SLOVENSKI STANDARD

SIST ISO 6490-2

prva izdaja
maj 1995

Krma - Določanje vsebnosti kalcija - 2. del: Metoda atomske absorpcijske spektrometrije (prevzet standard ISO 6490-2:1983 z metodo platnice)

Animal feeding stuffs - Determination of calcium content - Part 2: Atomic absorption spectrometric method

Aliments des animaux - Détermination de la teneur en calcium - Partie 2: Méthode par spectrométrie d'absorption atomique

Deskriptorji: krmila, preskus, določanje vsebnosti, kalcij, metoda atomske absorpcijske spektrometrije

ISC 71.040.40 * 65.120

Referenčna številka
SIST ISO 6490-2:1995 (en)

Nadaljevanje na straneh od II do III in 1 do 3

UVOD

Standard SIST ISO 6490-2, Krma - Določanje vsebnosti kalcija - 2. del: Metoda atomske absorpcijske spektrometrije, prva izdaja, 1995, ima status slovenskega standarda in je z metodo platnice prevzet mednarodni standard ISO 6490-2, Animal feeding stuffs - Determination of calcium content - Part 2: Atomic absorption spectrometric method, first edition, 1983-11-01.

PREDGOVOR

Mednarodni standard ISO 6490-2:1983 je pripravil tehnični odbor Mednarodne organizacije za standardizacijo ISO/TC 34 Kmetijski pridelki in živilski proizvodi.

Odločitev za prevzem tega standarda po metodi platnice je sprejela delovna skupina WG 10 Analitika krme v okviru tehničnega odbora USM/TC KŽP Kmetijski pridelki in živilski proizvodi.

Ta slovenski standard je dne 1995-05-08 odobril direktor USM.

ZVEZA S STANDARDI

S prevzemom tega mednarodnega standarda veljajo naslednje zveze:

SIST ISO 6651:1995 (en)	Krma - Določanje vsebnosti aflatoksina B_1
SIST ISO 6654:1995 (en)	Krma - Določanje vsebnosti sečnine
SIST ISO 6866:1995 (en)	Krma - Določanje vsebnosti prostega in skupnega gosipola
SIST ISO 6870:1995 (en)	Krma - Določanje vsebnosti zearalenona
SIST ISO 5498:1995 (en)	Kmetijski pridelki in živilski proizvodi - Določanje vsebnosti surove vlaknine - Splošna metoda
SIST ISO 5983:1995 (en)	Krma - Določanje vsebnosti dušika in izračun vsebnosti surovih beljakovin
SIST ISO 5984:1995 (en)	Krma - Določanje surovega pepela
SIST ISO 5985:1995 (en)	Krma - Določanje pepela, netopnega v klorovodikovi kislini
SIST ISO 6490-1:1995 (en)	Krma - Določanje vsebnosti kalcija - 1. del: Titrimetrična metoda
SIST ISO 6491:1995 (en)	Krma - Določanje vsebnosti skupnega fosforja - Spektrofotometrična metoda
SIST ISO 6495:1995 (en)	Krma - Določanje vsebnosti v vodi topnih kloridov
SIST ISO 6496:1995 (en)	Krma - Določanje vsebnosti vlage
SIST ISO 5506:1995 (en)	Sojini proizvodi - Določanje ureazne aktivnosti
SIST ISO 6541:1995 (en)	Kmetijski pridelki in živilski proizvodi - Določanje vsebnosti surove vlaknine - Modificirana Scharrerjeva metoda

OSNOVA ZA IZDAJO STANDARDA

- Prevzem standarda ISO 6490-2:1983

OPOMBI

- Povsod, kjer se v besedilu standarda uporablja izraz mednarodni standard , to pomeni v SIST ISO 6490-2:1995 slovenski standard .

- Uvod in predgovor nista sestavni del standarda.

Po mnenju Ministrstva za informiranje Republike Slovenije z dne 18. februarja 1992, štev. 23/96-92, spada ta publikacija med proizvode informativne narave iz 13. točke tarifne številke 3, za katere se plačuje 5-odstotni prometni davek.

