RESOLUTION OENO 21/2000

INTERNATIONAL OENOLOGICAL CODEX

CALCIUM PHYTATE

Calcium inositol hexaphosphate

Calcii phytas

$C_6H_6Ca_6O_{24}P_6, 3H_2O$= 942.11

1. OBJECTIVE, ORIGIN AND SCOPE OF APPLICATION

Calcium phytate is the salt of the inositol hexaphosphoric ester, or inositohexaphosphoric or phytic acid.

In its calcium and magnesium double salt forms, phytic acid composes phytin, a reserve form of phosphorous in plants.

Since it is an iron (III) complexing agent approved for removal of excess iron in wines, its use must be strictly monitored.

Any excess phytate with respect to the iron (III) content causes deposits to build up when the slightest oxidation occurs.

2. LABELING

The label should indicate product concentration even when used in mixtures, as well as its safety and storage conditions.

3. PROPERTIES

White powder with an acidulous taste, which is minimally soluble in water, soluble in dilute strong acids, and difficult to dissolve in wine, in which solubility is incomplete.

Aqueous calcium phytate solution possess an acidic nature, which is disclosed by movement of the indicator to litmus. It yields calcium reactions.

4. TESTS

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4.1. Desiccation Loss

Dry a 1 g sample of calcium phytate in an oven at 105 °C until a constant weight is obtained. Weight loss should be less than 12 pp 100.

Limits indicated below are for dry product.

4.2. Ash

Incinerate a 0.250 g test sample of calcium phytate at 550 °C. The residue should not be less than 65 pp 100 nor greater than 72 pp 100 of the dry product contained in the test sample.

4.3. Insoluble Substances

Prepare a first solution contaning 1 g of calcium phytate, 7 ml of 1M hydrochloric acid solution, and 93 ml of distilled water. Separately, prepare a solution of 1 g of calcium phytate with 50 ml of distilled water and 1.5 ml pure phosphoric acid (R). Filter each of the solutions separately and collect the deposit. Wash and dry the deposit at 100 °C. Each residue should be less than 1 part per 100 (10g/kg) of dried product at 105 °C.

4.4. Starch

Add several drops of iodinated water (R) to the residues obtained under Paragraph 4.3; no blue coloration should develop.

4.5. Sugars

Stir 3 g of calcium phytate with 15 ml of distilled water. Filter. The filtrate should not reduce the cupro-alkaline reagent (R) before or after the sucrose inversion.

4.6. Albumin

Dissolve 1 g of the product in a mixture of 1 ml of concentrated hydrochloric acid (R) and 3 ml of distilled water. Add 3 ml of 30% sodium hydroxide solution (R). Filter. When one drop of 4 pp 100 (m/v) copper (II) sulfate solution is added to the filtrate, no violet color should appear.

4.7. Preparing the Solution for Tests

Macerate a quantity of calcium phytate containing 5 g dry product with 100 ml of 10 g per liter citric acid (R) for 24 hours while agitating from time to time. Filter.

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4.8. Iron

Add 1 ml of concentrated hydrochloric acid (R) and 2 ml of 5 pp 100 potassium thiocyanate to 10 ml of test solution prepared under paragraph 4.7. The resulting colorating should be less intense than that produced by a control tube prepared with 2.5 ml solution in a concentration of of 0.010 g of iron per liter (R), 7.5 ml of distilled water, 1 ml of concentrated hydrochloric acid (R) and 2 ml of 5 pp 100 thiocyanate (R). (Iron content should be less than 50 mg/kg).

4.9. Lead

Using the method described in the Compendium, quantify lead analytically in the test solution prepared according to Par. 4.7. (Lead content should be less than 5 mg/kg).

4.10. Mercury

Using the method described in the annex, quantify mercury analytically in the test solution prepared according to Par. 4.7. (Mercury content should be less than 1 mg/kg).

4.11. Arsenic

Using the method described in the annex, quantify arsenic analytically in the test solution prepared according to Par. 4.7. (Arsenic content should be less than 3 mg/kg).

4.12. Mineral phosphates

Place 0.50 g calcium phytate in a 200 ml volumetric flask. Add 100 ml of distilled water and 5 ml of concentrated nitric acid (R). Agitate for 15 minutes at 20 °C and top off to 200 ml with distilled water. To 10 ml of this solution, add 10 ml of nitro-vanadomolybdic reagent (R). Leave in contact for 15 minutes at 20 °C. The resulting color should be less intense than that produced by adding 5 ml distilled water and 10 ml nitro-vanadomolybdic reagent (R) to 5 ml of a monopotassic phosphate solution containing 0.05 g phosphorous per liter (R). (Mineral phosphate content, expressed in terms of phosphorous, should be less than 1 pp 100).

4.13. Glycerophosphates

Heat 0.50 g of calcium phytate in the presence of monopotassic sulfate. No acrolein fumes (odor of burnt horn) should be released.

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4.14. Total Phosphorous Determination

Weigh precisely a 0.25 g sample of calcium phytate which has already been dried at 105 °C. Place it in a flask which is ground and polished so it can be fitted with a tube 8 mm in diameter and 1 m long which will serve as a reflux condenser. Add 5 ml of concentrated sulfuric acid (R) and 0.5 ml concentrated nitric acid (R). Bring to boiling under reflux for approximately 15 minutes. After cooling, decant the contents of the flask diluted with water in a 1 liter volumetric flask. Wash the condenser and flask with water by pouring these liquids in the volumetric flask, and fill to gauge line after bringing the temperature to 20 °C. Agitate.

Add 10 ml of nitro-vanadomolybdic reagent (R) to 10 ml of this solution. Agitate in a 20 °C water bath and let sit in the water bath for 15 minutes. The intensity of the resulting color should be equal to or greater than that of a control prepared under the same conditions using 8 ml of monopotassic phosphate solution in a concentration of 0.05 g of phosphorous per liter (R), 2 ml of water and 10 ml of nitro-vanadomolybdic reagent (R).

Total phosphorous analysis can also be determined using a spectrophotometer with a wavelength of 425 nm whose calibration curve was obtained based on 4-6-8-10 ml of solution in a concentration of 0.05 mg phosphorous per liter (R).

Calcium phytate should contain at least 15 parts of phosphorous per 100 , as compared with a product dried at 105 °C.

5. STORAGE

Calcium phytate should be stored in a dry place in hermetically sealed containers.

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