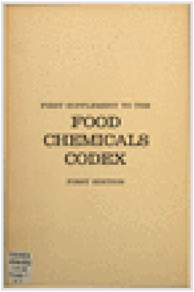

FOOD
CHEMICALS
CODEX

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FIRST SUPPLEMENT TO THE

FOOD CHEMICALS CODEX

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of the
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of the
Food Protection Committee

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1967

ADDITIONS, CHANGES, AND CORRECTIONS

Changes and additions listed herein constitute revisions in the Food Chemicals Codex, First Edition, effective October 1, 1967. Page numbers cited refer to F.C.C. I, unless otherwise indicated.

Benzyl Alcohol, page 75

Change the SPECIFICATION for *Specific gravity,* page 76, to read:

Specific gravity. Between 1.042 and 1.047.

Butyl Acetate

Insert the following new monograph to precede the monograph entitled *Butylated Hydroxyanisole,* page 91:

BUTYL ACETATE

n-Butyl Acetate

$CH_3COOCH_2CH_2CH_2CH_3$

$C_6H_{12}O_2$ Mol. wt. 116.16

DESCRIPTION

A clear, colorless, mobile liquid having a strong, characteristic, fruity odor. One ml. dissolves in about 145 ml. of water. It is miscible in all proportions with alcohol, with ether, and with propylene glycol.

SPECIFICATIONS

Assay. Not less than 98 per cent of $C_6H_{12}O_2$.

Distillation range. Between 120° and 128°.

Refractive index. Between 1.3930 and 1.3950 at 20°.

Specific gravity. Between 0.876 and 0.880.

Limits of Impurities

Acidity (as CH_3COOH). Not more than 0.01 per cent.

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

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

Nonvolatile residue. Not more than 60 parts per million (0.006 per cent).

TESTS

Assay. Transfer about 1.5 grams of the sample, accurately weighed, into a 250-ml. Erlenmeyer flask, add 25.0 ml. of 1 *N* potassium hydroxide and 25 ml. of anhydrous isopropanol, swirl to effect complete solution, and allow to stand at room temperature for 30 minutes. Add about 1 ml. of phenolphthalein T.S. to the mixture, and titrate with 0.5 *N* sulfuric acid to the disappearance of the pink color. Perform a residual blank determination (see page 2). Each ml. of 0.5 *N* sulfuric acid is equivalent to 58.08 mg. of $C_6H_{12}O_2$.

Distillation range. Distil 100 ml. as directed in the general method, page 737.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 4).

Acidity. Transfer 69.0 ml. (60 grams) into a 250-ml. Erlenmeyer flask, add phenolphthalein T.S., and titrate with 0.1 *N* alcoholic potassium hydroxide to a pink end-point that persists for at least 15 seconds. Not more than 1.0 ml. is required.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 720.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Nonvolatile residue. Evaporate 95 ml. (about 83.5 grams) to dryness in a tared platinum dish on a steam bath, and dry at 105° for 30 minutes. Cool in a desiccator and weigh. The weight of the residue does not exceed 5 mg.

Packaging and storage. Store in tight containers and avoid excessive temperatures.

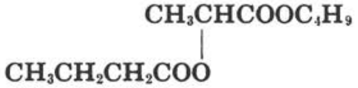
Functional use in foods. Flavoring agent.

Butyl Butyryllactate

Insert the following new monograph to precede the monograph entitled *1,3-Butylene Glycol*, page 96:

BUTYL BUTYRYLLACTATE

Butyryllactic Acid, Butyl Ester; Lactic Acid, Butyl Ester, Butyrate



$$\begin{array}{r}\mathrm{CH_3CHCOOC_4H_9}\\ |\quad\quad\quad\\ \mathrm{CH_3CH_2CH_2COO}\quad\quad\quad\end{array}$$

$C_{11}H_{20}O_4$ Mol. wt. 216.28

DESCRIPTION

A colorless liquid with a mild buttery odor. It is miscible with alcohol and most fixed oils, soluble in propylene glycol, and insoluble in glycerin and in water.

SPECIFICATIONS

Assay. Not less than 95 per cent of $C_{11}H_{20}O_4$.

Specific gravity. Between 0.970 and 0.974.

Solubility in alcohol. Passes test.

Refractive index. Between 1.4200 and 1.4230 at 20°.

Limits of Impurities

Acid value. Not more than 1.

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

TESTS

Assay. Weigh accurately about 1 gram, and proceed as directed under *Ester Determination*, page 742, using 54.07 as the equivalence factor (*e*) in the calculation.

Specific gravity. Determine by any reliable method (see page 4).

Solubility in alcohol. Proceed as directed in the general method, page 746. One ml. dissolves in 3 ml. of 70 per cent alcohol.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Acid value. Determine as directed in the general method, page 740.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight, glass or aluminum, or suitably lined containers in a cool place protected from light.

Functional use in foods. Flavoring agent.

Calcium Phosphate, Monobasic, page 133

Change the SPECIFICATION for *Assay* to read:

Assay. $Ca(H_2PO_4)_2$ (anhydrous), not less than 23.5 per cent and not more than 25.6 per cent of CaO; $Ca(H_2PO_4)_2.H_2O$ (monohydrate), not less than 22.2 per cent and not more than 24.7 per cent of CaO.

