

INTERNATIONAL STANDARD

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Tea — Determination of water extract

Thé — Détermination de l'extrait à l'eau



Reference number
ISO 9768:1990(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9768 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

This first edition of ISO 9768 cancels and replaces ISO 1574:1980, of which it constitutes a technical revision.

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Tea — Determination of water extract

1 Scope

This International Standard specifies a method for the determination of the water extract from unground tea.

NOTE 1 Special sample preparation for very large leaf green and black teas may be required. Further work to determine the precise method of sample preparation for these teas is being undertaken.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1573:1980, *Tea — Determination of loss in mass at 103 °C*.

3 Definition

For the purposes of this International Standard, the following definition applies.

water extract: The soluble matter extracted from a test portion by boiling water, under the conditions specified in this International Standard, expressed as a percentage by mass on the dry basis.

4 Principle

Extraction of soluble matter from a test portion of the product by means of water boiling under reflux. Filtration, washing, drying and weighing of the hot-water-insoluble residue. Calculation of the water extract.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Oven, constant-temperature and fan-assisted, capable of being controlled at $103\text{ °C} \pm 2\text{ °C}$.

5.2 Crucible, made of sintered borosilicate glass, of porosity grade P160, 40 mm in diameter and of 70 ml capacity.

5.3 Desiccator, containing an efficient desiccant.

5.4 Flask, of 500 ml capacity, fitted with a reflux condenser.

5.5 Filter flask, of 1 litre capacity, for vacuum filtration.

6 Test sample

Use an unground test sample of known dry matter content, determined using the method specified in ISO 1573.

7 Procedure

7.1 Preparation of the crucible

Heat the clean crucible (5.2) for 1 h in the oven (5.1) at $103\text{ °C} \pm 2\text{ °C}$. Cool in the desiccator (5.3) and weigh to the nearest 0,001 g.

7.2 Test portion

Weigh, to the nearest 0,001 g, 2 g of the unground test sample (clause 6) into the flask (5.4).

7.3 Determination

Add to the test portion (7.2) 200 ml of hot distilled water, or water of at least equivalent purity, and reflux over low heat for 1 h, rotating the flask oc-

asionally. Filter hot under vacuum through the prepared crucible (7.1) using the filter flask (5.5). Repeatedly wash out the flask with hot distilled water, transferring all the insoluble residue into the crucible. Finally, wash the residue with 200 ml of hot distilled water. Dry the residue by suction. Heat the crucible and its contents in the oven (5.1) controlled at $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 16 h (i.e. overnight). Cool in the desiccator (5.3) and weigh to the nearest 0,001 g.

7.4 Number of determinations

Carry out two determinations on the same unground test sample (clause 6).

8 Expression of results

The water extract yielded by the unground test sample, expressed as a percentage by mass on the dry basis, is given by the formula

$$\frac{(m_0 \times RS) - (m_1 \times 100)}{m_0 \times RS} \times 100$$

where

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in grams, of the dried insoluble residue;
- RS is the dry matter content, expressed as a percentage by mass, of the unground test sample. It is equal to 100 minus the loss in mass at $103\text{ }^{\circ}\text{C}$ determined using the method specified in ISO 1573.

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

9 Precision

9.1 Statistical results of inter-laboratory tests

Four inter-laboratory tests, carried out between 1984 and 1989 under the auspices of the International Organization for Standardization, gave the statistical results (evaluated in accordance with ISO 5725¹⁾) shown in table 1.

Table 1 — Statistical results of inter-laboratory tests

Year	1984	1986	1988	1989
Number of laboratories	7	21	16	10
Number of samples	3	6	6	3
Repeatability, r	0,877 to 1,259	0,677 to 1,114	1,37 to 1,60	0,50 to 0,63
Reproducibility, R	1,252 to 1,422	1,871 to 2,934	4,69 to 6,19	1,02 to 1,45

9.2 Repeatability

The difference between the values of two determinations, carried out in rapid succession (or simultaneously) by the same operator using the same apparatus on the same test sample, shall not exceed 1,0 % (m/m).

9.3 Reproducibility

The difference between the values of the final result obtained by two laboratories using this method for the analysis of the same laboratory sample is not expected to exceed 2,5 % (m/m) (95 % confidence level).

10 Test report

The test report shall specify the method used and the result obtained. It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result.

The test report shall include all information necessary for the complete identification of the sample.

1) ISO 5725:1986, Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.

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Steel and iron — Review of available methods of analysis

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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.

The main task of ISO technical committees is to prepare International Standards. In exceptional circumstances a technical committee may propose the publication of a technical report of one of the following types :

- type 1, when the necessary support within the technical committee cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development, requiring wider exposure;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical reports are accepted for publication directly by ISO Council. Technical reports types 1 and 2 are subject to review within three years of publication, to decide if they can be transformed into International Standards. Technical reports type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9769 was prepared by Technical Committee ISO/TC 17, *Steel*.

