharge to the neutral acetate, it becomes basic again by solution of litharge, making the process continuous.

A solution of lead acetate, of proper strength, is made as follows; First, boil a solution of neutral acetate with very finely divided litharge. When the litharge has dissolved, and a basic solution is well

saturated, the liquor is drawn off from the residues into a closed vessel. Then carbon dioxide is introduced, which is made by the combustion of coak, and and should be well washed. As soon as the basic excess of litharge has been transformed into carbonate, the liquors are allowed to settle. The white lead falls to the bottom, and the neutral acetate is drawn off and boiled with litharge to form basic acetate again. There is a small accidental loss of the neutral acetate. The white lead is wash until very clean, the first wash waters being returned to the neutral acetate solution, and dried in pots in a store room. This white lead is an impalpable powder, as white as snow, but has less density and body than white

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lead made by the "Dutch" and "English" processes.

The theory of these processes, as well as of many others, is as follows:--

1. The formation of basic acetate.
2. The decomposition of basic acetate into neutral and white lead.

This is illustrated in the following formulae

I. $2\{C_2H_3O\}_{H} + 3PbO = \{(C_2H_3O)_2\}_{Pb} O_2, 2Pb(HO)_2$

Acetic acid — Basic lead acetate

II. $\{(C_2H_3O)_2\}_{Pb} O_2, Pb(HO)_2 + 2CO_2 = 2Pb(CO)_3 +$ Lead carbonate

Basic lead acilate

$\{(C_2H_3O)_2\}_{Pb} O_2$

Neutral lead acetate

It is shown, by this, that a small quantity of lead acetate can produce a large quantity of white lead, and that the process would be endless but that the white lead retains some neutral acetate, and the loss of acetic acid cannot be avoided.

Adulteration of white lead is still practiced to some extent, generally by adding barium sulphate, either natural or artificial. White lead is also sometimes mixed with lead sulphate, chalk, barium carbonate, and pipe clay, which have no covering power & absorb oil causing a waste of this article.

Pure white lead ought to be soluble in dilute nitric acid, in which Solution KOH should not produce a precipitate. A residue on dissolving in

nitric acid indicates the presence of gypsum, heavy spar, or lead sulphate. Lead sulphate could be detected with the blowpipe. Barium sulphate may be detected by igniting on charcoal, treating residue with Hydrochloric acid, and adding a solution of gypsum which again precipitates the barium sulphate. Gypsum does not leave a residue with nitric acid but may be precipitated with ammonium oxalate. To remove the oil, thoroughly stir into the paint a mixture of equal parts of chloroform and alcohol, and work on a filter with a mixture of 2 parts of chloroform and 1 of alcohol. The quantity of acid is ascertained by evaporating this mixture. Wash twice with boiling water and dry, after which the lead may be tested by usual methods

Grinding white lead, with consideration of the effects of dust on the workmen.

Owing to the poisonous effect of the dust produced in grinding white lead, upon the health of the workmen engaged therein, many machines have been invented to prevent dust. One effective machine is known as the Ward machine. This comprises:--

I. A trough, 4 metres long, 2 metres wide, and 1.3 meters deep.

II. Two brass rollers, superposed, for grinding the substances. The lower one is immersed in water, so that their line of contact is under water. Motion is imparted to the upper roller by a pulley, and thence to lower by pinions. Counter weights are fixed to the

upper axis to prevent clogging where too large a piece of lead is introduced.

III A perforated oak platform, holes 15 or 16 millimetres in diameter serving as a sieve for the material which leaves the rolls. This platform is held, 8 centimetres below the rolls, by wooden block above and below it.

IV An inclined plane for feeding the rollers.

An outlet is left on one side of the trough for water. The white lead finely powdered falls through the holes in the platform, while the laminated metal always stays on top and is raked off.

As the white lead and non corroded metal are separated entirely under water, no dangerous dust escapes.

The metallic plates are allowed to drain an inclined trough before smelting.

