International Standard ISO 6466

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Tobacco and tobacco products — Determination of dithiocarbamate pesticides residues — Molecular absorption spectrometric method

Tabac et produits du tabac — Détermination des résidus de pesticides dithiocarbamates — Méthode par spectrométrie d'absorption moléculaire

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Foreword

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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 6466 was developed by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, and was circulated to the member bodies in March 1982.

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

Austria
Belgium
Brazil
Cuba
Czechoslovakia
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France
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No member body expressed disapproval of the document.

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Tobacco and tobacco products — Determination of dithiocarbamate pesticides residues — Molecular absorption spectrometric method

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1 Scope and field of application

This International Standard specifies a molecular absorption spectrometric method for the determination of residues from pesticides of the dithiocarbamate type on tobacco and tobacco products.

The method is applicable to the determination, on tobacco and tobacco products, of residues from the dithiocarbamate pesticides commonly used on tobacco crops.

2 References

ISO 1750, *Pesticides and other agrochemicals — Common names.*

ISO 4874, *Tobacco — Sampling of batches of raw material — General principles.*

ISO 6488, *Tobacco — Determination of water content (Reference method).*

3 Definition

dithiocarbamate pesticides residues content : The amount of carbon disulphide, in milligrams per kilogram of sample, determined according to the method specified.

NOTE — If required and if the identity of the dithiocarbamate pesticide present in the sample is known, the dithiocarbamate pesticides residues content may be expressed additionally as the dithiocarbamate by using the appropriate factor (see 9.3).

4 Principle

Decomposition of the dithiocarbamates in a test portion by heating with hydrochloric acid in the presence of tin(II) chloride. Distillation of the carbon disulphide formed and absorption in a methanolic solution of potassium hydroxide after removal of interfering substances by passing through sulphuric acid. Spectrometric determination of the potassium-*O*-methyl dithiocarbonate formed.

5 Reagents

All reagents shall be of analytical reagent grade. Distilled water, or water of at least equivalent purity, shall be used.

5.1 Sulphuric acid, of concentration 96 to 98 % (*m*/*m*).

5.2 Potassium hydroxide, approximately 1 mol/l solution in 95 % (*V*/*V*) methanol. If there is any sediment, filter the solution, using fluted filter paper, before it is used.

5.3 Tin(II) chloride (solid $SnCl_2$).

5.4 Hydrochloric acid, aqueous solution.

Add 75 ml of hydrochloric acid of concentration 37 to 38 % (*m*/*m*) to 150 ml of distilled water.

5.5 Sodium diethyldithiocarbamate, standard solution corresponding to 10 mg of carbon disulphide per litre.

Dissolve 29,6 mg of sodium diethyldithiocarbamate trihydrate in 1 000 ml of water. Prepare the solution on the day of use.

1 ml of this standard solution is equivalent to 10 µg of carbon disulphide.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Distillation apparatus (see the figure), comprising

6.1.1 Round bottom flask with three necks (A), of capacity 250 ml.

6.1.2 Condenser (B).

6.1.3 Reservoir (C).

6.1.4 Inlet tube, through which nitrogen is introduced into the flask (D).

6.1.5 Gas wash bottles (E and F), fitted with sintered glass (pore size index 160 to 250 µm) distribution tubes for absorption of the distillation products.

NOTE — Instead of the gas wash bottles shown in the figure, other absorption devices may be used, provided that they have been shown to be equally efficient.

6.2 Source of nitrogen, fitted with a three-way stopcock.

6.3 Spectrometer, capable of measurement in the ultraviolet region at 272, 302 and 332 nm, equipped with 10 mm silica cells.

7 Sampling

Carry out sampling in accordance with the procedure specified in ISO 4874.

8 Procedure

8.1 Preparation of the test sample

If possible, use a test sample that is in the form of cut tobacco or cigarette filler, without further preparation. If a test sample in one of these forms is not available, cut the test sample into pieces of a suitable size.

NOTE — Grinding and drying of tobacco lead to loss of dithiocarbamates.

8.2 Test portions

8.2.1 Weigh, to the nearest 10 mg, duplicate test portions, each of approximately 5 g, from the test sample. Carry out on each test portion the procedure specified in 8.3 and 8.4.

8.2.2 Proceeding as specified in 8.2.1, take another test portion and use this to determine the water content of the test sample in accordance with ISO 6488, if the result is to be calculated on the basis of water-free tobacco.