Diacetyl Tartaric Acid Esters of Mono- and Diglycerides and **Diethyl Malonate**

Insert the following two new monographs to precede the monograph entitled *Diethylpyrocarbonate*, page 224:

DIACETYL TARTARIC ACID ESTERS OF MONO- AND DIGLYCERIDES

DESCRIPTION

The reaction product of partial glycerides of edible oils, fats, or fat-forming fatty acids with diacetyl tartaric anhydride. The esters range in appearance from sticky, viscous liquids through a fat-like consistency to a waxy solid, depending upon the iodine value of the oils or fats used in their manufacture. The diacetyl tartroyl esters have a faint acid odor and are miscible in all proportions with oils and fats. They are soluble in most common fat solvents, in methanol, in acetone, and in ethyl acetate, but insoluble in other alcohols, in acetic acid, and in water. They are dispersible in water and resistant to hydrolysis by water for moderate periods of time. The pH of a 3 per cent dispersion in water is between 2 and 3.

IDENTIFICATION

To a solution of 500 mg. in 10 ml. of methanol add, dropwise, lead acetate T.S. A white, flocculent, practically insoluble precipitate forms.

SPECIFICATIONS

Assay for tartaric acid. Between 17 and 20 per cent of tartaric acid ($C_4H_6O_6$) after saponification.

Acetic acid. Between 14 and 17 per cent of CH_3COOH after saponification.

Acid value. Between 62 and 76.

Fatty acids, Total. Not less than 56 per cent of total fatty acids after saponification.

Glycerin. Not less than 12 per cent of $C_3H_8O_3$.

Saponification value. Between 62 and 76.

Limits of Impurities

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

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

Residue on ignition. Not more than 0.01 per cent.

TESTS

Assay for tartaric acid

Standard Reference Curve. Transfer 100 mg. of reagent grade tartaric acid, accurately weighed, into a 100-ml. volumetric flask, dissolve it in about 90 ml. of water, add water to volume, and mix well. Transfer 3.0, 4.0, 5.0, and 6.0-ml. portions into separate 19 × 150 mm. matched cuvettes, and add sufficient water to make 10.0 ml. To each cuvette add 4.0 ml. of a freshly prepared 1 in 20 solution of sodium metavanadate and 1.0 ml. of acetic acid. *Use these solutions within 10 minutes after being prepared.* Prepare a blank in the same manner using 10 ml. of water in place of the tartaric acid solutions. Set the instrument at zero with the blank, and then determine the absorbance of the four solutions of tartaric acid at 520 mμ with a suitable spectrophotometer or a photoelectric colorimeter equipped with a 520 mμ filter. From the data thus obtained, prepare a reference curve by plotting the absorbances on the ordinate against the corresponding quantities, in mg., of the tartaric acid on the abscissa.

Assay preparation. Transfer about 4 grams of the sample, accurately weighed, into a 250-ml. Erlenmeyer flask, and add 80 ml. of approximately 0.5 *N* potassium hydroxide and 0.5 ml. of phenolphthalein T.S. Connect an air condenser at least 65 cm. in length to the flask, and heat the mixture on a hot plate for about 2.5 hours. Add to the hot mixture approximately 10 per cent phosphoric acid until it is definitely acid to congo red test paper. Reconnect the air condenser, and heat until the fatty acids are liquified and clear. Cool and then transfer the mixture into a 250-ml. separator with the aid of small portions of water and chloroform. Extract the liberated fatty acids with three successive 25-ml. portions of chloroform, and collect the extracts in a second separator. Wash the combined chloroform extracts with two 25-ml. portions of water, and add the washings to the separator containing the water layer. Retain the combined chloroform extracts for the determination of *Total fatty acids.* Transfer the contents of the first separator to a 250-ml. beaker, heat on a steam bath to remove traces of chloroform, filter through acid-washed, fine texture filter paper into a 500-ml. volumetric flask, and finally dilute to volume with water (*Solution I*). Pipet 25.0 ml. of this solution into a 100-ml. volumetric flask, and dilute to volume with water (*Solution II*). Retain the rest of *Solution I* for the determination of *Glycerin.*

Procedure. Transfer 10.0 ml. of *Solution II* prepared under *Assay preparation* into a 19 × 150 mm. cuvette, and continue as directed under *Standard Reference Curve*, beginning with ". . . add 4.0 ml. of a freshly prepared 1 in 20 solution of sodium metavanadate . . ." From the reference curve determine the weight, in mg., of tartaric acid in the final dilution, multiply this by 20, and divide the result by the weight of the original sample to obtain the per cent of tartaric acid.

Acetic acid. Transfer about 4 grams of the sample, accurately weighed, into the inner tube of a modified Hortvet distillation apparatus for the determination of volatile acidity (see Fig. 22, page 143, Official Methods of Analysis of the A.O.A.C., 9th Edition, 1960). Insert the inner tube in the outer flask containing about 300 ml. of recently boiled hot water. To the sample add 10 ml. of approximately 4 *N* perchloric acid [35 ml. (60 grams) of 70 per cent perchloric acid in 100 ml. of water], and connect the inner tube to a water-cooled condenser through a large distillation trap. Distil by heating the outer flask so that 100 ml. of distillate is collected within 20 or 25 minutes. Collect the distillate in 100-ml. portions, add phenolphthalein T.S. to each portion, and titrate with 0.5 *N* sodium hydroxide. Continue the distillation until a 100-ml. portion of the distillate requires no more than 0.5 ml. of 0.5 *N* sodium hydroxide for neutralization. Each ml. of the 0.5 *N* sodium hydroxide consumed in the series of titrations is equivalent to 30.03 mg. of CH_3COOH.

Acid value. Transfer about 1 gram, accurately weighed, into a 125-ml. Erlenmeyer flask. Prepare a solvent by mixing 1 volume of benzene with 4 volumes of methanol, adding phenol red T.S., and neutralizing, if necessary. Dissolve the sample in about 25 ml. of this solvent by warming gently, if necessary. Titrate the solution with 0.1 *N* methanolic potassium hydroxide to a light red end-point. Perform a blank determination on a 25-ml. portion of the solvent, and make any necessary correction (see page 2). Calculate the acid value by the formula $56.1\ V \times N/W$, in which V is the volume, in ml., and N is the normality, respectively, of the methanolic potassium hydroxide, and W is the weight, in grams, of the sample taken.

