STATES PATENT OFFICE. UNITED

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MANUFACTURE OF ACETIC ANHYDRIDE.

No Drawing.

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(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that I, HENRY DREYFUS, of London, England, have invented certain new and useful Improvements Relating to 5 the Manufacture of Acetic Anhydride (for which I have filed applications in France, 478,951, June 30, 1914; in Switzerland Dec. 11, 1914; in Italy June 5, 1917, and in Great Britain Dec. 13, 1919), of which the follow-

10 ing is a specification. I have found that salts of pyrosulfuric anhydride, comprising bringing a salt of 65 acid, such for example as sodium prosulfate (Na₂S₂O₇) produced by the known in intimate mixture therewith in presence method of heating the bisulfate to decompany the proposulfate of a diluent. 15 pose it into pyrosulfate with elimination of water, can be employed for the manufacture of acetic anhydride by bringing them into reaction with salts of acetic acid. The reaction takes place in such a way that by 20 heating, the salt of pyrosulfuric acid combines with the salt of acetic acid, forming a neutral salt of sulfuric acid. By employing for example pyrosulfate of sodium and anhydrous acetate of sodium, preferably in powder, sulfate of sodium and acetic anhydride are obtained. The reaction is effected by mixing intimately, excluding any trace of moisture. The sodium acetate is believed to react in chemically equivalent 30 quantities according to the equation

 $\begin{array}{l} {\rm CH_3COONa} \\ {\rm CH_3COONa} \\ + {\rm Na_2S_2O_7 = 2Na_2SO_4} + \\ {\rm CH_3COONa} \end{array} \\ \\ + {\rm OCOONa} \\ \\$

but it is preferable to employ an excess of 35 the sodium pyrosulfate. The reaction takes place on heating. The acetic anhydride formed may distil off in porportion as the reaction proceeds.

The reaction is preferably performed in 40 the presence of diluents such as glacial acetic acid or acetic anhydride or mixtures thereof, in which case one has a still more intimate

mixture.

As compared with previously known proc-45 esses the present invention presents the advantage that it is not necessary to cool, but on the contrary it is necessary to heat, which acetic acid and applying heat to the mixsimplifies both the apparatus and the process of manufacture. Moreover pyrosulfate of 50 sodium is much cheaper than the materials hitherto employed for the manufacture of acetic anhydride. In particular I can employ the pyrosulfate of sodium obtained by the method of strongly heating acid sodium anhydride, comprising bringing a salt of acetic acid into reaction with pyrosulfate obtained by heating a bisulfate.

11. Process for the manufacture of acetic anhydride, comprising bringing a salt of

sulfate, NaHSO₄, which decomposes into pyrosulfate of sodium and water.

Instead of acetate of sodium other salts of acetic acid may be employed.

What I claim and desire to secure by Let-

ters Patent is:-1. Process for the manufacture of acetic anhydride comprising bringing a salt of acetic acid into reaction with a pyrosulfate.

2. Process for the manufacture of acetic

3. Process for the manufacture of acetic anhydride, comprising bringing a salt of 70 acetic acid into reaction with a pyrosulfate in intimate mixture therewith in presence of a substantially water-free acetic liquid incapable of reacting with acetic anhydride.

4. In a process for the manufacture of 75 acetic anhydride, mixing a salt of acetic acid and a pyrosulfate in presence of a diluent

and applying heat to the mixture.

5. In a process for the manufacture of acetic anhydride, mixing a salt of acetic acid 80 and pyrosulfate in presence of a substantially water-free acetic liquid incapable of reacting with acetic anhydride and applying heat to the mixture.

6. Process for the manufacture of acetic 85 anhydride, comprising bringing sodium acetate into reaction with sodium pyrosulfate.

7. In a process for the manufacture of acetic anhydride, mixing sodium acetate and sodium pyrosulfate in presence of a diluent 90 and applying heat to the mixture.

8. In a process for the manufacture of acetic anhydride, mixing sodium acetate and sodium pyrosulfate, in presence of a substantially water-free acetic liquid incapable 95 of reacting with acetic anhydride, and applying heat to the mixture.

9. In a process for the manufacture of acetic anhydride, mixing sodium acetate and sodium pyrosulfate in presence of glacial 100

10. Process for the manufacture of acetic anhydride, comprising bringing a salt of acetic acid into reaction with pyrosulfate 105

acetic acid into reaction with pyrosulfate anhydride, comprising bringing sodium aceobtained by heating a bisulfate, the reaction being performed in intimate mixture.

12. Process for the manufacture of acetic

5 anhydride, comprising bringing a salt of acetic acid into reaction with pyrosulfate obtained by heating a bisulfate, the reaction being performed in intimate mixture in

presence of a diluent.

13. Process for the manufacture of acetic anhydride, comprising bringing a salt of acetic acid into reaction with pyrosulfate obtained by heating a bisulfate, the reaction being performed in intimate mixture in 15 presence of a substantially water-free acetic liquid incapable of reacting with acetic anhydride.

14. Process for the manufacture of acetic anhydride, comprising bringing a salt of 20 acetic acid into reaction with pyrosulfate obtained by heating a bisulfate, the reaction being performed in intimate mixture in

presence of glacial acetic acid.

15. In a process for the manufacture of 25 acetic anhydride mixing a salt of acetic acid in presence of a diluent, with pyrosulfate obtained by heating bisulfate, and applying heat to the mixture.

16. In a process for the manufacture of 30 acetic anhydride, mixing a salt of acetic acid, in presence of a substantially waterfree acetic liquid incapable of reacting with acetic anhydride, with pyrosulfate obtained by heating bisulfate, and applying heat to 35 the mixture.

17. In a process for the manufacture of heat to the mixture. acetic anhydride, mixing a salt of acetic acid, in presence of glacial acetic acid, with pyrosulfate obtained by heating bisulfate,

40 and applying heat to the mixture. 18. Process for the manufacture of acetic

anhydride, comprising bringing sodium acetate into reaction with sodium pyrosulfate subscribed my name. obtained by heating sodium bisulfate.

19. Process for the manufacture of acetic

tate and sodium pyrosulfate obtained by heating sodium bisulfate into reaction in intimate mixture.

20. Process for the manufacture of acetic 50 anhydride, comprising bringing sodium acetate and sodium pyrosulfate obtained by heating sodium bisulfate into reaction in intimate mixture, the reaction being performed in presence of a diluent.

21. Process for the manufacture of acetic anhydride, comprising bringing sodium acetate and sodium pyrosulfate obtained by heating sodium bisulfate into reaction in intimate mixture, the reaction being per- so formed in presence of a substantially waterfree acetic liquid incapable of reacting with acetic anhydride.

22. Process for the manufacture of acetic anhydride, comprising bringing sodium ace- 65 tate and sodium pyrosulfate obtained by heating sodium bisulfate into reaction in intimate mixture, the reaction being performed in presence of glacial acetic acid.

23. In a process for the manufacture of 70 acetic anhydride, mixing sodium acetate in presence of a diluent with sodium pyrosulfate obtained by heating sodium bisulfate,

and applying heat to the mixture.

24. In a process for the manufacture of 75 acetic anhydride, mixing sodium acetate, in presence of a substantially water-free acetic liquid incapable of reacting with acetic anhydride, with sodium pyrosulfate obtained by heating sodium bisulfate, and applying 80

25. In a process for the manufacture of acetic anhydride mixing sodium acetate in presence of glacial acetic acid with sodium pyrosulfate obtained by heating sodium bi- 85 sulfate, and applying heat to the mixture.

In testimony whereof I have hereunto

HENRY DREYFUS.