8.3 Distillation

8.3.1 Transfer one of the test portions (8.2.1) to the flask (A) of the distillation apparatus (6.1) and add 2 g of the tin(II) chloride (5.3), followed by 50 ml of water. Shake the flask until all of the tobacco has been impregnated. Immediately this has been done, connect the flask to the condenser (B), which is connected to a gas wash bottle (E) containing 20 ml of the sulphuric acid (5.1) and then to a gas wash bottle (F) containing 25 ml of the potassium hydroxide solution (5.2). Put the reservoir (C) and inlet tube (D) in position. Make sure that there are no leaks.

Adjust the nitrogen supply to a flow rate of 50 ml/min, then connect this nitrogen flow to the apparatus via the three-way stopcock and inlet tube (D).

Heat the flask to between 30 and 40 °C and allow to remain as described for at least 10 min to ensure that all of the tobacco is well impregnated with tin(II) chloride and that any oxygen has been purged from the apparatus. The condenser (B) shall be well cooled to prevent water passing into the sulphuric acid in the first gas wash bottle (E).

Place 100 ml of the hydrochloric acid solution (5.4) into the reservoir (C). To prevent any suck back, turn the three-way stopcock so that the nitrogen supply is connected to the reservoir as well as passing into the flask through the inlet tube, then slowly add the hydrochloric acid solution in the reservoir to the flask. When all the hydrochloric acid solution has been added to the flask, turn the three-way stopcock so that the nitrogen flow of 50 ml/min passes totally through the inlet tube. Heat the contents of the flask to boiling point and boil gently for 30 min.

8.3.2 At the end of the 30 min period, stop heating, disconnect the gas wash bottles, and turn off the nitrogen supply. Transfer the contents of the second gas wash bottle (F) containing potassium hydroxide solution to a 50 ml one-mark volumetric flask. Wash the inside of the gas wash bottle and the distribution tube with water, adding the washings to the contents of the flask. Dilute to the mark with water, mix and allow to stand for 15 min.

8.4 Spectrometric measurement

Fill a 10 mm silica cell with the test solution obtained as specified in 8.3.2.

Measure the absorbance of the test solution using the spectrometer (6.3) at wavelengths of 272, 302 and 332 nm, using as the reference solution a mixture of 25 ml of the potassium hydroxide solution (5.2) diluted to 50 ml with water. The measured absorbance at 302 nm shall not be greater than 0,800, nor less than 0,100. If the absorbance is greater than 0,800, a further dilution or a smaller amount of tobacco shall be used. If the absorbance is less than 0,100, a silica cell of longer optical path length shall be used.

Calculate the corrected absorbance, A_{corr}, of the test solution from the formula

$$A_{\text{corr}} = A_{302} - \frac{A_{272} + A_{332}}{2}$$

where A_{272}, A_{302} and A_{332} are, respectively, the absorbances of the test solution at 272, 302 and 332 nm.

Repeat the determination on the duplicate test portion.

8.5 Calibration graph

Take, in turn, aliquot portions of 4, 6, 8, 10, 12 and 16 ml of the standard sodium diethyldithiocarbamate solution (5.5), corresponding to quantities between 40 and 160 µg of carbon disulphide, and treat as specified in 8.3, substituting the aliquot portion of the standard solution for the test portion.

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Measure the absorbance of the standard test solutions thus prepared, as specified in 8.4. Prepare a calibration graph of corrected absorbance values against the appropriate mass of carbon disulphide. The calibration graph has been found to be very reproducible and a full graph need not be prepared each day. A single point check is normally sufficient.

9 Expression of results

9.1 Method of calculation and formulae

9.1.1 Read from the calibration graph the amount of carbon disulphide present in the sample test solution.

9.1.2 The content of carbon disulphide, expressed in milligrams per kilogram of tobacco, is given by the formula

$$\mathrm{CS_2} = \frac{m}{m_0}$$

where

m is the mass, in micrograms, of carbon disulphide present in the sample test solution as read from the calibration graph;

m_0 is the mass, in grams, of tobacco taken as the test portion.

If the result is to be expressed on a water-free basis, use the formula

$$\mathrm{CS_2} = \frac{m}{m_0} \times \frac{100}{100 - H}$$

where H is the water content of the sample, expressed as a percentage by mass, determined in accordance with ISO 6488.

A correction shall be made for any further dilution (see 8.4).

Take as the result the mean of the two values, provided that the requirement for repeatability (9.2) is satisfied.

9.2 Repeatability

The difference between the results of duplicate determinations, carried out at the same time or rapidly one after another, by the same analyst, shall not exceed 7,5 % of their mean value.

