GOVERNMENT OF INDIA, THE PATENT OFFICE, 214, ACHARYA JAGADESH BOSE ROAD, CALCUTTA-17. Complete Specification No. 148273 dated 24th November, 1978. Application No. 844/bel/78 dated 24th November, 1978. Divided out of No. 1179/Cal/76 (Serial No. 146164) Ante-dated to 3rd July, 19767. Acceptance of the complete specification advertised on 27th December 1980.

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IMPROVED PROCESS FOR THE PRODUCTION OF ZINC PHOSPHATE USING ZINC CARBONATE.

OF
COUNCILISCIENTIFIC & INDUSTRIAL RESEARCH
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registered body incorporated under the
Registration of Societies Act (Act XXI of 1960)

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed:

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This invention relates to the improvements in or relating to the production of zinc phosphate by double decomposition of zinc carbonate with orthophosphoric acid for use as anti-corrosive primers.

Hitherto it has been the practice to prepare zinc phosphate by the interaction of zinc sulphate with disodium hydrogen phosphate to get zinc phosphate, sodium sulphate and sulphuric acid.

The objection to the procedure described above is that at involves in the formation of sodium sulphate and sulpharic acid. The presence of sulphate impurity is not desirable for the application of zinc phosphate in anti-corresive primers and unless proper precautions are taken in the pashing of the sample the sulphate may contaminate the product.

The object of this invention is to obviate these disadvantages and to prepare zinc phosphate of suitable purity needed for use as anti-corrosive primer.

Zinc carbonate is made into 15 to 25% but preferably
20% sturry with water. This sturry is allowed to react with
the steichiemetric quantity of orthophosphoric acid diluted
from 1:1 to 1:7 but preferably between 1:3 and 1:5 at
temperatures ranging from 30 to 60°C with stirring.

This mixture is kept for about 12 to 30 hrs with stirring. The crystallised salt after decantation of the supermatent liquor is washed twice or thrice with fresh cold water; then once or twice with hot water, centrifuged and finally dried at 100 to 120°C for 2 to 4 hours. This sample is subjected to the test as an anti-corrosive primer.

Accordingly the improved process for the production of zinc phosphate by the interaction of zinc carbonate and orthophosphoric acid is characterised in that the double decomposition reaction is carried out by reacting 15-25% slurry of zinc carbonate with water and stoichiometric amount of orthophosphoric acid diluted from 1:1 and 1:7 at a temperature range of 30 to 60° and isolating the solid zinc phosphate formed.

iected for use as anti-corrosive primer. For this, zinc phosphate prepared by the invented method was mixed with iron oxide and this mixture was incorporated in double boiled cil medium and linseed stand oil medium and ground well to form a paint. After adjusting the consistency the paints were coated on pickled m.s. panels and rusted m.s. panels and subjected to corrosion tests both by salt spray and exposing to the atmosphere preferably marine atmosphere at Mandapam. The results obtained with the zinc phosphate prepared by this invented method was comparable with the zinc phosphate pigment obtained from trade.

The present invention consists of a process for the production of zinc phosphate for use as anticorrosive primer, which comprises of double decomposition of zinc carbonate with orthophosphoric acid and isolating the solid zinc phosphate, wherein 15 to 25% but preferably 20% slurry of sinc carbonate is allowed to react with the stoichiometric quantity of orthophosphoric acid dilued from 1:1 to 1:7

out preferably 1:5 and 1:5 at temperatures ranging from 30 to 60°C with efficient stirring and finally centrifuging the solid zinc phosphate with cold and hot water wash to obtain pure product and drying the same. The zinc phosphate pigment prepared by this method proved to be comparable with that obtained from trade when subjected to corrosion trials.

Zinc phosphate prepared by this method is pure enough for its use in anti-corrosive primer.

## Typical example:

Zinc carbonate (20 g) .. 20% slurry

Orthophosphoric acid .. 1:3 dilution

Temperature of maction .. 35°C

Yaeld of sinc phosphate .. 98%

Purity: Zinc content 95.4% .. Phosphate content 95%

The following table gives some of the properties of sinc phosphate sample:

#### Properties of sinc phosphate

- 1. Volatile matter (\$) .. 7.1
- 2. Oil absorption .. 21 g/100 g pigment
- 3. Density g/ml .. 3.3
- 4. pH of extract .. 5.8
- 5. Extract inhibitive/ corrosive .. Corrosive

Zinc phosphate prepared by this invention was mixed with iron oxide and this mixture was incorporated in double boiled oil medium and linseed stand oil medium and ground well to form a paint. After adjusting the consistency the paints were coated on pickled m.s. panels and rusted m.s. panels to a thickness of 20 to 25 / . \*\* These panels were subjected to the usual tests like salt spray test with 3% sedium chloride solution; immersion in 3% sodium chloride solution and immersion in distilled water.

The painted panels were exposed in triplicate at Mandapan Camp. It was observed at the end of seven months that the performance of primer paint alone was similar to the performance of zinc phosphate primer obtained from trade. However, the performance of primer (developed) with finishing paint was better than that of commercial primer with finishing paint.

The following are the main advantages of this invention:

a) Zinc phosphate is obtained in pure form with good yields. b) This zinc phosphate pigment is comparable with the zinc phosphate pigment obtained from trade. c) The corresion resistant property of the zinc phosphate/red oxide primer is better than zinc chromate/red oxide primer of 18 2074.

The present invention consists of a process for the production of zinc phosphate for use in anti-corrosive primers wherein a slurry of zinc carbonate is reacted with stoichiometric quantity of orthophosphoric acid taken in dilution ranging from 1:1 to 1:7 but preferably between 1:3 and 1:5 at temperatures ranging from 30 to 60°C with efficient stirring.

Ziric phosphate pigment prepared by this method proved to be comparable with that obtained from trade when subjected to corresion trials.

#### Ve claim:

phosphate by interaction of zinc carbonate and orthophosphoric acid characterised in that the double-decomposition reaction is carried out by reacting 15-25% slurry of zinc carbonate with water and stoichiometric amount of orthophosphoric acid diluted from 1:1 to 1:7 at a temperature range of 30 to 60°C and institute with stirring and inclating the solid sine phosphate formed.

- 2. The process as claimed in claim I wherein the double-decomposition reaction is carried with stirring the reaction mixture for 12 to 30 hours and then centrifuging the solid zinc phosphate formed and wasning the same with cold and hot water to obtain a pure product.
- 3. The process as claimed in claim 1 and 2 wherein the zinc carbonate slurry with water used is preferably 20%.
- 4. The process as claimed in any of the proceeding claims wherein the concentration of orthophosphoric acid used is preferably between 1:3 and 1:5.
- 5. The process as claimed in claims 1 and 2 wherein the washings obtained after centrifuging solid zinc phosphate formed are reused in the process to prepare zinc carbonate slurry.
- 6. An improved process for the preparation of sinc phosphate by interaction of sinc carbonate and orthophosphoric acid substantially as herein described.

Dated this 26th day of July, 1977.

hue himmen

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