

UNITED STATES PATENT OFFICE.

HAROLD A. MORTON AND BEN H. NICOLET, OF PITTSBURGH, PENNSYLVANIA,
ASSIGNORS TO UNION CARBIDE COMPANY, OF NEW YORK, N. Y., A CORPORATION OF VIRGINIA.

METHOD OF PREPARING PARACETALDEHYDE.

1,300,451.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that we, HAROLD A. MORTON and BEN H. NICOLET, citizens of the United States, residing at Pittsburgh, in the county of Allegheny and State of Pennsylvania, have invented certain new and useful Improvements in Methods of Preparing Paracetaldehyde, of which the following is a specification.

This invention is a method of preparing paracetaldehyde (paraldehyde) substantially free from acetaldehyde.

In the manufacture of paraldehyde from acetaldehyde it has been proposed heretofore to treat acetaldehyde with a small proportion of sulfuric acid, to allow the reaction mixture to stand for several hours, and, after neutralization, to subject the mixture to fractional distillation. In practice, however, it is very difficult, even with very laborious fractionations, to free the paraldehyde from traces of acetaldehyde, the presence of which in the medicinal paraldehyde is not permitted by the *U. S. Pharmacopæia*, 9th edition. Other mechanical methods of separating the paraldehyde from the last traces of acetaldehyde have also been employed, including for example, freezing out the paraldehyde from the acetaldehyde; but these methods are tedious and do not give aldehyde-free paraldehyde without repeating the operation a number of times.

According to the present invention, paraldehyde prepared from acetaldehyde by any known or suitable method, is freed from residual traces of acetaldehyde by a chemical procedure which involves resinification of acetaldehyde in presence of a greatly predominating proportion of paraldehyde. This permits a simple and complete separation between the paraldehyde and the resin derived from the acetaldehyde, and may be made to yield directly paraldehyde conforming to the requirements of the *U. S. Pharmacopæia*.

Following is an example of a preferred method in accordance with our invention. The invention, however, is not restricted to the specific operating conditions and manipulations set forth in the example.

To about 45 pounds of acetaldehyde (CH_3CHO) maintained at 0°C. , we add about 30 grams of concentrated sulfuric acid, the solution being slowly agitated.

After the mixture has been allowed to stand for several hours the sulfuric acid is neutralized by an excess of sodium carbonate, and the mixture fractionally distilled. The fraction boiling below 120°C. is returned to the converter and subjected to a further treatment with additional acid. The paraldehyde fraction, boiling between 120° – 125°C. , is collected and submitted to a final purification, as follows: To the paraldehyde fraction we add a relatively small proportion, say 10% by weight or less, of a 35%–50% aqueous solution of caustic soda, and agitate the mixture for 30–60 minutes. The resinous alkali layer thus formed is preferably then separated from the paraldehyde, and the latter distilled with a somewhat smaller proportion, say 1%–5% by weight, of a similar aqueous solution of caustic soda. The fraction collected between 120° – 125°C. consists essentially of paraldehyde conforming to the requirements of the *U. S. Pharmacopæia*, 9th edition. In case the distillate or any portion thereof falls below the desired standard of purity it may be further purified by re-distillation with additional small proportions of caustic alkali.

The reaction involved in the resinification of acetaldehyde by means of alkali is, of course, well known, but so far as we are aware it has not been before recognized that the resinification of small proportions of acetaldehyde could be accomplished in presence of predominating proportions of paraldehyde, in such manner as to permit the recovery of the latter in a state of substantial purity.

We claim:—

1. The method of preparing paraldehyde substantially free from acetaldehyde, comprising treating acetaldehyde with sulfuric acid, neutralizing the acid, separating the bulk of the unchanged acetaldehyde by distillation, and purifying the paraldehyde fraction by treatment with caustic alkali and distillation.

2. The method of preparing paraldehyde substantially free from acetaldehyde, comprising effecting a partial conversion of acetaldehyde into paraldehyde by an acid treatment, neutralizing the acid, separating the bulk of the unchanged acetaldehyde by distillation, resinifying the residual acetaldehyde, in presence of paraldehyde, by an

alkali treatment and separating the paraldehyde by distillation.

3. The method of preparing paraldehyde substantially free from acetaldehyde, comprising effecting a partial conversion of acetaldehyde into paraldehyde by an acid treatment, neutralizing the acid, separating the bulk of the unchanged acetaldehyde by distillation, resinifying the residual acetaldehyde, in presence of paraldehyde, by an alkali treatment, separating the resinous product, and purifying the paraldehyde by distillation.

4. The method of preparing paraldehyde substantially free from acetaldehyde, comprising effecting a partial conversion of acetaldehyde into paraldehyde by an acid treatment, neutralizing the acid, separating the bulk of the unchanged acetaldehyde by distillation, resinifying the residual acetaldehyde, in presence of paraldehyde, by an alkali treatment, separating the resinous product, and purifying the paraldehyde by distillation in presence of a small proportion of additional alkali.

5. In a method of preparing paraldehyde substantially free from acetaldehyde, the

steps which consist in treating paraldehyde, contaminated by acetaldehyde, with an alkali under conditions to resinify the acetaldehyde, and recovering substantially pure paraldehyde by distillation.

6. In a method of preparing paraldehyde substantially free from acetaldehyde, the steps which consist in treating paraldehyde, contaminated by acetaldehyde, with an alkali under conditions to resinify the acetaldehyde, separating the resinous product, and purifying the paraldehyde by distillation.

7. In a method of preparing paraldehyde substantially free of acetaldehyde, the steps which consist in treating paraldehyde, contaminated by acetaldehyde, with an alkali under conditions to resinify the acetaldehyde, separating the resinous product, and purifying the paraldehyde by distillation in presence of a small proportion of additional alkali.

In testimony whereof, we affix our signatures.

HAROLD A. MORTON.
BEN H. NICOLET.