

Process for the preparation of L-ascorbic acid

The invention relates to a process for the preparation of L-ascorbic acid by reaction of 2-keto-L-gulonic acid with concentrated mineral acid at temperatures between 40 and 80°C.

The invention relates in particular to a process for the preparation of L-ascorbic acid, characterized in that the reaction is carried out at temperatures between 50 and 70°C.

Processes for the preparation of ascorbic acid in one step by reaction of 2-keto-L-gulonic acid with an acid are known. US 2,185,383 describes the reaction of 2-keto-L-gulonic acid with concentrated hydrochloric acid and acetic acid as a solvent. Japanese Unexamined Application 58-177986 describes a process for the preparation of L-ascorbic acid in which a solution of sodium 2-keto-L-gulonic acid [sic] in ethanol and acetone is first neutralized with hydrochloric acid. Japanese Examined Application 48-15931 describes the reaction of 2-keto-L-gulonic acid with a mineral acid in an inert solvent and in the presence of a surface-active substance. Likewise, the preparation of L-ascorbic acid starting from 2-keto-L-gulonic anhydride in the presence of a surface-active substance is described in WO 87/00839. EP 0 324 261 and GB 2,205,567 describe the reaction of 2-keto-L-gulonic acid with an acid in a mixture of inert solvents in the presence of a surface-active substance.

The reaction of 2-keto-L-gulonic acid with 36% hydrochloric acid is described in DE 2939052. Here, the L-ascorbic acid is obtained in a yield of 87% after a reaction at 100°C and after distilling off the hydrochloric acid.

Surprisingly, investigations in the context of L-ascorbic acid synthesis showed that high yields are obtained if the reaction is carried out at temperatures between 40 and 80°, in particular between 50 and 70°C.

A temperature range from 57 to 63°C has proven particularly preferable for carrying out the reaction.

The novel process according to the invention enables L-ascorbic acid to be obtained in high yields
5 by lactonization of 2-keto-L-gulonic acid with concentrated mineral acid.

It has proven particularly advantageous that at the lower temperatures the decomposition of the ascorbic acid obtained takes place significantly more slowly
10 than in the processes known from the prior art (e.g. DE 2939052; process at 100°C).

A further advantage proves to be the lower energy consumption associated therewith and thus lower environmental pollution.

15 Moreover, it proves advantageous in comparison with the prior art that the reaction can be carried out without organic solvents.

The process according to the invention proves to be advantageous if the 2-keto-L-gulonic acid and the
20 hydrochloric acid are employed in a quantitative ratio of 1:3.

The invention thus also relates to a process in which the 2-keto-L-gulonic acid and the mineral acid are employed in the ratio 1:3.

25 However, the concentration can be varied within wide ranges. A more concentrated and also a more dilute procedure can be carried out.

The weight ratio 2-keto-L-gulonic acid/concentrated mineral acid can be, for example, between 10:1 and
30 1:10. The range is particularly preferably between 1:2 and 1:4, and a ratio of 1:3 is very particularly advantageous.

Mineral acid means, for example, hydrochloric acid, phosphoric acid or sulfuric acid. The
35 concentrated mineral acids are preferably employed.

The invention also relates to a process, as described, characterized in that the mineral acid used is concentrated hydrochloric acid.

The invention also relates to a process, as described, characterized in that a mixture consisting of 2-keto-L-gulonic acid and concentrated mineral acid is heated to the desired reaction temperature.

5 The reaction time is customarily between 1 and 5 hours, in particular between 2 and 4 hours, and a reaction time of approximately 3 hours is particularly preferred.

10 Working up is carried out by methods known per se, e.g. by active carbon treatment, filtration, removal of the solvent and crystallization. In accordance with the process according to the invention, the yields of L-ascorbic acid are, as a rule, over 90%, such that complicated purification steps can be
15 dispensed with.

Above and below, all temperatures are indicated in °C.

Example 1

100 g of 2-keto-L-gulonic acid are introduced into 300 g of 37% hydrochloric acid. The batch is then heated to 58° and kept at this temperature for 3 hours.

5 After removal of the hydrochloric acid and work-up, a yield of L-ascorbic acid of 91.4% of theory is obtained.

Example 2

10 100 g of 2-keto-L-gulonic acid are introduced into 300 g of 37% hydrochloric acid. The batch is then heated to 59-60° and kept at this temperature for 3 hours.

After removal of the hydrochloric acid and work-up, a
15 yield of L-ascorbic acid of 90.2% of theory is obtained.

Example 3

20 100 g of 2-keto-L-gulonic acid are introduced into 300 g of 37% hydrochloric acid. The batch is then heated to 59-60° and kept at this temperature for about 1.5 hours.

After removal of the hydrochloric acid and work-up, a
25 yield of L-ascorbic acid of 88.4% of theory is obtained.

Patent Claims

1. Process for the preparation of L-ascorbic acid, characterized in that 2-keto-L-gulonic acid is reacted
5 with concentrated mineral acid at temperatures between 40 and 80°C.
2. Process according to Claim 1, characterized in that the reaction is carried out at temperatures between 50 and 70°C.
- 10 3. Process according to Claim 1 or 2, characterized in that the 2-keto-L-gulonic acid and the mineral acid are employed in the ratio 1:3.
4. Process according to one of Claims 1-3, characterized in that the mineral acid used is
15 concentrated hydrochloric acid.
5. Process according to one of Claims 1-4, characterized in that a mixture consisting of 2-keto-L-gulonic acid and concentrated mineral acid is brought to the desired reaction temperature.

