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D4947

December 27, 1939

Mr. Frederick Laist, Vice-President
International Smelting & Refining Co.
25 Broadway, (Room 1720)
New York, N. Y.

Dear Sir: Re: Johnson et al. Application, Ser. No.
178,352, Filed December 6, 1937,
For: Method of Producing White Lead

Enclosed herewith are two copies of a supplemental
amendment and affidavit of William J. Knox, Jr. which was
forwarded to the Patent Office today for filing in the above-
mentioned application.

Very truly yours,

James W. Laist

Encs.

copy sent to Mr. Case - 28 Dec. 1939

r

IN THE UNITED STATES PATENT OFFICE

In re application of

GUNNARD E. JOHNSON and
REGINALD G. BOWMAN and
WILLIAM J. KNOX, JR.

METHOD OF PRODUCING WHITE LEAD

Serial No. 178,352

Filed December 6, 1937

Div. 56 - Room 4725

New York, N. Y., December 27, 1939

SUPPLEMENTAL AMENDMENT

S i r:

Supplemental to the amendment filed in the above-identified application on October 14, 1939, an affidavit of William J. Knox, Jr., one of the above-named applicants, is being filed herewith.

The principal basis on which the claims were rejected in the Office action of April 22, 1939, was lack of invention over the disclosures made in the Johnson et al. patent No. 1,845,713. The Examiner stated that this reference clearly teaches the advantages of controlling bicarbonate ion concentration in the anolyte, and that in view of this disclosure there is no invention in the particular method of controlling bicarbonate ion concentration by the steps of excess carbonation of the catholyte and adding hydroxyl ions to the anolyte in sufficient amount to establish therein a desired bicarbonate ion concentration.

Actually the process of the present invention is not suggested by the disclosures of the reference. The accompanying affidavit shows that the control method of the present invention is greatly superior to the control method

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of the Johnson et al. patent, and that it leads to extremely beneficial results which could not be predicted on the basis of the disclosures of the references. In the affidavit it is shown, for example, that the chemical composition of white lead must be uniform in order to obtain a pigment having uniform oil-absorption or oil-taking properties, and that prior to the present invention the chemical composition of white lead pigment produced electrolytically, as indicated by its CO_2 content, varied materially (on the average from 11.40% to 12.10% CO_2 , a range of 0.70% CO_2). The method of the present invention made it possible to control the chemical composition of the pigment much more closely (from 11.72% to 11.76% CO_2 , a normal range of only 0.04%). This product, being much more uniform than the product previously made, is a markedly superior product. The fact that this superior product was made possible by the control method of the invention is shown by the fact that the process by which the improved product is made differs from the process by which the former product was made only by the substitution of the control method of the invention for the control methods formerly used, among which was the control method described in the Johnson et al. patent.

A further remarkable and unpredictable advantage of the method of the invention is the speed with which it permits the composition of the white lead product to be adjusted during commercial operation of the process. By following control methods formerly used, a period amounting on the average to about 50 minutes elapsed between the time when it first became apparent that some adjustment should be made to correct the composition of the cell product, and the time by which the adjustment actually was reflected in the composition in the product. In accordance with the control

method of the invention, this period amounts on the average only to about 8 minutes.

The control method of the invention has proven much more economical in commercial operation than the control methods formerly known and employed. For example, it is stated in the affidavit that a tank room containing 48 cells required the attendance of four men for its successful operation under the control method described in the Johnson et al. patent, whereas under the new control method the same tank room may be operated by two men.

In view of the decided advantages of the control method of the invention, which advantages could not have been predicted from the disclosures of the references relied upon, it is submitted that the claimed process represents a marked advance in the art and constitutes a patentable invention of a high order. It is therefore believed that the claims appearing in the case should be allowed.

