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Testing Specifications

Copper 8-Quinolinolate
(Oxine Copper)

CAS No. 10380-28-6
M.F.:$C_{18}H_{12}CuN_20_2$
M.W.: 351.85

Unless otherwise specified, these specifications are made in accordance with General Notices of Japanese Pharmacopoeia XI (JP XI).

Appearance:

Copper 8-Quinolinolate occurs as yellow-green crystalline powder. It has no odor. It is very slightly soluble in chloroform and practically insoluble in water, absolute ethanol, methanol and benzene.

Sodium Sulfate (Na_2So_4):

Sodium sulfate: Weigh accurately about 10*g* of the sample in a 500 *ml* Erlenmeyer's flask, add 200 *ml* of water, shake for 30 minutes and filtrate. For the filtrate, follow the testing procedures for sodium sulfate as directed under JP XI (not more than 2%).

Water (Moisture):

Perform the test for loss on drying as directed under General Tests of JP XI (1%, 105°C, 2 hours, not more than 0.5%).

Assay:

(1) Preparation of 8-Quinolinol Standard Solution

Dissolve 125 mg of 8-Quinolinol Reference Standard, [#1)] accurately weighed, in 0.5 M phosphoric acid in a 250 ml volumetric flask and add the same acid to volume. Measure 10 *ml* of the solution into a 100 *ml* volumetric flask and 10 *ml* of 0.5 M phosphoric acid and dilute to volume with water. 1 *ml* of the solution contains 50 μ *g* of 8-quinolinol.

(2) Preparation of the standard curve of 8-quinolinol

To each of 5 *ml* volumetric flask containing, 0, 1, 2, 3, 4 and 5 *ml* of 8-Quinolinol Standard Solution respectively, add 0.1 M of phosphoric acid to volume, and transfer the contents of the flask to the separatory funnels respectively. To each separatory funnel, add 25 *ml* of water, 3 *ml* of 0.01% cupric sulfate solution,

[#2] and 2 *ml* of 2 N ammonium acetate, [#3] Then add 10 *ml* of chloroform exactly, shake vigorously for 1 minute, and allow to stand 20-30 minutes.

Determine the absorbances of the chloroform layers in 1 cm cells at the wavelength of 410 nm with a suitable spectrophotometer, using chloroform as a blank. Perform a blank determination in the same manner, using 5 *ml* of 0.1 M phosphoric acid, and make any necessary corrections.

(3) Preparation of sample solution

Dissolve a portion of the sample, equivalent to 180 mg of copper 8-quinolinolate, accurately weighed, in a 10 *ml* of 0.5 M phosphoric acid in a flask and heat on a water bath for 30 minutes. Filter the solution with a filter paper, wash the residue and the filter with water. Combine the filtrate with the washings, transfer into a 250 ml volumetric flask, allow to cool and dilute with water to volume.

Measure 5 ml of the solution into a 100 *ml* volumetric flask, add 15 *ml* of 0.5 M phosphoric acid and dilute to volume with water. Then measure 5 *ml* of the solution into a 100 *ml* separatory funnel, add 25 *ml* of water, 2 *ml* of 2 N ammonium acetate, 3 *ml* of 0.01% cupric sulfate solution and 10 *ml* of chloroform in order, shake vigorously for 1 minute and allow to stand for 20-30 minutes.

(4) Determination of absorbance

Determine the absorbance of the chloroform layer in 1 cm cell at the wavelength of 410 nm with a suitable spectrophotometer, using chloroform as a blank. Read the amount of 8-quinolinol from the standard curve. Calculate the percent assay of copper 8-quinolinolate by the following equation:

Percent assay of copper 8-quinolinolate =

$$\frac{\text{Amount, in } \mu g, \text{ of 8-quinolinol}}{\text{Weight, in mg, of sample taken}} \times 1.2119 \times 50$$

(not less than 96%)

[#1] 8-Quinolinol Reference Standard

Recrystallize 8-Quinolinol JIS (Japanese Industrial Standards).
Reagent grade Special class from water.

[#2] 0.01% cupric sulfate solution

Dissolve 100 mg of $CuSO_4$?$5H_2$) (Reagent grade Special class) in 1 liter of water.

[#3] 2 N ammonium acetate solution

Reagent grade Special class.