International Standard 6490/2

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Animal feeding stuffs — Determination of calcium content — Part 2: Atomic absorption spectrometric method

Aliments des animaux — Détermination de la teneur en calcium — Partie 2: Méthode par spectrométrie d'absorption atomique

First edition — 1983-11-01

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Descriptors: animal feeding products, tests, determination of content, calcium, atomic absorption spectrometric method.

Price based on 3 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6490/2 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in July 1982.

It has been approved by the member bodies of the following countries:

Australia	Ireland	Romania
Austria	Israel	South Africa, Rep. of
Canada	Italy	Sri Lanka
Chile	Korea, Rep. of	Tanzania
Egypt, Arab Rep. of	Malaysia	Thailand
Ethiopia	Netherlands	United Kingdom
France	New Zealand	USA
Hungary	Peru	USSR
India	Philippines	Yugoslavia
Iran	Poland	
Iraq	Portugal	

No member body expressed disapproval of the document.

Printed in Switzerland

Animal feeding stuffs — Determination of calcium content — Part 2: Atomic absorption spectrometric method

1 Scope and field of application

This part of ISO 6490 specifies an atomic absorption spectrometric method for the determination of the calcium content of animal feeding stuffs.

The limit of detection is in the region of 10 mg/kg.

2 References

ISO 6497, *Animal feeding stuffs — Sampling.*[1)]

ISO 6498, *Animal feeding stuffs — Preparation of test samples.*[1)]

3 Principle

Following any necessary destruction of organic matter by incineration of a test portion, dissolution of the calcium by treatment with hydrochloric acid and dilution of the solution obtained, in the presence of lanthanum which is used as a spectral buffer. Determination of the calcium content by atomic absorption spectrometry.

4 Reagents

All reagents shall be of recognized analytical quality and the water used shall be double-distilled, deionized and distilled or double deionized water.

4.1 Hydrochloric acid, concentrated (ϱ_{20} = 1,18 to 1,19 g/ml).

4.2 Hydrochloric acid, 6 mol/l solution.

4.3 Lanthanum chloride, solution prepared as follows:

In a 1 000 ml one-mark volumetric flask, dissolve 25 g of lanthanum oxide of low calcium content in 75 ml of the hydrochloric acid (4.1). Following reaction, allow to cool, add a little water, agitate, dilute to the mark with water and mix.

4.4 Calcium, standard solution corresponding to 40 mg/l.

4.4.1 Stock solution corresponding to 1 g/l.

Weigh 2,497 g of calcium carbonate which has been previously dried at 105 °C for 1 h. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, rinsing with about 100 ml of water. Add 50 ml of the hydrochloric acid (4.1) to dissolve the carbonate, dilute to the mark with water and mix.

4.4.2 Standard solution corresponding to 40 mg/l.

By means of a pipette, transfer 10 ml of the stock solution (4.4.1) into a 250 ml one-mark volumetric flask. Add several drops of the hydrochloric acid (4.1), dilute to the mark with water and mix.

1 ml of this standard solution corresponds to 40 µg of calcium.

5 Apparatus

Usual laboratory equipment, and in particular

5.1 Electrically heated muffle furnace, preferably of silica, capable of being controlled at 550 ± 10°C.

5.2 Incineration dishes, of platinum, or, failing this, silica or porcelain.

5.3 Atomic absorption spectrometer, equipped to determine calcium, using an air/acetylene flame.

5.4 Ashless filter paper.

5.5 Beakers, of capacity 250 ml.

5.6 Volumetric flasks, of capacities 100 and 250 ml.

5.7 Pipettes, to deliver 5, 10, 15, 20 and 25 ml.

5.8 Sand bath or **hot plate,** capable of being controlled at approximately 150 °C.

5.9 Analytical balance.

1) At present at the stage of draft.

6 Sampling

Take the laboratory sample in accordance with ISO 6497.

7 Procedure

7.1 Preparation of the test sample

Prepare the test sample in accordance with ISO 6498.

7.2 Test portion

7.2.1 Samples containing organic matter

Weigh, to the nearest 1 mg, 1 to 5 g of the test sample, according to the expected calcium content, into an incineration dish (5.2).

7.2.2 Samples not containing organic matter

Weigh, to the nearest 1 mg, 1 to 5 g of the test sample, according to the expected calcium content, into a 250 ml beaker (5.5).

