SLOVENSKI STANDARD
SIST EN 12396-1:1999

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Živila brez maščob - Določevanje ostankov ditiokarbamatov in tiuram-disulfidov - 1. del: Spektrometrijska metoda

Non-fatty foods - Determination of dithiocarbamate and thiuram disulfide residues - Part 1: Spectrometric method

Fettarme Lebensmittel - Bestimmung von Dithiocarbamat- und Thiuramdisulfid-Rückständen - Teil 1: Spektralphotometrisches Verfahren

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Aliments non gras - Détermination des résidus de dithiocarbamates et de bisulfures de thiurame - Partie 1: Méthode spectrométrique

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Ta slovenski standard je istoveten z: **EN 12396-1:1998**

ICS:

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

SIST EN 12396-1:1999 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD
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EN 12396-1

October 1998

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Descriptors: food products, chemical analysis, determination of content, pesticides, fungicides, pesticide residues, spectrophotometric analysis

English version

Non-fatty foods - Determination of dithiocarbamate and thiuram disulfide residues - Part 1: Spectrometric method

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This European Standard was approved by CEN on 2 October 1998.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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This European Standard has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 1999, and conflicting national standards shall be withdrawn at the latest by April 1999.

This European Standard EN 12396 „Non-fatty foods - Determination of dithiocarbamate and thiuram disulfide residues“ consists of three parts:

Part 1: Spectrometric method

Part 2: Gas chromatographic method

Part 3: Xanthogenate method

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European Standard specifies a spectrometric method for the determination of residues of dithiocarbamates and thiuram disulfides, which release carbon disulfide under the described conditions (e.g. mancozeb, maneb, propineb, thiram, zineb). It is applicable to such compounds in and on fruits and many vegetables and also in and on cereals and other foodstuffs of plant origin.

Only the quantification of the whole group is possible using this method but not the identification of individual compounds. Generally the maximum residue limits (MRLs) are expressed in terms of carbon disulfide.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 1750		Pesticides and other agrochemicals - Common names.
EN 12393-1	1998	Non-fatty foods - Multiresidue methods for the gas chromatographic determination of pesticide residues - Part 1:General considerations.
EN 12396-2		Non-fatty foods – Determination of dithiocarbamate and thiuram disulfide residues – Part 2:Gas chromatographic method.

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3 Principle

The sample is heated with hydrochloric acid and tin(II)chloride to release carbon disulfide from any dithiocarbamates and/or thiuram disulfide present. The carbon disulfide is separated and purified by distillation and collected in an ethanolic solution of copper(II)acetate and diethanolamine. With copper(II)acetate and diethanolamine, the carbon disulfide forms two yellow copper(II)-N,N-bis(2- hydroxy-ethyl)-dithiocarbamate complexes with the molar ratio $Cu:CS_2$ = 1:1 and 1:2. The absorption of these reaction products is measured in a spectrometer at a wavelength of 435 nm and the concentration of dithiocarbamate and/or thiuram disulfide residues is calculated and expressed in terms of milligrams of carbon disulfide per kilogram of foodstuff. For further information on the principle of this method, see [1] to [4].

4 Reagents

4.1 General

Unless otherwise stated, use reagents of recognized analytical grade, preferably for pesticide residue analysis, and only distilled or demineralized water.

Label all standard containers with name and purity of all pesticides. For the full chemical names and structures, see ISO 1750.

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Take every precaution to avoid possible contamination of water, solvents, inorganic salts etc. by plastics and rubber materials. Use only glass containers for storage and handling of all water and reagents.

4.2 Carbon disulfide, colourless, mass concentration of at least 99 %. If stored at - 20 °C it is stable for 2 years to 3 years.

4.3 Diethanolamine, at least 99 % of mass concentration.

4.4 Ethanol, mass concentration of at least 95 %.

4.5 Hydrochloric acid, concentrated, ρ_{20}(HCl) = 1,16 g/ml.

4.6 Sulfuric acid (optional), concentrated, ρ_{20} (H_2SO_4) = 1,84 g/ml

4.7 Sodium hydroxide solution, ρ (NaOH) = 100 g/l [1]

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4.8 Copper(II) acetate solution, ρ ($Cu(CH_3COO)_2 \cdot H_2O$) = 400 mg/l

Weigh 400 mg of copper(II)acetate monohydrate, to the nearest 1 mg, with gentle warming only, in 250 ml of ethanol (4.4). Dilute 25 ml of this solution to 100 ml with ethanol.

