USP 41

Reagents / Reagent Specifications 5697

[NOTE—A suitable grade can be obtained from Roche Diagnostics Corporation, www.roche-diagnostics.com.]

Fluorene,  $C_{13}H_{10}$ —166.22 [86-73-7]—White to off-white crystals or powder. Soluble in benzene, in carbon disulfide, in ether, and in hot alcohol; freely soluble in glacial acetic acid.

**SOLUBILITY TEST:** One g dissolves in 10 mL of acetone to yield a clear and complete solution.

MELTING RANGE (741): between 113° and 117°, within a 2° range.

**9-Fluorenylmethyl Chloroformate**, C<sub>15</sub>H<sub>11</sub>ClO<sub>2</sub>—**258.70** [28920-43-6]—Clear, colorless solid. Melts at about 62°.

Fluorescamine,  $C_{17}H_{10}O_4$ —278.26 [38183-12-9]—White to off-white powder. Very slightly soluble in water; freely soluble in methylene chloride; soluble in alcohol; slightly soluble in chloroform.

Assay: Dissolve about 600 mg in 75 mL of dimethylformamide, and titrate with 0.1 N lithium methoxide to a blue endpoint, using 1% thymol blue in dimethylformamide as the indicator. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N lithium methoxide is equivalent to 27.83 mg of  $C_{12}H_{10}O_4$ . Not less than 99% is found.

Loss on DRYING (731): Dry it at 105° for 4 hours: it loses not more than 0.5% of its weight.

4'-Fluoroacetophenone, FC<sub>6</sub>H<sub>4</sub>COCH<sub>3</sub>—138.14 [403-42-9]—Colorless liquid.

ASSAY: Inject an appropriate specimen into a suitable gas chromatograph (see *Chromatography* (621)) equipped with a flame-ionization detector, helium being used as the carrier gas. The following conditions have been found suitable: a 25-mm  $\times$  30-m capillary column coated with a 1-µm layer of phase G2; the injection port temperature is maintained at 200°; the detector temperature is maintained at 250°; the column temperature is maintained at 100° and programmed to rise 10° per minute to 250°. The area of the FC<sub>6</sub>H<sub>4</sub>COCH<sub>3</sub> peak is not less than 99% of the total peak area.

REFRACTIVE INDEX (831): 1.510 at 20°.

Formaldehyde Solution, HCHO—(30.03) and water—Use ACS reagent grade.

Formamide, HCONH<sub>2</sub>—45.04 [75-12-7]—Use ACS reagent grade.

**PREPARATION FOR DIGITOXIN ASSAY:** To ensure freedom from ammonia, treat Formamide as follows. Shake a suitable quantity of formamide with about 10% of its weight of anhydrous potassium carbonate for 15 minutes, and filter. Distill the filtrate in an all-glass apparatus under vacuum at a pressure of about 25 mm of mercury or less. Reject the first portion of distillate containing water, and collect the fraction that boils at about 115° at a pressure of 25 mm of mercury. Store in tight containers, protected from light.

Formamide, Anhydrous, HCONH<sub>2</sub>—45.04 [75-12-7]— Use formamide that has a water content of less than 0.1%.

Formic Acid, HCOOH—46.03 [64-18-6]—Use ACS reagent grade Formic Acid, 88 Percent.

Formic Acid, 96 Percent, HCOOH—46.03 [64-18-6]— Use ACS reagent grade Formic Acid, 96 Percent.

Formic Acid, 98 Percent, HCOOH—46.03 [64-18-6]— Use a suitable grade with a content of NLT 98%.

Formic Acid, Anhydrous—Use ACS reagent grade Formic Acid, 96 Percent.

Fuchsin, Basic (Basic Red 9, Parafuchsin Hydrochloride),  $C_{19}H_{17}N_3 \cdot HCI$ —323.82 [569-61-9]—Use a suitable grade.

Fuller's Earth, Chromatographic—(Very Fine and Moderately Coarse)—Gray or grayish-white powder or granules consisting mainly of hydrous aluminum-magnesium silicate.

POWDER FINENESS: see Powder Fineness (811)

SOLUBLE MATTER: Twenty g, treated with 50 mL of cold water and filtered, yields not more than 60 mg of residue upon evaporation of the filtrate (0.3%). A second 20-g portion, treated with 50 mL of cold alcohol and filtered, yields not more than 14 mg upon evaporation of the filtrate (0.07%).

Loss ON DRYING (731): Dry it at 105° for 6 hours: it loses between 7.0% and 10.0% of its weight. [NOTE—Adjust the water content, if necessary, by drying

[NOTE—Adjust the water content, if necessary, by drying in vacuum at room temperature, restoring the water required, and equilibrating by shaking for 2 hours.]

Fuming Nitric Acid—See Nitric Acid, Fuming.

Fuming Sulfuric Acid—See Sulfuric Acid, Fuming.

**Furfural** (2-Furancarboxyaldehyde; 2-Furaldehyde), C₄H<sub>3</sub>OCHO**—96.08** [98-01-1]—Use ACS reagent grade.

**G Designations**—See phases for gas chromatography under *Reagents, Chromatographic Columns.* 

Gadolinium (Gd III) Acetate Hydrate,  $(CH_3CO_2)_3Gd \cdot xH_2O$ —334.38 [100587-93-7]—White, crystalline, hygroscopic powder. Irritant. Use a suitable grade.

**Gadolinium Sulfate**,  $Gd_2(SO_4)_3 \cdot 8H_2O$ —746.83 [13628-54-1]—Use a suitable grade with a content of NLT 99.9%. [NOTE—A suitable grade is available as catalog number 41111 from www.gfschemicals.com.]

## Add the following:

▲Gelatin—Use Gelatin (NF monograph). ▲USP41

Geneticin (G418; O-2-Amino-2,7-dideoxy-D-glycero-alpha-Dglucoheptopyranosyl-(1-4)-O-(3-deoxy-4-C-methyl-3-(methylamino)-beta-L-arabinopyranosyl-(1-6)-D-streptamine),  $C_{20}H_{40}N_4O_{10}$ —496.55 [49863-47-0]—Use a suitable grade, cell culture tested.

Girard Reagent T—See Trimethylacethydrazide Ammonium Chloride.

**Gitoxin**,  $C_{41}H_{64}O_{14}$ —**780.94** [4562-36-1]—White, crystalline powder. Practically insoluble in water, in chloroform, and in ether; slightly soluble in pyridine and in diluted alcohol. Melts at about 250°, with decomposition.

SPECIFIC ROTATION (781): between +3.8° and +4.8°, determined in a solution of pyridine containing 10 mg per mL, with the use of a mercury light at 546.1 nm; between +21° and +25°, determined in a solution of equal parts of chloroform and methanol containing 5 mg per mL, with the use of sodium light.

SUITABILITY: Dissolve 10 mg each of USP Digitoxin RS, previously dried, USP Digoxin RS, previously dried, and gitoxin, respectively, in separate 5-mL portions of a mixture of 2 parts of chloroform and 1 part of methanol, and dilute each with additional solvent mixture to 10 mL. Then proceed as directed in the *Identification test* under *Digoxin*. The chromatogram of gitoxin shows one fluorescent spot, located between the digoxin and digitoxin spots.

Glacial Acetic Acid—See Acetic Acid, Glacial.