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THIRD SUPPLEMENT TO THE

FOOD CHEMICALS CODEX

SECOND EDITION

(Third Supplement to F.C.C. II)

Subcommittee on Codex Specifications
Committee on Food Protection
Food and Nutrition Board
Division of Biological Sciences
Assembly of Life Sciences
National Research Council

NATIONAL ACADEMY OF SCIENCES
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NOTICE

The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the Councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the Committee responsible for the report were chosen for their special competences and with regard for appropriate balance.

This report has been reviewed by a group other than the authors according to procedures approved by a Report Review Committee consisting of members of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine.

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Compliance with Federal Statutes. The fact that an article appears in this Food Chemicals Codex does not exempt it from compliance with requirements of Acts of Congress or with regulations and rulings issued by agencies of the United States Government under authority of these Acts.

Revisions of the federal requirements that affect the Codex standards will be included in Codex Supplements as promptly as practicable.

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ADDITIONS, CHANGES, AND CORRECTIONS

Additions, changes, and corrections listed in this THIRD SUPPLEMENT constitute revisions in the Food Chemicals Codex, Second Edition (F.C.C. II). Page numbers refer to the F.C.C. II hard-bound volume, unless references to the FIRST SUPPLEMENT, SECOND SUPPLEMENT, or THIS SUPPLEMENT are specifically indicated.

MONOGRAPHS

Alginic Acid, page 26

Change the SPECIFICATION for *Assay*, page 27, to read:

Assay. It yields not less than 20 percent and not more than 23 percent of carbon dioxide (CO_2), corresponding to between 91 and 104.5 percent of alginic acid (equiv. wt. 200.00), calculated on the dried basis.

Change the SPECIFICATION (*Limits of Impurities*) for *Ash*, page 27, to read:

Ash. Not more than 4 percent after drying.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter*, page 27.

Change the paragraph entitled *Ash*, page 27, to read:

Ash. Weigh accurately about 3 grams in a tared crucible, and incinerate at about 650° until free from carbon. Cool the crucible and its contents in a desiccator, weigh, and determine the weight of the ash.

Delete the paragraph entitled *Insoluble matter*, page 27. [NOTE: The revised paragraph on pp. 1–2 of the FIRST SUPPLEMENT should also be deleted.]

Ammonium Alginate, page 43

Change the SPECIFICATION for *Assay* to read:

Assay. It yields not less than 18 percent and not more than 21 percent of carbon dioxide (CO_2), corresponding to between 88.7 and 103.6 percent of ammonium alginate (equiv. wt. 217.00), calculated on the dried basis.

Change the SPECIFICATION (*Limits of Impurities*) for *Ash*, page 44, to read:

Ash. Not more than 4 percent after drying.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter*, page 44.

Change the SPECIFICATION (*Limits of Impurities*) for *Loss on drying*, page 44, to read:

Loss on drying. Not more than 15 percent.

Delete the paragraph entitled *Insoluble matter*, page 44. [NOTE: The revised paragraph on p. 3 of the FIRST SUPPLEMENT should also be deleted.]

Ammonium Chloride, page 4, FIRST SUPPLEMENT

Delete the revision indicated for the *Assay*. [*Note:* The intent of this further revision is to reinstate the original SPECIFICATION for *Assay* (Not less than 99.0 percent of NH_4Cl after drying).]

Ammonium Saccharin, page 4, FIRST SUPPLEMENT

Change the SPECIFICATION (*Limits of Impurities*) for *Toluenesulfonamides* to read:

Toluenesulfonamides. Not more than 25 parts per million (0.0025 percent).

Angelica Seed Oil, page 58

Change the paragraph entitled *Solubility in alcohol*, page 59, to read:

Solubility in alcohol. Proceed as directed in the general method, page 899. One ml. dissolves in 4 ml. of 90 percent alcohol, often with considerable turbidity, and remains in solution on further addition of alcohol to a total of 10 ml.

Butadiene-Styrene 75/25 Rubber, page 102

Change the last two sentences of the *Description* to read:

The solid form is supplied by the manufacturer (prewashed by an equivalent procedure) either in slab form or as a uniform, free-flowing crumb, and may contain a suitable food-grade antioxidant. The crumb form, in addition, may contain a suitable food-grade partitioning agent.

Change the SPECIFICATION (*Limits of impurities*) for *Residual styrene* to read:

Residual styrene. Not more than 20 parts per million (0.002 percent).

Change the paragraph entitled *Residual styrene*, page 103, to read:

Residual styrene. Determine as directed in the revised method for *Residual Styrene*, under *Chewing Gum Base*, page 41 of THIS SUPPLEMENT.

Butadiene-Styrene 50/50 Rubber, page 103

Change the last two sentences of the *Description* to read:

The solid form is supplied by the manufacturer (prewashed by an equivalent procedure) either in slab form or as a uniform, free-flowing crumb, and may contain a suitable food-grade antioxidant. The crumb form, in addition, may contain a suitable food-grade partitioning agent.

Change the SPECIFICATION (*Limits of impurities*) for *Residual sytrene* to read:

Residual styrene. Not more than 30 parts per million (0.003 percent).

Change the paragraph entitled *Residual styrene*, page 104, to read:

Residual styrene. Determine as directed in the revised method for *Residual Styrene*, under *Chewing Gum Base*, page 41 of THIS SUPPLEMENT.

Calcium Alginate, page 117

Change the section entitled IDENTIFICATION, page 118, to read:

IDENTIFICATION

A. Calcium alginate meets the requirements of *Identification Test C* under *Alginic Acid*, page 26.

B. Extract the *Ash* from calcium alginate with diluted hydrochloric acid T.S. and filter. The filtrate gives positive tests for *Calcium*, page 926.

Change the SPECIFICATION for *Assay*, page 118, to read:

Assay. It yields not less than 18 percent and not more than 21 percent of carbon dioxide (CO_2), corresponding to between 89.6 and 104.5 percent of calcium alginate (equiv. wt. 219.00), calculated on the dried basis.

Change the SPECIFICATION for *Ash*, page 118, to read:

Ash. Between 12 and 18 percent after drying.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter*, page 118.

Delete the paragraph entitled *Insoluble matter*, page 118. [NOTE: The revised paragraph on p. 9 of the FIRST SUPPLEMENT should also be deleted.]

Calcium Chloride, page 123

Change the SPECIFICATION (*Limits of Impurities*) for *Magnesium and alkali salts* to read:

Magnesium and alkali salts. Not more than 4 percent.

Change the last sentence of the paragraph entitled *Magnesium and alkali salts*, page 124, to read:

The weight of the residue does not exceed 20 mg.

Calcium Phosphate, Dibasic, page 146

Change the SPECIFICATION for *Assay* to read:

Assay. Not less than 30.0 percent and not more than 31.7 percent of calcium (Ca), calculated on the ignited basis.

Replace the paragraph entitled *Assay* with the following:

Assay. Transfer about 200 mg. of the sample, accurately weighed, to a 250-ml. beaker equipped with a magnetic stirrer, and dissolve it, with the aid of gentle heat if necessary, in a mixture of 5 ml. of hydrochloric acid and 3 ml. of water. Cautiously add 125 ml. of water. With constant stirring, add in the order named 0.5 ml. of triethanolamine, 300 mg. of hydroxy naphthol blue indicator, and (from a 50-ml. buret) about 23 ml. of 0.05 *M* disodium ethylenediaminetetraacetate. Add sodium hydroxide solution (45 in 100) until the initial red color changes to clear blue, then continue to add it dropwise until the color changes to violet, and then add an additional 0.5 ml. The pH should be between 12.3 and 12.5. Continue the titration dropwise with the 0.05 *M* disodium ethylenediaminetetraacetate to the appearance of a clear blue end-point that persists for not less than 60 seconds. Each ml. of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 2.004 mg. of calcium (Ca).

Calcium Phosphate, Monobasic, page 147

Delete the SPECIFICATION, page 148, and the test procedure, page 149, for *Neutralizing value*.

Calcium Phosphate, Tribasic, page 149

Delete *Identification test B*, page 150.

Change the SPECIFICATION for *Assay*, page 150, to read:

Assay. Not less than 34.0 percent and not more than 40.0 percent of calcium (Ca). [NOTE: This revision supercedes the revision in the *Assay* as shown on page 12 of the SECOND SUPPLEMENT.]

Delete the SPECIFICATION and test for *Titration value*, page 150. [NOTE: This revision supercedes the revision in the *Titration value* procedure as shown on page 13 of the SECOND SUPPLEMENT.]

Change the SPECIFICATION (*Limits of Impurities*) for *Fluoride*, page 150, to read:

Fluoride. Not more than 75 parts per million (0.0075 percent).

Replace the paragraph entitled *Assay*, page 150, with the following:

Assay. Proceed as directed in the revised *Assay* under *Calcium Phosphate, Dibasic*, page 4 of THIS SUPPLEMENT, using a 150-mg. sample, accurately weighed. Each ml. of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 2.004 mg. of Ca.

Change the paragraph entitled *Fluoride*, page 151, to read:

Fluoride. Weigh accurately 670 mg., and proceed as directed in the *Fluoride Limit Test*, page 917.

Calcium Pyrophosphate, page 153

Change *Identification test B* to read:

B. Dissolve 100 mg. of the sample in 100 ml. of diluted nitric acid T.S. Add 0.5 ml. of this solution to 30 ml. of quimociac T.S. (see page 43 of the SECOND SUPPLEMENT). A yellow precipitate does not form. Heat the remaining portion of the sample solution for 10 minutes at 95°, and then add 0.5 ml. of the solution to 30 ml. of quimociac T.S. A yellow precipitate forms immediately.

Calcium Saccharin, page 12, FIRST SUPPLEMENT

Change the SPECIFICATION (*Limits of Impurities*) for *Toluenesulfonamides* to read:

Toluenesulfonamides. Not more than 25 parts per million (0.0025 percent).

Calcium Silicate, page 156

Change the SPECIFICATION for *Fluoride* (*Limits of Impurities*) to read:

Fluoride. Not more than 10 parts per million (0.001 percent).

Replace the paragraph entitled *Fluoride*, page 157, with the following new section:

Fluoride. Prepare a slurry consisting of 5 grams of the sample and 45 ml. of 0.1 *N* hydrochloric acid, stir for 15 minutes at room temperature, and filter through a 0.45-micron membrane filter into a 50-ml. volumetric flask. Wash the filter with five 1-ml. portions of 0.1 *N* hydrochloric acid, collecting the washings in the flask, then dilute to volume with 0.1 *N* hydrochloric acid, and mix. Transfer 5.0 ml. of this solution into a 25-ml. volumetric flask, add 5.0 ml. of a 10% solution of Amadac-F* in 60% isopropanol, dilute to volume with water, mix, and allow to stand for 1 hour in diffuse light at room temperature. Determine the absorbance of this solution in a 1-cm. cell with a suitable spectrophotometer, at the wavelength of maximum absorption at about 620 mμ, against a blank consisting of 5.0 ml. of 0.1 *N* hydrochloric acid, 5.0 ml. of the Amadac indicator solution, and 15.0 ml. of water. The absorbance is not greater than that produced by 5.0 ml. of a solution containing 2.21 mcg. of NaF per ml. of 0.1 *N* hydrochloric acid, when treated in the same manner as the sample.

