

(f) *Distillation range.* No distillate should come over below 123 °C. and none above 129 °C.

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

#### § 21.119 Nicotine solution.

(a) *Composition.* Five gallons of an aqueous solution containing 40 percent nicotine; 3.6 avoirdupois ounces of methylene blue, U.S.P.; water sufficient to make 100 gallons.

(b) *Color.* One ml of the nicotine solution (previously agitated in the presence of air) is measured into 100 ml of water and thoroughly mixed. Fifty ml of this colored solution is compared, using Nessler tubes, with 50 ml of a standard color solution containing 5 grams of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , C.P. in 100 ml of water. The color intensity of the solution tested should be equal to or greater than that of the standard solution.

(c) *Nicotine content.* The above solution must contain not less than 1.88 percent of nicotine determined by the following process: 20 ml of the solution are measured into a 500 ml Kjeldahl flask provided with a suitable bulb tube, 50 ml of 0.1 N NaOH added and the 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  $\text{H}_2\text{SO}_4$  using rosolic acid or methyl red as indicator. Not less than 23.2 ml should be required for neutralization.

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#### § 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.

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#### § 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.

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#### § 21.122 Pyridine bases.

(a) *Alkalinity.* One ml of pyridine bases dissolved in 10 ml of water is titrated with 1 N  $\text{H}_2\text{SO}_4$  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  $\text{CaCl}_2$  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