It was decided to publish this document in the form of a technical report type 3.

Aciers et fontes — Vue d'ensemble des méthodes d'analyse disponibles

L'ISO (Organisation internationale de normalisation) est une fédération mondiale d'organismes nationaux de normalisation (comités membres de l'ISO). L'élaboration des Normes internationales est en général confiée aux comités techniques de l'ISO. Chaque comité membre intéressé par une étude a le droit de faire partie du comité technique créé à cet effet. Les organisations internationales, gouvernementales et non gouvernementales, en liaison avec l'ISO participent également aux travaux.

La tâche principale des comités techniques de l'ISO est d'élaborer les Normes internationales. Exceptionnellement, un comité technique peut proposer la publication d'un rapport technique de l'un des types suivants :

- type 1: lorsque, en dépit de maints efforts au sein d'un comité technique, l'accord requis ne peut être réalisé en faveur de la publication d'une Norme internationale;
- type 2: lorsque le sujet en question est encore en cours de développement technique et requiert une plus grande expérience;
- type 3: lorsqu'un comité technique a réuni des données de nature différente de celles qui sont normalement publiées comme Normes internationales (ceci pouvant comprendre des informations sur l'état de la technique, par exemple).

La publication des rapports techniques dépend directement de l'acceptation du Conseil de l'ISO. Les rapports techniques des types 1 et 2 font l'objet d'un nouvel examen trois ans au plus tard après leur publication afin de décider éventuellement de leur transformation en Normes internationales. Les rapports techniques du type 3 ne doivent pas nécessairement être révisés avant que les données fournies ne soient plus jugées valables ou utiles.

L'ISO/TR 9769 a été élaboré par le comité technique ISO/TC 17, *Acier*.

Il a été décidé de publier le présent document sous forme de rapport technique du type 3.

1 Scope and field of application

This Technical Report aims to facilitate reference to the available international standard method(s) for the determination of required element(s) in steel and iron.

In this Technical Report, field of application, method of determination (principle) and precision (see annex B) of each standard are stated.

1 Objet et domaine d'application

Le présent Rapport technique vise à faciliter la référence à la (aux) norme(s) internationale(s) disponible(s) pour le dosage de l'(des) élément(s) requis dans les aciers et fontes.

Dans ce présent Rapport technique sont exposés le domaine d'application, la méthode de dosage (principe) et les données de fidélité (voir annexe B) de chaque norme.

2 List of International Standards Liste de Normes internationales

2.1 Carbon Carbone

Reference	Ed Pages	Title	Titre
ISO 437-1982	4	Steel and cast iron - Determination of total carbon content - Combustion gravimetric method	Aciers et fontes - Dosage du carbone total - Méthode gravimétrique après combustion
ISO/TR 4830/1 -1978	6	Steel - Determination of low carbon contents - Part I : Manometric (low-pressure) method after combustion	Acier - Dosage du carbone en faibles teneurs - Partie I : Méthode manométrique (basse pression) après combustion
ISO/TR 4830/2 -1978	10	Steel - Determination of low carbon contents - Part II : Titrimetric method after combustion	Acier - Dosage du carbone en faibles teneurs - Partie II : Méthode titrimétrique après combustion
ISO/TR 4830/3 -1978	8	Steel - Determination of low carbon contents - Part III : Conductimetric measurement after combustion	Acier - Dosage du carbone en faibles teneurs - Partie III : Méthode conductimétrique après combustion
ISO/TR 4830/4 -1978	8	Steel - Determination of low carbon contents - Part IV : Coulometric method after combustion	Acier - Dosage du carbone en faibles teneurs - Partie IV : Méthode coulométrique après combustion

2.2 Chromium Chrome

Reference	Ed Pages	Title	Titre
ISO 4936-1984	3	Steel and cast iron - Determination of chromium content - Diphenylcarbazide spectrophotometric method	Aciers et fontes - Dosage du chrome - Méthode spectrophotométrique à la diphénylcarbazide
ISO 4937-1986	8	Steel and iron - Determination of chromium content - Potentiometric or visual titration method	Aciers et fontes - Dosage du chrome - Méthode par titrage potentiométrique ou visuel

2.3 Copper Cuivre

Reference	Ed Pages	Title	Titre
ISO 4943-1985	6	Steel and cast iron - Determination of copper content - Flame atomic absorption spectrometric method	Aciers et fontes - Dosage du cuivre - Méthode par spectrométrie d'absorption atomique dans la flamme
ISO 4946-1984	6	Steel and cast iron - Determination of copper content - 2,2'-Diquinolyl spectrophotometric method	Aciers et fontes - Dosage du cuivre - Méthode spectrophotométrique au 2,2'-biquinolyle

