

UNITED STATES PATENT OFFICE.

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MANUFACTURE OF ALUMINIUM-ACETATE COMPOUNDS.

1,132,709.

Specification of Letters Patent.

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No Drawing.

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To all whom it may concern:

Be it known that I, GEORG EICHELBAUM, doctor of philosophy and chemist, a subject of the King of Prussia, and residing at 5 Augsburgstrasse 8, Berlin, W. 50, Germany, have invented certain new and useful Improvements in the Manufacture of Aluminium-Acetate Compounds, of which the following is a specification.

10 When solutions of aluminium acetate are heated or concentrated, insoluble basic salts of aluminium are precipitated. Attempts have been made (see German Patents 9790 and 10488) to avoid this precipitation by 15 the addition of certain acids. The residue obtained after evaporation of such solutions to dryness is however no longer an acetate of aluminium as the acetic acid is displaced by the acid added and evaporated, because 20 of its easy volatility, an aluminium salt of the acid added (tartaric acid, lactic acid, etc.,) being left which no longer possesses the special therapeutic action of the original acetate, and only small traces of the 25 acetic acid remaining.

This invention relates to a process, by means of which it is possible to obtain compounds which, without the acetate of aluminium being substantially affected, can be 30 evaporated to dryness and yet remain soluble. This is effected by combining the acetic acid contained in the solution of acetate of aluminium, whether it be combined with the aluminium or in free state, with hexamethylene-tetramin. In this way a double salt, 35 namely hexamethylene-tetramin-aluminium-acetate is obtained. The acetic acid is so to say protected. At the same time there is utilized the well known property of certain hydroxyl compounds, such as mannite, 40 glycerin, or oxy-acids, such as citric acid, tartaric acid or their salts, of maintaining in solution compounds of aluminium (as well as of iron). It is advisable in practice 45 not to use the said oxy compounds in a free state, but combined or mixed with an equivalent quantity of hexamethylene-tetramin. Hexamethylene-tetramin is a monoacidic base, the molecular weight of which is 140. 50 Equivalent quantities of hexamethylene-tetramin and citric acid for instance are therefore 140 parts of the former to 210/3=70 parts of the latter (citric acid being a tribasic acid). Owing to the above

named formation of the compound hexa- 55 methylene-tetramin-aluminium-acetate and the further combination of this product with for instance hexamethylene-tetramin-lactate, compounds are produced, which, owing to the fact that they dissociate very 60 readily, combine in an advantageous manner the efficacious action both of acetate of aluminium and of the hexamethylene-tetramin which is also antiseptic. At the same time the flavor is improved and rendered 65 less sharp, which is of importance for internal administration of the products.

The process is carried out by causing aluminium acetate, one of the aforementioned compounds which increase the solu- 70 bility of aluminium acetate, and hexamethylene-tetramin, to react on each other either simultaneously or consecutively. The hexamethylene-tetramin is employed in such a quantity as to neutralize completely 75 the acetic acid contained in the aluminium acetate titrable with sodium-hydroxid (NaOH), as well as the second acid added. By titrating aluminium acetate with sodium-hydroxid until phenolphthalein, added as 80 an indicator becomes red, the free acetic acid as well as about one molecule of the acetic acid combined in the aluminium acetate is neutralized, as is well known. The quantity of hexamethylene-tetramin 85 used according to the present process should be sufficient to combine with the acetic acid determined in this way, as well as with the second acid added, the hexamethylene-tetramin being calculated as a mono-acidic 90 base, as above indicated. The solution thus obtained is then evaporated to dryness, preferably in a vacuum.

Example 1: 1 kg. of officinal aluminium acetate solution (containing 0.085 gr. acetic 95 acid in 1 cbc. cm.) is mixed at the ordinary temperature with 50 gr. citric acid and 300-310 gr. hexa-methylene-tetramin and left standing for some time. The solution thus obtained is then evaporated to dryness 100 in a vacuum at about 60-70°. The salt obtained in this manner, is a white crystalline mass of sweet taste, which can be easily reduced to powder. The well known harsh, tart taste of soluble aluminium compounds 105 is no longer perceptible. The preparation is easily soluble in water. The solution does not coagulate when boiled, and does not

precipitate any alumina. The salt is almost insoluble in ether. Warm dilute alcohol dissolves it almost completely, while it does not completely dissolve in absolute alcohol.

