XYLITOL

Prepared at the 46th JECFA (1996), published in FNP 52 Add 4 (1996) superseding specifications prepared at the 33rd JECFA (1988), published in FNP 38 (1988). Metals and arsenic specifications revised at the 57th JECFA (2001). An ADI 'not specified' was established at the 27th JECFA (1983)

SYNONYMS INS No. 967

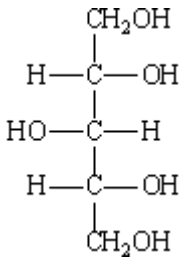
DEFINITION

Chemical names Xylitol

C.A.S. number 87-99-0

Chemical formula $C_5H_{12}O_5$

Structural formula



Formula weight 152.15

Assay Not less than 98.5% and not more than 101.0% on the anhydrous basis

DESCRIPTION White, crystalline powder, practically odourless

FUNCTIONAL USES Sweetener, humectant

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Very soluble in water, sparingly soluble in ethanol

Melting range (Vol. 4) 92 - 96°

Infrared absorption The infrared spectrum of a potassium bromide dispersion of the sample corresponds with the reference infrared spectrum below

PURITY

Water (Vol. 4) Not more than 0.5% (Karl Fischer Method)

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

Nickel (Vol. 4) Not more than 2 mg/kg
Proceed as directed under *Nickel in Polyols*

Reducing sugars (Vol. 4) Not more than 0.2%
Dissolve about 500 mg of the sample, accurately weighed, in 2 ml of water in a 10 ml conical flask. To a second conical flask add 2 ml of a dextrose solution containing 0.5 mg per ml. Add 2 ml of cupric tartrate, alkaline TS to each flask, heat to boiling, and cool. The sample solution is less turbid than the dextrose solution, in which a reddish brown precipitate is formed.

Other polyols Not more than 1.0%
Proceed as described under Method of Assay and calculate the percentage of each polyol (L-arabinitol, galactitol, mannitol, and sorbitol) by the formula therein given, in which W_S refers to the weight, in mg, of the respective polyol taken for the standards solution; R_S is the peak response ratio of the corresponding polyol obtained from the Standard solution; and R_U is the peak response ratio of the corresponding polyol obtained from the Sample preparation. Sum the four individual polyol percentages to obtain the total.

Lead (Vol. 4) Not more than 1 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."

METHOD OF ASSAY

Internal standard solution:
Transfer about 500 mg of erythritol, accurately weighed, into a 25 ml volumetric flask, dilute to volume with water, and mix.

Standard solution:
Transfer about 25 mg each of L-arabinitol, galactitol, mannitol, and sorbitol, accurately weighed, to a 100-ml volumetric flask, dilute to volume with water, and mix. To an accurately measured volume of this solution, add an accurately weighed amount of Reference Standard Xylitol (available from US Pharmacopeial Convention, Inc. 12601 Twinbrook Parkway, Rockville, MD 20852, USA) to obtain a solution with a known concentration of about 49 mg/ml.

Sample preparation:
Transfer about 5 g of the sample, accurately weighed, into a 100-ml volumetric flask, dilute to volume with water, and mix.

Chromatography
Use a gas chromatograph equipped with a flame-ionization detector and a 2-m x 2-mm glass column packed with 3% liquid phase of 25% phenyl-25% cyanopropylmethylsilicone (OV-225 or equivalent) on silanized siliceous earth support (Chromosorb W-HP or equivalent). The carrier gas is nitrogen flowing at about 30 ml/min. The injector port temperature is 250°, the column temperature 200° and the detector temperature 250°. Chromatograph the derivatized Standards Solution prepared as directed under Procedure, and record the peak responses. The relative retention times corresponding to erythritol, L-arabinitol, xylitol, galactitol, mannitol, and sorbitol are usually about 1.0, 2.77, 3.90, 6.96, 7.63 and 8.43,

