

LIBRARY COPY

E. I. DU PONT DE NEMOURS & COMPANY  
KREBS PIGMENTS DEPARTMENT  
256 VANDERPOOL STREET  
NEWARK, NEW JERSEY

NEWARK PLANT  
PIGMENT COLOR RESEARCH REPORT

Final Report

Sick List Study - Lead Nitrate Process

Period Covered: October 1941 - May 1942.

FILE: 121.1-1

DATE: 7-25-44

Serial No. KN-44-46

Copy No. /

Copy to:

- #1 - Numerical File
- 2 - Research Office (121.1-1)
- 3 - Library File (121.1-1)
- 4 - #10 Building File
- 5 - # 6 " "
- 6 - Imperial Chemical Industries, Ltd.
- 7 - " " " "
- 8 - " " " "
- 9 - Mr. C. H. Rupprecht, Wilmington (ICI File)

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Title: Sick List Study - Lead Nitrate Process

Period Covered: October 1941 - May 1942.

SUBMITTED BY: *L. Stewart*  
L. Stewart

DATE SUBMITTED: 7-8-44

APPROVED BY: *V. Chalupski 7/25/44*  
V. Chalupski

DATE ISSUED: 7-25-44

## INTRODUCTION

Early in October 1941 the plant issued a Sick List Request stating that chrome yellows were running dirty in masstone and poor in lightfastness. This was readily traced to impure lead nitrate.

At that time, lead nitrate was made by dissolving pig lead and/or lead sludge from the tri-basic lead acetate process in a dilute nitric acid solution; purifying by precipitating the impurities; and clarifying by filtering out the various impurities. The purified lead nitrate solution was pumped to the color making area as needed thru iron pipes. Details may be found in the following formulas, notes, and reports:-

SF-10, K-8983 - the manufacture of lead nitrate from pig lead.  
SF-9 , K-8984 - the manufacture of lead nitrate from TBL sludge.  
SF-7 , K-9028 - the removal of iron from lead nitrate solutions.  
KN-38-718 - the manufacture of lead nitrate.  
KN-36-596 - the purification of lead nitrate.

## SUMMARY AND CONCLUSIONS

The initial investigation showed that the poor quality of the lead nitrate was due to the following conditions:-

1. Large quantities of brown sludge (high in iron and other impurities) were found in all the scale and storage tanks. This indicated lack of control of the lead nitrate acidity and equipment failure.
2. The filter press which was supposed to remove solid impurities was in very poor condition and as a consequence large amounts of these impurities were passing thru the press into the finished lead nitrate storage tanks. The electric eye which is located on the filtrate discharge as a protection against press leakage was not functioning because of its poor mechanical condition.
3. Standard operating procedures were not being observed by the operators. In several instances it was found that the operators were using methods which differed from standard and from operator to operator.
4. The standard operating procedures were found to be in need of revision at several points because of changes in equipment and methods which had been made during the past several years without being incorporated into standard procedure.

The general condition of the lead nitrate equipment and operation is quite clearly shown by the plant records which indicated that approximately one-third of the lead nitrate manufactured at that time was unsatisfactory for use in the manufacture of chrome yellows.

The following steps were taken to eliminate these substandard conditions:-

1. The impurities (brown sludge) were removed from the scale tanks, storage tanks, etc., and attention called to the fact that the cleaning schedule was not being adhered to.
2. The operating procedures were revised, where necessary, and copies posted on the bulletin board in the leadshop for the operators guidance. This was followed by a period of training during which the details of the various operations were carefully explained to each of the operators.
3. All of the defective equipment was replaced as rapidly as possible and several rearrangements made to give smoother and more efficient operation.

Details concerning the substandard conditions found and the corrective measures applied are fully covered in the various memoranda, letters, etc., issued during this period. Copies of these are to be found in DSN-44-13.

In general, standard process as it existed at that time was satisfactory provided the proper supervision was supplied and standard procedures observed. This is clearly shown by the following table:-

| <u>Period</u>            | <u>% of lead nitrate mfg.<br/>unsatisfactory</u> |
|--------------------------|--------------------------------------------------|
| Oct. 12 to Oct 31 (1941) | 27.5                                             |
| Nov. 1 to Nov. 15        | 21.3                                             |
| Nov. 15 to Nov. 30       | 7.5                                              |
| Dec. 1 to Dec. 15        | 10.4                                             |
| Dec. 15 to Dec. 28       | 3.9                                              |
| January 1942             | 0.8 - a mechanical failure.                      |

#### ELIMINATION OF PURIFICATION-SETTLING METHOD

Experience has shown that lead nitrate made from pig lead does not contain sufficient impurities to require purification. For economic reasons, however, it is necessary at times to consume certain lead sludges which accumulate over a period of time in the lead liquor and lead nitrate processes. At such times it is necessary to purify the lead nitrate solution because of the relatively large amounts of impurities present in the lead sludges.

Early in January 1942 the plant proposed that a program be organized to study the elimination of the purification step in order to effect labor and yield savings and free the purification equipment for use in the manufacture of copperas. This was to be accomplished as follows:- the lead sludges were to be consumed uniformly in all batches of lead nitrate, a small amount being added to each lead nitrate "make", so that at no time would there be the possibility of more than a slight increase in impurities. The lead nitrate solution would then be run to a settling tank to remove the undissolved sludge, and after settling, run off thru a standpipe to the storage tanks.

A trial run was arranged for the period January 12-January 18, 1942 under the supervision of the Research Department. During this period 30 batches or 115,000 lbs. of lead nitrate were made from pig lead and 7410 lbs. of TEL sludge. After the run, 795 lbs. of this sludge were found in the settling tank and 53 lbs. in the finished liquor storage tanks. The results indicated that the method was a workable one but required very close supervision and adherence to standard procedure. Although the Research Department was skeptical that these conditions would be met, the process was written up (SF-10, K-1720) and supervision of the process turned over to the plant in May 1942.

#### EXPERIMENTAL DETAILS

DSN-44-13  
SF-10, K-1720  
KCR-120