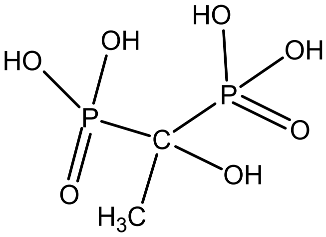
1-HYDROXYETHYLIDENE-1,1-DIPHOSPHONIC ACID

New specifications prepared at 63rd JECFA (2004) and published in FNP 52 Add 12 (2004). Levels of residue that are expected to remain on foods do not pose a safety concern (63rd JECFA, 2004).

SYNONYMS	HEDP, ethane-1-hydroxy-1,1-diphosphonic acid, EHDP, editronic acid
DEFINITION	1-Hydroxyethylidene-1,1-diphosphonic acid (HEDP) is manufactured commercially by the reaction of phosphorous acid with one or more acetylating agents; specifically acetic anhydride, acetyl chloride and/or acetic acid. The final product is typically a 60% solution of HEDP in water.
Chemical name	1-hydroxyethylidene-1,1-diphosphonic acid
C.A.S. number	2809-21-4
Chemical formula	$CH_3C(OH)[PO(OH)_2]_2$
Structural formula	
Empirical formula	$C_2H_8O_7P_2$
Formula weight	205.02
Assay	Total active acid 58 – 62%
DESCRIPTION	Clear pale yellow liquid, free of suspended matter
FUNCTIONAL USES	Sequestrant (for use in antimicrobial washing solutions)
CHARACTERISTICS	
IDENTIFICATION	
<u>Solubility</u> (Vol. 4)	Miscible with water, phosphoric acid and ethylene glycol; soluble in most organic solvents
<u>pH</u> (Vol. 4)	Not more than 2.0 (1% soln)
<u>Specific gravity</u> (Vol. 4)	1.430- 1.471 at 20º
<u>Freezing point</u>	-25º
PURITY	
<u>Chloride</u>	Not more than 40 mg/kg See description under TESTS

Phosphorous acid

Not more than 4.0%
See description under TESTS

Acetic acid

Not more than 1.0%
See description under TESTS

Iron (Vol. 4)

Not more than 10 mg/kg
Determine using an atomic absorption technique appropriate to the specified level

Arsenic ((Vol. 4)

Not more than 5 mg/kg

Lead (Vol. 4)

Not more than 5 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

Chloride

Determine by potentiometric titration by placing 25 g of the sample, accurately weighed, into a titration vessel and adding sufficient water to cover the electrodes. Add 3 ml of concentrated nitric acid. Titrate with 0.005 mol/l silver nitrate to first inflection point and record the titre in ml (A). Calculate the chloride content (mg/kg) from:

$$\text{Chloride (mg/kg)} = [A \times M \times 3.55 \times 10000] / W$$

where
M = concentration of silver nitrate solution (mol/l)
W = weight of sample taken (g)

Phosphorous acid

Determined by iodometric titration. Iodine oxidizes the phosphorous acid present to phosphate, excess iodine is determined and the Phosphorous acid calculated.

Buffer solution pH 7.3: Dissolve 138 g of sodium dihydrogen phosphate in 800 ml of water and adjust pH to 7.3 with 50% sodium hydroxide solution. Make up to 1000 ml with water.

Add 1.5 g of the sample to 20 ml of water in a 250 ml beaker. Add 50 ml of pH 7.3 phosphate buffer. Adjust pH to 7.3 using 50% sodium hydroxide. Transfer the solution to an iodine flask and add 25.0 ml of 0.1 N iodine. Stopper and swirl the solution and place in the dark immediately. After 15 min, remove the flask and add 5 ml acetic acid to flask. Titrate with 0.1 N sodium thiosulfate until a light straw yellow colour. Add starch indicator and continue titration until the end point "black to colourless" is observed and record the titre in ml (B). Repeat titration with a reagent blank determination omitting the sample and record the titre in ml (A).

Calculate the percentage of phosphorus acid from:

$$\text{Phosphorous acid (\%)} = [(A-B) \times N \times E \times 100] / [w \times 1000]$$

where

N = normality of sodium thiosulfate solution
E = equivalent weight of H_3PO_3 (40.99)
w = weight of sample taken (g)

The accuracy has been determined as +/- 0.01% at phosphorous acid level of 1.28%

Acetic acid

Determine by ion chromatography using a Dionex ICE-ASI column with weak acid eluent. Set up the system in line with the instrument manufacturer's operation procedure.

The signal from the acetate ion is quantified against a calibration standard using Formic acid as the internal standard.
Equipment: Dionex ICE-AS1 column
Reagents: Acetic acid (analytical grade) and formic acid (analytical grade)
Procedure: Carry out the determination according to the instrument manufacturer's operation procedure

METHOD OF ASSAY

Place about 3 g of the sample, accurately weighed (w) into a beaker and add 100-150 ml of water. Stir the solution with a magnetic stirrer (maintain throughout titration). Insert pH electrode(s) and record the pH value. Titrate with 1 mol/l sodium hydroxide and record pH (or millivolts) after every 1ml added. Stop the titration at pH 10. Plot the pH as a function of added sodium hydroxide and manually draw the titration curve. Two inflection points will be observed at around pH 3 and pH 8. Take only into account the inflection point at around pH 8. Trace the tangent to this inflection point in order to determine the end-point. Calculate the total active acid from

$$\text{Total active acid (\%)} = [A \times 206 \times N] / [30 \times w] - [1.676 \times P]$$

Where

A = ml of N NaOH from start of titration to end point at pH 8-85
N = concentration of sodium hydroxide used
P = concentration of phosphorous acid (%) (Determined as above)
1.676 = [MW of HEDP x 2] / [MW of phosphorous acid x 3]

Using auto-titration for end-point detection, accuracy has been determined as +/- 0.2% at total active acid level of 63.5%