GLYCEROL

Prepared at the 20th JECFA (1976), published in FNS 1B (1977) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004). An ADI 'not specified' was established at the 20th JECFA (1976)

SYNONYMS Glycerin; INS No. 422

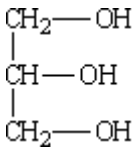
DEFINITION

Chemical names 1,2,3-Propanetriol, glycerol, trihydroxypropane

C.A.S. number 56-81-5

Chemical formula $C_3H_8O_3$

Structural formula



Formula weight 92.10

Assay Not less than 99% of on the anhydrous basis

DESCRIPTION Clear, colourless, hygroscopic, syrupy liquid, having a not more than a slight characteristic odour, which is neither harsh nor disagreeable

FUNCTIONAL USES Humectant, solvent, bodying agent, plasticizer

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Miscible with water and with ethanol; immiscible with ether

Test for glycerol (Vol. 4) Passes test

PURITY

Water (Vol. 4) Not more than 5% (Karl Fischer Method)

Colour The colour of the sample, when viewed downward against a white surface in a 50-ml Nessler tube, is not darker than the colour of a standard made by diluting 0.4 ml of ferric chloride TSC with water to 50 ml and similarly viewed in a Nessler tube of approximately the same diameter and colour as that containing the sample.

Sulfated ash Not more than 0.01%
Heat 50 g in a tared, open dish, and ignite the vapours, allowing them to burn until the sample has been completely consumed. After cooling, moisten the residue with 0.5 ml of concentrated sulfuric acid, and complete

the ignition by heating for 15 min periods at 800±25° to constant weight.

Chlorides (Vol. 4)

Not more than 10 mg/kg
Test 10 g of the sample as directed in the Limit Test using 0.1 mg of chloride ion in the control

Chlorinated compounds

Not more than 30 mg/kg (as chloride ion)
Transfer 5 g of the sample into a dry 100 ml round bottom ground joint flask and add 15 ml of morpholine. Connect the flask with a ground joint reflux condenser, and reflux the mixture gently for 3 h. Rinse the condenser with 10 ml of water, receiving the washing into the flask, and cautiously acidify with nitric acid. Transfer the solution to a suitable comparison tube, add 0.5 ml of silver nitrate TS, dilute with water to 50 ml, and mix thoroughly. Any turbidity does not exceed that produced by 150 µg of chloride ion (Cl) in an equal volume of solution containing the quantities of reagents used in the test, omitting the refluxing.

Fatty acids and esters

Not more than 30 mg/kg
To a 40 ml (50 g) of the sample add 50 ml of recently boiled water and 5 ml of 0.5 N sodium hydroxide, then mix. Boil the mixture for 5 min, cool, add phenolphthalein TS, and titrate the excess alkali with 0.5 N hydrochloric acid. Not more than 1 ml of 0.5 N sodium hydroxide is consumed.

Readily carbonizable substances

Rinse a glass-stoppered, 25-ml cylinder with sulfuric acid TS, and allow to drain for 10 min. Add 5 ml of the sample and 5 ml of sulfuric acid TS, shake vigorously for 1 min, and allow to stand for 1 h. The mixture is no darker than *Matching Fluid H.*

Butanetriols

Not more than 0.2%
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

Butanetrioles

Reagents
- Chromatographic Siliceous Earth: Use 80- to 100-mesh Chromosorb W or other comparable grade of purified chromatographic siliceous earth.
- 1,4-Butanediol: Purify the commercial product by vacuum distillation, collecting the portion distilling between 120° and 121° at 8 mm of mercury.
- 1,2,4-Butanetriol: Purify the commercial product by vacuum distillation, collecting the portion distilling between 151° and 153° at 2 mm of mercury.

Preparation of Column Material
Place about 500 g of chromatographic siliceous earth in a large beaker, add sufficient 6 N hydrochloric acid to cover the material, and allow to

stand overnight. Decant the acid, wash the siliceous earth on a Buchner funnel with water until the wash water is neutral to pH indicator paper then wash with acetone until free from water, and spread out to dry in the air. Transfer the washed and dried material to a sintered glass funnel, cover with chloroform, stir, and remove the chloroform, by aspiration. Repeat the washing with chloroform, and again dry in the air at room temperature. Weigh 87.5 g of the dried siliceous earth into a dish, and add sufficient acetone to form a slurry. Transfer 12.5 g of polyoxyethylene-(8)-ethylenediamine into a beaker, and dissolve in acetone. Place the dish on a steam bath, and heat gently, with stirring, while adding the solution of polyoxyethylene-(8)-ethylenediamine. Continue heating until enough acetone has evaporated to cause the mixture to become free-flowing, and spread out to dry at room temperature.