9.3 Conversion factors

If required, the results expressed in terms of carbon disulphide may also be expressed in terms of any particular dithiocarbamate pesticide by multiplying by the appropriate factor. The following factors have been established :

a)	maneb :	1,74
b)	zineb :	1,81
c)	propineb :	1,90

NOTE — These three common names are included in ISO 1750, together with the systematic names and structures.

10 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all details required for the complete identification of the sample.

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Dimensions in millimetres

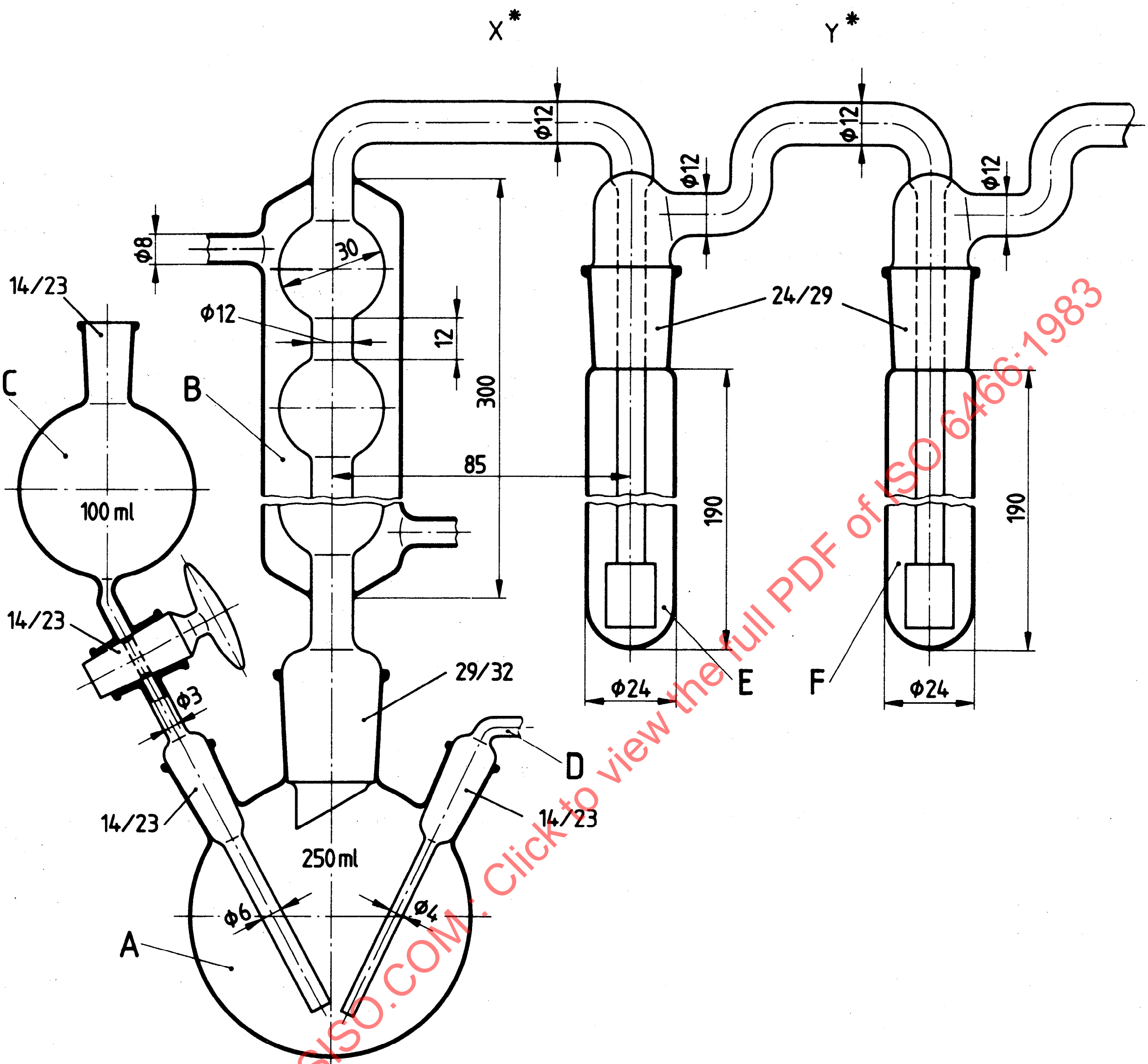

Figure — Distillation apparatus

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* To assist in washing the apparatus, joints may be fitted at points X and Y, provided that they are completely leak-proof and free of grease.