

RESOLUTION OENO 36/2000

INTERNATIONAL OENOLOGICAL CODEX

POTASSIUM HEXACYANOFERRATE (II)

Potassium ferrocyanide
Cianuretum ferroso - Kalium
 $K_4[Fe(CN)_6] \cdot 3H_2O = 422.40$
SIN NO. 536

1. OBJECTIVE, ORIGIN AND SCOPE OF APPLICATION

Potassium hexacyanoferrate (II) is found in the form of yellow, monoclinic crystals having no odor and with a bitter, salty flavor. Density is 1.935 at 20 °C.

This salt is slightly efflorescent and begins to lose its water of crystallization at approximately 60 °C. In an oven at 100 °C, it dehydrates completely, becoming white and hygroscopic.

Freshly prepared aqueous solutions are yellow and decay slowly in light with the release of alkalinity. They take on a greenish color by forming a small quantity of Prussian blue.

Potassium hexacyanoferrate (II) is used to remove iron (III) and iron (II) ions in wines, which could cause iron breakdown. It is also used to avoid copper breakdown. It is used, more generally, to reduce the heavy metal content.

Its use must be strictly controlled by mandatory monitoring.

2. LABELING

The label should indicate the product's purity as well as its safety and storage conditions.

3. IDENTIFYING PROPERTIES

The aqueous solution at 1 pp 100 (m/v) yields hexacyanoferrate (II) ion and potassium reactions, particularly with the iron (III) cation, resulting in a dark blue iron (III) hexacyanoferrate (II) (Prussian blue) precipitate which is insoluble in dilute mineral

acids. With the copper cation, it forms a purple copper (II) hexacyanoferrate (II) precipitate that is insoluble in dilute mineral acids.

4. SOLUBILITY

Water at 20 °C	265 g/l
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Water at 100 °C	740 g/l
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5. TESTS

5.1. Desiccation Loss

Place 1 g of powdered potassium hexacyanoferrate (II) in a calibrated dish and dry in an oven at 100 °C until it has a constant weight. Weight loss should be between 12 and 13 pp 100.

5.2. Insoluble Products

Dissolve 10 g of potassium hexacyanoferrate (II) in 100 ml of water. The solution should be clear.

5.3. Preparing the Test Solution

Calcine 1 g of potassium hexacyanoferrate (II) in a silica dish, but without exceeding 550 °C. Take up the residue with 10 ml of water and 2 ml of concentrated nitric acid (R). Decant in a 50 ml volumetric flask. Add 5 ml concentrated ammonium hydroxide (R). Fill to 50 ml with distilled water. Filter.

5.4. Chlorides

To 2.5 ml of this test solution (5.3), add 5 ml nitric acid diluted to 10 pp 100 (R), 12.5 ml of distilled water and 0.5 ml of 5 pp 100 silver nitrate (R). If any opalescence develops, it should be less intense than that observed in a control prepared as indicated in the Annex. (Chloride content, expressed in terms of hydrochloric acid content, should be less than 1 g/kg).

5.5. Sulfates

Add 2 ml hydrochloric acid diluted to 10 pp 100 (R) to a 5 ml test solution (5.3), increase to 20 ml with distilled water and add 2 ml of a barium chloride solution (R). The mixture should be clear ; or else, any opalescence observed after 15 minutes should be less intense than that of the control prepared as indicated in the annex. (Sulfates content, as expressed for sulfuric acid, should be lower than 1 g/kg.)

5.6. Sulfides

In the 100 ml flask of a distilling apparatus equipped with a small rectifying column or other anti-priming device (designed to prevent the direct flow of liquid fractions in the flask into the distillate), dissolve 1 g of potassium hexacyanoferrate (II) in 10 ml of hydrochloric acid diluted to 10 pp 100 (R) and 10 ml of distilled water. Distill and collect 5 ml of distillate in 5 ml of 1M sodium hydroxide.

Take 0.5 ml of this distillate and add 18.0 ml of distilled water and 1 ml of a lead nitrate solution in a concentration of 1 g per liter (R). The resulting brown coloration should be less intense than that of a control prepared by adding 0.5 ml of hydrogen sulfide solution in a concentration of 1 g of sulfur per liter (R), 18 ml of distilled water and 1 ml of lead nitrate in a concentration of 1 g per liter (R). (Sulfide content, expressed in terms of sulfur, should be less than 100 mg/kg).

5.7. Cyanides

In a 40 ml volumetric flask containing 25 ml of distilled water and 2.5 ml of pH 7.5 buffer solution (R), place 40 mg of potassium hexacyanoferrate (II). After dissolving, add immediately 0.3 ml of 0.1 pp 100 T chloramine solution (R). Wait 90 seconds, then add 6 ml of pyridine-pyrazolone reagent (R).

Fill to 40 ml with distilled water and mix. The resulting coloration should not be more intense than that obtained by treating in the same way 4 ml of freshly prepared potassium cyanide solution assayed at 1 mg of hydrogen cyanide per liter (R). (Free cyanide content, expressed in terms of hydrogen cyanide, should be less than 100 mg/kg).

5.8. Lead

Using the technique described in the Compendium, determine the lead content in the solution (5.3). (Lead content should be less than 5 mg/kg.)

5.9. Mercury

Using the technique described in the annex, determine the mercury content in the test solution (5.3). (Mercury content should be less than 1 mg/kg.)

5.10. Arsenic

Using the technique described in the annex, determine the arsenic content in the test solution (5.3). (Arsenic content should be less than 3 mg/kg.)

5.11. Ammonia

Place 2 g of potassium hexacyanoferrate (II), 25 ml of distilled water and 5 ml of 30 pp 100 sodium hydroxide (R) in the flask of a distilling apparatus. Distill and collect 20 ml of distillate in 40 ml of 4 pp 100 boric acid (R) in the presence of methyl red. 1.2 ml of 0.1M hydrochloric acid should be sufficient to turn the indicator. (Total ammonia content should be less than 100 mg/kg).

6. STORAGE

Potassium hexacyanoferrate (II) should be stored in airtight bags away from moisture.