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4.9 Lead acetate solution (optional), ρ ($Pb(CH_3COO)_2 \cdot 3H_2O$) = 300 g/l.

4.10 Tin(II) chloride solution, ρ ($SnCl_2 \cdot 2H_2O$) = 40 g/100 ml in concentrated hydrochloric acid (4.5).

4.11 Tin(II) chloride - hydrochloric acid solution, ρ ($SnCl_2 \cdot 2H_2O$) = 3,3 g/100 ml.

Mix 20 ml of tin(II)chloride solution (4.10) with 20 ml of concentrated hydrochloric acid (4.5) and add carefully 200 ml of water.

4.12 Zinc acetate solution (optional), ρ ($Zn(CH_3COO)_2$) = 20 g/100 ml.

4.13 Carbon disulfide stock solution

Weigh to the nearest 10 mg a stoppered 50 ml volumetric flask with a ground glass neck containing 40 ml of ethanol (4.4). Add approximately 1 ml of carbon disulfide (4.2) (equivalent to approximately 1,25 g) using a pipette, close the flask at once and re-weigh to the nearest 10 mg to obtain the exact mass of carbon disulfide by difference. Dilute to the mark with ethanol and mix well. Prepare freshly for each calibration curve.

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[1] ρ is the mass concentration

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4.14 Carbon disulfide standard solution

Pipette 5 ml of carbon disulfide stock solution (4.13) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.4). Pipette 5 ml of this dilution into a 250 ml volumetric flask and dilute to the mark with ethanol. 1 ml of this standard solution is equivalent to approximately 50 µg of carbon disulfide. Prepare freshly for each calibration curve.

4.15 Colour reagent

Successively introduce 100 ml of ethanol (4.4), 30 ml of copper(II)acetate solution (4.8) and 25 g of diethanolamine (4.3) into a 250 ml volumetric flask, and dilute to the mark with ethanol.

5 Apparatus

5.1 General

Thoroughly clean glassware shall be used.

See 5.1 of EN 12393-1:1998 for the cleaning of glassware.

Usual laboratory equipment and, in particular, the following:

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5.2 Decomposition and distillation apparatus, consisting of a 1 l or 2 l round bottomed three necked flask or cylindrical flask with a three necked adapter, a dropping funnel, a gas inlet tube, an ascending Liebig condenser, three absorption tubes, the last two preferably fitted with a Widmer helix, connected by spherical socket joints, the first being attached to the Liebig condenser; (see figure 1).

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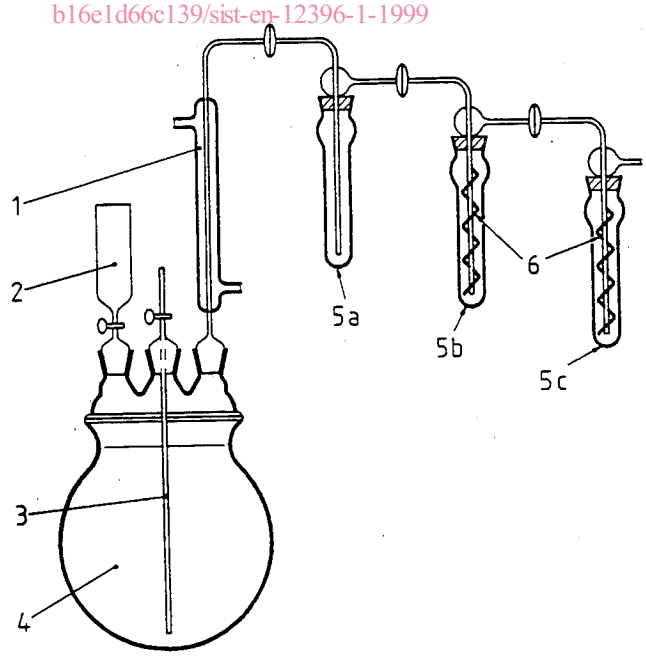

1 Liebig condenser
2 dropping funnel
3 gas inlet tube
4 cylindrical flask or round bottomed flask
5 absorption tubes
6 Widmer helix

Figure 1:Decomposition and distillation apparatus

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5.3 Flowmeter

5.4 Heating mantle, electrically operated, at least 450 W; or gas burner fitted with a Babo funnel and flask holder

5.5 Spectrometer, suitable for measurements at a wavelength of 435 nm, with a 1 cm glass or quartz cell. A double beam spectrometer should preferably be used.

