

# PATENT SPECIFICATION



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## PROVISIONAL SPECIFICATION.

### Improvements in or relating to the Manufacture of Acetic Anhydride and Acetic Acid.

We, BRITISH CELANESE LIMITED, of 8, Waterloo Place, London, S.W. 1, a company incorporated in accordance with the laws of Great Britain, and  
5 WALTER BADER, a citizen of the Swiss Confederation, of the works of British Celanese Limited, Spondon, near Derby, do hereby declare the nature of this invention to be as follows:—

10 This invention relates principally to the manufacture of acetic anhydride. It also relates to making acetic acid or mixtures thereof with acetic anhydride.

15 It has been found that acetic anhydride can be produced by combining acetone with carbon dioxide. This is a remarkable reaction and provides a simple and advantageous method of producing acetic anhydride. The reaction  
20 may be performed with or without pressure, with or without heat and with or without catalysts. Preferably it is performed under pressure and/or with heat. A catalyst or catalysts may be  
25 employed.

30 In carrying out the invention for the production of acetic anhydride the acetone and carbon dioxide may be employed free from water, but water or moisture might also be present.

By way of example a mixture of

acetone vapour and carbon dioxide, obtained for instance by bubbling carbon dioxide through acetone, and containing some excess of carbon dioxide, may be led, preferably under pressure, through one or more heated tubes or chambers containing or not containing a catalyst. Untransformed acetone and carbon dioxide can be returned again through the tube or chamber, if necessary after adjustment of their relative proportions. It is of course understood that we in no way confine ourselves to this example which may be varied widely.

45 Acetic acid may be made from the anhydride by subsequent treatment with water, or acetic acid or a mixture of acetic acid may even be obtained by introducing water in the acetone-carbon dioxide reaction, for example employing acetone or carbon dioxide containing water, or admitting steam together with the mixture of acetone and carbon dioxide to the reaction.

Dated this 23rd day of January, 1924.

T. L. WHITEHEAD,  
Chartered Patent Agent,  
Patent Department,  
British Celanese Ltd.,

8, Waterloo Place, London, S.W. 1.

## COMPLETE SPECIFICATION.

### Improvements in or relating to the Manufacture of Acetic Anhydride and Acetic Acid.

65 We, BRITISH CELANESE LIMITED, of 8, Waterloo Place, London, S.W. 1, a company incorporated in accordance with the laws of Great Britain, and

WALTER BADER, a citizen of the Swiss Confederation, of the works of British Celanese Limited, Spondon, near Derby, do hereby declare the nature of this

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invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

5 This invention relates principally to the manufacture of acetic anhydride. It also relates to making acetic acid or mixtures thereof with acetic anhydride.

10 It is known that acetic anhydride is decomposed into acetone and carbon dioxide by heat. This reaction is catalysed by a great number of substances. The literature (see Sabatier-Reid, Catalysis in Organic Chemistry, 15 London 1923) mentions the oxides and carbonates of lithium, calcium, barium, strontium, magnesium, zinc, cadmium, iron, aluminium, chromium, uranium, manganese, thorium, zirconium, 20 titanium, cerium and tin. Many other bodies and mixtures, like charcoal, silica, Portland cement, metallic copper have the same effect.

25 It has been found that this reaction is reversible, that is to say that acetic anhydride may be formed by reacting on acetone with carbon dioxide in excess, preferably under pressure. The same substances which catalyse the decom- 30 position of acetic anhydride also catalyse its synthesis from acetone and carbon dioxide. Catalysts may, however, be omitted, though less advantageously.

35 While the temperature for the manufacture of acetone from acetic acid or anhydride is usually near 500° C., it may be much lower for the reversed reaction, for instance about 350° C., but there 40 is no marked higher or lower limit of temperature. The reaction of the present invention is preferably performed at temperatures of from about 250° C. to 450° C.

45 The invention may be carried out according to one way, to which we in no wise confine ourselves, by saturating a stream of carbon dioxide with acetone vapours at about 20° C., and leading it through a tube filled with wood char- 50 coal and heated to about 350° C. The liquid which can be condensed out of the carbon dioxide after the passage, by cooling, contains acetic anhydride.

55 The following example illustrates the manner in which the invention may practically be carried out, but the invention is in no way limited to this particular method.

#### EXAMPLE.

60 In a closed system of tubes and con- tainers carbon dioxide is circulated by a pump. The system is connected with a container of liquid carbon dioxide, so that the pressure is everywhere about 750 lb. per square inch. The carbon dioxide 65 gas is made to bubble through acetone at about 100° C., then to pass through a tube heated to 350° C. The tube is filled with pieces of pumice stone on to which manganese carbonate has been precipitated. After passing the tube 70 the gas current is cooled to about 0° C. and the condensate separated. The un- condensed gas re-enters the circulation. The condensate is a mixture of acetone 75 with up to about 10—15% or more of acetic anhydride which can be separated in any convenient way, e.g. by fractional distillation.

80 If water is present in this reaction, be it by the use of an aqueous acetone solution, or by the injection of steam into the system, the acetic anhydride produced is partially or totally converted 85 into acetic acid.

Having now particularly described and ascertained the nature of our said inven- tion and in what manner the same is to be performed, we declare that what we 90 claim is:—

1. Process for the manufacture of acetic anhydride, characterised in that acetone is acted upon with carbon dioxide in excess, preferably under 95 pressure.

2. Process according to Claim 1 char- acterised in that the reaction is per- formed in presence of catalysts such as are known to promote the formation of acetone from acetic acid or anhydride. 100

3. Process according to Claim 1 or 2 characterised in that the reaction is effected in presence of water vapour, whereby the acetic anhydride is partially 105 or entirely converted to acetic acid.

4. A process substantially as described for the manufacture of acetic anhydride or acetic acid.

Dated this 17th day of October, 1924.

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8, Waterloo Place, London, S.W. 1.