AMENDED SPECIFICATION.

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# PATENT SPECIFICATION

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COMPLETE SPECIFICATION (AMENDED).

## Improved Process for the Production of Ketene.

We, KODAK LIMITED, a Company registered under the Laws of Great Britain, of Kodak House, Kingsway, London, W.C. 2, (Assignees of HANS THACHER CLARKE, citizen of the United State of America, and CHARLES EDWARD WARING, citizen of the United States of America, both of Kodak Park, Rochester, New York, United States of America) do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to a process for the manufacture of ketene from acetone and has as its object to provide a process in which the heat is efficiently and economically transferred to the acetone vapours, all particles of the vapour reach the required decomposition temperature, and the vapours are maintained at a specified temperature for the optimum reaction period.

It was shown in 1910 by Schmidlin and Bergmann (Berichte der Deutschen Chemischen Gesellschaft, Vol. 43, p. 2821) that acetone vapour, when passed through a combustion tube, reacted to form ketene. Hurd and Cochran (J. Amer. Chem. Soc. 45, page 515, 1923) repeated the work of Schmidlin and Bergmann, using a combustion tube filled with broken porcelain and obtained, at a temperature of 600° C., slightly less than 11% of available ketene. These and other investigators believed that a much larger amount of ketene had been initially formed and subsequently decomposed, as they found the resulting decomposition products, ethylene and carbon monoxide in considerable quantities.

British Patent Specification No. 237,573 described the use of a preventive catalyst, which counteracts the degenerative decomposition, noted above. By the use of this catalyst and a temperature of between 600° C. and 675° C. a yield of more than 80% of the theoretical amount of ketene was obtained, while without the catalyst less than 20% was produced.

From the prior work it would, therefore, be supposed that the essential procedure to be followed in order to realise a high yield in manufacturing ketene comprises accurate temperature control, and the use of some preventive catalyst It has been found, on the other hand, that the catalyst is not necessary. The precautionary measures of excluding from the apparatus certain metals that promote the degenerative decomposition, and also of ensuring the optimum time of contact at the correct operating temperature will result in an equally high yield.

In the course of an investigation of this subject, it was determined that certain metals apparently catalyse an entirely different reaction in which carbon is deposited in large quantities and the yield of ketene is enormously decreased if not completely inhibited. Certain metals, such as iron or nickel, or their salts, if present even in minute quantities, cause a very marked reduction in the yield.

The previous work on the manufacture of ketene has been carried out on a laboratory scale in which combustion tubes of small diameter have been used. In working on a semi-production basis employing larger tubes it was found that the percentage of conversion was considerably decreased with a large quantity of the starting material appearing in the final product unchanged. The filling material used for the combustion tubes was an inert material which had been leached with nitric and hydrochloric acids to remove all traces of the decomposing catalysts. It was evident that all points

within the combustion tube were not at the correct reaction temperature, the centre, naturally, being cooler than the wall. It was then found that by packing the combustion tube with fragments or turnings of a good heat conductor, such as copper, it was possible to bring the efficiency to a much higher value.

The effect of using copper as a filling material does not increase the percentage yield but, because of its excellent heat conductivity, raises the temperature of the vapours quickly to the reaction temperature. This prevents undue degenerative decomposition of the ketene which results when the ketene is retained at the operating heat for any appreciable time.

The number of metals that can replace copper are necessarily limited. They should have a higher melting point than the operating temperature used and should be free from iron or other metals that promote the breaking down of the ketene. Metals other than iron and nickel having a melting point above 700° C. and a coefficient of heat conductivity of more than .150 C.G.S. system, may be used. Copper, its alloys, brass, bronze, and the like, and silver, have been found to work entirely satisfactorily.

The time of contact of the starting material at the operating temperature is of first importance,—over-exposure at this temperature will greatly decrease the useful yield and under-exposure will permit an unnecessarily large proportion of the starting material to pass through the combustion chamber unchanged, the optimum time of contact for acetone vapour in the heated zone being not more than five seconds.

The invention will now be described by way of example, but it will be understood that it is not limited to the details thus given, except as indicated in the appended claims.

Acetone is vapourised by boiling at a predetermined rate, and the vapour is passed into the copper reaction tube having a diameter of the order of a half-inch or more, filled with copper turnings, and heated to 650—670° C. The acetone vapour is conducted into the reaction chamber at such a rate that the acetone is at the optimum temperature, during a period of time less than five seconds. The unchanged acetone is condensed and returned to the boiling vessel, and the gaseous reaction products are condensed or absorbed in any desired manner. After about eight hours operation a yield of approximately 80% is realised.

It is to be understood that while the use of metal filings or turnings has been described as the preferred filler, other forms may be used giving an equivalent cellular effect, that is, providing a considerable area of unimpeded air space with comparatively thin walls or masses of heat conductive material in a sponge or network throughout the space.

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

1. The method of manufacturing ketene from acetone which comprises passing acetone vapours through a heated chamber containing a network or sponge-like structure of a metal which is free from iron and nickel and which has a high thermal conductivity and a melting point temperature greater than the reaction temperature.

2. The method as claimed in Claim 1 in which the metal has a melting point above 700° C. and a coefficient of thermal conductivity greater than 0.150 C.G.S. units.

3. The method as claimed in Claim 1 or Claim 2 in which the chamber is maintained at a temperature of 650—700° C. and the vapours are so passed therethrough that the period of contact of the vapour and the network is less than five seconds.

4. The method of manufacturing ketene from acetone substantially as described.

Dated this 15th day of March, 1929.

KILBURN & STRODE,
Agents for the Applicants.

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