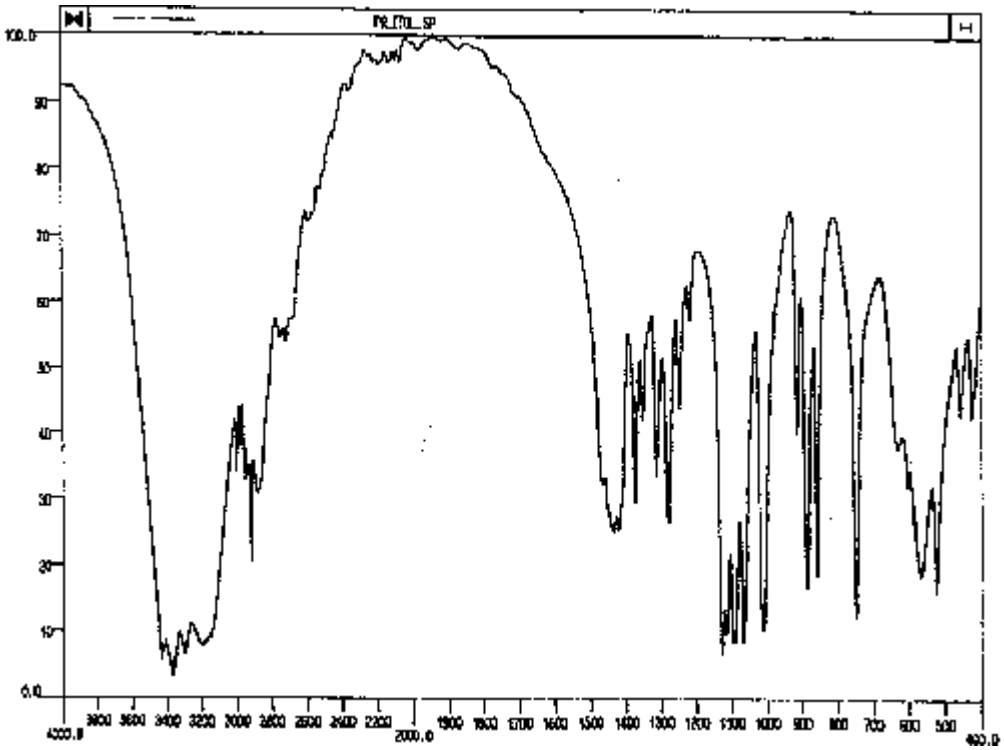
respectively. The relative standard deviation of the response ratios of the derivatized Xylitol to the derivatized erythritol from three replicate injections does not exceed 2.0%.

Procedure:
Pipet 1 ml portions of the standards solution and the sample preparation into separate 100-ml, round-bottom boiling flasks. To each flask, add 1.0 ml of internal standard solution, and evaporate the respective mixtures to dryness on a water bath at 60^{o} with the aid of a rotary evaporator. Dissolve each dry residue in 1 ml of pyridine, and add 1 ml of acetic anhydride to each flask. Boil each solution under reflux for 1 h to complete the acetylation. Separately inject 1-µl portions of the derivatized solutions from the sample preparation and the standard solution into the gas chromatograph and measure the peak responses. Calculate the percentage of xylitol, on the as-is basis, by the formula:

$$100 \times \frac{W_S}{W_U} \times \frac{R_U}{R_S}$$

where
W_S = the weight, in mg, of Reference Standard Xylitol used for the Standard solution
W_U = the weight, in mg, of the sample taken for the Assay preparation
R_U and R_S = the ratios of peak responses of the derivatized analyte to the derivatized erythritol from the Internal Standard solution obtained from the Sample Preparation and the Standard Solution, respectively. Using the value obtained in the water determination, correct the percentage to the anhydrous basis.

Infrared spectrum — Xylitol


PR_1701_SP
100.0
90
80
70
60
50
40
30
20
10
0.0
4000.0
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000.0
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400.0