dl-MALIC ACID

Prepared at the 57th JECFA (1999) and published in FNP 52 Add 9 (2001), superseding specifications prepared at the 53rd JECFA (1999), published in FNP 52 Add 7 (1999). ADI "not specified", established at the 13th JECFA in 1969.

SYNONYMS   2-Hydroxybutanedioic acid; INS No. 296

DEFINITION

Chemical names   dl-Malic acid, 2-Hydroxybutanedioic acid, Hydroxysuccinic acid

C.A.S. number   617-48-1

Chemical formula $C_4H_6O_5$

Structural formula


```
COOH
|
CHOH
|
CH₂
|
COOH
```

Formula weight   134.09

Assay   Not less than 99.0%

DESCRIPTION   White or nearly white crystalline powder or granules

FUNCTIONAL USES   Acidity regulator, flavouring agent (See 'Flavouring agents' monograph JECFA no. 619)

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)   Very soluble in water; freely soluble in ethanol

Melting range (Vol. 4)   127 - 132°

Test for malate (Vol. 4)   Passes test
Test 5 ml of a 1 in 20 solution of the sample, neutralized with ammonia TS

PURITY

Sulfated ash (Vol. 4)   Not more than 0.1%
Test 2 g of the sample (Method I)

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, $K_2HPO_4 \cdot H_2O$, 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 out accurately about 20 g of the sample, 200 mg of fumaric acid and 10 mg of maleic acid, both of the highest purity available, and transfer into a 500-ml volumetric flask. Add 300 ml of sodium hydroxide TS, a few drops of phenolphthalein TS and continue adding 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, accurately weighed, to a 100-ml volumetric flask and dissolve in 25 ml of water. Add phenolphthalein TS, and neutralize with sodium hydroxide TS as directed for 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 24.5-25.5º, 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 min,

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 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 2 g of the sample, accurately weighed, in 40 ml of recently boiled and cooled water, add 2 drops of phenolphthalein TS and titrate with 1 N sodium hydroxide to the first appearance of a faint pink colour which persists for at least 30 sec. Each ml of 1 N sodium hydroxide is equivalent to 67.04 mg of $C_4H_6O_5$.