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PATENT APPLICATION

PROCESS FOR THE MANUFACTURE OF HYDROGEN PEROXIDE

It is known that hydrogen peroxide forms compounds with a number of organic substances. For instance, alkyl and acyl peroxides are formed by replacing one or both hydrogen atoms of the hydrogen peroxide by one or two alkyl or acyl radicals. Acetone reacts with hydrogen peroxide under formation of cyclic addition compounds. By reacting Caro acid, H_2SO_5, with acetone, dicycloacetonperoxide is formed

$$(CH_3)_2\ C\begin{matrix}\diagup O\text{-}O\diagdown \\ \diagdown O\text{-}O\diagup\end{matrix} C\ (CH_3)_2$$, melting point 132°C.

A second compound of this class is the tricycloacetonperoxide of the probable structure

$$(CH_3)_2\ C\begin{matrix}\diagup O\text{-}O\text{-}C\ (CH_3)_2O \\ \diagdown O\text{-}O\text{-}C\ (CH_3)_2O\end{matrix}$$

and a melting point of 94 - 95°C. It is formed when concentrated hydrochloric acid is added to a mixture of about equal parts by weight of a hydrogen peroxide solution (50%) and acetone under cooling. The compound is insoluble in water, acids and alkalis but is easily soluble in ether, benzene and other organic solvents; it is well crystallized, volatile and explodes violently when struck or on heating.

Whereas the hydrogen peroxide cannot be recovered as such from very many of its organic compounds, the addition compounds of acetone and hydrogen peroxide which will be called here acetone peroxide for the sake of brevity can be more or less completely decomposed into its components. Acetone peroxide decomposes on heating with dilute sulphuric acid into acetone and hydrogen peroxide; the trimeric compound is more easily decomposed than the dimer.

This behavior makes it possible to recover the hydrogen peroxide, for instance, by distillation.

A considerable part of the hydrogen peroxide, however, is decomposed in this procedure so that when known methods of preparation are followed the yield of hydrogen peroxide is only about 30%.

It has been found that the losses which occur in the course of the usual acidic decomposition of acetone peroxide can be, to a large extent, avoided if the decomposition is carried out in the presence of a mutual solvent. Water soluble organic solvents, as e.g. methanol, ethanol or acetone itself are suitable for this purpose. Mixtures of two or more compounds also can be used as mutual solvents. The presence of these mutual solvents also considerably shortens the time required for the hydrolysis of acetone peroxide and hydrolysis can be carried out at a much lower temperature. In this way yields of 80 - 90% of hydrogen peroxide are obtained.

Example 1.

12 parts by weight of tricycloacetonperoxide are refluxed for 10 minutes with 300 parts by weight of ethyl alcohol and 150 parts by weight of 10% sulphuric acid. Subsequently, the ethanol is distilled off under vacuum. The aqueous residue contains, besides sulphuric acid, 4.8 parts by weight of hydrogen peroxide corresponding to 87% of the active oxygen charged.

Example 2.

A mixture of 12 parts by weight of acetone peroxide, 400 parts by weight of methanol and 100 parts by weight of 5% sulphuric acid is refluxed for 15 minutes. Subsequently, the methanol is distilled off under reduced pressure. The residue contains 4.6 parts by weight of hydrogen peroxide corresponding to 82% of the active oxygen charged.

Example 3.

12 parts by weight of acetone peroxide are refluxed with 200 parts by weight of acetone and 50 parts by weight of 10% sulphuric acid for 30 minutes. The acetone is then distilled off under vacuum. The residue contains 3.5 parts by weight of hydrogen peroxide corresponding to 65% of the active oxygen charged.

Claims

Process for the manufacture of hydrogen peroxide by acid hydrolysis of acetone peroxide characterized by working in the presence of mutual solvents.