5.6 Water jet pump, attached to the last absorption tube, or a **source of nitrogen** under pressure, attached to the gas inlet tube.

6 Sampling

Prepare the laboratory sample according to generally recommended methods of sampling to achieve a representative part of the product to be analysed.

NOTE:Sampling procedures for the official control of pesticide residues in and on fruits and vegetables are given in EEC directive 79/700/EEC [5].

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7 Preparation of the samples

7.1 Test sample

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If the sample reaches the laboratory frozen, store it at - 20 °C before analysis.

Where possible, carry out the analysis of fresh samples immediately after their arrival in the laboratory. Do not analyse a laboratory sample which is wholly or extensively spoiled.

For analysis take only the portion of the laboratory sample to which the maximum residue limit applies. No further plant-parts may be removed. A record of the plant-parts which have been removed shall be kept. The sample thus prepared is the analytical sample.

If the sample cannot be analyzed immediately, it may be stored at 0 °C to 5 °C for no longer than 2 days before analysis.

The reduction of the analytical sample shall be carried out in such a way that representative portions are obtained (e.g. by division into four and selection of opposite quadrants). When the samples are in small units (e.g. small fruits, vegetables, cereals), the analytical sample shall be thoroughly mixed before weighing out the test portion. When the samples are in larger units, take wedge-shaped sections (e.g. large fruits and vegetables) or cross sections (e.g. cucumbers) which include the outer surface from each unit.

NOTE:The residues of dithiocarbamate and thiuram disulfide, which are on the surface of the plant-parts and are not systemic, decompose rapidly especially in chopped samples. Therefore precautions should be taken to avoid decomposition.

If samples have to be stored for more than 2 days, they shall be deep-frozen at - 20 °C. To ensure that even after thawing representative samples can be taken, prepare portions of the product which are each sufficient for one analysis.

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7.2 Test portion

Weigh out test portions of masses up to 200 g to an accuracy of ± 1 %. After weighing out the test portion, remove any parts which would interfere with the analytical procedure. In the case of stone fruits, the stones are removed after weighing out. The basis for the calculation of the residue mass fraction is the mass of the original test portion (with stones).

The test portion shall not be cut or reduced to smaller pieces than can just pass the neck of the reaction flask, as the residues of dithiocarbamate and thiuram disulfide fall the more the test portion is cut.

Analyse the test portion immediately after cutting.

8 Procedure

8.1 Safety aspects

WARNING:Many dithiocarbamates, thiuram disulfides, and carbon disulfide are toxic by various routes of exposure, especially in concentrated form. When working with dithiocarbamates, thiuram disulfides and carbon disulfide, consult safety data sheets of the manufacturer for information.

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Vapours from some volatile solvents are toxic. Several of these solvents can easily be absorbed through the skin. Use effective fume hoods to remove vapours of these solvents as they are set free.

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8.2 Preparation of blanks

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Prepare reagent and matrix blanks. Spiked recovery tests at levels appropriate to the maximum residue limits shall be carried out and shall lead to satisfying results.

The absorption for the reagent blanks against a solution made of 15 ml of colour reagent (4.15) and 10 ml of ethanol (4.4) in the reference cell shall be zero or near zero at a wavelength of 435 nm.

NOTE 1:Analysts should thoroughly familiarize themselves with the method before starting the analysis.

NOTE 2:Some vegetables (e.g. those of the family *Cruciferae*) contain naturally occurring compounds which release carbon disulfide under the conditions described in this draft European Standard. Therefore the analysis of such vegetables can lead to false positive results.

8.3 Preparation of the calibration curve

Add 15 ml of colour reagent (4.15) to each of ten 25 ml volumetric flasks. Then add 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of the carbon disulfide standard solution (4.14) (equivalent to 50 μg, 100 μg, 150 μg, 200 μg and 250 μg of CS_2) from a graduated pipette or a burette to each of two volumetric flasks. Dilute to the mark with ethanol, mix well, and let the 10 mixtures stand for 60 min. Perform the spectrometric measurement as described in 8.4.3.