* Amadac-F is a product of Burdick & Jackson Laboratories, Inc., Muskegon, Michigan 494442, consisting of a blended solid mixture of partially hydrated sodium acetate, acetic acid, stabilizers, lanthanum nitrate, and 3-amino-methylalizarin-*N,N*-diacetate (alizarin complexan), the lanthanum and complexan being equimolar.

Calcium Sulfate, page 163 (see also FIRST SUPPLEMENT, page 13)

Change the paragraph entitled *Assay* to read:

Assay. Dissolve 250 mg., accurately weighed, in 100 ml. of water and 4 ml. of diluted hydrochloric acid T.S., boil to effect solution, and cool. While stirring, preferably with a magnetic stirrer, add about 30 ml. of 0.05 *M* disodium ethylenediaminetetraacetate from a 50-ml. buret, then add 25 ml. of sodium hydroxide T.S. and 300 mg. of hydroxy naphthol blue indicator, and continue the titration to a blue end-point. Each ml. of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 6.807 mg. of $CaSO_4$.

Caramel, page 13, SECOND SUPPLEMENT

Change the last sentence of the paragraph entitled *Mercury*, page 15, to read: Any absorbance produced by the *Sample preparation* is not more than half that produced by the *Standard preparation*, indicating not more than 0.1 part per million of Hg in the sample taken.

Carmine, page 18, SECOND SUPPLEMENT

Change the paragraph entitled *Carminic acid*, page 19 of the supplement, to read:

Carminic acid. Weigh accurately 100 mg. of carmine and dissolve in 30 ml. of boiling 2 *N* hydrochloric acid, and cool. Transfer quantitatively to a 1000-ml. volumetric flask, dilute to volume with water, and mix. Determine the absorbance of the solution in a 1-cm. cell at the wavelength of maximum absorbance at about 494 mμ with a suitable spectrophotometer, using 3 ml. of 2 *N* hydrochloric acid per 100 ml. aqueous solution as the blank. Calculate the quantity, in mg., of carminic acid in the carmine taken for analysis by the formula (1)(100)(A/1.39), in which 1 is one liter, 100 is one hundred mg. per liter, A is the absorbance of the sample solution, and 1.39 is the absorbance of a solution of carminic acid having a concentration of 100 mg. per liter.

Carrageenan

Replace the entire monograph for *Carrageenan*, pages 172–74, with the following:

CARRAGEENAN

DESCRIPTION

Carrageenan is obtained by extraction with water or aqueous alkali from certain members of the class Rhodophyceae (red seaweeds). It is a hydrocolloid consisting mainly of the potassium, sodium, magnesium, calcium, and ammonium sulfate esters of galactose and 3,6-anhydrogalactose copolymers. These hexoses are alternately linked α-1,3 and β-1,4 in the polymer. The relative proportion of cations existing in carrageenan may be changed during processing to the extent that one may become predominant.

The prevalent copolymers in the hydrocolloid are designated *kappa-*, *iota-*, and *lambda*-carrageenan. *Kappa*-carrageenan is mostly the alternating polymer of D-galactose-4-sulfate and 3,6-anhydro-D-galactose; *iota*-carrageenan is similar, except that the 3,6-anhydrogalactose is sulfated at carbon 2. Between *kappa*-carrageenan and *iota*-carrageenan, there is a continuum of intermediate compositions differing in degree of sulfation at carbon 2. In *lambda*-carrageenan, the alternating monomeric units are mostly D-galactose-2-sulfate (1,3-linked) and D-galactose-2,6-disulfate (1,4-linked).

The ester sulfate content of carrageenan ranges from 18% to 40% (see SPECIFICATIONS). In addition, it contains inorganic salts that originate from the seaweed and the process of recovery from the extract. Carrageenan is recovered by alcohol precipitation, by drum drying, or by freezing. The alcohols used during recovery and purification are restricted to methanol, ethanol, and isopropanol. When carrageenan is recovered by drum roll drying, it may contain mono- and diglycerides or up to 5% polysorbate 80 used as roll-stripping agents.

Carrageenan is a yellowish or tan to white, coarse to fine powder that is practically odorless and has a mucilaginous taste. It is soluble in water at a temperature of about 80°, forming a viscous, clear or slightly opalescent solution that flows readily. It disperses in water more readily if first moistened with alcohol, glycerin, or a saturated solution of sucrose in water.

IDENTIFICATION

A. Add 4 grams of sample to 200 ml. of water, and heat the mixture in a water bath at 80°, with constant stirring, until dissolved. Replace any water lost by evaporation, and allow the solution to cool to room temperature. It becomes viscous and may form a gel.

B. To 50 ml. of the solution or gel obtained in *Identification test A* add 200 mg. of potassium chloride, then reheat, mix well, and cool. A short-textured ("brittle") gel indicates a carrageenan of a predominantly *kappa* type; a compliant ("elastic") gel indicates a predominantly *iota* type. If the solution does not gel, the carrageenan is of a predominantly *lambda* type.

C. To 5 ml. of the solution obtained in *Identification test A* add 1 drop of a 1 in 100 solution of methylene blue. A fibrous precipitate forms.

D. Obtain infrared absorption spectra on the gelling and non-gelling fractions of the sample by the following procedure: Disperse 2 grams of the sample in 200 ml. of 2.5% potassium chloride solution, and stir for 1 hour. Let stand overnight, stir again for 1 hour, and transfer into a centrifuge tube. (If the transfer cannot be made because the dispersion is too viscous, dilute with up to 200 ml. of the potassium chloride solution.) Centrifuge for 15 minutes at approximately 1000 *g*'s.

Remove the clear supernatant, resuspend the residue in 200 ml. of 2.5% potassium chloride solution, and centrifuge again. Coagulate the combined supernatants by adding 2 volumes of 85% ethanol or isopropanol. (*Note:* Retain the sediment for use as directed below.) Recover the coagulum, and wash it with 250 ml. of the alcohol. Press the excess liquid from the coagulum, and dry it at 60° for 2 hours. The product obtained is the non-gelling fraction (*lambda* carrageenan).

Disperse the sediment (retained above) in 250 ml. of cold water, heat at 90° for 10 minutes, and cool to 60°. Coagulate the mixture, and then recover, wash, and dry the coagulum as described above. The product obtained is the gelling fraction (*kappa* and *iota* carrageenan).

Prepare a 0.2% aqueous solution of each fraction, cast films 0.0005-cm. thick (when dry) on a suitable non-sticking surface such as Teflon, and obtain the infrared absorption spectrum of each film. (Alternatively, the spectra may be obtained on potassium bromide pellets if care is taken to avoid moisture.)

Carrageenan has strong, broad absorption bands, typical of all polysaccharides, in the 1000–1100 $cm.^{-1}$ region. Absorption maxima are 1065 and 1020 $cm.^{-1}$ for gelling and non-gelling types, respectively. Other characteristic absorption bands and their intensities relative to the absorbance at 1050 $cm.^{-1}$ are as follows:

Wave Number cm^{-1}	Molecular Assignment	Absorbance relative to 1050 cm^{-1}		
		Kappa	Iota	Lambda
1220–1260	Ester sulfate	0.7–1.2	1.2–1.6	1.4–2.0
928– 933	3,6 anhydrogalactose	0.3–0.6	0.2–0.4	0–0.2
840– 850	Galactose-4-sulfate	0.3–0.5	0.2–0.4	–
825– 830	Galactose-2-sulfate	–	–	0.2–0.4
810– 820	Galactose-6-sulfate	–	–	0.1–0.3
800– 805	3,6 anhydrogalactose-2-sulfate	0–0.2	0.2–0.4	–

SPECIFICATIONS

Ash (Total). Not more than 35.0 percent.

Ash (Acid-insoluble). Not more than 1.0 percent.

Loss on drying. Not more than 12 percent.

Sulfate. Between 18 percent and 40 percent on the dry weight basis.

Viscosity of a 1.5 percent solution. Not less than 5 centipoises at 75°.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 40 parts per million (0.004 percent).

Lead. Not more than 10 parts per million (0.001 percent).

TESTS

Ash (Total). Transfer about 2 grams, accurately weighed, into a previously ignited, tared, silica or platinum crucible. Heat the sample with a suitable infrared heat lamp, increasing the intensity gradually, until it is completely charred, and then continue for an additional 30 minutes. Transfer the crucible and charred sample into a muffle furnace and ignite at about 550° for 1 hour, then cool in a desiccator and weigh. Repeat the ignition in the muffle furnace until a constant weight is attained. If a carbon-free ash is not obtained after the first ignition, moisten the charred spots with a 1 in 10 solution of ammonium nitrate and dry under an infrared heat lamp before reigniting.

Ash (Acid-insoluble). Proceed as directed in the general method, page 869.

Loss on drying, page 931. Dry at 105° for 4 hours.

Sulfate. Transfer about 500 mg., previously dried at 105° for 12 hours and accurately weighed, into a 100-ml. Kjeldahl flask. Add 10 ml. of nitric acid and heat gently for 30 minutes, adding more of the acid, if necessary, to prevent evaporation to dryness, and to yield a volume of about 3 ml. at the end of the heating. Cool the mixture to room temperature and decompose the excess nitric acid by the addition of formaldehyde T.S., dropwise, heating, if necessary, until no brown fumes continue to be evolved. Continue the heating until the volume of the reaction mixture is reduced to about 5 ml., then cool, and transfer the

residue quantitatively with the aid of water into a 400-ml. beaker, dilute it to about 100 ml., and filter, if necessary, to produce a clear solution. Dilute the solution to about 200 ml., and add 1 ml of hydrochloric acid. Heat to boiling and add, dropwise, with constant stirring, an excess (about 6 ml.) of hot barium chloride T.S. Heat the mixture for 1 hour on a steam bath, collect the precipitate of barium sulfate on a filter, wash it until free from chloride, dry, ignite, and weigh. The weight of the barium sulfate so obtained, multiplied by 0.4116, gives the equivalent of sulfate (SO_4).

