Prussian Blue Insoluble

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Manufacturers, regulatory authorities, health authorities, researchers, and other stakeholders are invited to provide their feedback and comments on this draft proposal. Manufacturers are also invited to submit samples of their products to the IPC to ensure that the proposed monograph adequately controls the quality of the product(s) they manufacture. Comments and samples received after the last date will not be considered by the IPC before finalizing the monograph.

Please send any comments you may have on this draft document to lab.ipc@gov.in, with a copy to Dr. Gaurav Pratap Singh (email: gpsingh.ipc@gov.in) before the last date for comments.

Document History and Schedule for the Adoption Process

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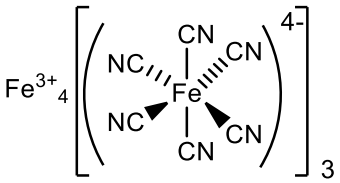

$C_{18}Fe_7N_{18}$ Mol. Wt. 859.2

Prussian blue insoluble is ferric ferrocyanide.

Prussian blue insoluble contains not less than 30.0 per cent of Fe, calculated on the dried basis.

Category. Antidote for radioactive and nonradioactive cesium and thallium.

Description. A dark blue granular powder.

Identification

A. Determine by infrared absorption spectrophotometry (2.4.6). Compare the spectrum with that obtained with *prussian blue IPRS* or with the reference spectrum of prussian blue.

B. Moisten 0.1 g of the substances with 1 ml of *water* and add gradually while shaking 1 ml of *1 M sodium hydroxide*; A reddish brown precipitate is formed within 10 minutes of incubation.

Tests

Solution A. Dissolve 100.0 g in 40 ml of *hydrochloric acid*, heat to boiling, cool and add 80 ml of *water*, filter, wash the filter and residue with *water* and dilute the filtrate and washing to 200.0 ml with *water*.

Chlorides (2.3.12). 25 ml of solution A complies with the limit test for chlorides (20 ppm).

Potassium (2.3.16). To 2 ml of solution A complies with the limit test for potassium (20 ppm).

Sulphates (2.3.17). To 30 ml of solution A complies with the limit test for sulphates (10 ppm).

Ferricyanide. Not more than 200 ppm.

Transfer 8.0 ml of solution A to three separate 50-ml volumetric flasks, marked as test, reference and blank solution, add 1.0 ml of *ferricyanide standard solution* (50 ppm) to the volumetric flask marked as reference solution. Add 1.0 ml of a 0.5 per cent w/v solution of *ferrous ammonium sulphate* to each volumetric flask and dilute to volume with *water*. Allow to stand for 30 minutes. Measure the absorbance of the resulting solutions at the maximum at about 720 nm (2.4.7) using blank solution as compensation liquid. The absorbance obtained with the test solution is more than the absorbance obtained with the reference solution.

Ferrocyanide. Not more than 200 ppm.

Transfer 20.0 ml of solution A to three separate 50-ml volumetric flasks, marked as test, reference and blank solution, add 2.0 ml of *ferrocyanide standard solution* (100 ppm) to the volumetric flasks marked as reference solution. Add 1.0 ml of *ferric chloride solution* to the volumetric flasks marked as test solution and reference solution and dilute to volume with *water*. Allow to stand for 30 minutes. Measure the absorbances of the resulting solutions at the maximum at about 695 nm (2.4.7) using blank solution as compensation liquid. The absorbance obtained with the test solution is not more than the absorbance obtained with the reference solution.

Maximum Binding Capacity. Not less than 300 mg per g for Cs. Determine by atomic absorption spectrophotometry (2.4.2), measuring at 852.12 nm using caesium lamp.

Buffer solution. Dissolve 12.8 g of *dipotassium hydrogen orthophosphate* and 3.6 g of *potassium dihydrogen orthophosphate* in 1000 ml with *water*, adjusted to pH 7.5 with *dilute orthophosphoric acid* or dilute *sodium hydroxide* solution.

Reference solution (a) (Caesium (Cs) 1500 ppm). Dissolve 1.9005 g of *caesium chloride* in 1000.0 ml of the buffer solution.

Reference solutions (b) (Caesium (Cs) 600 ppm). Dilute 20.0 ml of reference solution (a) to 50.0 ml with the buffer solution.

Reference solutions (c) (Caseium (Cs) 750 ppm). Dilute 25.0 ml of reference solution (a) to 50.0 ml with the buffer solution.

Reference solutions (d) (Caesium (Cs) 900 ppm). Dilute 30.0 ml of reference solution (a) to 50.0 ml with the buffer solution.

Reference solutions (e) (Caesium (Cs) 1200 ppm). Dilute 40.0 ml of reference solution (a) to 10.0 ml with the buffer solution.