There are three reasons that the substances are not [netted?] before passing through the rolls.

1. They would be composty, and their passage through the rolls difficult.
2. This poste would not be well sifted.
3. A certain proportion of metallic lead is necessary to the operation, and should not be removed before the substances are poured through the rolls.

In Southwest Missouri a so called
white lead is made by collecting the
fumes from the smelting of galena
in ore hearths, but differs very much
in composition from other white
leads.
There are two operations involved: first,
cooling and collecting of dark ore hearth
fumes, called blue powder, in the first
bag house, or blue room; second, refining
blue powder in a slag eye followed by
cooling and collecting of white paint
in the second bag room or paint house.
The fumes from the ore hearth are
drawn off by a suction fan six feet
in diameter, three feet wide, running
at 290 revolutions per minute. They
are first drawn through a dust
chamber of brick four feet long,

nineteen feet high and six and one half feet wide. This catches the heavy particles. The dust passes out at the top of the chamber by a five foot pipe of sheet iron 350 feet long, to a fan and thence through a four foot pipe to the blue room. This room is built of brick, 95 feet long, 50 wide and 45 feet high. It is in two compartments, so that one may be shut when it is necessary to examine the bags. Each of these compartments is in two stories, the lower 12 feet high. The four foot pipe is divided into four branches in each room, corresponding to four rows of nine hoppers each. These are 12 feet wide at the top and 1.5 at the bottom, and are 10 feet high. The hopper cover is made of 1/16 inch sheet iron with sixteen holes 18 in in diameter,

ctia, with thimbles 12 inches long projecting upwards. To these are tied unwashed woolen bags 20 inches in diameter and 33 feet long. The upper ends of the bags are tied shut and are suspended from rods of gas pipe. There are 800 bags in one house. The gases entering the hoppers and bags, under low pressure, are filtered.

The hoppers, which serve to collect the fumes, are connected together by short lengths of pipe. The bags are shaken every two days.

The composition of the blue powder in the hoppers varies as the distance of the hopper from the furnace increases. Diminution in amounts of Fe_2O_3, Al_2O_3, CaO, SiO_2, CO_2, and SO_2, being particularly noticeable.

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The blue powder is mixed with waste
oil and waste and fired. It burns about
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ing a porous pink residue, which is quite
friable, and is free from carbonaceous
matter and lead sulphide. It is now
refined in the slag eye. As the object of this
process is to produce as much oxide as possi-
ble, very little lead is produced.

Charge

Slag - - - - - - - - - - - 2800#
Blue powder - - - - - - - - 1000#
Dry bone (carbonate) - - - - 600#
Dust from cooling chamber & pipes 450#
With these are also mixed tin scrap and
coak. The ratio of lead to paint produ-
duced is 1 ÷ 1.6. It is necessary to keep
a hot top and a very liquid slag
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five hours is blue powder and good paint is not made until furnace has run about twenty four hours.

The paint fumes are drawn from the furnace by a fan, as before, first through a seven foot pipe, 25 ft vertically downward then into a brick chamber 95 sq. feet in area, thence upwards 20 feet through a 3 ½ foot pipe curving downwards to a second brick chamber 75 sq feet in area, and in the same manner it passes through a third and fourth chambers.

The fumes then pass to the cooling room or second bag room. This is 40 x 90 feet, built in two stories 9 ft. high. It is similar in all other respects to the first bag room, except that the hoppers are made of wood lined with sheet iron, below which

are wooden bins also lined with iron.
The following analysis gives the
average composition of the paint
produced by this process.

PbO	25.85
$PbSO_4$	65.46
ZnO	5.95
Fe_2O_3	0.03
Al_2O_3	0.02
SiO_2	0.10 to trace
SO_2	0.04
CO_2	1.53
Na_2O	1.69
Total	100.67

The zinc oxide contained in this paint
rather improves its quality than otherwise,
because it prevents the paint
being blackened by sulphuretted
hydrogen.