Viscosity of a 1.5 percent solution. Transfer 7.5 grams of the sample into a tared, 600-ml. tall-form (Berzelius) beaker, and disperse with agitation for 10–20 minutes in 450 ml. of deionized water. Add sufficient water to bring the final weight to 500 grams, and heat in a water bath, with continuous agitation, until a temperature of 80° is reached (20–30 minutes). Add water to adjust for loss by evaporation, cool to 76–77°, and place in a constant-temperature bath at 75°. Pre-heat the bob and guard of a Brookfield LVF or LVT viscometer to approximately 75° in water, then dry the bob and guard and attach them to the viscometer, which should be equipped with a No. 1 spindle (19 mm. in diameter, approx. 65 mm. in length) and capable of rotating at 30 rpm. Adjust the height of the bob in the sample solution, start the viscometer rotating at 30 rpm, and, after six complete revolutions, take the reading on the 0–100 scale. Record the results in centipoises by multiplying the reading by 2. [NOTE: Some samples of carrageenan may be too viscous to be read when a No. 1 spindle is used. Such samples obviously pass the specification, but if a viscosity reading is desired for other reasons, use a No. 2 spindle, take the reading on the 0–100 scale, and multiply the reading by 10 to obtain the viscosity in centipoises, or read on the 0–500 scale and multiply by 2. If the viscosity is very low, increased precision may be obtained by using the Brookfield UL (ultra low) adapter, in which case the viscometer reading on the 0–100 scale should be multiplied by 0.2 to obtain the viscosity in centipoises.]

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Lead. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Lead Limit Test*, page 929, using 10 mcg. of lead ion (Pb) in the control.

Packaging and storage. Store in well-closed containers.

Functional use in foods. Emulsifier; stabilizer; thickener; gelling agent.

Cellulose, Powdered

Replace the entire monograph for *Cellulose, Powdered*, pages 21–22 of the SECOND SUPPLEMENT, with the following:

CELLULOSE, POWDERED

DESCRIPTION

Powdered cellulose is purified, mechanically disintegrated cellulose prepared by processing bleached cellulose obtained as a pulp from such fibrous materials as wood or cotton. It occurs as a white, odorless substance and consists of fibrous particles that may be compressed into self-binding tablets that disintegrate rapidly in water. It exists in various grades, exhibiting degrees of fineness ranging from a dense, free-flowing powder to a coarse, fluffy, non-flowing material. It is insoluble in water, in dilute acids, and in nearly all organic solvents. It is slightly soluble in sodium hydroxide T.S.

IDENTIFICATION

A. Mix approximately 30 grams of the sample with 270 ml. of water in a high-speed (approx. 12,000 r.p.m.) power blender for 5 minutes. The mixture will be either a free-flowing suspension or a heavy, lumpy suspension which flows poorly (if at all), settles only slightly, and contains many trapped air bubbles. The mixture is not slimy. If a free-flowing suspension is obtained, transfer 100 ml. of it into a 100-ml. graduate, and allow to settle for 1 hour: the solids settle in the cylinder and a supernatant liquid appears above the layer of the cellulose.

B. To 5 ml. of the clear supernatant liquid from test *A*, add a few drops of iodine T.S. and mix. No purplish to blue or blue color is produced.

C. To 20 ml. of a 0.1% solution of anthrone in 75% sulfuric acid add from 2 to 5 mg. of the sample, and heat on a steam bath. The solution turns blue-green within 5 minutes.

D. Place a few drops of the mixture from test *A* on a microscope slide and insert a coverglass. Observe at 100 magnifications with a microscope. Fibers and fiber fragments are visible, regardless of the degree of fineness of the sample.

E. Dilute 10 ml. of the mixture from test *A* to 1000 ml. with water, and filter 125 ml. of the dilution through a Buchner funnel. Rinse the pad with 25 ml. of acetone, and dry (paper included) at 105°. Transfer the powder to a tared weighing bottle, weigh, then transfer to a 50-ml. Erlenmeyer flask and seal with a rubber stopper. Record the weight of the sample as w, in mg. Dissolve the sample in 0.167 *M* and 1.0 *M* solutions of cupriethylenediamine (CED), the volumes of which are determined as follows: $0.12 \times w$ = ml. of 0.167 *M* CED, and $0.08 \times w$ = ml. of 1.0 *M* CED. Add a few 3-mm. glass beads and the calculated volume of 0.167 *M* CED, blow nitrogen over the surface of the solution, and shake for 2 minutes. Add the calculated volume of 1.0 *M* CED, again introduce the nitrogen, and shake vigorously for at least 3 minutes. A dark blue solution, clear under microscopic examination, is produced.

SPECIFICATIONS

Assay. Not less than 97.0 percent and not more than the equivalent of 102.0 percent of carbohydrate, calculated as cellulose.

Loss on drying. Not more than 7 percent.

pH. Between 5.0 and 7.5.

Limits of Impurities

Arsenic (as As). Not more than 1 part per million (0.0001 percent).

Ash (total). Not more than 0.3 percent.

Chloride. Not more than 0.05 percent.

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

Sulfur (total). Not more than 0.01 percent.

Water-soluble substances. Not more than 1.5 percent.

TESTS

Assay. Transfer about 125 mg. of the sample, accurately weighed, to a 300-ml. Erlenmeyer flask, using about 25 ml. of water. Add 50.0 ml. of 0.5 N potassium dichromate, mix, then carefully add 100 ml. of sulfuric acid, and heat to boiling. Remove from heat, allow to stand at room temperature for 15 minutes, then cool in a water bath, and transfer the solution to a 250-ml. volumetric flask. Dilute with water almost to volume, cool to 25°, dilute to volume with water, and mix. Titrate a 50-ml. aliquot with 0.1 N ferrous ammonium sulfate, using 2 or 3 drops of orthophenanthroline T.S. (see page 43 of THIS SUPPLEMENT). Perform a blank determination, and calculate the normality, N, of the ferrous ammonium sulfate solution by the formula $(0.1 \times 50)/B$, in which B is the volume, in ml., of ferrous ammonium sulfate solution required in the blank titration. Calculate the percent of cellulose in the sample by the formula $6.75(B - S) \times N/2W$, in which S is the volume, in ml., of ferrous ammonium sulfate solution used in the sample titration, and W is the weight of the sample taken, in grams, corrected for moisture content (see *Loss on drying*).

Loss on drying, page 931. Dry to constant weight at 105°.

pH. Mix 10 grams of the sample with 90 ml. of water, allow to stand with occasional stirring for 1 hour, and determine the pH of the supernatant liquid by the *Potentiometric Method*, page 941.

Arsenic. A *Sample Solution* prepared from a 3-gram sample as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Ash (total). Heat 3 grams at 550 ± 50° until completely charred, then ignite at 800 ± 25° until free from carbon, cool in a desiccator, and weigh.

Chloride. Transfer about 5 grams of the sample, accurately weighed, to a 500-ml. conical flask, add 250 ml. of water, and reflux the mixture for 1 hour. Filter through paper and again reflux the sample with 200 ml. of water for 30 minutes. Filter and combine the filtrates and hot water rinses. Add 1 ml. of nitric acid, heat to boiling, and slowly add 5 ml. of a 5% solution of silver nitrate. After the precipitate has coagulated, cool and filter through a Gooch crucible. Wash with nitric acid solution (1 in 100) until free from silver nitrate, then rinse with water, dry at 130°, and weigh. Perform a blank determination to obtain the corrected weight of the sample precipitate, each mg. of which is equivalent to 0.247 mg. of chloride.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Sulfur (total). Transfer about 5 grams of the sample, previously dried at 105° to constant weight and accurately weighed, to a 300-ml. conical flask, and add 50 ml. of a 2:3 mixture, v/v, of perchloric acid and nitric acid. Heat on a hot plate under a hood, and boil until all organic matter has been destroyed and copious fumes of perchloric acid are evolved. If the organic matter chars and cannot be destroyed quickly by further heating for a short time, add 10 to 20 ml. of the acid mixture and continue the treatment until a clear, syrupy residue is obtained. (NOTE: It is absolutely necessary that all of the nitric acid be driven from the flask, as it will form a double salt with the barium sulfate formed later). Allow the mixture to cool for a few minutes, then add 200 ml. of hot water, and heat again to boiling. (If the solution is cloudy, filter and rinse the filter with a small amount of hot water before boiling.) As soon as the mixture is boiling gently, carefully run in 20 ml. of barium chloride T.S., boil for a few minutes longer, and allow to stand for at least 12 hours on a steam bath. Filter any barium sulfate onto an ashless filter paper, and rinse with five portions of boiling water to remove traces of perchloric acid. Place the paper in a tared platinum dish, dry in an oven at 105°, and ignite at 800 ± 25° for 1 hour. Perform a blank determination to obtain the corrected weight of the sample precipitate, each mg. of which is equivalent to 0.137 mg. of sulfur.

Water-soluble substances. Mix 6 grams of the sample with 90 ml. of recently boiled and cooled water, and allow to stand with occasional stirring for 10 minutes. Filter, discard the first 10 ml. of filtrate, and pass the filtrate through the same filter a second time, if necessary, to obtain a clear filtrate. Evaporate a 15-ml. portion of the filtrate to dryness in a tared evaporating dish on a steam bath, dry at 105° for 1 hour, cool in a desiccator, and weigh.

Packaging and storage. Store in well-closed containers.

Functional use in foods. Anticaking agent; binding agent; bulking agent; disintegrating agent; dispersing agent; filtering aid; texturizing agent; thickening agent.

Citric Acid, page 204

Insert the following new SPECIFICATION (*Limits of Impurities*) for *Tridodecyl amine:*

Tridodecyl amine. Not more than 0.1 part per million (0.00001 percent).

Insert the following new test procedure following the paragraph entitled *Residue on ignition*, page 205:

Tridodecyl amine

Buffered Indicator Solution. Prepare a mixture consisting of 700 ml. of 0.1

M citric acid (anhydrous, reagent grade), 200 ml. of 0.2 *M* disodium phosphate, and 50 ml. each of 0.2 percent bromophenol blue and of 0.2 percent bromocresol green in spectro-grade methanol.

No-Indicator Buffer Solution. Prepare a mixture consisting of 700 ml. of 0.1 *M* citric acid (anhydrous, reagent grade), 200 ml. of 0.2 *M* disodium phosphate, and 100 ml. of spectro-grade methanol.

Amine Stock Solution. Transfer between 40 and 45 mg. of tridodecyl (trilauryl) amine, accurately weighed, into a 500-ml. volumetric flask, dilute to volume with isopropyl alcohol, and mix. Discard after three weeks.

Standard Amine Solution. Using a graduated 5-ml. pipet, transfer into a 100-ml. volumetric flask an amount of *Amine Stock Solution* equivalent to 400 mcg. of tridodecyl amine, dilute to volume with isopropyl alcohol, and mix. Prepare this solution fresh on the day of use.