7.3 Preparation of the test solution

7.3.1 Incineration and rendering the silica insoluble (only in the case of samples containing organic matter)

Place the incineration dish in the muffle furnace (5.1) while cold, close the furnace and gradually increase the temperature so as to reach 550 ± 10 °C in approximately 1 h 30 min. Maintain this temperature until the ash is free of carbon particles (if necessary, maintain this temperature for 16 h, i.e. overnight), then remove the incineration dish and leave to cool. Transfer the ash to a 250 ml beaker (5.5), moisten the ash with water, then rinse the incineration dish with a total of approximately 5 ml of the hydrochloric acid (4.1), collecting the rinsings in the beaker; take care during the addition as a violent reaction may occur.

Evaporate the contents of the beaker to dryness on the sand bath or hot plate (5.8) controlled at approximately 150 °C.

7.3.2 Dissolution of calcium

7.3.2.1 Add to the beaker containing the test portion (see 7.2.2), or the ash after incineration and rendering the silica insoluble (see 7.3.1), 15 ml of the hydrochloric acid solution (4.2) and 120 ml of water, and bring to the boil.

Filter through the filter paper (5.4) and collect the filtrate in a 250 ml one-mark volumetric flask (5.6).

7.3.2.2 If the residue on the filter shows only traces of carbon, ignore this, and wash the filter with 5 ml of the hydrochloric acid solution (4.2) and a little hot water, collecting the washings in the volumetric flask. Dilute to the mark with water and mix.

7.3.2.3 If the residue on the filter appears black (presence of carbon), place the filter containing the residue in a dish (5.2) and incinerate again in the muffle furnace, controlled at 550 ± 10 °C, until all the carbonaceous matter has completely disappeared (this operation usually requires 3 to 5 h). Allow to cool, add 2 ml of the hydrochloric acid (4.1) and evaporate to dryness on the sand bath or hot plate (5.8) controlled at approximately 150 °C, then add 5 ml of the hydrochloric acid solution (4.2). Heat, filter through a filter paper, collecting the solution in the 250 ml volumetric flask containing the filtrate previously collected (see 7.3.2.1), wash the filter paper with water, and dilute to the mark with water. Mix.

NOTE — For the analysis of products such as alumino-calcium phosphates which are not readily soluble in hydrochloric acid, it is recommended that an alkaline fusion be carried out as follows:

Mix the sample for analysis in a platinum crucible with 5 times its mass of a mixture of equal parts of potassium carbonate and sodium carbonate. Heat carefully until the mixture has completely melted. Cool and carefully dissolve the residue in the hydrochloric acid solution as described in 7.3.2.

7.4 Blank test

Prepare a solution in the same way as for the test solution, carrying out all the operations specified in 7.3 and using all the reagents, but omitting the test portion.

7.5 Preparation of the calibration graph

7.5.1 Preparation of calibration solutions

Prepare a series of six 100 ml one-mark volumetric flasks (5.6), and by means of a pipette (5.7), introduce, respectively, 0 — 5 — 10 — 15 — 20 and 25 ml of the standard calcium solution (4.4).

Add to each flask 20 ml of the lanthanum chloride solution (4.3), dilute to the mark with water and mix.

These solutions correspond respectively to 0 — 2 — 4 — 6 — 8 and 10 µg of calcium per millilitre.

This series of calibration solutions is given as a guide, with the understanding that it may be displaced slightly depending on the sensitivity of the instrument. If this is the case, the operator should determine the final dilution of the test portion to obtain the optimum conditions.

NOTE — For the analysis of mineral compounds which are rich in alkali metals, it is recommended that the calibration solutions be prepared using a solution containing sodium and potassium ions in the same proportions as those of the product for analysis.

7.5.2 Spectrometric measurements

Measure the absorbances of the calibration solutions (7.5.1), using the atomic absorption spectrometer (5.3), set at a wavelength of 422,7 nm, with an air/acetylene flame.

7.5.3 Plotting the graph

Plot a graph having, for example, the calcium contents, in micrograms per millilitre, as abscissae, and the corresponding absorbances of the calibration solutions as ordinates.