Fatty acids, Total. Dry the combined chloroform extracts of fatty acids obtained in the *Assay for tartaric acid* by shaking with a few grams of anhydrous sodium sulfate. Filter the solution into a tared 250-ml. beaker, evaporate the chloroform on a steam bath, cool, and weigh.

Glycerin. Transfer 5.0 ml. of *Solution I* prepared in the *Assay for tartaric acid* into a 250-ml. glass-stoppered Erlenmeyer or iodine flask. Add to the flask 15 ml. of glacial acetic acid and 25.0 ml. of periodic acid solution, prepared by dissolving 2.7 grams of periodic acid (H_5IO_6) in 50 ml. of water, adding 950 ml. of glacial acetic acid, and mixing thoroughly; protect this solution from light. Shake the mixture for 1 or 2 minutes, allow it to stand for 15 minutes, add 15 ml.

of potassium iodide solution (15 in 100) and 15 ml. of water, swirl, let stand 1 minute, and then titrate the liberated iodine with 0.1 *N* sodium thiosulfate using starch T.S. as the indicator. Perform a *residual blank titration* (see page 2) using water in place of the sample. The corrected volume is the number of ml. of 0.1 *N* sodium thiosulfate required for the glycerin and the tartaric acid in the sample represented by the 5 ml. of *Solution I*. From the per cent determined in the *Assay for tartaric acid* calculate the volume of 0.1 *N* sodium thiosulfate required for the tartaric acid in the titration. The difference between the corrected volume and the calculated volume required for the tartaric acid is the number of ml. of 0.1 *N* sodium thiosulfate consumed due to the glycerin in the sample. One ml. of 0.1 *N* sodium thiosulfate is equivalent to 2.303 mg. of glycerin and to 7.505 mg. of tartaric acid.

Saponification value. Determine as directed in the general method, page 760, using about 2 grams, accurately weighed. Add 5 to 10 ml. of water to samples and blanks before saponification; otherwise sufficient salts precipitate during saponification to cause serious bumping and spattering.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 720.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

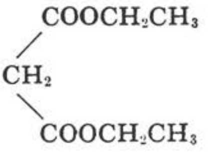
Residue on ignition. Ignite 10 grams as directed in the general method, page 786.

Packaging and storage. Store in well-closed containers.

Functional use in foods. Emulsifier.

DIETHYL MALONATE

Ethyl Malonate; Malonic Ester



$C_7H_{12}O_4$ Mol. wt. 160.17

DESCRIPTION

A colorless liquid with a slight, fruitlike odor. It is soluble in most fixed oils and in propylene glycol. It is insoluble in glycerin and in mineral oil.

SPECIFICATIONS

Assay. Not less than 98 per cent $C_7H_{12}O_4$.

Specific gravity. Between 1.053 and 1.056.

Solubility in alcohol. Passes test.

Refractive index. Between 1.4130 and 1.4157 at 20°.

Limits of Impurities

Acid value. Not more than 1.

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

TESTS

Assay. Weigh accurately about 1 gram, and proceed as directed under *Ester Determination*, page 742, using 40.05 as the equivalence factor (*e*) in the calculation.

Specific gravity. Determine by any reliable method (see page 4).

Solubility in alcohol. Proceed as directed in the general method, page 746. One ml. dissolves in 1.5 volumes of 60 per cent alcohol.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Acid value. Determine as directed in the general method, page 740.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight, preferably glass, tin, or suitably lined containers in a cool place, protected from light.

Functional use in foods. Flavoring agent.

Diethyl Sebacate

Insert the following new monograph to precede the monograph entitled *Dilauryl Thiodipropionate*, page 225:

DIETHYL SEBACATE

Ethyl Sebacate

$C_2H_5OOC(CH_2)_8COOC_2H_5$

$C_{14}H_{26}O_4$ Mol. wt. 258.36

DESCRIPTION

A colorless to light yellow liquid with a faint odor; insoluble in water; miscible with alcohol, ether, and other organic solvents.

SPECIFICATIONS

Assay. Not less than 98 per cent $C_{14}H_{26}O_4$.

Refractive index. Between 1.4350 and 1.4380 at 20°.

Specific gravity. Between 0.960 and 0.965.

Limits of Impurities

Acid value. Not more than 1.

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

TESTS

Assay. Weigh accurately about 1 gram, and proceed as directed under *Ester Determination*, page 742, using 64.59 as the equivalence factor (*e*) in the calculation.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 4).

Acid value. Determine as directed in the general method, page 740.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in amber glass, aluminum, or suitably lined containers in a cool place protected from light.

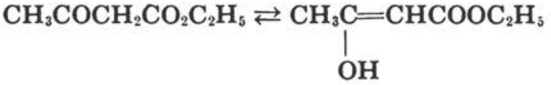
Functional use in foods. Flavoring agent.

Ethyl Acetoacetate

Insert the following new monograph to precede the monograph entitled *Ethyl Anisate*, page 247:

ETHYL ACETOACETATE

Acetoacetic Ester; Ethyl 3-Oxobutanoate



$$CH_3COCH_2CO_2C_2H_5 \rightleftarrows CH_3\underset{\underset{\textstyle OH}{|}}{C}{=}CHCOOC_2H_5$$

$C_5H_{10}O_3$ Mol. wt. 130.14

DESCRIPTION

An equilibrium mixture of the keto- and enol-forms of ethyl acetoacetate. It is a clear, colorless to very light yellow, mobile liquid having a characteristic, agreeable odor. It is miscible in all proportions with alcohol, with ether, and with ethyl acetate. One ml. dissolves in about 12 ml. of water.