Procedure. Dissolve 160 grams of anhydrous reagent-grade citric acid (not the sample to be tested) in 320 ml. of water, and divide the solution equally between two 250-ml. separators, S_1 and S_2. To S_1 add 5 ml. of *No-Indicator Buffer Solution.* To S_2 add 2.0 ml. of *Standard Amine Solution* and 5 ml. of *Buffered Indicator Solution.*

To prepare solutions to the sample being tested, dissolve 160 grams of anhydrous citric acid sample in 320 ml. of water (or 174 grams of citric acid monohydrate sample in 306 ml. of water). Divide the test solution equally between two 250-ml. separators, S_3 and S_4. Add 5 ml. of *No-Indicator Buffer Solution* to S_3, and 5 ml. of *Buffered Indicator Solution* to S_4.

To each of the four separators add 20 ml. of a 1:1 mixture (v/v) prepared from spectro-grade chloroform and *n*-heptane, shake for 15 minutes on a mechanical shaker, and allow the phases to separate for 45 minutes. Drain all except the last few drops of the lower (aqueous) phases, and discard. Hand-shake the organic phases with 25 ml. each of 0.05 *N* sulfuric acid for 30 seconds, and allow the phases to separate for 30 minutes. Drain all except the last few drops of the lower (organic) phases through dry Whatman No. 40 (or equivalent) paper, and collect the aqueous filtrates in separate small glass-stoppered containers.

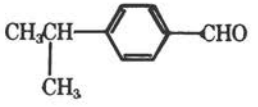
Determine the absorbance of each solution in a 5-cm. cell at 400 mμ, with a suitable spectrophotometer standardized prior to analysis, against chloroform-heptane (1:1 v/v). The net absorbance of the sample ($S_4 - S_3$) is not greater than that of the standard ($S_2 - S_1$).

Cuminic Aldehyde

Insert the following new monograph to precede the monograph entitled *Cumin Oil*, page 224:

CUMINIC ALDEHYDE

p-Cuminic Aldehyde; Cumaldehyde; *p*-Isopropylbenzaldehyde; Cuminal



$C_{10}H_{12}O$ Mol. wt. 148.21

DESCRIPTION

A colorless to pale yellow liquid having the strong, pungent odor of cumin oil. It is soluble in alcohol and in ether but is practically insoluble in water.

SPECIFICATIONS

Assay. Not less than 95.0 percent of $C_{10}H_{12}O$.

Acid value. Not more than 5.0.

Refractive index. Between 1.529 and 1.534 at 20°.

Solubility in alcohol. Passes test.

Specific gravity. Between 0.976 and 0.980.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Halogens. Passes test.

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Assay. Weigh accurately about 1 gram of the sample, and proceed as directed under *Aldehydes and Ketones—Hydroxylamine/Tert-Butyl Alcohol Method*, page 43 of THIS SUPPLEMENT, using 74.11 as the equivalence factor (*E*) in the calculation. Allow the mixture to stand at room temperature for 1 hour before titrating.

Acid value. Determine as directed in the general procedure, page 893.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Solubility in alcohol. Proceed as directed in the general method, page 899. One ml. dissolves in 4 ml. of 70 percent alcohol.

Specific gravity. Determine by any reliable method (see page 5).

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Halogens. Proceed as directed in the test for *Chlorinated Compounds*, page 895.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

Diatomaceous Silica, page 242

Insert, beneath the title of the monograph, the following synonyms:

Diatomite; Diatomaceous Earth (D.E.)

Delete the last sentence of the *Description*.

Insert the following section on *Identification* to precede the section on *Specifications:*

IDENTIFICATION

When examined with a 100- to 200-power microscope, typical diatom shapes are observed.

Enzyme Preparations, page 18, FIRST SUPPLEMENT

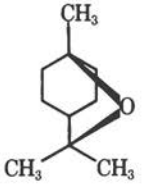
Delete the SPECIFICATION (*Limits of Impurities*), page 23, and the test procedure, page 24, for *Pseudomonas aeruginosa*.

Eucalyptol

Insert the following new monograph to precede the monograph entitled *Eucalyptus Oil*, page 305:

EUCALYPTOL

1:8 cineol; anhydride of menthane 1:8 diole; 1:8 oxido-*p*-menthane; 1:8 epoxy-*p*-menthane



$C_{10}H_{18}O$ Mol. wt. 154.25

DESCRIPTION

Eucalyptol is obtained from several varieties of eucalyptus oil and other sources. It can be separated from essential oils by freezing or by a combination of distilling and freezing. It is a colorless liquid having a characteristic odor and a pungent, cooling taste. It is soluble in all proportions in benzyl benzoate, diethyl phthalate, glycerin, mineral oil, propylene glycol, and in most fixed oils.

SPECIFICATIONS

Angular rotation. Between −0.5° and +0.5°.

Refractive index. Between 1.455 and 1.460 at 20°.

Solidification point. Not lower than 0°.

Solubility in alcohol. Passes test.

Specific gravity. Between 0.921 and 0.924.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Angular rotation. Determine in a 100-mm. tube as directed under *Optical rotation*, page 939.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Solidification point. Determine as directed in the general method, page 945.

Solubility in alcohol. Proceed as directed in the general method, page 899. One ml. dissolves in 5 ml. of 60 percent alcohol.

Specific gravity. Determine by any reliable method (see page 5).

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

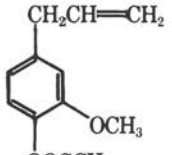
Functional use in foods. Flavoring agent.

Eugenyl Acetate

Insert the following new monograph to precede the monograph entitled *Fennel Oil*, page 307:

EUGENYL ACETATE

4-Allyl-2-methoxyphenyl Acetate; Eugenol Acetate; Acetyl Eugenol; Aceteugenol



$C_{12}H_{14}O_3$ Mol. wt. 206.24

DESCRIPTION

A fused solid melting at warm room temperature to a pale yellow liquid. It has a mild odor resembling that of clove. It is soluble in alcohol and in ether but is practically insoluble in water.

SPECIFICATIONS

Assay. Not less than 98.0 percent of $C_{12}H_{14}O_3$.

Acid value. Not more than 1.0.

Solidification point. Not lower than 25°.

Solubility in alcohol. Passes test.

Specific gravity. Between 1.077 and 1.082.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Assay. Weigh accurately about 1.7 grams, and proceed as directed under *Ester Determination* (High Boiling Solvent), page 896, using 103.12 as the equivalent factor (*e*) in the calculation.

Acid value. Determine as directed in the general procedure, page 893.

Solidification point. Determine as directed in the general method, page 954.

Solubility in alcohol. Proceed as directed in the general method, page 899. One ml. dissolves in 5 ml. of 70 percent alcohol.

Specific gravity. Determine by any reliable method (see page 5), using a sample that has been melted and then supercooled.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light. Do not use iron containers.

Functional use in foods. Flavoring agent.

Ferric Pyrophosphate, page 312

In the paragraph entitled *Arsenic*, page 313, change the page reference (to the *Ferric Phosphate* monograph) to read:

. . . page 310.

Food Starch, Modified, page 324

On page 327, under the section entitled *Monofunctional and/or polyfunctional etherification*, and further under the subsection entitled *Treatment to produce hydroxypropyl distarch glycerol*, replace the statement, "Epichlorohydrin, not to exceed 0.1%, combined with propylene oxide, not to exceed 10%" with the following:

Epichlorohydrin, not to exceed 0.1%, and propylene oxide, not to exceed 10%, added in combination or in any sequence.

Gibberellic Acid, page 343

Change the SPECIFICATION (*Limits of Impurities*) for *Loss of drying*, page 344, to read:

Loss on drying. Not more than 3 percent.

Glycerin, page 350

Delete the SPECIFICATION (*Limits of Impurities*) for *Butanetriols*.

Delete the section entitled *Butanetriols*, beginning on page 352.

Iron, Electrolytic, page 393

In the SPECIFICATION (*Limits of Impurities*) for *Arsenic,* change "(0.004 percent)" to read:

. . . (0.0004 percent).

Isobutyric Acid, page 411

In the second line of the paragraph entitled *Reducing substances,* change ". . . 50 ml. of sulfuric acid . . ." to read:

. . . 5 ml. of sulfuric acid . . .

Kaolin, page 421

Insert, beneath the title of the monograph, the following synonym:

China Clay

Lactic Acid, page 428

Insert the following new SPECIFICATION (*Limits of Impurities*) for *Cyanide:*

Cyanide. Passes test (approx. 5 parts per million).

Insert the following new section to precede the paragraph entitled *Heavy Metals,* page 429:

Cyanide

p-Phenylenediamine-Pyridine Mixed Reagent. Dissolve 200 mg. of *p*-phenylenediamine hydrochloride in 100 ml. of water, warming to effect solution. Cool, allow the solids to settle, and use the supernatant liquid to make the mixed reagent. Dissolve 128 ml. of pyridine in 365 ml. of water, add 10 ml. of hydrochloric acid, and mix. To prepare the mixed reagent, mix 30 ml. of the *p*-phenylenediamine solution with all of the pyridine solution, and allow to stand for 24 hours before using. The mixed reagent is stable for about three weeks when stored in an amber bottle.

Sample Solution. Transfer an accurately weighed quantity of the sample, equivalent to 20.0 grams of 100% lactic acid, into a 100-ml. volumetric flask, dilute to volume with water, and mix.

Cyanide Standard Solution. Dissolve 2.5 grams of potassium cyanide in 1000 ml. of 0.1 *N* sodium hydroxide. Transfer a 1-ml. aliquot into a 100-ml. volu-

metric flask, dilute to volume with 0.1 *N* sodium hydroxide, and mix. Each ml. of this solution contains 10 mcg. of CN.

Procedure. Pipet a 10-ml. aliquot of the *Sample Solution* into a 50-ml. beaker. Into a second 50-ml. beaker pipet 1.0 ml. of the *Cyanide Standard Solution*, and add 10 ml. of water. Place the beakers in an ice bath, and adjust the pH to between 9 and 10 with 20% sodium hydroxide, stirring slowly and adding the reagent slowly to avoid overheating. Allow the solutions to stand for 3 minutes, and then slowly add 10% phosphoric acid to a pH between 5 and 6. Transfer the solutions into 100-ml. separators containing 25 ml. of cold water, and rinse the beakers and pH meter electrodes with a few ml. of water, collecting the washings in the respective separator. Add 2 ml. of bromine T.S., stopper, and mix. Add 2 ml. of 2% sodium arsenite solution, stopper, and mix. To the clear solutions add 10 ml. of *n*-butanol, stopper, and mix. Finally, add 5 ml. of *p-Phenylenediamine-Pyridine Mixed Reagent*, mix, and allow to stand for 15 minutes. Remove and discard the aqueous phases, and filter the alcohol phases into 10-mm. cells. The absorbance of the sample, determined at 480 mμ with a suitable spectrophotometer, is no greater than that of the standard.

