

mixture distilled in a current of steam until the distillate is no longer alkaline (about 500 ml). The distillate is then titrated with 0.1 N H₂SO₄ using rosolic acid or methyl red as indicator. Not less than 23.2 ml should be required for neutralization.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.120 Nitropropane, mixed isomers of.

(a) *Nitropropane content.* A minimum of 94 percent by weight.

(b) *Total nitroparaffin content.* A minimum of 99 percent by weight.

(c) *Distillation range.* 119° to 113 °C.

(d) *Specific gravity at 20°/20 °C.* 0.992 to 1.003.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.121 Phenyl mercuric benzoate.

(a) *Assay (as phenyl mercuric benzoate).* Not less than 99.0 percent by weight.

(b) *Melting point.* Not less than 94 °C.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.122 Pyridine bases.

(a) *Alkalinity.* One ml of pyridine bases dissolved in 10 ml of water is titrated with 1 N H₂SO₄ until a drop of the mixture placed upon Congo paper shows a distinct blue border, which soon disappears. A minimum of 9.5 ml of the acid must be required for the end point. (Congo paper: filter paper treated with 0.1 percent aqueous solution of Congo red and dried.)

(b) *Distillation range.* One hundred ml of the denaturant are distilled in the following manner: The sample is placed in a short-necked glass flask of about 200 ml capacity which is rested on an asbestos plate having a circular opening of 30 mm in diameter. The neck of this flask is fitted with a fractionating tube 12 mm in diameter and 170 mm long and having a bulb just 1 cm below the side tube which is connected with a Liebig condenser having a water jacket not less than 400 mm in length. A standardized thermometer is placed in

the fractionating tube so that the mercury bulb is suspended in the center of the fractionating bulb. Heat is applied slowly and in such manner that 5 ml of distillate is collected per minute in a graduated cylinder. At least 50 ml must distill at or below 140 °C. and at least 90 ml below 160 °C.

(c) *Reactions.* Dissolve 1 ml of pyridine bases in 100 ml of water.

(1) Ten ml of this solution are treated with 5 ml of 5 percent aqueous solution of anhydrous fused CaCl₂ and the mixture vigorously shaken. An abundant crystalline separation should occur within 10 minutes.

(2) Ten ml of the pyridine solution mixed with 50 ml of Nessler's reagent must give a white precipitate.

(d) *Water content.* Twenty ml of pyridine bases are shaken with 20 ml of a caustic soda solution having a specific gravity of 1.40 (15.56 °/15.56 °C.) and the mixture allowed to stand until completely separated into two layers. The amount of pyridine base layer should be 18.5 ml, minimum.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.123 Pyronate.

Pyronate is a product of the destructive distillation of hardwood meeting the following requirements:

(a) *Acidity (as acetic acid).* Not more than 0.1 percent by weight, determined as follows:

Add 5.0 ml sample to 100 ml distilled water in an Erlenmeyer flask and titrate with 0.1 N NaOH to a bromthymol blue endpoint.

(b) *Color.* The color shall be no darker than the color produced by 2.0 grams of potassium dichromate in 1 liter of water. The comparison shall be made in 4-ounce oil sample bottles viewed crosswise.

(c) *Distillation range.* When 100 ml are distilled not more than 5 ml shall distill below 70 °C., not less than 50 ml below 160 °C., and not less than 90 ml below 205 °C.

NOTE. Any material submitted as pyronate must agree in color, odor, taste and denaturing value with a standard sample furnished by the Alcohol and Tobacco Tax and Trade

§ 21.124

Bureau to chemists authorized to examine samples of denaturants.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.124 Quassin.

(a) Quassin is the bitter principle of quassia wood (occurring as a mixture of two isomeric forms). It shall be a good commercial grade of purified amorphous quassin, standardized as to bitterness.

(b) *Bitterness*. An aqueous solution of quassin shall be distinctly bitter at a 1 to 250,000 dilution. To test: Dissolve 0.1 gram of quassin in 100 ml of 95 percent alcohol, then dilute 4 ml of the solution to 1,000 ml with distilled water, mix well and taste.

(c) *Identification test*. Dissolve about 0.5 gram of quassin in 10 ml of 95 percent alcohol and filter. To 5 ml of the filtrate, add 5 ml of concentrated hydrochloric acid and 1 mg of phloroglucinol and mix well. A red color develops.

(d) *Optical assay*. When 1 gram of quassin (in solution in a small amount of 95 percent alcohol) is dissolved in 10,000 ml of water, the absorbance of the solution in a 1 cm cell at a wavelength of 258 millimicrons shall not be less than 0.400.

(e) *Solubility*. When 0.5 gram of quassin is added to 25 ml of 190 proof alcohol, it shall dissolve completely.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.125 Rubber hydrocarbon solvent.

(a) Rubber hydrocarbon solvent is a petroleum derivative.

(b) *Distillation range*. When 10 percent of the sample has been distilled into a graduated receiver, the thermometer shall not read more than 170 °F. nor less than 90 °F. When 90 percent has been recovered in the receiver the thermometer shall not read more than 250 °F.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.126 Saffrole.

(a) *Congealing point*. 10.0° to 11.2 °C.

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(b) *Refractive index at 20 °C*. 1.5363 to 1.5385.

(c) *Specific gravity at 15 °/15 °C*. 1.100 to 1.107.

(d) *Odor*. Characteristic odor.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.127 Shellac (refined).

(a) *Arsenic content*. Not more than 1.4 parts per million as determined by the Gutzeit Method (AOAC method 25.020; for incorporation by reference, see § 21.6(c)).

(b) *Color*. White or orange.

(c) *Rosin content*. None when tested by the following method: Add 20 ml of absolute alcohol or glacial acetic acid (m. p. 13° to 15 °C.) to 2 grams of the shellac and thoroughly dissolve. Add 100 ml of petroleum ether and mix thoroughly. Add approximately 2 liters of water and separate a portion of the ether layer (at least 50 ml) and filter if cloudy. Evaporate the petroleum ether and test as follows: Solution A—5 ml of phenol dissolved in 10 ml of carbon tetrachloride. Solution B—1 ml of bromine dissolved in 4 ml of carbon tetrachloride. To the residue obtained above add 2 ml of Solution A and transfer the mixture to a porcelain spot plate, filling one cavity. Immediately fill an adjacent cavity with solution B. Cover the plate with a watch glass and observe any color formation in Solution A. A decided purple or deep indigo blue color is an indication of the presence of rosin.

[T.D. ATF-133, 48 FR 24673, June 2, 1983. Redesignated by T.D. ATF-442, 66 FR 12854, Mar. 1, 2001]

§ 21.128 Sodium (metallic).

(a) *Color*. Silvery-white (metallic luster) when freshly cut.

(b) *Identification test*. Clean a platinum wire by dipping it in concentrated hydrochloric acid and holding it over a Bunsen burner until the flame is no longer colored. Moisten the wire loop with hydrochloric acid and dip it into the sample. Hold the wire in the Bunsen flame and note the color. Sodium produces a golden yellow flame; not observed when viewed through a cobalt glass.