(Note: The column prepared with polyoxyethylene-(8)-ethylene-diamine does not have long-term stability, particularly when used with a flame-ionization detector; it is more stable, however, when used with a thermal-ionization detector. To prolong stability in either case, the column should be kept sealed against exposure to air when not in use.)

Weigh accurately about 10 g of the sample, add 1 drop of 1,4-butanediol, accurately weighed, as internal standard, dilute with 5 ml of methanol, and mix. Inject a 10- portion of this solution into a gas chromatographic apparatus equipped with a linear temperature programming device. The operating conditions of the apparatus may vary, depending upon the particular instrument used, but a suitable chromatogram is obtained with a copper column, 1.5 m in length and 6.3 mm in outside diameter, packed with the column material previously described. In addition, the carrier is helium, flowing at the rate of 100 ml per min; the injector block temperature is 320^{o}, the detector block temperature is 250^{o}, and the column temperature is programmed to rise from 150^{o} to 180^{o} at a rate of 5.60 per min. The detector bridge current should be maintained at 250 mA when the operating conditions described are employed.

The resolution factor, R. should be not less than 1.9 between the threo- and the erythro-butanetriols peaks, not less than 2.5 between the erythro-1,2,3-butanetriol and the glycerol peaks, and not less than 4.5 between the glycerol and the 1,2,4-butanetriol peaks. (These values for R are obtained when mixtures of equal quantities of glycerol and the butanetriols are determined in an apparatus programmed as described above).

Prepare a 1 in 1,000 solution in glycerol of 1,2,4-butane-triol, accurately weighed, and calculate the percent (P) of 1,2,4-butanetriol in the standard mixture. Weigh accurately about 10 g of the standard mixture, add 1 drop of 1,4-butane-diol, accurately weighed, as the internal standard, and dilute with 5 ml of methanol. Inject about 10 µl of this solution, and obtain a standard chromatogram under the same operating conditions as employed for the sample, applying attenuation of the detector signal as necessary. Under the conditions described, the 1,4-butanediol is eluted in about 8 min, and an area of about 10 cm^2 is generated as compared to an area of 1.0-1.5 cm^2 for the butanetriols when present in a concentration of about 0.1%. In addition, the following retention times have been obtained: 1.00 for 1,4-butanediol, 2.14 for threo-1,2,3-butanetriol, 2.52 for erythro-1,2,3-

butanetriol, and 5.26 for 1,2,4-butanetriol. Retention times will vary if programming different from that described is used.

Calculation

Measure the areas of the peaks produced by the 1,4-butanediol (a) and by the 1,2,4-butanetriol (A), and calculate the response factor (f) by the formula:

$$\frac{W \times P \times a}{100 \times w \times A}$$

where
W = the exact weight of the standard mixture used for dilution with the methanol
w = the exact weight of the drop of 1,4-butanediol internal standard added to the standard mixture.
Calculate the percent of each butanetriol in the sample by the formula:

$$\frac{100 \times f \times W' \times Ax}{A' \times W}$$

where
f = the response factor previously determined
W' = the exact weight of 1,4-butanediol internal standard added to the sample solution
Ax = the area of the peak produced by each butanetriol
A' = the area of the 1,4-butanediol peak
W = the weight of the sample
The sum of the percents found does not exceed 0.2.

METHOD OF ASSAY

Weigh accurately about 1 g of the sample and dissolve in water to make 100 ml. Add 100 ml of 0.3% potassium periodate solution to a 5 ml portion of the solution, shake thoroughly, and allow to stand for 1 h. Add 1 ml of propylene glycol, allow to stand for 10 min, and titrate with 0.05 N sodium hydroxide, using 3 drops of phenol red TS as the indicator, until a pink colour persists. Perform a blank test in the same manner as the sample. Each ml of 0.05 N sodium hydroxide is equivalent to 4.605 mg of $C_3H_8O_3$.