Lemon Oil, Distilled

Insert the following new monograph to precede the monograph entitled DL-*Leucine*, page 450:

LEMON OIL, DISTILLED

DESCRIPTION

The volatile oil obtained by distillation from the fresh peel or juice of the fruit of *Citrus limon* L. Burmann filius (Fam. *Rutaceae*), with or without the previous separation of the juice, pulp, and peel. It is a colorless to pale yellow liquid having the characteristic odor of fresh lemon peel. It is soluble in most fixed oils, in mineral oil, and in alcohol (with haze). It is insoluble in glycerin and in propylene glycol.

SPECIFICATIONS

Aldehydes. Between 1.0 percent and 3.5 percent of aldehydes, calculated as citral ($C_{10}H_{16}O$).

Angular rotation. Between +55° and +75°.

Refractive index. Between 1.470 and 1.475 at 20°.

Specific gravity. Between 0.842 and 0.856.

Ultraviolet absorbance. Not more than 0.01.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).
Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Aldehydes. Weigh accurately about 5 ml. of the sample, and proceed as directed under *Aldehydes and Ketones—Hydroxylamine/Tert-Butyl Alcohol Method,* page 43 of THIS SUPPLEMENT, using 76.12 as the equivalence factor (*E*) in the calculation. Allow the mixture to stand at room temperature for 1 hour before titrating.

Angular rotation. Determine in a 100-mm. tube as directed under *Optical Rotation,* page 939.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 5).

Ultraviolet absorbance. Proceed as directed under *Ultraviolet Absorbance of Citrus Oils,* page 900, using about 250 mg. of sample, accurately weighed. The maximum absorbance occurs at 315 ± 5 mμ.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test,* page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test,* page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

L-Lysine Monohydrochloride, page 467

Change the paragraph entitled *Assay,* page 468, to read:

Assay. Transfer about 150 mg., accurately weighed, into a 150-ml. beaker, and dissolve in 8 ml. of mercuric acid T.S., heating on a steam bath to effect solution. Cool, add 100 ml. of glacial acetic acid, and titrate with 0.1 *N* perchloric acid, determining the end-point potentiometrically. Each ml. of 0.1 *N* perchloric acid is equivalent to 9.133 mg. of $C_6H_{14}N_2O_2.HCl$.

Magnesium Phosphate, Tribasic, page 477

Delete the SPECIFICATION, page 477, and the test procedure, page 478, for *Titration value.*

Magnesium Silicate, page 479

Insert, beneath the title of the monograph, the following synonym:

Synthetic Magnesium Silicate

Change the *Description* to read:

DESCRIPTION

A synthetic, usually amorphous, form of magnesium silicate in which the molar ratio of magnesium oxide to silicon dioxide is approximately 2:5. It occurs as a very fine, white, odorless, tasteless powder, free from grittiness. It is insoluble in water and in alcohol, but is readily decomposed by mineral acids. The pH of a 1 in 10 slurry is between 7.0 and 10.8.

Change the SPECIFICATION for *Fluoride* (*Limits of Impurities*) to read:

Fluoride. Not more than 10 parts per million (0.001 percent).

Change the SPECIFICATION for *Soluble salts* (*Limits of Impurities*) to read:

Soluble salts. Not more than 3 percent.

Delete the SPECIFICATION for *Water* (*Limits of Impurities*) and replace it with the following two new specifications:

Loss on drying. Not more than 15 percent.

Loss on ignition. Not more than 15 percent, determined on a dried sample.

Replace the paragraph entitled *Fluoride,* page 480, with the following:

Fluoride. Determine as directed in the revised test for *Fluoride* under *Calcium Silicate,* page 6 of THIS SUPPLEMENT.

Delete the test procedure for *Water,* page 480, and replace it with the following new tests:

Loss on drying, page 931. Dry at 105° for 2 hours. Retain the sample for determination of *Loss on ignition.*

Loss on ignition. Ignite the sample, retained from the test for *Loss on drying,* at 900°–1000° for 20 minutes.

Malic Acid, page 484

Add the following sentence at the end of the paragraph entitled *Description:*

It melts at about 130°.

Delete the SPECIFICATION, page 484, and test procedure, page 485, for *Melting range*.

Manganese Gluconate, page 491

Change the line formula (shown beneath the title of the monograph) to indicate "$2H_2O$" (instead of $3H_2O$), i.e.:

$$[CH_2OH(CHOH)_4COO]_2Mn.2H_2O$$

Marjoram Oil, Sweet

Insert the following new monograph to precede the monograph entitled *Masticatory Substances, Natural,* page 501:

MARJORAM OIL, SWEET

DESCRIPTION

The volatile oil obtained by steam distillation of the dried herb of the marjoram shrub, *Marjoram hortensis* L. (Fam. *Labiatae*), which is cultivated in Germany, Hungary, France, Tunis, and to a small extent in the United States. It is a yellow or greenish yellow oil having a spicy or cardamon note. It is soluble in most fixed oils and in mineral oil (with turbidity). It is only partly soluble in propylene glycol and is insoluble in glycerin.

SPECIFICATIONS

Acid value. Not more than 2.5.

Angular rotation. Between +14° and +24°.

Refractive index. Between 1.470 and 1.475 at 20°.

Saponification value. Between 23 and 40.

Saponification value after acetylation. Between 68 and 86.

Solubility in alcohol. Passes test.

Specific gravity. Between 0.890 and 0.906.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Acid value. Determine as directed in the general method, page 893.

Angular rotation. Determine in a 100-mm. tube as directed under *Optical Rotation*, page 939.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Saponification value. Determine as directed in the general method, page 896, using about 5 grams of sample, accurately weighed.

Saponification value after acetylation. Proceed as directed under *Total Alcohols*, page 893, using about 2.5 grams of acetylated oil, accurately weighed. Calculate the saponification value by the formula $(28.05 \times A)/B$, in which A is the number of ml. of 0.5 N alcoholic potassium hydroxide consumed in the titration, and B is the weight of the acetylated oil, in grams.

Solubility in alcohol. Proceed as directed in the general method, page 899. One ml. dissolves in 2 ml. of 80 percent alcohol.

Specific gravity. Determine by any reliable method (see page 5).

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

Mentha Arvensis Oil, Dementholized

Insert the following new monograph to precede the monograph entitled *Menthol*, page 502:

MENTHA ARVENSIS OIL, DEMENTHOLIZED

Cornmint Oil, Dementholized

DESCRIPTION

The portion of oil remaining after the partial removal of menthol, by freezing operations only, from the oil of *Mentha arvensis* var. *piperascens* Holmes (forma piperascens Malinvaud). It is a colorless to yellow liquid having a characteristic minty odor. It is soluble in most fixed oils, in mineral oil, and in propylene glycol. It is insoluble in glycerin.

SPECIFICATIONS

Assay. Not less than 40.0 percent and not more than 60.0 percent of total alcohols, calculated as menthol ($C_{10}H_{20}O$).

Angular rotation. Between −20° and −35°.

Esters, Total. Between 5.0 percent and 20.0 percent, calculated as menthyl acetate ($C_{12}H_{22}O_2$).

Ketones, Total. Between 30.0 percent and 50.0 percent, calculated as menthone ($C_{10}H_{18}O$).

Refractive index. Between 1.458 and 1.465 at 20°.

Solubility in alcohol. Passes test.

Specific gravity. Between 0.888 and 0.908.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Assay. Proceed as directed under *Total Alcohols*, page 893, using about 1.5 grams of the acetylated oil, accurately weighed, for the saponification. Calculate the percent of alcohol, as menthol, in the sample by the formula $[A \times 7.813(1 - 0.0021E)]/(B - 0.021A)$, in which A is the number of ml. of 0.5 N alcoholic potassium hydroxide consumed in the saponification; B is the weight of the acetylated oil taken, in grams; and E is the percentage of esters, as menthyl acetate, determined as directed under *Total esters* below.

Angular rotation. Determine in a 100-mm. tube as directed under *Optical Rotation*, page 939.

Total esters. Weigh accurately about 10 grams, and proceed as directed under *Ester Determination*, page 896, using 99.15 as the equivalence factor (e) in the calculation.

Total ketones. Weigh accurately about 1 gram, and proceed as directed for ketones under *Aldehydes and Ketones—Hydroxylamine Method*, page 894, using 77.12 as the equivalence factor (E) in the calculation.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Solubility in alcohol. Proceed as directed in the general method. One ml. dissolves in 2.5 to 4 ml. of 80 percent alcohol and may become hazy upon further dilution.

Specific gravity. Determine by any reliable method (see page 5).

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

DL-Methionine, page 504

Change the last sentence of *Identification test B* to read:

A red or orange-red color appears. [NOTE: The L-*Methionine* monograph, page 506, is automatically changed by this revision.]

Monosodium L-Glutamate, page 544

Replace *Identification test C* with the following:

C. Prepare a 10 percent solution of the sample in 1 *N* hydrochloric acid. To 1 ml. of this solution add 5 ml. of cobalt-uranyl acetate T.S., and agitate on a vortex mixer for 3 minutes. A golden-yellow precipitate forms, indicating the presence of sodium.

Niacinamide, page 551

Change the paragraph entitled *Heavy metals,* page 552, to read:

Heavy metals. Prepare and test a 670-mg. sample as directed in *Method II* under the *Heavy Metals Test,* page 920, using 20 mcg. of lead ion (Pb) in the solution (*Solution A*).

Orange Oil, Distilled

Insert the following new monograph to precede the monograph entitled *Origanum Oil, Spanish,* page 568:

ORANGE OIL, DISTILLED

DESCRIPTION

The volatile oil obtained by distillation from the fresh peel or juice of the fruit of *Citrus sinesis* L. Osbeck (Fam. *Rutaceae*), with or without the previous separation of the juice, pulp, or peel. It is a colorless to pale yellow liquid having the characteristic odor of fresh orange peel. It is soluble in most fixed oils, in mineral oil, and in alcohol (with haze). It is insoluble in glycerin and in propylene glycol.

SPECIFICATIONS

Aldehydes. Between 1.0 percent and 2.5 percent of aldehydes, calculated as decyl aldehyde ($C_{10}H_{20}O$).

Angular rotation. Between +94° and +99°.

Refractive index. Between 1.471 and 1.474 at 20°.

Ultraviolet absorbance. Not more than 0.01.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Aldehydes. Weigh accurately about 5 ml. of the sample, and proceed as directed under *Aldehydes and Ketones—Hydroxylamine/Tert-Butyl Alcohol Method,* page 43 of THIS SUPPLEMENT, using 78.14 as the equivalence factor (*E*) in the calculation. Allow the mixture to stand at room temperature for 1 hour before titrating.

