FIRST SUPPLEMENT TO THE FIFTH EDITION

FOOD CHEMICALS CODEX

INSTITUTE OF MEDICINE
OF THE NATIONAL ACADEMIES

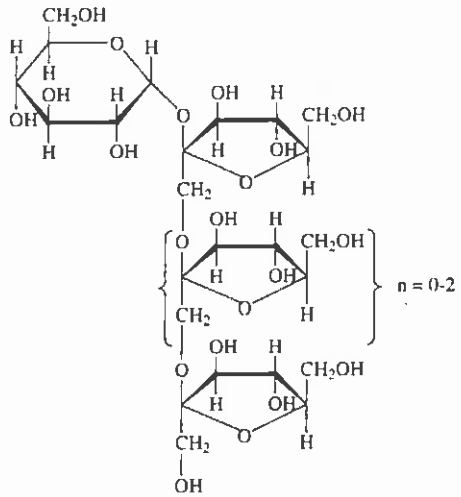
Peroxide Value Determine as directed under *Peroxide Value*, Appendix VII.
Residue on Ignition Determine as directed under *Residue on Ignition*, Appendix IIC.
Saponification Value Determine as directed under *Saponification Value*, Appendix VII.
Unsaponifiable Matter Determine as directed under *Unsaponifiable Matter*, Appendix VII.
Water Determine as directed in *Method Ib* under *Water Determination*, Appendix IIB.

Packaging and Storage Store in well-closed containers.

This is a new monograph.

Fructooligosaccharides, Short Chain

scFOS



DESCRIPTION

Fructooligosaccharides, Short Chain (scFOS), are indigestible carbohydrates synthesized from sucrose and fructose through an enzymatic process or from Inulin by partial enzymatic hydrolysis. These carbohydrates are a mixture of polysaccharides consisting of a sucrose molecule (glucose–fructose disaccharide, GF_1) linked to one (GF_2), two (GF_3), or three (GF_4) additional fructose units added by β2-1 glycosidic linkages to the fructose unit of sucrose for the synthesized scFOS. The scFOS from Inulin consists of oligosaccharides with the same structure but a slightly larger range of polymerization (from GF_2 to GF_9, and from F_3 to F_9). They are very soluble in hot and cold water, and almost insoluble in most organic solvents.

Function Bulking agent; source of dietary fiber; sweetener; **prebiotic.**

REQUIREMENTS

Identification

Acetate Buffer (pH 4.5 ± 0.05) Transfer 22 mL of 0.2 *M* sodium acetate and 28 mL of 0.2 *M* acetic acid into a 100-mL volumetric flask, and dilute to volume with water.

Procedure Transfer 10 mL of *Acetate Buffer* and 10 mL of *Sample Preparation* (see the *Assay* test, below) into a 25-mL volumetric flask. Add 150 units of Fructozyme SP230 enzyme (Novozymes, Denmark), or equivalent. Digest for 30 min at 60°, cool, and dilute to volume with water. Inject 20 µL of the digested solution into the chromatograph, obtain the chromatogram, and calculate the percentage of fructose and glucose as indicated in the *Assay* test. Correct the results for mono- and disaccharide content (from the *Assay* test) and for moisture. scFOS releases >67% fructose and <33% glucose upon enzymatic digestion.

Assay Not less than 85.0% scFOS (≥30.0% trimer, ≥45.0% tetramer, and ≥5.0% pentamer and larger), with the remainder being glucose, fructose, and sucrose, after drying.
Lead Not more than 1 mg/kg.
Residue on Ignition Not more than 0.1%.
Total Solids Not less than 95.0%.

TESTS

Assay

Standard Solution Transfer 300 mg of GF_2, 450 mg of GF_3, and 50 mg of GF_4 scFOS Reference Standards (Wako Pure Chemical Industries, Ltd., Osaka, Japan, or equivalent) and 50 mg each of fructose, glucose, and sucrose, accurately weighed, into a 100-mL volumetric flask, and dissolve in and dilute to volume with water.

Sample Preparation Accurately weigh 1.0 g of sample, previously dried to constant weight, transfer it into a 100-mL volumetric flask, and dilute to volume with water.

Chromatographic System Use a high-performance liquid chromatograph equipped with a 20-µL sample loop, a refractive index detector (sensitivity of 8×10^{-5}), and a 25-cm × 4-mm (id), 5-µm LiChrospher 100 NH2 column (Merck Corp.), or equivalent. Maintain the column temperature at 35°. Use a 65–70%:35–30% acetonitrile:water mixture as mobile phase at a flow rate of 1.0 mL/min and a run time of 12 min.

Procedure Inject 20 µL of the *Standard Solution* into the chromatograph, and record the area responses for each scFOS. Repeat the procedure with the *Sample Preparation*. Calculate the percentage of each scFOS, from trimers to nonamers, in the sample taken, using the formula

$$100\,(C_{ST} \times A_{SA})/(A_{ST} \times W),$$

in which C_{ST} is the concentration, in milligrams per 100 mL, of each scFOS in the *Standard Preparation*; A_{SA} is the area response for the corresponding peak in the *Sample Preparation*; A_{ST} is the area response for the corresponding peak in the *Standard Preparation* (for oligomers without a specific standard, use the average area response of the peaks of the standards); and W is the weight, in grams per 100 mL, of the sample taken. Calculate the total percentage of scFOS in the sample by adding the individual percentages of each scFOS, from trimers to nonamers.