SPECIFICATIONS

Assay. Not less than 97.5 per cent of $C_6H_{10}O_3$.

Refractive index. Between 1.4180 and 1.4205 at 20°.

Specific gravity. Between 1.022 and 1.027.

Limits of Impurities

Acidity (as CH_3COOH). Not more than 0.2 per cent.

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

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

Ignition residue. Not more than 0.01 per cent.

TESTS

Assay. Introduce 50 ml. of freshly distilled pyridine into a 250-ml. Erlenmeyer flask, and add about 450 mg. of the sample, accurately weighed. Stopper the flask, and swirl the mixture to effect complete solution. Add a few drops of thymolphthalein T.S., and titrate with 0.1 *N* sodium methoxide in pyridine to the first appearance of a blue end-point. Perform a blank determination (see page 2). During the titration direct a gentle stream of nitrogen into the flask through a short piece of 6-mm. glass tubing attached near the tip of the buret. Each ml. of 0.1 *N* sodium methoxide in pyridine is equivalent to 13.01 mg. of $C_6H_{10}O_3$.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 4).

Acidity. Transfer 10 ml. of water, recently boiled and then cooled to about 5°, into a 250-ml. Erlenmeyer flask containing 50 ml. of alcohol, add about 0.5 ml. of bromocresol purple T.S., and neutralize the mixture with 0.1 *N* sodium hydroxide to the appearance of a blue end-point. Introduce 24.0 ml. of the sample, previously cooled to about 5°, into the flask, and titrate with 0.1 *N* sodium hydroxide to a blue end-point that persists for at least 30 seconds. Not more than 8.3 ml. is required.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 720.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Ignition residue. Transfer a 50-gram sample into a tared 125-ml. platinum dish, heat until the vapors are ignited, withdraw the flame, protect the combustion from drafts, and allow the vapors to continue to burn spontaneously. Transfer the dish into a muffle furnace maintained at about 900° until all carbonaceous material has been removed, then cool in a desiccator, and weigh.

Packaging and storage. Store in tight, light-resistant containers.

Functional use in foods. Flavoring agent.

Eucalyptus Oil, page 269

In the paragraph entitled *Assay*, second line on top of page 270, change "crystallizatiln" to read:

. . . crystallization . . .

In the paragraph entitled *Assay*, third line on top of page 270, change "Stire" to read:

. . . Stir . . .

Ferric Phosphate, page 273

Insert the following new SPECIFICATION (*Limits of Impurities*), page 274, for *Arsenic:*

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

Insert the following new paragraph to precede the paragraph entitled *Fluoride*, page 274:

Arsenic. Assemble the special distillation apparatus as shown in Fig. 3a on page 25 of the *First Supplement.* Transfer 2 grams of the sample, 50 ml. of hydrochloric acid, and 5 grams of cuprous chloride into the distilling flask (*B*). Reassemble the distillation apparatus and apply gentle suction to flask *F* to produce a continuous stream of bubbles. Heat the solution in flask *B* to boiling and distil until between 30 and 35 ml. of distillate has been collected in flask *D*. Quantitatively transfer the distillate to a 100-ml. volumetric flask with the aid of water, dilute to volume with water, and mix (*Sample Solution*). Prepare *Standard* and *Blank Solutions* in the same manner, using 6.0 ml. of *Standard Arsenic Solution* (page 720) in place of the sample in the *Standard Solution*, and 6.0 ml. of water in the *Blank Solution*. Transfer 50.0 ml. of the *Sample Solution* into the generator flask (Fig.

2, page 720), add 2 ml. of potassium iodide solution (15 in 100), and continue as directed in the *Procedure* under *Arsenic Test,* page 720, beginning with "(add) 0.5 ml. of *Stannous Chloride Solution,* and mix." Modify the *Procedure* by using 5.0 grams of Devarda's metal in place of the 3.0 grams of 20-mesh granular zinc, and maintain the temperature of the reaction mixture in the generator flask between 25° and 27°. Treat 50.0 ml. each of the *Standard Solution* and of the *Blank Solution* in the same manner and under the same conditions. Determine the absorbance at 525 mμ produced by each solution as directed under *Procedure.* Calculate the arsenic content (in parts per million) of the sample by the formula $3 \times (A_U - A_B)/(A_S - A_B)$, in which A_U is the absorbance produced by the *Sample Solution,* A_S is the absorbance produced by the *Standard Solution,* and A_B is the absorbance produced by the *Blank Solution.* [*Note*—If A_B exceeds 0.300, different samples of reagent grade cuprous chloride and Devarda's metal should be tested for arsenic content by the procedure described herein, and lots of these reagents should be selected which will give blank readings that do not exceed 0.300.]

Ferric Pyrophosphate, page 276

Change the paragraph entitled *Arsenic,* page 277, to read:

Arsenic. Prepare and test a 2-gram sample as directed in the test for *Arsenic* under *Ferric Phosphate,* page 11 of the *First Supplement.*

Ferrous Sulfate, Dried, page 285

Change the first sentence of the DESCRIPTION to read:

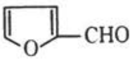
A grayish white to buff-colored powder . . .

Furfural

Insert the following new monograph to precede the monograph entitled *Garlic Oil*, page 292:

FURFURAL

2-Furaldehyde; Pyromucic Aldehyde



$C_5H_4O_2$ Mol. wt. 96.09

DESCRIPTION

Colorless to yellow oily liquid, which turns to reddish brown on prolonged storage. Odor typical of cyclic aldehydes. It is soluble in water and miscible with alcohol.

SPECIFICATIONS

Assay. Not less than 96 per cent of $C_5H_4O_2$.

Refractive index. Between 1.5220 and 1.5275 at 20°.