Angular rotation. Determine in a 100-mm. tube as directed under *Optical Rotation,* page 939.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 5).

Ultraviolet absorbance. Proceed as directed under *Ultraviolet Absorbance of Citrus Oils,* page 900, using about 250 mg. of sample, accurately weighed. The maximum absorbance occurs at 330 ± 3 mμ.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test,* page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test,* page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

Parsley Herb Oil

Insert the following new monograph to precede the monograph entitled *Parsley Seed Oil,* page 576:

PARSLEY HERB OIL

DESCRIPTION

The oil obtained by the steam distillation of the aboveground parts of the plant *Petroselinium sativum* Hoffm. (Fam. *Umbelligerae*), including the immature

seed. It is a yellow to light brown liquid, having the odor of parsley herb. It is soluble in most fixed oils, in mineral oil, and in alcohol (with opalescence). It is slightly soluble in propylene glycol, but it is insoluble in glycerin.

SPECIFICATIONS

Acid value. Not more than 2.0.

Angular rotation. Between +1° and −9°.

Refractive index. Between 1.503 and 1.530 at 20°.

Specific gravity. Between 0.908 and 0.940.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 10 parts per million (0.001 percent).

TESTS

Acid value. Determine as directed in the general method, page 893.

Angular rotation. Determine in a 100-mm. tube as directed under *Optical Rotation*, page 939.

Refractive index, page 945. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 5).

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

Peppermint Oil, page 589

Change the first sentence of the paragraph entitled *Assay for total menthol*, page 590, to read:

Proceed as directed under *Total Alcohols*, page 893, using a 2.5-gram sample of the acetylized oil.

Potassium Alginate, page 641

Change the SPECIFICATION for *Assay* to read:

Assay. It yields not less than 16.5 percent and not more than 19.5 percent of carbon dioxide (CO_2), corresponding to between 89.2 and 105.5 percent of potassium alginate (equiv. wt. 238.00).

Change the SPECIFICATION for *Ash* to read:

Ash. Between 22 and 33 percent after drying.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter.*

Delete the paragraph entitled *Insoluble matter,* page 642. [NOTE: The revised paragraph on p. 46 of the FIRST SUPPLEMENT should also be deleted.]

Potassium Hydroxide, page 652

Change the SPECIFICATION (*Limits of Impurities*) for *Mercury* to read:

Mercury. Not more than 0.1 part per million (0.00001 percent).

Replace the paragraph entitled *Mercury,* page 653, with the following:

Mercury. Determine as directed under *Mercury Limit Test,* page 934, preparing the *Standard preparation* and the *Sample preparation* as follows:

Standard preparation. Prepare the stock solution and the dilutions to obtain a solution containing 1 mcg. of Hg per ml., as directed on page 934. Transfer 1.0 ml. of the final solution (1 mcg. of Hg) to a 50-ml. beaker, and add 20 ml. of water, 1 ml. of dilute sulfuric acid solution (1 in 5), and 1 ml. of potassium permanganate solution (1 in 25). Cover the beaker with a watch glass, boil for a few seconds, and cool.

Sample preparation. Transfer 10.0 grams of the sample into a 100-ml. beaker, dissolve in 15 ml. of water, add 2 drops of phenolphthalein T.S., and slowly neutralize, with constant stirring, with dilute hydrochloric acid solution (1 in 2). Add 1 ml. of dilute sulfuric acid solution (1 in 5) and 1 ml. of potassium permanganate solution (1 in 25), cover the beaker with a watch glass, boil for a few seconds, and cool.

Propylene Glycol, page 678

Change the SPECIFICATION for *Assay,* page 679, to read:

Assay. Not less than 99.5 percent, by weight, of $C_3H_8O_2$.

Replace the paragraph entitled *Assay*, page 679, with the following:

Assay. Inject a 10-µl. portion of the sample into a suitable gas chromatograph in which the detector is the thermal conductivity type and the column is 1-meter × ¼-inch stainless steel tubing packed with 4% Carbowax compound 20 M on 40/60-mesh Chromosorb T, or equivalent materials. The carrier is helium flowing at 75 ml. per minute. The injection port temperature is 240°; the column temperature is 120° to 200°, programmed at a rate of 5° per minute; and the block temperature is 250°. Under the conditions described, the approximate retention time for propylene glycol is 5.7 minutes, and for the three isomers of dipropylene glycol, 8.2, 9.0, and 10.2 minutes. Measure the area under all peaks by any convenient means, and calculate the area percent of propylene glycol and report as weight percent.

Propylene Glycol Alginate, page 680

Change the section entitled IDENTIFICATION to read:

IDENTIFICATION

Transfer 20 ml. of the saponified solution obtained in the determination of *Esterified carboxyl groups* into a 250-ml. Erlenmeyer flask, add 50 ml. of 0.1 *M* periodic acid, swirl, and allow to stand for 30 minutes. Add 2 grams of potassium iodide, titrate with 0.1 *N* sodium thiosulfate to a faint yellow color, and then dilute the mixture to 200 ml. with water. To 10 ml. of this solution add 5 ml. of hydrochloric acid and 10 ml. of modified Schiff's reagent. A blue to blue-violet color develops in about 20 minutes (*formaldehyde*). To another 10-ml. portion of the solution add 1 ml. of a saturated solution of piperazine hydrate and 0.5 ml. of sodium nitroferricyanide T.S. A green color develops (*acetaldehyde*). *Note:* Oxidation of propylene glycol alginate yields formaldehyde and acetaldehyde.

Change the SPECIFICATION for *Assay*, page 681, to read:

Assay. It yields not less than 16 percent and not more than 20 percent of carbon dioxide (CO_2), calculated on the dried basis.

Change the SPECIFICATION for *Ash*, page 681, to read:

Ash. Not more than 10 percent after drying.

Change the SPECIFICATION for *Free carboxyl groups*, page 681, to read:

Free carboxyl groups. Not more than 35 percent, calculated on the dried basis.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter*, page 681.

Change the paragraph entitled *Free carboxyl groups*, page 681, to read:

Free carboxyl groups. Transfer about 1 gram, accurately weighed, into a 600-ml. beaker. Dissolve the sample in 200 ml. of water, stirring mechanically for a minimum of 30 minutes, and titrate with 0.1 *N* sodium hydroxide to a pH of 7.0, determining the end-point potentiometrically. Calculate the percent of free carboxyl groups by the formula $(V \times 44)/(\%CO_2 \times W)$, in which V is the volume of 0.1 *N* sodium hydroxide consumed, in ml.; $\%CO_2$ is the percent of carbon dioxide in the sample as determined by the *Assay;* and W is the weight of the sample taken, in grams, calculated on the dried basis.

Delete the paragraph entitled *Insoluble matter*, page 682. [*Note:* The revised paragraph on p. 48 of the FIRST SUPPLEMENT should also be deleted.]

Pyridoxine Hydrochloride, page 689

Change the paragraph entitled *Heavy metals*, page 690, to read:

Heavy metals. Prepare and test a 670-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Riboflavin, page 697

Add the following sentence at the end of the paragraph entitled *Description:*

In aqueous sodium hydroxide solutions it is levorotatory, and in aqueous hydrochloric acid it is dextrorotatory.

Delete the SPECIFICATION for *Specific rotation*.

Saccharin, page 49, FIRST SUPPLEMENT

Change the SPECIFICATION (*Limits of Impurities*) for *Toluenesulfonamides* to read:

Toluenesulfonamides. Not more than 25 parts per million (0.0025 percent).

Silicon Dioxide, page 716

Replace the entire monograph for *Silicon Dioxide* with the following:

SILICON DIOXIDE

Synthetic Amorphous Silica

SiO_2 Mol. wt. 60.09

DESCRIPTION

Silicon dioxide for food use is an amorphous substance which shows a noncrystalline pattern when examined by x-ray diffraction. It is produced synthetically by either a vapor-phase hydrolysis process, yielding *fumed* (or colloidal) *silica*, or by a wet process, yielding either *precipitated silica* or *silica gel*. *Fumed silica* is produced in essentially an anhydrous state, whereas *precipitated silica* and *silica gel* are obtained as hydrates.

Fumed silica occurs as a white, fluffy, nongritty powder of extremely fine particle size. *Precipitated silica* and *silica gel* occur as white, fluffy powders or as white, microcellular beads or granules. These forms of silicon dioxide, all of which are hygroscopic, are insoluble in water and in organic solvents but are soluble in hydrofluoric acid and in hot, concentrated solutions of alkalies.

SPECIFICATIONS

Assay

Fumed silica: Not less than 99.0 percent of SiO_2 after ignition.

Precipitated silica and *silica gel:* Not less than 94.0 percent of SiO_2 after ignition.

Loss on drying

Fumed silica: Not more than 2.5 percent.

Precipitated silica and *silica gel:* Not more than 7 percent.

Loss on ignition

Fumed silica: Not more than 2 percent after drying.

Precipitated silica and *silica gel:* Not more than 8.5 percent after drying.

Limits of Impurities

Arsenic (as As). Not more than 3 parts per million (0.0003 percent).

Heavy metals (as Pb). Not more than 30 parts per million (0.003 percent).

Lead. Not more than 10 parts per million (0.001 percent).

Soluble ionizable salts (as Na_2SO_4).

Precipitated silica and *silica gel:* Not more than 5 percent.

Assay. Transfer about 1 gram of the sample, previously dried at 105° for 2 hours and accurately weighed, into a tared platinum crucible, ignite as directed

in the test for *Loss on ignition*, cool in a desiccator, and weigh to obtain the ignited sample weight (W). Moisten the residue with 3 or 4 drops of alcohol, add 2 drops of sulfuric acid, and then add enough hydrofluoric acid to cover the wetted sample. Evaporate to dryness on a hot plate, using medium heat (95°–105°), then add 5 ml. of hydrofluoric acid, swirl the dish carefully to wash down the sides, and again evaporate to dryness. Ignite the dried residue to a red heat over a Meker burner, cool in a desiccator, and weigh to obtain the residual weight (w). The difference between the ignited sample weight and the residual weight ($W - w$) represents the weight of SiO_2 in the ignited sample.

Loss on drying, page 931. Dry at 105° for 2 hours.

Loss on ignition. Transfer into a suitable tared crucible about 1 gram of an accurately weighed sample that has been previously dried at 105° for 2 hours. Place the crucible in a cold muffle furnace, and bring the temperature to 900°–1000° during a one-hour period. Ignite at this temperature for 1 hour, cool in a desiccator, and weigh.