- 5 It is also largely soluble in benzol. The salt dissolves completely in warm diluted hydrochloric acid (5%) also in acetic acid or glacial acetic acid, and also in diluted sulfuric acid but in the latter case a slight opalescence is noticeable, (due to calcium acetate of the acetate of alumina). Dilute soda lye also dissolves it, and also in this case only a little lime is left as a sediment at the bottom. Acetone dissolves it only partially.

- 15 Example 2: 1 kg. aluminium acetate (compare Example 1) is mixed with 50 gr. tartaric acid and about 285 gr. hexa-methylene-tetramin, and then treated in the same way as in the foregoing example. In this way solid white transparent crystals are obtained, the properties of which are the same as those of the salts obtained in the Example 1.

- 25 Example 3: 1 kg. aluminium acetate (see Example 1) is mixed with 50 gr. lactic acid and about 265 gr. hexa-methylene-tetramin and treated as stated in Example 1. This gives a white mass of very thick pasty consistency which can be readily pressed in tubes. The properties correspond otherwise to those of the product described in the Example 1.

- 35 In place of the solid hexamethylene-tetramin, formaldehyde and ammonia can be used, these reagents combining to form hexamethylene-tetramin in solution, as is well known. Accordingly the dry hexamethylene-tetramin and the formaldehyde-ammonia solutions which form hexamethylene-tetramin are equivalents for purposes of the present invention. The use of such formaldehyde-ammonia solutions is illustrated by the following example.

- 45 Example 4: 1 kg. of solution of officinal aluminium acetate (compare Example 1) is mixed with 50 gr. citric acid, and the liquid evaporated after adding a mixture of formalin and ammonia in quantities calculated for 300 gr. of hexa-methylene-tetramin. This mixture of formalin and ammonia must be left standing 24 hours before use, until, after a slight heating, the ammonia smell has disappeared. The properties of the end product obtained in that manner, correspond to those described in Example 1.

I claim:

1. The process of producing water soluble compounds of aluminium acetate which comprises treating the latter with hexamethylene-tetramin.
2. The process of producing water soluble

compounds of aluminium acetate which comprises treating aluminium acetate solutions with hexamethylene-tetramin and evaporating to dryness.

3. The process of producing water soluble compounds of aluminium acetate which comprises treating the same with hexamethylene-tetramin in the presence of compounds increasing the solubility of aluminium compounds.

4. The process of producing water soluble compounds of aluminium acetate which comprises treating aluminium acetate solutions with hexamethylene-tetramin and evaporating to dryness in the presence of compounds increasing the solubility of aluminium compounds.

5. The process of producing water soluble compounds of aluminium acetate which comprises treating solutions of aluminium acetate with a solution of hexamethylene-tetramin, and evaporating to dryness.

6. The process of producing water soluble compounds of aluminium acetate which comprises treating solutions of aluminium acetate with a solution of hexamethylene-tetramin, and evaporating to dryness in the presence of compounds increasing the solubility of aluminium compounds.

7. The process of producing water soluble compounds of aluminium acetate which comprises treating solutions of aluminium acetate with a solution of hexamethylene-tetramin, and evaporating to dryness in a vacuum and at a temperature of about 60 to 70°.

8. The process of producing water soluble compounds of aluminium acetate which comprises treating solutions of aluminium acetate with a solution of hexamethylene-tetramin, and evaporating to dryness in the presence of hydroxyl compounds increasing the solubility of aluminium compounds.

9. A new product comprising a water soluble compound of aluminium acetate and hexamethylene-tetramin.

10. A new product comprising a water soluble compound of aluminium acetate and hexamethylene-tetramin combined with a compound increasing the solubility of aluminium compounds.

11. A new product comprising a water soluble compound of aluminium acetate and hexamethylene-tetramin combined with a hydroxyl compound increasing the solubility of aluminium compounds.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

GEORG EICHELBAUM.

Witnesses:

HENRY HASPER,
WOLDEMAR HAUPT.