Respectfully submitted,

Attorneys for Applicants

IN THE UNITED STATES PATENT OFFICE

In re application of	)	
GUNNARD E. JOHNSON and	)	
REGINALD G. BOWMAN and	)	
WILLIAM J. KNOX, JR.	)	
Serial No. 178,352	)	METHOD OF PRODUCING WHITE LEAD
Filed December 6, 1937	)	
Div. 56 - Room 4725	)	

AFFIDAVIT

STATE OF INDIANA)
) ss:
 COUNTY OF LAKE)

William J. Knox, Jr., being duly sworn deposes
 and says:

I am one of the applicants in the above-mentioned
 application.

I am employed by the International Smelting and
 Refining Co., and I am thoroughly familiar with the White
 Lead Plant of that Company at East Chicago, Indiana, where
 white lead is produced electrolytically. I am and have been
 for the past 14 years Superintendent in charge of this
 White Lead Plant.

Since the invention described and claimed in the
 above-mentioned application was made, it has been employed
 in the White Lead Plant and has resulted in considerable
 savings as compared with the processes previously employed
 there, including the process described in United States
 patent No. 1,845,713, and in the production of a superior
 white lead product.

Variations in the chemical composition of white
 lead pigment have a marked effect on the oil-absorption or

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oil-taking property of such pigments. The magnitude of this effect is shown in the following table, in which is set forth the oil-absorption or oil-taking property of the pigment as determined by the three methods most commonly used by paint technologists in determining the amount of oil required to produce a paint of brushing consistency.

	Product "A"	Product "B"	Product "C"
Analysis - Percent CO ₂	11.15	11.75	12.40
" PbCO ₃	67.70	71.20	75.30
" Pb(OH) ₂	32.30	28.81	24.80
Primary Oil Absorption (gms/100cc)	13.5	11.5	8.5
Consistency (Stromer- Krebs Units 35% oil)	9.2K	9. N	7.4N
Flow (Gardner flow meter, 35% oil)	2.2"	2.75"	3.6"

Up until the time the invention described in the above-mentioned application was made and put into use, the CO₂ content of the white lead product produced at the White Lead Plant varied in normal operation of the plant from 11.40% to 12.10%. The CO₂ content of the white lead product made at the White Lead Plant since the said invention was there put into use varies in normal operation of the plant from 11.72 % to 11.76 %. This product is more uniform than and hence superior to the product formerly made. The process by which the improved product is made differs from the process by which the former product was made only in that the method described in the above-mentioned application for controlling composition of the white lead product has been substituted for the methods previously employed, among which was the method of exact neutralization or accurate deficient carbonation described in United States patent No. 1,845,713.

According to the process of the United States patent No. 1,845,713, a considerable period of time was

required to effect adjustment of the composition of the white lead product. This period, which amounted on an average to about 50 minutes, was a result of the time required for the following chain of events to take place before actual adjustment of the composition of the product could occur: -

- (1) Titration of catholyte at frequent intervals.
- (2) Adjustment of carbonation fan damper to increase or decrease degree of carbonation of catholyte.
- (3) Time required to displace old catholyte in storage tanks, piping, and cells by new catholyte of adjusted carbonate-to-hydroxide ratio.
- (4) Time of adjustment of composition of anolyte by flow of catholyte ions of new ratio from catholyte to anolyte compartment of cell.

In accordance with the method described in the above-mentioned application, steps (2), (3) and (4) set forth above are eliminated, as it is only necessary to change the setting of one valve to vary the volume of alkaline solution flowing into the anolyte and so to adjust the bicarbonate ion concentration of the anolyte, and thereby to adjust the composition of the white lead product. Moreover, the number of control titrations that are required have been greatly reduced. As a result of these savings the period required to effect adjustment of the composition of the white lead product has been reduced to about 8 minutes on the average.

The method of the invention described in the above-mentioned application has resulted in substantial operating savings. For example, to control operation of a tank room containing forty-eight cells by the process described in United States patent No. 1,845,713 required one circulation man, one titration man, and two cell men. Under

the method described in the above-mentioned application only one circulation man and one cell man are required to control operation of the same tank room.

William J. Knox, Jr.

Subscribed and sworn to before me this day
of December, 1939.

Notary Public