Sample Solution for the Determination of Arsenic, Heavy Metals, and Lead. Transfer 3.3 grams of the sample into a 250-ml. beaker, add 50 ml. of 0.5 *N* hydrochloric acid, cover with a watch glass, and heat slowly to boiling. Boil gently for 15 minutes, cool, and let the undissolved material settle. Decant the supernatant liquid through a Whatman No. 3 filter paper, or equivalent, into a 100-ml. volumetric flask, retaining as much as possible of the insoluble material in the beaker. Wash the slurry and beaker with three 10-ml. portions of hot water, decanting each washing through the filter into the flask. Finally, wash the filter paper with 15 ml. of hot water, cool the filtrate to room temperature, dilute to volume with water, and mix.

Arsenic. A 30-ml. portion of the *Sample Solution* meets the requirements of the *Arsenic Test*, page 865.

Heavy metals. A 20-ml. portion of the *Sample Solution* meets the requirements of the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Lead. A 30-ml. portion of the *Sample Solution* meets the requirements of the *Lead Limit Test*, page 929, using 10 mcg. of lead ion (Pb) in the control.

Soluble ionizable salts. Weigh accurately 12.5 grams of the sample, and stir it with 240 ml. of water for at least 5 minutes with a high speed mixer. Transfer the mixture into a 250-ml. graduate, and wash the mixer container with water, adding the washings to the graduate to make 250 ml. Stopper the graduate, and invert it several times to mix the slurry. The conductivity of the slurry, determined with a suitable conductance bridge assembly, is not greater than that produced by a control solution containing 750 mg. of anhydrous sodium sulfate in each 250 ml.

Packaging and storage. Store in well-closed containers.

Functional use in foods. Anticaking agent; defoaming agent; carrier; conditioning agent.

Sodium Acid Pyrophosphate, page 719

Delete the SPECIFICATION for *Neutralizing value*, page 720.

Change the SPECIFICATION (*Limits of Impurities*) for *Fluoride*, page 720, to read:

Fluoride. Not more than 50 parts per million (0.005 percent).

Delete the paragraph entitled *Neutralizing value*, page 720.

Change the paragraph entitled *Fluoride*, page 720, to read:

Fluoride. Weigh accurately 1 gram, and proceed as directed in *Method I* under the *Fluoride Limit Test*, page 917.

[*Note*—These revisions in the *Fluoride* limit and test procedure supersede those made via the FIRST SUPPLEMENT.]

Sodium Alginate, page 721

Change the SPECIFICATION for *Assay* to read:

Assay. It yields not less than 18 percent and not more than 21 percent of carbon dioxide (CO_2), corresponding to between 90.8 and 106 percent of sodium alginate (equiv. wt. 222.00).

Change the SPECIFICATION for *Ash* to read:

Ash. Between 18 percent and 27 percent after drying.

Delete the SPECIFICATION (*Limits of Impurities*) for *Insoluble matter*.

Delete the paragraph entitled *Insoluble matter*, page 722. [NOTE: The revised paragraph on p. 50 of the FIRST SUPPLEMENT should also be deleted.]

Sodium Aluminum Phosphate, Acid, page 722

Delete the SPECIFICATION, page 722, and the test procedure, page 723, for *Neutralizing value*.

Sodium Chloride, page 52, FIRST SUPPLEMENT

Change the SPECIFICATION for *Assay* to read:

Assay

Evaporated salt with up to 2 percent of suitable free-flowing or conditioning agents and anticaking agents such as sodium ferrocyanide: Not less than 97.5 percent of NaCl after drying at 625° for 2 hours.

Evaporated salt with only anticaking agents such as sodium ferrocyanide: Not less than 99.0 percent after drying at 625° for 2 hours.

Rock or solar salt: Not less than 97.5 percent of NaCl after drying at 625° for 2 hours, the remainder consisting chiefly of minor amounts of naturally occurring components such as alkaline and/or alkaline earth sulfates and chlorides.

Sodium Hydroxide, page 743

Change the SPECIFICATION (*Limits of Impurities*) for *Mercury* to read:

Mercury. Not more than 0.1 part per million (0.00001 percent).

Replace the paragraph entitled *Mercury*, page 744, with the following:

Mercury. Determine as directed under *Mercury Limit Test*, page 934, preparing the *Standard preparation* and the *Sample preparation* as follows:

Standard preparation. Prepare the stock solution and dilutions to obtain a solution containing 1 mcg. of Hg per ml., as directed on page 934. Transfer 1.0 ml. of the final solution (1 mcg. of Hg) to a 50-ml. beaker, and add 20 ml. of water, 1 ml. of dilute sulfuric acid solution (1 in 5), and 1 ml. of potassium permanganate solution (1 in 25). Cover the beaker with a watch glass, boil for a few seconds, and cool

Sample preparation. Transfer 10.0 grams of the sample into a 100-ml. beaker, dissolve in 15 ml. of water, add 2 drops of phenolphthalein T.S., and slowly neutralize, with constant stirring, with dilute hydrochloric acid solution (1 in 2). Add 1 ml. of dilute sulfuric acid solution (1 in 5) and 1 ml. of potassium permanganate solution (1 in 25), cover the beaker with a watch glass, boil for a few seconds, and cool.

Sodium Metaphosphate, page 749

Change the SPECIFICATION (*Limits of Impurities*) for *Fluoride*, page 750, to read:

Fluoride. Not more than 50 parts per million (0.005 percent).

Change the *Fluoride* test procedure, page 750, to read:

Fluoride. Weigh accurately 1 gram, and proceed as directed in *Method I* under the *Fluoride Limit Test*, page 917.

Change the paragraph entitled *Heavy metals*, page 750, to read:

Heavy metals. Dissolve 20 grams of the sample in 80 ml. of water in a 250-ml. beaker, cautiously add 20 ml. of sulfuric acid, and boil for 1 hour. Cool the solution, dilute it to 100 ml. with water, mix, and filter through a fritted-disk funnel. Dilute a 10-ml. aliquot to 25 ml. with water, and adjust the pH to between 3.0 and 4.0 with ammonium hydroxide. Dilute to 40 ml. with water, mix, and add 10 ml. of freshly prepared hydrogen sulfide T.S. Allow to stand for 5 minutes, and view downward over a white surface. The color of the sample solution is no darker than that of a standard prepared with 20 mcg. of lead ion (Pb), treated in the same manner as the sample.

Sodium Saccharin, page 56, FIRST SUPPLEMENT

Change the SPECIFICATION (*Limits of Impurities*) for *Toluenesulfonamides* to read:

Toluenesulfonamides. Not more than 25 parts per million (0.0025 percent).

Sodium Sulfate, page 775

In the *Description*, replace the fifth sentence, page 776, with the following:

A 1 in 20 solution is neutral or slightly alkaline to litmus paper.

Sodium Tartrate, page 777

Replace the paragraph entitled *Assay*, page 778, with the following:

Assay. Weigh accurately about 250 mg., previously dried at 150° for 3 hours, and transfer it to a 250-ml. beaker. Add 150 ml. of glacial acetic acid, heat to near boiling, stir until the sample is dissolved (preferably with a magnetic stirrer), and cool to room temperature. Titrate with 0.1 *N* perchloric acid in glacial acetic acid, determining the end-point potentiometrically. Each ml. of 0.1 *N* perchloric acid is equivalent to 9.703 mg. of $C_4H_4Na_2O_6$.

Sodium Tripolyphosphate, page 780

Change the SPECIFICATION for *Assay* to read:

Assay. Not less than 85.0 percent of $Na_5P_3O_{10}$, calculated on the dried basis.

Change the SPECIFICATION for *Loss on drying* to read:

Loss on drying. *Anhydrous*, not more than 0.7 percent; *hexahydrate*, between 17.0 and 22.7 percent.

Sorbitol, page 786

Change the paragraph entitled *Assay*, page 787, to read:

Assay. Proceed as directed in the revised *Assay* for *Sorbitol Solution* (see below).

Sorbitol Solution, page 788

Replace the section entitled *Assay*, page 789, with the following:

Assay

Reagent-internal standard preparation. Dissolve a suitable quantity of *n*-butylboronic acid in pyridine to obtain a solution having a concentration of about 10 mg. per ml. This is the reagent solution. Add a suitable quantity of methyl nonadecanoate, the internal standard, to the reagent solution to obtain a solution having a concentration of about 2 mg. per ml. This is the *Reagent-internal standard preparation.*

Standard preparation. Dissolve an accurately weighed quantity of USP Sorbitol Reference Standard in water to obtain a solution having a concentration of about 1.5 mg. per ml.

Assay preparation. Dissolve an accurately weighed quantity of the sample, equivalent to about 150 mg. of anhydrous sorbitol, in water in a 100-ml. volumetric flask, dilute to volume with water, and mix.

Procedure. Pipet 1-ml. portions of the *Standard preparation* and of the *Assay preparation* into separate 25-ml. vials, and heat the vials in a vacuum oven at 50° to dryness. Add 1.0 ml. of the *Reagent-internal standard preparation* to each residue, and mix. Inject a 1.0-µl. portion of the solution from the *Assay preparation* into a suitable gas chromatograph in which the detector is the hydrogen flame-ionization type and the column is 4 mm. × 1.8 m., packed with 3 percent cyanopropylphenyl silicone liquid phase on 80- to 100-mesh silanized diatomaceous earth. The carrier is nitrogen flowing at 50 ml. per minute. The injection port temperature is 245°, the column temperature is 205°, and the detector temperature is 260°. The retention time of the internal standard is

about 4 minutes, and that of sorbitol about 9 minutes. In a suitable chromatogram, the resolution factor, R, is not less than 5.0 between the peaks for sorbitol and the internal standard, and six replicate injections of the Standard preparation show a relative standard deviation of not more than 1.5 percent. Similarly, inject a 1.0-µl. portion of the solution from the *Standard preparation*. Calculate the quantity, in mg., of $C_6H_{14}O_6$ in the sample taken by the formula $100C(R_U/R_S)$, in which C is the concentration of USP Sorbitol Reference Standard in the *Standard preparation*, in mg. per ml., and R_U and R_S are the ratios of the peak areas of sorbitol to those of the internal standard of the *Assay preparation* and *Standard preparation*, respectively.

Tartaric Acid, page 808

Insert the following new SPECIFICATION for *Specific rotation:*

Specific rotation, $[\alpha]_D^{25°}$. Between +12.0° and +13.0°.

Insert the following new test procedure to precede the paragraph entitled *Arsenic*, page 809:

Specific rotation, page 939. Determine in a solution containing 2 grams in each 10 ml.

L-Tyrosine, page 842

Change the paragraph entitled *Heavy metals*, page 843, to read:

Heavy metals. Prepare and test a 670-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 920, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Xanthan Gum, page 856

Replace the paragraph entitled *Ash*, page 857, with the following:

Ash. Weigh accurately about 3 grams, previously dried at 105° for 4 hours, in a tared crucible, and incinerate at about 650° until free from carbon. Cool the crucible and its contents in a desiccator, weigh, and determine the weight of the ash.