Specific gravity. Between 1.154 and 1.158.

Limits of Impurities

Acid value. Not more than 1.

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

TESTS

Assay. Weigh accurately about 1.5 grams, and proceed as directed under *Aldehydes*, page 741, using 48.05 as the equivalence factor (*E*) in the calculation.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 4).

Acid value. Determine as directed in the general method, page 740.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in tight containers, preferably glass, protected from light. Any material which has been stored for a prolonged period should be redistilled.

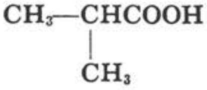
Functional use in foods. Flavoring agent.

Isobutyric Acid

Insert the following new monograph to precede the monograph entitled *Isoeugenol*, page 353:

ISOBUTYRIC ACID

2-Methyl Propanoic Acid; Isopropylformic Acid



$$\begin{array}{l} CH_3\text{—}CHCOOH \\ \qquad\quad | \\ \qquad\; CH_3 \end{array}$$

$C_4H_8O_2$ Mol. wt. 88.11

DESCRIPTION

Isobutyric acid is a colorless liquid having a strong, penetrating odor suggestive of rancid butter. It is miscible with alcohol, propylene glycol, glycerin, mineral oil, and most fixed oils. It is soluble in water.

SPECIFICATIONS

Assay. Not less than 99 per cent and not more than the equivalent of 101 per cent of $C_4H_8O_2$.

Refractive index. Between 1.3920 and 1.3945 at 20°.

Specific gravity. Between 0.944 and 0.948.

Limits of Impurities

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

Reducing substances. Passes test.

TESTS

Assay. Transfer about 1.5 grams, accurately weighed, into a 250-ml. Erlenmeyer flask containing about 75 ml. of water, add phenolphthalein T.S., and titrate with 0.5 *N* sodium hydroxide. Each ml. of 0.5 *N* sodium hydroxide is equivalent to 44.06 mg. of $C_4H_8O_2$.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Specific gravity. Determine by any reliable method (see page 4).

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Reducing substances. Dilute 2 ml. in a glass-stoppered flask with 50 ml. of water and 50 ml. of sulfuric acid, shaking the flask during the addition. While the solution is still warm, titrate with 0.1 *N* potassium permanganate. Not more than 1 ml. is required to produce a pink color that persists for 1 minute.

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

Functional use in foods. Flavoring agent.

L-Lysine Monohydrochloride

Insert the following new monograph to precede the monograph entitled *Mace Oil*, page 395:

L-LYSINE MONOHYDROCHLORIDE

2,6-Diaminohexanoic Acid Hydrochloride

$NH_2(CH_2)_4CH(NH_2)COOH.HCl$

$C_6H_{14}N_2O_2.HCl$ Mol. wt. 182.65

DESCRIPTION

A white or nearly white, practically odorless, free-flowing, crystalline powder. It is freely soluble in water, but is almost insoluble in alcohol and in ether. It melts at about 255°.

IDENTIFICATION

A. Heat 5 ml. of a 1 in 100 solution with 1 ml. of ninhydrin T.S. A violet color is produced.

B. A 1 in 20 solution gives positive tests for *Chloride*, page 769.

SPECIFICATIONS

Assay. Not less than 98 per cent of $C_6H_{14}N_2O_2.HCl$, calculated on the dried basis.

Specific rotation, $[\alpha]_D^{25°}$. Between +25.0° and +26.0°.

Limits of Impurities

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

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

Loss on drying. Not more than 0.6 per cent.

Residue on ignition. Not more than 0.1 per cent.

TESTS

Assay. Transfer about 275 mg., accurately weighed, into a 150-ml. beaker, and dissolve in 10 ml. of mercuric acetate T.S., heating on a steam bath to effect solution. Cool, add 75 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 $C_6H_{14}N_2O_2.HCl$.

Specific rotation, page 780. Determine in a solution containing 1.25 grams of sample in sufficient 6 *N* hydrochloric acid to make 50 ml.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test,* page 720.

Heavy metals. Prepare and test a 2-gram sample as directed in *Method II* under the *Heavy Metals Test,* page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Loss on drying, page 774. Dry in vacuum at 60° for 4 hours.

Residue on ignition. Ignite 1 gram as directed in the general method, page 786.

Packaging and storage. Store in well-closed containers.

Functional use in foods. Nutrient; dietary supplement.

Manganese Sulfate

Insert the following new monograph to precede the monograph entitled *Mannitol,* page 419:

MANGANESE SULFATE

$MnSO_4.H_2O$ Mol. wt. 169.02

DESCRIPTION

A pale pink, granular, odorless powder. It is freely soluble in water and is insoluble in alcohol.

IDENTIFICATION

A 1 in 10 solution gives positive tests for *Manganese,* page 771, and for *Sulfate,* page 771.

SPECIFICATIONS

Assay. Not less than 98 per cent and not more than the equivalent of 102 per cent of $MnSO_4.H_2O$.

Loss on ignition. Between 10 and 13 per cent.

Limits of Impurities

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

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

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

TESTS

Assay. Transfer about 4 grams, accurately weighed, into a 250-ml. volumetric flask, dissolve in water, dilute to volume with water, and mix. Transfer a 25.0-ml. portion of this solution into a 400-ml. beaker, and add 10 ml. of a 1 in 10 solution of hydroxylamine hydrochloride, 25 ml. of 0.05 *M* disodium ethylenediaminetetraacetate measured from a buret, 25 ml. of ammonia-ammonium chloride buffer T.S., and 5 drops of eriochrome black T.S. Heat the solution to between 55° and 65°, and titrate from the buret to a blue end-point. Each ml. of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 8.451 mg. of $MnSO_4.H_2O$.

