HYDROGEN PEROXIDE

Prepared at 63rd JECFA (2004) and published in FNP 52 ADD 12 (2004) superseding specifications prepared at the 29th JECFA (1985), published in FNP 52 (1992). No ADI was allocated by the 24th JECFA (1980) with comment "may be used only where better methods of milk preservation are not available". Small residues of hydrogen peroxide on food (which has been treated with antimicrobial washing solutions) at the time of consumption would not pose a safety concern (63rd JECFA, 2004).

DEFINITION

The principal method for the manufacture of hydrogen peroxide is the catalytic reduction by hydrogen of a substituted anthraquinone dissolved in a mixed aromatic hydrocarbon solvent, to anthraquinol. The hydrogenation catalyst is removed and the anthraquinol solution is subjected to aerial oxidation, to yield anthraquinone and hydrogen peroxide. The anthraquinone is recycled and the hydrogen peroxide, extracted with water, is purified and concentrated. The dilution of the concentrate is adjusted and a tin based stabilizer added.

Chemical name	Hydrogen peroxide
C.A.S. number	7722-84-1
Chemical formula	H_2O_2
Structural formula	H-O-O-H
Formula weight	34.01
Assay	Not less than the labelled concentration or within the range stated on the label

DESCRIPTION

An odourless, or nearly odourless, transparent and colourless liquid
Caution: Powerful oxidizing agent. Avoid contact with eyes and skin.

FUNCTIONAL USES

Antimicrobial agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)	Miscible with water
Test for peroxide (Vol. 4)	Passes test
Acidity	Acid to litmus

PURITY

Non-volatile residue (Vol. 4)	Not more than 60 mg/kg
Acidity	Not more than 0.03% (as sulfuric acid) Dilute 9 ml of the sample with 90 ml of carbon dioxide-free water, add methyl red TS, and titrate with 0.02 N sodium hydroxide. The volume of sodium hydroxide solution should not be more than 3 ml greater than the volume required for a blank test on 90 ml of water used for dilution.
Phosphate	Not more than 50 mg/kg

See description under TESTS

Iron — Not more than 0.5 mg/kg
See description under TESTS

Tin — Not more than 10 mg/kg
See description under TESTS

Lead (Vol. 4) — Not more than 4 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of the sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental methods

TESTS

PURITY TESTS

Phosphate — Evaporate 400 mg of the sample to dryness on a steam bath. Dissolve the residue in 25 ml of approximately 0.5 N sulfuric acid. Add 1 ml of ammonium molybdate solution (500 mg of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 10 ml of water) and 1 ml of p-methylaminophenol sulfate TS, and allow to stand for 2 h. Any blue colour produced should not exceed that of a control solution made the same way as the test solution, using 2.0 ml of Phosphate Standard Solution (20 µg PO_4) in an equal volume of solution containing equal quantities of reagents used in the test.

Iron — Evaporate 20 g of the sample to dryness on a steam bath with 10 mg of sodium chloride, dissolve the residue in 2 ml of hydrochloric acid, and dilute to 50 ml with water. Add about 40 mg of ammonium persulfate crystals and 10 ml of ammonium thiocyanate TS, and mix. Any red or pink colour does not exceed that produced by 1.0 ml of *Iron Standard Solution* (10 µg Fe) in an equal volume of solution containing the quantities of the reagents used in the test.

Tin — *Aluminium Chloride Solution*: Dissolve 8.93 g of aluminium chloride, $AlCl_3 \cdot 6H_2O$, in sufficient water to make 1000 ml.

Gelatin Solution: On the day of use, dissolve 100 mg of gelatin in 50 ml of boiled water that has been cooled to between 50^o and 60^o.

Tin Stock Solution: Dissolve 250.0 mg of lead-free tin foil in 10 to 15 ml of hydrochloric acid, and dilute to 250.0 ml with dilute hydrochloric acid (1 in 2).

Standard Solution
On the day of use, transfer 5.0 ml of Tin Stock Solution into a 100-ml volumetric flask, dilute to volume with water, and mix. Transfer 2.0 ml of this solution (100 µg Sn) into a 250-ml Erlenmeyer flask, and add 15 ml of water, 5 ml of nitric acid, and 2 ml of sulfuric acid.

Place a small stemless funnel in the mouth of the flask, and heat until strong fumes of sulfuric acid are evolved. Cool, add 5 ml of water, evaporate again to strong fumes, and cool. Repeat the addition of water and heating to strong fumes, then add 15 ml of water, heat to boiling, and cool. Dilute to about 35 ml with water, add 1 drop of methyl red TS and 2.0 ml of the Aluminium Chloride Solution, and mix. Make the solution just

alkaline by the dropwise addition of stronger ammonia TS, stirring gently, and then add 0.1 ml in excess. (Caution: To avoid dissolving the aluminium hydroxide precipitate, do not add more than 0.1 ml in excess of the ammonia solution.) Centrifuge for about 15 min at 4000 rpm, and then decant the supernatant liquid as completely as possible without disturbing the precipitate. Dissolve the precipitate in 5 ml of dilute hydrochloric acid (1 in 2), add 1.0 ml of the Gelatin Solution, and dilute to 20.0 ml with a saturated solution of aluminium chloride.

Sample Solution

Transfer 10 g of the sample into a 250-ml Erlenmeyer flask, and add 15 ml of water, 5 ml of nitric acid, and 2 ml of sulfuric acid. Mix and heat gently on a hot plate to initiate and maintain a vigorous decomposition.

When decomposition is complete, place a small stemless funnel in the mouth of the flask, and continue as directed for the Standard Solution (above), beginning with "... and heat until strong fumes of sulfuric acid are evolved."

Procedure

Rinse a polarographic cell or other vessel with a portion of the Standard Solution, then add a suitable volume to the cell, immerse it in a constant temperature bath maintained at 35 ± 0.2^{o}, and deaerate by bubbling oxygen-free nitrogen or hydrogen through the solution for at least 10 min. Insert the dropping mercury electrode of a suitable polarograph, and record the polarogram from -0.2 to -0.7 V and at a sensitivity of 0.0003 µA per mm, using a saturated calomel reference electrode. In the same manner, record a polarogram of a portion of the Sample Solution at the same current sensitivity. The height of the wave produced by the Sample Solution is not greater than that produced by the Standard Solution at the same half-wave potential.

METHOD OF ASSAY

Accurately weigh a volume of the sample equivalent to about 300 mg of H_2O_2 into a 100-ml volumetric flask, dilute to volume with water, and mix thoroughly. To a 20-ml portion of this solution add 25 ml of diluted sulfuric acid TS, and titrate with 0.1 N potassium permanganate. Each ml of 0.1 N potassium permanganate is equivalent to 1.701 mg of H_2O_2.