§ 21.97 Benzene.

(a) *Distillation range.* (For applicable ASTM method, see 1980 Annual Book of ASTM Standards, Part 29, page 573, Standard No. D 836–77; for incorporation by reference, see § 21.6(b).) When 100 ml of benzene are distilled by this method, not more than 1 ml should distill below 77 °C., and not less than 95 ml below 85 °C.

(b) *Odor.* Characteristic odor.

(c) *Specific gravity at 15.6 °/15.6 °C.* 0.875 to 0.886.

(d) *Water solubility.* When 10 ml of benzene are shaken with an equal volume of water in a glass-stoppered bottle, graduated to 0.1 ml, and allowed to stand 5 minutes to separate, the upper layer of liquid shall measure not less than 9.5 ml.

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

§ 21.98 Bone oil (Dipple's oil).

(a) *Color.* The color shall be a deep brown.

(b) *Distillation range.* When 100 ml are distilled in the manner described for pyridine bases, not more than 5.0 ml should distill below 90 °C.

(c) *Pyrrol reaction.* Prepare a 1.0 percent solution of bone oil in 95 percent alcohol. Prepare a second solution containing 0.025 percent bone oil by diluting 2.50 ml of the first solution to 100 ml with 95 percent alcohol. Dip a splinter of pine, previously moistened with concentrated hydrochloric acid, into 10 ml of the 0.025 percent bone oil solution. After a few minutes the splinter should show a distinct red coloration.

(d) *Reaction with mercuric chloride.* Add 5 ml of the 1.0 percent bone oil solution above to 5 ml of a 2 percent alcoholic solution of mercuric chloride. A turbidity is formed at once which separates into a flocculent precipitate on standing several minutes. Add 5.0 ml of the 0.025 percent bone oil solution to 5.0 ml of a 2.0 percent alcoholic solution of mercuric chloride. A faint turbidity appears after several minutes.

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

§ 21.99 Brucine alkaloid.

(a) *Identification test.* Add a few drops of concentrated nitric acid to about 10 mg of brucine alkaloid. A vivid red color is produced. Dilute the red solution with a few drops of water and add a few drops of freshly made dilute stannous chloride solution. A reddish purple (violet) color is produced.

(b) *Melting point.* 178 °±1 °C. Dry the alkaloid in an oven for one hour at 100 °C., increase the temperature to 110° and dry to a constant weight before taking melting point.

NOTE. Brucine alkaloid tetrahydrate melts at 105 °C. while the anhydrous form melts at 178 °C.

(c) *Strychnine test.* Brucine alkaloid shall be free of strychnine when tested by the method listed under Brucine Sulfate, N.F. IX.

NOTE. If the brucine contains as much as 0.05 percent strychnine, a clear distinctive violet color, characteristic of strychnine, will be obtained.

(d) *Sulfate test.* No white precipitate is formed that is not dissolved by hydrochloric acid when several drops of a 1 N barium chloride solution are added to 10 ml of a solution of the alkaloid.

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

§ 21.100 n-Butyl alcohol.

(a) *Acidity (as acetic acid).* 0.03 percent by weight maximum.

(b) *Color.* Colorless.

(c) *Dryness at 20 °C.* Miscible without turbidity with 10 volumes of 60° Bé1. gasoline.

(d) *Odor.* Characteristic odor.

(e) *Specific gravity at 20 °/20 °C.* 0.810 to 0.815.

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

§ 21.101 tert-Butyl alcohol.

(a) *Acidity (as acetic acid).* 0.003 percent by weight maximum.

(b) *Color.* Colorless.

(c) *Distillation range.* When 100 ml of tertiary butyl alcohol are distilled, none should distill below 78 °C. and none above 85 °C. More than 95 percent should distill between 81 °−83 °C.

(d) *Dryness at 20 °C.* Miscible without turbidity with 19 volumes of 60° Bé1. gasoline.

(e) *Freezing point (first needle).* Above 20 °C.

(f) *Identification test.* Place five drops of a solution containing approximately 0.1 percent tertiary butyl alcohol in ethyl alcohol in a test tube. Add 2 ml of Denige's reagent (dissolve 5 grams of red mercuric oxide in 20 ml of concentrated sulfuric acid; add this solution to 80 ml of distilled water, and filter when cool). Heat the mixture just to the boiling point and remove from the flame. A yellow precipitate forms within a few seconds.

(g) *Nonvolatile matter.* Less than 0.005 percent by weight.

(h) *Odor.* Characteristic odor.

(i) *Residual odor after evaporation.* None.

(j) *Specific gravity at 25 °/25 °C.* 0.780 to 0.786.

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

§ 21.102 Caustic soda, liquid.

(a) The liquid caustic soda may consist of either 50 percent or 73 percent by weight sodium hydroxide in aqueous solution. The amount of caustic soda used shall be such that each 100 gallons of alcohol will contain not less than 8.76 pounds of sodium hyroxide, anhydrous basis.

(b) *Color.* A 2 percent solution of the sodium hydroxide in water shall be water-white.

(c) *Assay.* The sodium hydroxide content of the caustic soda solution shall be determined by the following procedure:

Accurately weigh 2 grams of liquid caustic soda into a 100 ml volumetric flask, dissolve, and dilute to the mark with distilled water at room temperature. Transfer a 25 ml aliquot of the solution to a titration flask, add 10 ml of 1 percent barium chloride solution, 0.2 ml of 1 percent phenolphthalein indicator, and 50 ml of distilled water. Titrate with 0.25 N hydrochloric acid to the disappearance of the pink color. Not less than 25 ml of the hydrochloric acid shall be required to neutralize the sample of diluted 50 percent caustic soda, and not less than 36.5 ml of the hydrochloric acid shall be required to neutralize the sample of diluted 73 percent caustic soda.

One ml of 0.25 N hydrochloric acid equals 0.01 gram of sodium hydroxide (anhydrous).

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

§ 21.103 Chloroform.

(a) *Odor.* Characteristic odor.

(b) *Specific gravity at 25 °/25 °C.* Not less than 1.400.

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

§ 21.104 Cinchonidine.

(a) *Melting point.* 208° to 210 °C.

(b) *Color.* White powder.

(c) *Taste.* Bitter.

(d) *Test.* A solution of cinchonidine in dilute sulfuric acid shall not have more than a faint blue fluorescence (to distinguish from quinine and quinoidine).

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

§ 21.105 Citronella oil, natural.

(a) *Java type:*

(1) *Alcohol content (as Geraniol).* Not less than 85 percent by weight.

(2) *Aldehyde content (as Citronellal).* Not less than 30 percent by weight.

(3) *Refractive index at 20 °C.* 1.4660 to 1.4745.

(4) *Specific gravity at 25 °/25 °C.* 0.875 to 0.893.

(5) *Odor.* Characteristic odor.

(b) *Ceylon type:*

(1) *Alcohol content (as Geraniol).* Not less than 55 percent by weight.

(2) *Aldehyde content (as Citronellal).* Not less than 7 percent by weight.

(3) *Refractive index at 20 °C.* 1.4790 to 1.4850.

(4) *Specific gravity at 25 °/25 °C.* 0.891 to 0.904.

(5) *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]

§ 21.106 Diethyl phthalate.

(a) *Refractive index at 25 °C.* 1.497 to 1.502.

(b) *Color.* Colorless.

(c) *Odor.* Practically odorless.

(d) *Solubility.* Soluble in 20 parts of 60 percent alcohol.