

ANACONDA LEAD PRODUCTS COMPANY



EAST CHICAGO, INDIANA

September 28th, 1933

Mr. Frederick Laist, Vice-President,
Anaconda Lead Products Company,
Room 1720 - 25 Broadway,
New York, N. Y.

Dear Mr. Laist:-

I am enclosing a description of a "Process for the Manufacture of Basic Sulphate Pigment" with attached memorandum by Mr. Bowman which outlines the process we are now using for the production of basic lead sulphate for use in the manufacture of leaded zinc oxide at East Chicago. This information has been prepared for use by the patent attorneys in preparing an application for a patent on the process.

The inventor of the process is Mr. Stephen W. Stockdale and the patent, if obtained, should be assigned to the Anaconda Lead Products Company.

It is our understanding that it is doubtful whether a patent will be granted on this process; however we desire to have the application made in order to prevent anyone else from obtaining a patent on the particular features which we have found necessary to produce a satisfactory product.

Very truly yours,

Superintendent.

GEJ's
encls.

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ANACONDA LEAD PRODUCTS COMPANY
EAST CHICAGO, INDIANA

MEMORANDUM

TO: Mr. G. E. Johnson, Superintendent,
(Building)

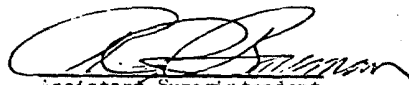
DATE September 17th, 1933

SUBJECT:

The attached description of a "Process for Manufacture of Basic Sulphate Pigment" describes the method of producing Basic Lead Sulphate as developed in the Research Department and now used at the White Lead Plant.

This description was prepared by Mr. Stockdale to be forwarded to Mr. Laist for use by the attorneys in preparing an application for patent on the process.

The idea and the development were Mr. Stockdale's and application should be filed in his name as Inventor.


Assistant Superintendent.

RGB/s

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PROCESS FOR MANUFACTURE OF BASIC LEAD SULPHATE PIGMENT

This invention relates to a process of manufacturing basic lead sulphate pigment from basic lead carbonate by treatment of the latter with sulphuric acid and caustic soda, and particularly concerns itself with the control of various factors which directly affect the important physical properties of the product and its worth as a pigment. The following is a clear and exact description of the process together with data showing the influence of various factors affecting the physical properties of the pigment such as will allow others skilled in the art to carry out the same.

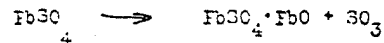
Basic lead sulphate is an important commodity, and finds many applications in the arts especially as a pigment, and, as such, it possesses peculiar advantages not common to other pigments. Previous practice in the manufacture of this pigment has been to subject lead sulphide or galena concentrates to the action of heat in a suitable furnace wherein the material is sublimed in the presence of oxygen and the resulting product, consisting essentially of lead sulphate, is recovered in settling chambers and bags. A modification of this method, and one which produces a much superior product, is to sublime lead or lead oxide in an atmosphere of sulphur dioxide and oxygen in a furnace of suitable design and recover the product in settling chambers and bags. These processes are fully described in patents and the technical press, suffice it to say that, due to impurities in the raw materials used and the difficulties in controlling furnace conditions, the products made by such processes are not always of uniform composition and will be

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found to vary somewhat in their physical properties. Due to these difficulties, a considerable proportion of the product must finally be returned for retreatment which fact constitutes a major objection to this method of manufacture.

From a practical viewpoint, the actual constitution of basic sulphate white lead is immaterial. The quality depends more upon its physical form than its chemical composition. A large number of basic sulphate formulae are known, ranging in composition from almost PbO , on the one hand, through the so called true basic sulphate, $\text{PbSO}_4 \cdot \text{PbO}$, to normal lead sulphate, PbSO_4 , on the other hand. Until further researches have settled the point it cannot be stated whether the various basic sulphates are definite chemical compounds or solid solutions of two or more constituents. It is probable that the true nature is a solution of PbSO_4 in PbO or, what is the same thing, the result of the partial decomposition of PbSO_4 according to the equation



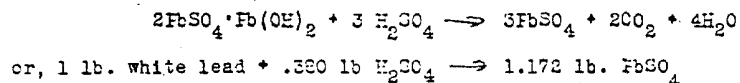
and that the constitution of the product depends upon the conditions of the reaction. It is believed that a relationship exists between basicity and opacity as if a small sphere of PbSO_4 were covered with a coating of amorphous PbO . The less basic sulphates are distinctly much whiter in the dry powder but are less opaque in oil. The optimum appears to be at a point about $2\text{PbSO}_4 \cdot \text{PbO}$; on increasing the basicity the product is liable to assume a yellowish cast of free PbO .

The present invention concerns itself with and has for its object a process for making basic lead sulphate of uniform physical properties which are capable of close control by regulating the conditions under which the same is produced. Having now described

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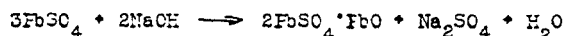
briefly the previous practice and shortcomings of this art, I will now describe my own invention the advantages of which will be obvious to those familiar with such matters. In order to simplify the disclosure of this invention, one preferred application will be described, but it is understood that the same result may be achieved by certain variations in the conditions without departing from the spirit of the invention or the claims thereto.