Replace the paragraph entitled *Isopropyl alcohol*, page 858, with the following:

Isopropyl alcohol

IPA standard solution. Transfer 500.0 mg. of chromatographic quality isopropyl alcohol into a 50-ml. volumetric flask, dilute to volume with water, and mix. Pipet 10 ml. of this solution into a 100-ml. volumetric flask, dilute to volume with water, and mix.

TBA standard solution. Transfer 500.0 mg. of chromatographic quality *tert*-butyl alcohol into a 50-ml volumetric flask, dilute to volume with water, and mix. Pipet 10 ml. of this solution into a 100-ml. volumetric flask, dilute to volume with water, and mix.

Mixed standard solution. Pipet 4 ml. each of the *IPA standard solution* and of the *TBA standard solution* into a 125-ml. graduated Erlenmeyer flask, dilute to about 100 ml. with water, and mix. This solution contains approximately 40 mcg. each of isopropyl alcohol and of *tert*-butyl alcohol per ml.

Sample preparation. Disperse 1 ml. of a suitable antifoam emulsion, such as Dow-Corning G-10, or equivalent, in 200 ml. of water contained in a 1000-ml. 24/40 round-bottom distilling flask. Add about 5 grams of the sample, accurately weighed, and shake for 1 hour on a wrist-action mechanical shaker. Connect the flask to a fractionating column, and distill about 100 ml., adjusting the heat so that foam does not enter the column. Add 4.0 ml. of *TBA standard solution* to the distillate to obtain the *Sample preparation.*

Procedure. Inject about 5 µl. of the *Mixed standard solution* into a suitable gas chromatograph equipped with a flame ionization detector and a 1.8 m. × 3.2 mm. stainless steel column packed with 80/100-mesh Porapak QS, or equivalent. The carrier is helium flowing at 80 ml. per minute. The injection port temperature is 200°, the column temperature is 165°, and the detector temperature is 200°. The retention time of isopropyl alcohol is about 2 minutes, and that of *tert*-butyl alcohol about 3 minutes.

Determine the areas of the IPA and TBA peaks, and calculate the response factor, f, by the formula A_{IPA}/A_{TBA}, in which A_{IPA} is the area of the isopropyl alcohol peak, and A_{TBA} is the area of the *tert*-butyl alcohol peak.

Similarly, inject about 5 µl. of the *Sample preparation*, and determine the peak areas, recording the area of the isopropyl alcohol peak as a_{IPA}, and that of the *tert*-butyl alcohol peak as a_{TBA}. Calculate the isopropyl alcohol content, in parts per million, in the sample taken by the formula

$$(a_{IPA} \times 4000)/(f \times a_{TBA} \times W),$$

in which W is the weight of the sample taken, in grams.

GENERAL TESTS AND APPARATUS

Alginates Assay, page 863

In the first sentence of the paragraph entitled *Procedure*, delete the phrase, ". . . previously dried in vacuum for 4 hours at 60° . . ." Add the following sentence at the end of the paragraph:

Calculate the results on the dried basis.

Chewing Gum Base, page 873

Replace the procedure for *Residual Styrene*, page 877, with the following:

RESIDUAL STYRENE

Standard Preparation. Place 25 ml. of carbon disulfide in a 100-ml. volumetric flask, cap with a serum stopper, and tare the flask to the nearest 0.1 mg. Inject, with 50-µl. syringes, 15 µl. each of styrene and of alpha-methylstyrene (AMS), reweighing after each addition to obtain the weight of each solution injected. Record the weight, in mg., of styrene as w_1 and that of AMS as w_2. Dilute to volume with carbon disulfide, and mix. Pipet 2 ml. of this solution into a second 100-ml. volumetric flask, dilute to volume with carbon disulfide, and mix. Finally, pipet 25 ml. of the diluted solution into a third 100-ml. volumetric flask, dilute to volume with carbon disulfide, and mix.

AMS-Solvent Solution. Place 25 ml. of carbon disulfide in a 100-ml. volumetric flask, cap with a serum stopper, and tare the flask to the nearest 0.1 mg. Using a 50-µl. syringe, inject 15 µl. of AMS, and reweigh to obtain the weight of AMS injected. Dilute to volume with carbon disulfide, and mix. Pipet 2 ml. of this solution into a second 100-ml. volumetric flask, dilute to volume with carbon disulfide, and mix. Finally, pipet 25 ml. of the diluted solution into a third 100-ml. volumetric flask, dilute to volume, and mix. Calculate the weight, in grams, of AMS in each ml. of the final solution, and record the result as w' (approximately 7.5×10^{-7}).

Sample Preparation

Latex Samples. Add, with agitation, 100 ml. of the latex to a mixture consisting of 15 ml. of glacial acetic acid and 10 grams of sodium chloride in 500 ml. of hot water. Coagulation starts almost immediately. When coagulation is complete, collect the coagulum on a coarse filter or cheesecloth, and wash with 1000 ml. of a hot solution prepared with 5.6 grams of sodium hydroxide and 1000 ml. of water. Wash with hot water until the wash water is free of alkali, then cut the coagulum into small pieces, and dry at 105° for 4 hours. Continue as

directed under *Solid Samples*, beginning with "Transfer 1.5 grams, accurately weighed . . ."

Solid Samples. Cut a piece approximately 2″ × 3″ × 5″ from the corner of a polymer bale, and pass it through a cold mill, set at least ¼″ open, four times, reversing the sample on each pass. Cut the sample into two pieces at least 1 inch from the edge to expose clean polymer, and then dice or cut into small strips approximately 2 grams of the clean polymer. Transfer 1.5 grams, accurately weighed, into a 4-ounce bottle fitted with a polyethylene cap, add 25.0 ml. of the *AMS-Solvent Solution*, cap tightly, and agitate on a mechanical shaker until the polymer dissolves. (NOTE: Some polymers tend to swell and form viscous cements instead of dissolving cleanly. If this occurs, add 5- to 10-ml. increments of carbon disulfide to obtain a mobile slurry, and in the next step increase the volume of methanol by a proportional amount.) Add 25 ml. of methanol, cap the bottle, and shake vigorously on the shaker for 30 minutes. After the contents have settled, decant 10 ml. of the coagulant serum into a 1-ounce bottle, add 10 ml. of water, and stopper with a serum cap. Shake vigorously for 1 minute, then turn the bottle upside down, and allow the layers to separate. Withdraw by syringe 1 to 2 ml. of the lower (carbon disulfide) layer, and transfer it into a 10-dram vial filled ¼″ with anhydrous sodium sulfate. Seal with a polyethylene cap, shake to mix, and allow to settle.

Procedure. Inject a 10-µl. portion of the *Sample Preparation* into a suitable gas chromatograph in which the detector is the hydrogen flame-ionization type and the column is 10′ × 3/16″ stainless steel tubing packed with 25% Ucon 50 HB 2000 on 60/80-mesh acid-washed DMCS Chromosorb W, or with equivalent packing materials. The carrier is nitrogen or helium flowing at 40 ml. per minute. The injection port temperature is 240°, the column temperature 170° isothermal, and the detector temperature 250°. Adjust the sensitivity of the instrument to give as large a signal as possible for styrene and AMS as is consistent with an acceptable background level. Measure the styrene and AMS peaks by any convenient method, recording the area of the styrene peak as A_1 and that of the AMS peak as A_2.

In the same manner, inject a 10-µl. portion of the *Standard Preparation* into the chromatograph, obtain the chromatogram, and record the area of the styrene peak as a_1 and that of the AMS peak as a_2. Calculate the styrene factor, F, by the formula $(w_1/w_2) \times (a_2/a_1)$.

Calculate the content of residual styrene in the sample taken, in parts per million, by the formula $(A_1/A_2) \times F \times 25 \times (w'/W) \times 10^6$, in which W is the weight of the sample taken, in grams.

Essential Oils and Related Substances, page 892

Insert the following new subsection to precede the subsection entitled *Aldehydes and Ketones—Neutral Sulfite Method*, page 895:

ALDEHYDES AND KETONES—HYDROXYLAMINE/TERT-BUTYL ALCOHOL METHOD

Hydroxylamine Solution. Dissolve 45 grams of reagent grade hydroxylamine hydrochloride in 130 ml. of water, add 850 ml. of *tert*-butyl alcohol, mix, and neutralize to a pH of 3.0 to 3.5 with sodium hydroxide, using a pH meter.

Caution: Do not heat the solution.

Procedure. Weigh accurately the quantity of the sample specified in the individual monograph, and transfer it into a 250-ml. glass-stoppered flask. Add 50 ml. of the *Hydroxylamine Solution*, or the volume specified in the monograph, mix thoroughly, and allow to stand at room temperature for the time specified in the monograph. Titrate with 0.5 *N* sodium hydroxide to the same pH as the *Hydroxylamine Solution* used. Calculate the percent of aldehyde or ketone by the formula:

$$AK = \frac{(S)(100E)}{W}$$

in which AK = percent of aldehyde or ketone, S = the number of ml. of 0.5 *N* sodium hydroxide consumed in the titration of the sample, E = the equivalence factor given in the monograph, and W = the weight of the sample in mg.

In the subsection entitled *Solubility in Alcohol*, page 899, change the second sentence to read:

Maintain the temperature at 25° and shake the cylinder thoroughly after each addition of alcohol.

SOLUTIONS AND INDICATORS

Orthophenanthroline T.S., page 993

In the first sentence, change "1.48 grams" (of clear crystals of ferrous sulfate) to read:

. . . 700 mg. . . .

Insert the following new volumetric solution to precede *Hydrochloric Acid, 1 N*, page 998:

Ferrous Ammonium Sulfate, 0.1 N [39.21 grams $Fe(NH_4)_2(SO_4)_2.6H_2O$ per liter]. Dissolve 40 grams of ferrous ammonium sulfate hexahydrate in a previously cooled mixture of 40 ml. of sulfuric acid and 200 ml. of water, dilute to 1000 ml. with water, and mix. On the day of use, standardize the solution as follows:

Transfer from 25 to 30 ml. of the solution, accurately measured, into a flask, add 2 drops of orthophenanthroline T.S., and titrate with 0.1 *N* ceric sulfate until the red color is changed to pale blue. From the volume of 0.1 *N* ceric sulfate consumed, calculate the normality.

Oxalic Acid, 0.1 N, page 999

Change the first sentence to read:

Dissolve 6.45 grams of oxalic acid, $H_2C_2O_4.2H_2O$, in sufficient water to make 1000 ml.

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Individuals who wish to cut apart the corrections and additions in this THIRD SUPPLEMENT and paste them in the main volume of the *Food Chemicals Codex Second Edition* will need both sides of each page. Additional copies of the Supplement are available at no charge and may be secured on request to:

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