Loss on heating. Heat about 1 gram, accurately weighed, in a crucible tared in a stoppered weighing bottle, to constant weight at 400°–500°. Cool in a desiccator, transfer to the stoppered weighing bottle, and weigh.

Arsenic. A solution of 1 gram in 35 ml. of water meets the requirements of the *Arsenic Test*, page 720.

Heavy metals. A solution of 500 mg. in 25 ml. of water meets the requirements of the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Lead. Dissolve 1 gram in 3 ml. of dilute nitric acid (1 in 2) and 10 ml. of water, and boil for 2 minutes. Cool, and dilute to 100 ml. with water. A 25-ml. portion of this solution meets the requirements of the *Lead Limit Test*, page 772, using 25 ml. of *Ammonium Citrate Solution*, 1 ml. of *Potassium Cyanide Solution*, 0.5 ml. of *Hydroxylamine Hydrochloride Solution*, and 2.5 mcg. of lead ion (Pb).

Packaging and storage. Store in well-closed containers.

Functional use in foods. Nutrient; dietary supplement.

DL-Methionine and L-Methionine

Insert the following two new monographs to precede the monograph entitled *Methyl Acetophenone*, page 424:

DL-METHIONINE

α-Amino-γ-methylmercaptobutyric Acid

$$CH_3SCH_2CH_2\underset{\displaystyle NH_2}{\underset{|}{C}}HCOOH$$

$C_5H_{11}NO_2S$ Mol. wt. 149.21

DESCRIPTION

White, crystalline platelets or powder having a characteristic odor. One gram dissolves in about 30 ml. of water. It is soluble in dilute acids and in solutions of alkali hydroxides. It is very slightly soluble in alcohol, and practically insoluble in ether. It is optically inactive. The pH of a 1 in 100 solution is between 5.6 and 6.1.

IDENTIFICATION

A. Add 25 mg. of a previously dried sample to 1 ml. of a saturated solution of anhydrous cupric sulfate in sulfuric acid. A yellow color appears.

B. To 10 ml. of a 1 in 1000 solution add successively, shaking after each addition, 1 ml. of a 1 in 5 solution of sodium hydroxide, 1 ml. of a 1 in 100 glycine solution, and 0.3 ml. of a freshly prepared 1 in 10 solution of sodium nitroferricyanide. Keep the mixture at about 40° for 10 minutes, cool in an ice bath for 2 minutes, then add 2 ml. of 20 per cent hydrochloric acid and shake the mixture. A reddish-purple color appears.

C. To 1 ml. of a 1 in 30 solution add 1 ml. of ninhydrin T.S. and 100 mg. of sodium acetate, and heat to boiling. An intense violet-blue color is formed (distinction from hydroxy analog).

SPECIFICATIONS

Assay. Not less than 99 per cent of $C_5H_{11}NO_2S$, calculated on the dried basis.

Limits of Impurities

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

Heavy metals (as Pb). Not more than 20 parts per million (0.002 per cent).

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

Loss on drying. Not more than 0.5 per cent.

Residue on ignition. Not more than 0.1 per cent.

TESTS

Assay. Transfer about 300 mg., accurately weighed, into a glass-stoppered flask. Add 100 ml. of water, 5 grams of dibasic potassium phosphate, 2 grams of monobasic potassium phosphate, and 2 grams of potassium iodide, and mix well to dissolve. Add exactly 50 ml. of 0.1 *N* iodine, stopper the flask, mix well, allow to stand for 30 minutes, and then titrate the excess iodine with 0.1 *N* sodium thiosulfate. Perform a blank determination (see page 2) and make any necessary correction. Each ml. of 0.1 *N* iodine is equivalent to 7.461 mg. of $C_5H_{11}NO_2S$.

Arsenic. A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 720.

Heavy metals. Prepare and test a 1-gram sample as directed in *Method II* under the *Heavy Metals Test*, page 763, 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 772, using 10 mcg. of lead ion (Pb) in the control.

Loss on drying, page 774. Dry at 105° for 2 hours.

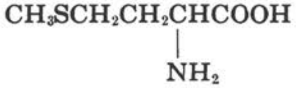
Residue on ignition, page 786. Ignite 1 gram as directed in the general method.

Packaging and storage. Store in well-closed, light-resistant containers.

Functional use in foods. Nutrient; dietary supplement.

L-METHIONINE

α-Amino-γ-methylmercaptobutyric Acid



$$CH_3SCH_2CH_2\underset{\displaystyle NH_2}{\underset{|}{C}}HCOOH$$

$C_5H_{11}NO_2S$ Mol. wt. 149.21

DESCRIPTION

Colorless or white lustrous plates, or a white crystalline powder. It has a slight, characteristic odor. It is soluble in water, in alkali solutions, and in dilute mineral acids. It is slightly soluble in alcohol and practically insoluble in ether.

IDENTIFICATION

L-Methionine responds to *Identification Tests A*, *B* and *C* under DL-*Methionine*, page 17 (*First Supplement*).

SPECIFICATIONS

Assay. Not less than 99 per cent of $C_5H_{11}NO_2S$, calculated on the dried basis.

Specific rotation, $[\alpha]_D^{25^\circ}$. Between −6.8° and −8.2°.

Limits of Impurities

L-Methionine complies with the tolerance limits specified for **Arsenic, Heavy metals, Lead, Loss on drying,** and **Residue on ignition** in the monograph for DL-*Methionine*, page 17 (*First Supplement*).

TESTS

Assay. Proceed as directed in the *Assay* under DL-*Methionine*, page 17 (*First Supplement*).

Specific rotation, page 780. Determine in a solution containing 1 gram of a previously dried sample in sufficient water to make 25 ml.

Arsenic, Heavy metals, Lead, Loss on drying, and **Residue on ignition.** Determine as directed in the monograph for DL-*Methionine*, page 17 (*First Supplement*).

