SODIUM DL-MALATE

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SYNONYMS Malic acid sodium salt; INS No. 350(ii)

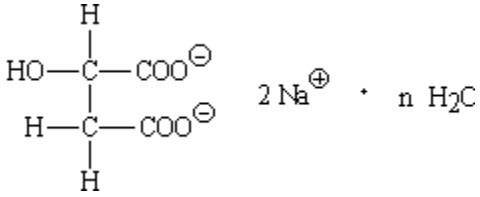
DEFINITION

Chemical names: Disodium DL-malate, hydroxybutanedioic acid disodium salt

C.A.S. number: 676-46-0

Chemical formula: Hemihydrate: $C_4H_4Na_2O_5 \cdot 1/2\ H_2O$
Trihydrate: $C_4H_4Na_2O_5 \cdot 3\ H_2O$

Structural formula:



Formula weight: Hemihydrate: 187.05
Trihydrate: 232.10

Assay: Not less than 98% and not more than 102% on the dried basis

DESCRIPTION Odourless white crystalline powder or lumps

FUNCTIONAL USES Acidity regulator, flavouring agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Freely soluble in water

Test for malate (Vol. 4): Passes test
Test a 1 in 20 solution

Test for sodium (Vol. 4): Passes test

Test for 1,2-dicarboxylic acid: Heat a mixture of 1 ml of a 1 in 20 solution of the sample, 2 to 3 mg of resorcinol and 1 ml of sulfuric acid in a test tube at 120° to 130° for 5 min, cool, and add water to 5 ml. Add sodium hydroxide solution (2 in 5) dropwise while cooling to make alkaline, and add water to 10 ml. A pale blue fluorescence is observed under the ultraviolet light.

PURITY

Loss on drying (Vol. 4): Hemihydrate: Not more than 7% (130°, 4 h)

Trihydrate: 20.5% - 23.5% (130^{o}, 4 h)

Sulfated ash (Vol. 4) — Between 78.2% and 81.4% on the dried basis
Test 0.5 g of the sample (Method I)

Alkalinity — Not more than 0.2% as Na_2CO_3
Dissolve 1 g of the sample in 20 ml of freshly boiled and cooled water, and add 2 drops of phenolphthalein TS. If a pink colour is produced, add 0.4 ml of 0.1 N sulfuric acid. The colour of the solution disappears.

Fumaric and maleic acid — Not more than 1.0% of fumaric acid and not more than 0.05% of maleic acid
See description under TESTS

Lead (Vol. 4) — Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of 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

Fumaric and maleic acid

Buffer solution A:
In a 1000-ml volumetric flask dissolve 74.5 g of potassium chloride in 500 ml of water, add 100 ml of concentrated hydrochloric acid, and dilute to volume with water.

Buffer solution B:
Dissolve 171.0 g of dipotassium hydrogen phosphate, $K_2HPO_4 \cdot 3H_2O$, in 1000 ml of water, and add potassium dihydrogen phosphate, KH_2PO_4, until the pH is exactly 7.0.

Maxima suppressor:
Dissolve, with the aid of a magnetic stirrer, 1 g of gelatin in 65 ml of hot, boiled water. After cooling, add 35 ml of ethanol as a preservative.

Standard solution:
Weigh accurately about 20 g of the sample, previously dried at 130^{o} for 4 h, 100 mg of fumaric acid of the highest purity available, and 10 mg of maleic acid of the highest purity available and transfer into a 500-ml volumetric flask. Add 300 ml of water, 1.5 ml of sodium hydroxide TS, and a few drops of phenolphthalein TS and then continue the neutralization with sodium hydroxide TS to a faint pink colour that persists for at least 30 sec. Dilute to volume with water and mix.

Sample solution:
Transfer about 4 g of the sample, previously dried at 130^{o} for 4 h and accurately weighed, into a 100 ml volumetric flask, and dissolves in 25 ml of water. Add phenolphthalein TS, and neutralize with sodium hydroxide TS if necessary, as directed for the Standard solution. Dilute to volume with water, and mix.

Procedure:
Transfer two 25-ml portions of the "Sample solution" into separate 100-ml volumetric flasks. Dilute one flask (Sample A) to volume with "Buffer solution A". To the other flask (Sample B) add 50 ml of "Buffer solution B" and dilute to volume with water. Rinse a polarograph cell with a portion of "Sample A", add a suitable volume of the solution to the cell, immerse it in a water bath regulated at 24.5 - 25.5°, add 2 drops of the "Maxima suppressor", and then de-aereate by bubbling nitrogen through the solution for at least 5 min. Insert the dropping mercury electrode (negative polarity) of a suitable polarograph, adjust the current sensitivity as necessary, and record the polarogram from -0.1 to -0.8 volt at the rate of 0.2 volt per minute, using a saturated calomel electrode as the reference electrode. Transfer 25 ml of the "Standard solution" into a 100-ml volumetric flask, and dilute to volume with "Buffer solution A". Obtain the polarogram of this solution (Standard A) in the same manner as directed for "Sample A". In each polarogram, determine the height of the maleic acid plus fumaric acid wave occurring at the half-wave potential near -0.56 volt, recording that for the sample as i_U and that for the standard as i_S. In the same manner, obtain polarograms from "Sample B" and a "Standard B", except record the polarogram from -1.05 to -1.7 volts at the rate of 0.1 volt per minute. In each polarogram, determine the height of the maleic acid wave occurring at the half-wave potential near -1.33 volts, recording that for the sample as i_U' and that for the standard as i_S'.

Calculation
Calculate the weight in mg, p, of combined maleic acid and fumaric acid in the sample taken by the formula:

$$500C \times [i_U/(i_S - i_U)]$$

where
C = the concentration, in mg per ml, of combined maleic acid and fumaric acid in the Standard solution.

Similarly, calculate the weight in mg, q, of maleic acid in the sample taken by the formula:

$$500C' \times [i_U'/(i_S' - i_U')]$$

where
C'= the concentration, in mg per ml of maleic acid in the Standard solution. Calculate the weight of fumaric acid in mg, r, in the sample taken from the difference in these values, i.e. (r = p - q).

Finally, calculate the percentage of fumaric and maleic acids present by multiplying r and q, respectively, by 0.025.

METHOD OF ASSAY

Dissolve about 0.25 g of the dried sample, accurately weighed, in 50 ml of glacial acetic acid, and titrate with 0.1 N perchloric acid, determining the endpoint potentiometrically. Each ml of 0.1 N perchloric acid is equivalent to 8.903 mg of $C_4H_4Na_2O_5$.