I start out with basic carbonate white lead from any source, preferably, but not necessarily, in the form of a water pulp of from 35 to 40% solids. After sampling and determining accurately the percentage of solids, I transfer a predetermined volume of this pulp to a treatment tank of suitable size and shape and add sufficient water to reduce the pulp to about 30% solids. Based upon the amount by dry weight of white lead contained, a calculated quantity of concentrated sulphuric acid, H_2SO_4 , is added to the pulp and the whole mass agitated by any suitable means to secure good contact of acid and white lead. To assist and speed the reaction the pulp is heated by steam or other means for about 16 hours after which time the following reaction will have taken place



It is desirable to use a slight excess of acid for two reasons, namely, to insure maximum CO_2 elimination within a reasonable time, and to provide an acid solution containing all the iron which may have come into the system from the acid or from other sources, in a soluble form capable of being separated from the solids by filtration or decantation. Iron present in the water or acid used, if allowed to precipitate as

ferric hydrate, harmfully affects the color of the final product even though present in minute quantities; by maintaining an acid pulp this precipitation is prevented and a large proportion of the iron may be removed from the system at this point by separation of solution from solids. For practical purposes it has been found that for every pound of dry basic carbonate contained in the pulp to be treated, 0.40 pounds of 100% H_2SO_4 is sufficient for these purposes. Upon completion of the sulphating operation, the pulp is allowed to settle and the clear solution decanted off. The pulp is again thinned with water and the sulphated pulp carefully adjusted to 23-26 percentage solids and again agitated. The temperature of the pulp is then raised to about 62°C and a calculated quantity of caustic soda as a 20% solution is slowly run into the tank. The pulp concentration, temperature, and quantity of caustic soda added at this point are important factors and each influences the physical character of the product. The following reaction takes place.



or, 1 lb $PbSO_4$ + .088 lb NaOH $\longrightarrow$ 0.912 lb. basic sulphate

or, 1 lb. white lead + .40 lb. acid + .103 lb. NaOH $\longrightarrow$ 1.07 lb Basic Sulphate

For all practical purposes it has been found that from 0.09 to 0.125 pounds NaOH per pound of dry white lead contained in the starting pulp is sufficient, according to the basicity desired. The pulp is allowed to agitate until it becomes just slightly acid to test paper when it is run into another tank, filtered, and washed. The washed filter cake is dried and pulverized ready for use.

The product formed by these reactions is capable of considerable variation in its physical character and is dependent upon

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three important factors, namely the basicity, the temperature of the pulp, and the pulp concentration at the time the caustic soda is added. The control of these variables as specified above comprises part of the basis for this invention. To illustrate, as examples of the effect of these variables, the following data are tabulated. In order to clarify the comparison and avoid the use of comparative numerical standards, the values are shown as relative, one to another. For example, under the heading "Effect of Basicity" opposite "Tinting Strength", 1 refers to a higher tinting strength value than 2, 2 higher than 3, 4 being lowest, and so on throughout the table, the object being not to confuse the reader with actual numerical values which themselves require further definition.

Effect of Basicity

	Percentage PbSO_4 in Product			
	57.77%	64.81%	69.71%	73.37%
Calculated Composition	$\text{PbSO}_4 \cdot \text{PbO}$	$4\text{PbSO}_4 \cdot 3\text{PbO}$	$5\text{PbSO}_4 \cdot 3\text{PbO}$	$2\text{PbSO}_4 \cdot \text{PbO}$
Oil Absorption, Rating *	1	2	3	4
Tinting Strength, Rating	1	2	3	4
Hiding Power, Rating	1	2	3	4
Color, Rating	4	1	2	3
Brightness, Rating	1	2	3	4

Effect of Pulp Concentration

	Percentage Solids in Pulp			
	5%	15%	25%	40%
Apparent Density, Rating *	1	2	3	4
Oil Absorption, Rating	4	3	2	1
Tinting Strength, Rating	4	3	2	1

Effect of Pulp Temperature

	Temperature			
	30°C	50°C	75°C	98°C
Tinting Strength, Rating *	4	1	3	2
Brightness, Rating	3	1	2	4
Color, Rating	4	2	1	3

* Rating: 1 Highest, 4 Lowest

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It is understood that the above is only one form of the application of this invention. Others are, of course, possible without departing from the scope of the invention. Basic carbonate from various sources may be used either in dry or in pulp form, sulphuric acid of satisfactory purity of any source or strength, or other bases such as caustic potash or ammonia may also be used, those specified in the above application being given merely as the preferred use. I am fully aware of the fact that the chemical reactions involved in this process have been well known for a long time. No claim is made for them.

What I claim and desire to secure by patent, is:

Claims. The wording of the claims of this specification is left to the discretion of the attorneys but they should specifically include the following:

1. Conversion of basic lead carbonate to normal lead sulphate by treatment of the former with sulphuric acid and the subsequent conversion of the normal lead sulphate to basic lead sulphate by treatment with caustic soda.
2. The control of the physical properties of the basic lead sulphate by careful control of conditions such as basicity, pulp dilution and pulp temperature.

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