Packaging and storage. Store in well-closed, light-resistant containers.

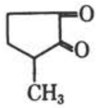
Functional use in foods. Nutrient; dietary supplement.

Methyl Cyclopentenolone

Insert the following new monograph to precede the monograph entitled *Methyl Ethylcellulose*, page 436:

METHYL CYCLOPENTENOLONE

3-Methylcyclopentane-1,2-dione



$C_6H_8O_2$ Mol. wt. 112.13

DESCRIPTION

A white crystalline powder with a characteristic nutty odor, and suggestive of a maple-licorice aroma in dilute solution. Soluble in alcohol and in propylene glycol; slightly soluble in most fixed oils. One part dissolves in about 72 parts water.

SPECIFICATIONS

Melting range. Between 104° and 108°.

Solubility in alcohol. Passes test.

Limits of Impurities

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

TESTS

Melting range. Determine as directed in the general procedure, page 775.

Solubility in alcohol. Proceed as directed in the general method, page 746. One gram dissolves in 5 ml. of 90 per cent alcohol.

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test*, page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in tight containers. Avoid contact with iron.

Functional use in foods. Flavoring agent.

Mono- and Diglycerides, page 452

Change the paragraph entitled *Residue on ignition*, page 453, to read:

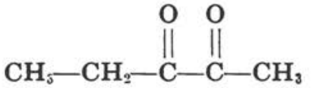
Residue on ignition. Ignite 5 grams as directed in the general method, page 786.

2,3-Pentanedione

Insert the following new monograph to precede the monograph entitled *Peppermint Oil*, page 493:

2,3-PENTANEDIONE

Acetyl Propionyl



$$CH_3{-}CH_2{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}CH_3$$

$C_5H_8O_2$ Mol. wt. 100.12

DESCRIPTION

A clear yellow to yellowish green liquid with a strong, pungent odor, which becomes typically buttery upon dilution. It is miscible with alcohol, with propylene glycol, and with fixed oils. It is soluble in mineral oil, but practically insoluble in glycerin and water.

SPECIFICATIONS

Assay. Not less than 93 per cent of $C_5H_8O_2$.

Refractive index. Between 1.4020 and 1.4060 at 20°.

Solubility in alcohol. Passes test.

Specific gravity. Between 0.952 and 0.962.

Limits of Impurities

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

TESTS

Assay. Weigh accurately about 400 mg., and proceed as directed under *Aldehydes and Ketones-Hydroxylamine Method*, page 741, using 50.06 as the equivalence factor (E) in the calculation.

Refractive index, page 785. Determine with an Abbé or other refractometer of equal or greater accuracy.

Solubility in alcohol. Proceed as directed in the general method, page 746. One ml. dissolves in 3 ml. of 50 per cent alcohol.

Specific gravity. Determine by any reliable method (see page 4).

Heavy metals. Prepare and test a 500-mg. sample as directed in *Method II* under the *Heavy Metals Test,* page 763, using 20 mcg. of lead ion (Pb) in the control (*Solution A*).

Packaging and storage. Store in full, tight containers, preferably glass, aluminum, tin, or tin-lined, in a cool place protected from light.

Functional use in foods. Flavoring agent.

Propyl Gallate, page 568

Change the SPECIFICATION for *Melting range* to read:

Melting range. Between 146° and 150°.

Sodium Nitrate, page 630

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

Total chlorine. Not more than 0.2 per cent.

Change the paragraph entitled *Total chlorine*, page 631, to read:

Total chlorine. Dissolve 1 gram in 100 ml. of water, add enough 6 per cent sulfurous acid to give the solution a distinct odor of sulfur dioxide, boil gently until the odor of the sulfur dioxide is no longer apparent, and adjust the volume to 100 ml. by the addition of water. Add 1.0 ml. of 0.1 *N* silver nitrate followed by 3 ml. of nitric acid and 3 ml. of nitrobenzene, and shake vigorously. Add ferric ammonium sulfate T.S., and titrate the excess silver nitrate with 0.1 *N* ammonium thiocyanate. No more than 0.6 ml. of the 0.1 *N* silver nitrate is consumed.

Sodium Stearyl Fumarate, page 646

Change the paragraph entitled *Assay* to read:

Assay. Transfer about 250 mg., accurately weighed, into a 50-ml. Erlenmeyer flask, mix it with 1 ml. of chloroform, and add 20 ml. of glacial acetic acid to dissolve the sample. Add quinaldine red T.S., and titrate with 0.1 *N* perchloric acid in glacial acetic acid. Each ml. of 0.1 *N* perchloric acid is equivalent to 39.05 mg. of $C_{22}H_{39}NaO_4$.

Change the paragraph entitled *Saponification value* to read:

Saponification value. Transfer about 450 mg. of sodium stearyl fumarate, accurately weighed, into a 300-ml. Erlenmeyer flask, and add 50.0 ml. of ethanolic potassium hydroxide solution, rinsing down the inside of the flask during the addition. [Prepare the ethanolic potassium hydroxide solution as follows: dissolve about 5.5 grams of potassium hydroxide in absolute ethanol, heating if necessary to effect solution, and dilute to 1000 ml. with absolute ethanol. Prepare fresh daily, and filter if necessary to remove carbonate.] Reflux the mixture gently on a steam bath for at least 2 hours, swirling gently occasionally but avoid splashing the mixture up into the condenser. Rinse the condenser with 10 ml. of 70 per cent alcohol, followed by three 10-ml. portions of water, collecting the rinsings in the flask. Cool, rinse the sides of the flask with two 10-ml. portions of 70 per cent alcohol, add phenolphthalein T.S., and titrate with 0.1 *N* hydrochloric acid to the disappearance of any pink color. Perform a blank determination using the same amount of the ethanolic potassium hydroxide solution (see page 2). Calculate the saponification value by the formula $56.1 (B - S) \times N/W$, in which $B - S$ represents the difference between the volumes of 0.1 *N* hydrochloric acid required for the blank and the sample, respectively, *N* is the exact normality of the hydrochloric acid, and *W* is the weight, in grams, of the sample taken.