Lead Determine as directed for *Method I* in the *Atomic Absorption Graphite Furnace Spectrophotometric Method* under *Lead Limit Test*, Appendix IIIB.

Residue on Ignition Determine as directed under *Loss on Ignition*, Appendix IIC, igniting a sample at 525° for 2 h.

Total Solids Determine the water content as directed in the *Karl Fischer Titrimetric Method* under *Water Determination*, Appendix IIB, using an accurately weighed sample. Calculate the percent total solids by the formula

$$(W_u - W_w) \times 100/W_u,$$

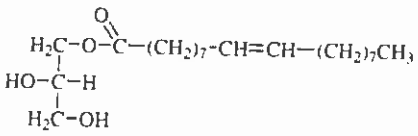
in which W_u is the weight, in milligrams, of the sample taken; and W_w is the weight, in milligrams, of water determined.

Packaging and Storage Store in tightly closed containers in a cool, dry place.

Revision: ***Synonym*** deleted; ***Lead Requirement*** corrected.

Glyceryl Monooleate

Monoolein



$C_{21}H_{40}O_4$ Formula wt 356.54

INS: 471 CAS: [25496-72-4]

FEMA: 2526

DESCRIPTION

Glyceryl Monooleate occurs as a clear liquid at room temperature. It has a mild, fatty taste. It is prepared by esterifying glycerin with food-grade oleic acid in the presence of a suitable catalyst such as aluminum oxide. It also occurs in many animal and vegetable fats such as tallow and cocoa butter. It is soluble in hot alcohol and in chloroform; very slightly soluble in cold alcohol, in ether, and in petroleum ether; and insoluble in water. It melts at around 15°. It may also contain tri- and diesters.

Function Emulsifier; flavoring agent.

REQUIREMENTS

Identification Glyceryl Monooleate exhibits the following typical composition profile of fatty acids determined as directed under *Fatty Acid Composition*, Appendix VII:

Fatty Acid:	≤12	12:0	14:0	16:0	16:1
Weight % (Range):	0	0	<4	1–5	<9
Fatty Acid:	18:0	18:1	18:2	≥20	
Weight % (Range):	<3.0	≥82	3–7	<1.5	

Assay Not less than 35.0% monoglycerides, calculated on the anhydrous basis.

Acid Value Not more than 6.

Free Glycerin Not more than 6.0%.

Hydroxyl Value Between 300 and 330.

Iodine Value Between 58 and 80.

Lead Not more than 1 mg/kg.

Residue on Ignition Not more than 0.1%.

Saponification Value Between 160 and 176.

Water Not more than 1.0%.

TESTS

Assay

Propionating Reagent Mix 10 mL of pyridine with 20 mL of propionic anhydride.

Internal Standard Solution Transfer about 400 mg of hexadecyl hexadecanoate, accurately weighed, into a 100-mL volumetric flask, dilute with chloroform to volume, and mix.

Standard Preparation Transfer about 50 mg of USP Monoglycerides Reference Standard, accurately weighed, into a 25-mL flask, add 5 mL of *Internal Standard Solution* by pipet, and mix. When solution is complete, immerse the flask in a water bath maintained at a temperature between 45° and 50°, and volatilize the chloroform with the aid of a stream of nitrogen. Add 3.0 mL of *Propionating Reagent*, and heat on a hot plate at 75° for 30 min. Evaporate the reagents with the aid of a stream of nitrogen and gentle steam heat. Add 15 mL of chloroform, and swirl to dissolve the residue.

Assay Preparation Transfer about 50 mg of sample, accurately weighed, into a 25-mL conical flask, and proceed as directed for *Standard Preparation*, beginning with "add 5 mL of *Internal Standard Solution*. . . ."

Chromatographic System (See *Chromatography*, Appendix IIA.) Use a gas chromatograph equipped with a flame-ionization detector, and containing a 2.4-m × 4-mm (id) borosilicate glass column, or equivalent, packed with 2% liquid phase, 5% phenyl methyl silicone on 80- to 100-mesh support (Supelcoport, or equivalent). Maintain the column isothermally at a temperature between 270° and 280°, and the injection port and detector block at about 310°. Use helium as the carrier gas at a flow rate of about 70 mL/min.

System Suitability Chromatograph 6 to 10 injections of the *Standard Preparation* as directed under *Procedure*. The resolution factor, R, between the peaks for the derivatized glyceryl hexadecanoate and glyceryl octadecanoate is not less than 2.0, and the relative standard deviation of the ratio of the peak area of the derivatized glyceryl *cis*-9-octadecanoate to that of the hexadecyl hexadecanoate is not more than 2.0%.

Procedure Inject a suitable portion of the *Standard Preparation* into the gas chromatograph, and record the chromatogram. Measure the areas under the peaks, and record the values of the sum of the areas under the derivatized monoglyceride peaks and of the area under the hexadecyl hexadecanoate peak as A_S and A_D, respectively. Calculate the response factor, F, using the formula

$$(A_S/A_D)(W_D/W_S),$$

in which W_D and W_S are the weights, in milligrams, of hexadecyl hexadecanoate and of USP Monoglycerides Reference Standard,