Procedure. Transfer 50.0 ml, each of, reference solution (a), reference solution (b), reference solution (c), reference solution (d) and reference solution (e) to five separate 250-ml volumetric flasks. Add 100 mg of substance under examination to each of the volumetric flask. Incubate the mixture for 24 hours with constant shaking at 25°. After 24 hours, filter the solutions and aspirate to AAS.

Measure caesium (Cs) concentrations in all the solutions using caesium lamp at 852.12 nm (2.4.2).

Calculate maximum binding capacity by Langmuir adsorption isotherm model. Ce and qe is calculated and a graph is plotted.

Where,

Ce (mg/litre) = Concentration of caesium (Cs) at equilibrium,
qe (mg/litre) = Amount of caesium (Cs) adsorbed at equilibrium,
qm (mg/g) = Maximum capacity of adsorbent,
KL (l/mg) = Constant related to energy of adsorption.

Plot the line between Ce/qe vs Ce and from which the constants qm and KL are calculated.

Cyanide. Not more than 400 µg per g at pH 1 with 24 hours of dwelling time.

Buffer solution. Dissolve 12.8 of *dipotassium hydrogen orthophosphate* and 3.6 g of *potassium dihydrogen orthophosphate* in 1000 ml of *water*, adjusted to pH 7.5 with *dilute orthophosphoric acid* or *dilute sodium hydroxide solution.*

Pyridine barbituric acid reagent. Dissolve 6 g of *barbituric acid* in minimum volume of *water*, add 30 ml of *pyridine* and mix. Add 6 ml of *hydrochloric acid*, mix and cool to room temperature, dilute to 100 ml with *water.*

Solution A. Dissolve 410 g of *sodium acetate* in 500 ml of *water*, adjusted to pH 4.5 with *glacial acetic acid* and dilute to 1000 ml with *water.*

Test solution. Weigh and transfer 1 g of the substance under examination to a 100-ml volumetric flask, add 50 ml of the buffer solution and incubate in a shaking water-bath at 37° with 75 shakes per minute for 24 hours, filter. Transfer 10.0 ml of the filtrate to 50-ml volumetric flask.

Reference solution. Dissolve 0.251 g of *potassium cyanide* in *water* and dilute to 100.0 ml with *water*. This solution contains 1 µg cyanide (CN) per ml.

Transfer 1.0 ml, 2.0 ml, 3.0 ml, 4.0 ml, 5.0 ml, 6.0 ml, 7.0 ml, 8.0 ml, 9.0 ml and 10.0 ml of the solution to separate 50-ml volumetric flasks (contains cyanide (CN) from 0.02 to 0.2 µg per ml).

Blank solution. Pipette 10 ml of *water* into a 50-ml volumetric flask.

Procedure. To each volumetric flask of the reference solution, test solution and blank solution, add 1.0 ml of solution A and 1.0 ml *chloramine-T solution*, mix. Add 5.0 ml of pyridine-barbituric acid reagent and dilute to volume with *water*. Stand for 10 minutes. Measure the absorbances of reference solutions, test solution at 578 nm (2.4.7) using blank solution as compensation liquid.

Plot the reference solution curve on the abscissa and concentration of the cyanide on the ordinate.

Calculate the cyanide concentration as follows from slope and intercept of reference solution drawn.

$$CN \text{ (mg per litre)} = (\text{Slope} \times A_1 + \text{intercept}) \times 50/W \times 250/Y$$

Where,

A_1 = Absorbance of test solution,
W = Weight of the substance under examination,
Y = Volume of test solution taken for colourimetric.

Loss on drying (2.4.19). Not more than 10.0 per cent, determined on 1.0 g by drying in an oven at 105° for 3 hours.

Assay. Determine by atomic absorption spectrophotometry (2.4.2), measuring at 248 nm using iron lamp.

Test solution. Weigh and transfer 10 mg of the substance under examination to a 25-ml volumetric flask, add 10 ml of *1 M sodium hydroxide* and mix with constant stirring followed by 10 minutes of incubation at room temperature, a reddish-brown precipitate is formed, allow for sedimentation. Collect the clear supernatant liquid in to a measuring cylinder (solution A).

To the sedimented precipitate, add 10 ml of *hydrochloric acid* and heat until solution turns clear (solution B).

Both solution A and solution B are analyzed for Iron content after appropriate dilution using Atomic absorption spectrophotometry (2.4.2).

The sum of the Iron content in solution A and solution B gives the total iron content.

Storage. Store at a temperature not exceeding 30°.

Solubility. Insoluble in *water,* in diluted acid and in most common organic solvents.