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dl-Alpha Tocopherol, page 686

Change the second sentence of the DESCRIPTION to read:

. . . It occurs as a yellow to amber, nearly odorless . . .

In *Identification test B*, change "between 71 and 76" to read:

. . . between 7.1 and 7.6.

Change the SPECIFICATION for *Assay* to read:

Assay. Not less than 97 per cent of $C_{29}H_{50}O_2$.

d-Alpha Tocopheryl Acetate, page 690

In *Identification test B*, change "between 40 and 44" to read:

. . . between 4.0 and 4.4.

Change the SPECIFICATION for *Assay* to read:

Assay. Not less than 97 per cent of $C_{31}H_{52}O_3$.

dl-Alpha Tocopheryl Acetate, page 691

In *Identification test B*, page 692, change "between 40 and 44" to read:

. . . between 4.0 and 4.4.

Change the SPECIFICATION for *Assay*, page 692, to read:

Assay. Not less than 97 per cent of $C_{31}H_{52}O_3$.

d-Alpha Tocopheryl Acid Succinate, page 695

In *Identification test B*, page 696, change "between 35 and 40" to read:

. . . between 3.5 and 4.0.

Change the SPECIFICATION for *Assay*, page 696, to read:

Assay. Not less than 97 per cent of $C_{33}H_{54}O_5$.

GENERAL TESTS AND APPARATUS

Arsenic Test, page 720

Insert the following new figure to precede the paragraph entitled *Interferences*, page 722:

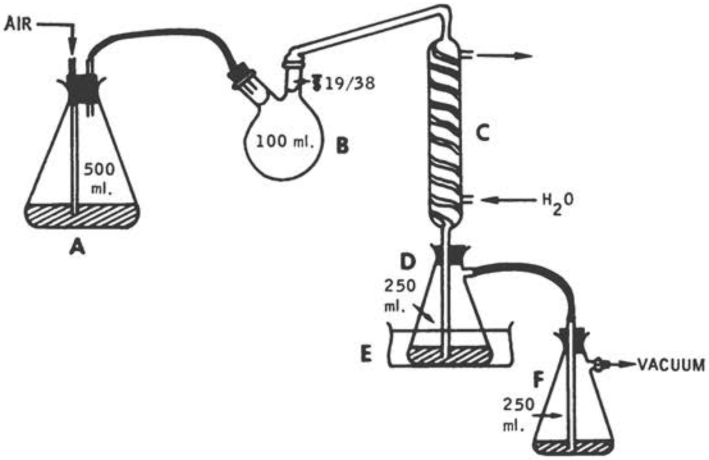

Fig. 3a—Special Apparatus for the Distillation of Arsenic Trichloride. Flask *A* contains 150 ml. of hydrochloric acid; flasks *D* and *F* contain 20 ml. of water. Flask *D* is placed in an ice water bath, *E*.

Note—This special apparatus is to be used only when specified in certain monographs.

Oxyethylene Determination, page 756

In the paragraph entitled *Procedure*, fourth line on top of page 758, change "tube *D*" to read:

. . . tube *C* . . .

Heavy Metals Test, page 763

In the third line of the paragraph entitled *Method II* under *Procedure*, page 764, change "nitric acid" to read:

. . . sulfuric acid . . .

TEST SOLUTIONS

Insert the following new test solution to precede *Sodium Carbonate T.S.*, page 819:

Sodium Borate T.S. Dissolve 2 grams of sodium borate, $Na_2B_4O_7.10H_2O$, in water to make 100 ml.

VOLUMETRIC SOLUTIONS

Insert the following new volumetric solution to precede *Sodium Nitrite, 0.1 M*, page 826:

Sodium Methoxide, 0.1 N, in Pyridine (5.40 grams CH_3ONa per liter). Weigh 14 grams of freshly cut sodium metal, and cut into small cubes. Place about 0.5 ml. of anhydrous methanol in a round-bottom 250-ml. flask equipped with a ground-glass joint, add 1 cube of the sodium metal, and, when the reaction subsides, add the remaining sodium metal to the flask. Connect a water-cooled condenser to the flask, and slowly add 100 ml. of anhydrous methanol, in small portions, through the top of the condenser. Regulate the addition of the methanol so that the vapors are condensed and do not escape through the top of the condenser. After addition of the methanol is complete, connect a drying tube to the top of the condenser, and allow the solution to cool. Transfer 17.5 ml. of this solution (approximately 6 *N*) into a 1000-ml. volumetric flask containing 70 ml. of anhydrous methanol, and dilute to volume with freshly distilled pyridine. Store preferably in the reservoir of an automatic buret suitably protected from carbon dioxide and moisture. Standardize the solution as follows: Weigh accurately about 400 mg. of primary standard benzoic acid, transfer it into a 250-ml. wide-mouth Erlenmeyer flask, and dissolve it in 50 ml. of freshly distilled pyridine. Add a few drops of thymolphthalein T.S., and titrate immediately with the sodium methoxide solution to a blue end-point. During the titration, direct a gentle stream of nitrogen into the flask through a short piece of 6-mm. glass tubing fastened near the tip of the buret. Perform a blank determination (see page 2), correct for the volume of sodium methoxide solution consumed by the blank, and calculate the normality. Each 12.21 mg. of benzoic acid is equivalent to 1 ml. of 0.1 *N* sodium methoxide in pyridine.