2.4 Manganese Manganèse

Reference	Ed Pages	Title	Titre
ISO 629-1982	4	Steel and cast iron - Determination of manganese content - Spectrophotometric method	Aciers et fontes - Dosage du manganèse - Méthode spectrophotométrique

2.5 Molybdenum Molybdène

Reference	Ed Pages	Title	Titre
ISO 4941-1978	4	Steels and cast irons - Determination of molybdenum content - Photometric method	Aciers et fontes - Dosage du molybdène - Méthode photométrique

2.6 Nickel Nickel

Reference	Ed Pages	Title	Titre
ISO/DIS 4938		Steel and iron - Determination of nickel content - Gravimetric or titrimetric method	Aciers et fontes - Dosage du nickel - Méthode gravimétrique ou titrimétrique
ISO 4939-1984	6	Steel and cast iron - Determination of nickel content - Dimethylglyoxime spectrophotometric method	Aciers et fontes - Dosage du nickel - Méthode spectrophotométrique à la diméthylglyoxime
ISO 4940-1985	7	Steel and cast iron - Determination of nickel content - Flame atomic absorption spectrometric method	Aciers et fontes - Dosage du nickel - Méthode par spectrométrie d'absorption atomique dans la flamme

2.7 Niobium Niobium

Reference	Ed Pages	Title	Titre
ISO/DIS 9441		Steel - Determination of niobium content - PAR spectrophotometric method	Aciers - Dosage du niobium - Méthode spectrophotométrique au PAR

2.8 Nitrogen Azote

Reference	Ed Pages	Title	Titre
ISO 4945-1977	6	Steel - Determination of nitrogen content - Spectrophotometric method	Acier - Dosage de l'azote - Méthode spectrophotométrique

2.9 Phosphorus Phosphore

Reference	Ed Pages	Title	Titre
ISO 2732-1984	2 3	Steel and cast iron - Determination of phosphorus content - Phosphovanadomolybdate spectrophotometric method	Aciers et fontes - Dosage du phosphore - Méthode spectrophotométrique au phosphovanadomolybdate

2.10 Silicon Silicium

Reference	Ed Pages	Title	Titre
ISO 439-1982	3	Steel and cast iron - Determination of total silicon - Gravimetric method	Aciers et fontes - Dosage du silicium total - Méthode gravimétrique
ISO 4829/1-1986	7	Steel and cast iron - Determination of total silicon content - Reduced molybdsilicate spectrophotometric method - Part 1 : Silicon contents between 0,05 and 1,0 %	Aciers et fontes - Dosage du silicium total - Méthode spectrophotométrique au molybdsilicate réduit - Partie 1 : Teneurs en Silicium entre 0,05 et 1,0 %
ISO/DIS 4829/2		Steel and iron - Determination of total silicon content - Reduced molybdsilicate spectrophotometric method - Part 2 : Silicon contents between 0,01 and 0,05 %	Aciers et fontes - Dosage du silicium total - Méthode spectrophotométrique au molybdsilicate réduit - Partie 2 : Teneurs en Silicium entre 0,01 et 0,05 %

2.11 Sulfur Soufre

Reference	Ed	Pages	Title	Titre
ISO 671-1982		5	Steel and cast iron - Determination of sulphur content - Combustion titrimetric method	Aciers et fontes - Dosage du soufre - Méthode titrimétrique après combustion
ISO 4934-1980		5	Steel and cast iron - Determination of sulphur content - Gravimetric method	Aciers et fontes - Dosage du soufre - Méthode gravimétrique

2.12 Vanadium Vanadium

Reference	Ed	Pages	Title	Titre
ISO/DIS 4942			Steel and iron - Determination of vanadium content - N-BPHA spectrophotometric method	Aciers et fontes - Dosage du vanadium - Méthode spectrophotométrique au N-BPHA
ISO 4947-1986		6	Steel and cast iron - Determination of vanadium content - Potentiometric titration method	Aciers et fontes - Dosage du vanadium - Méthode par titrage potentiométrique

(1) CARBON (total)

Reference	Field of application		Method of determination (Principle)
		Concentration range	
ISO 437-1982	Steel and cast iron	not less than 0,1 % (<u>m/m</u>)	<p>Combustion gravimetric method</p> <p>Combustion of a test portion at a high temperature (1 200 to 1 350°C) in a current of pure oxygen, if necessary in the presence of a flux and transformation of carbon into carbon dioxide.</p> <p>Absorption of the carbon dioxide carried by current of oxygen in soda asbestos contained in a weighed absorption bulb, and determination of the increase in mass.</p>
ISO/TR 4830/1-1978	Steel	0,002 to 0,1 % (<u>m/m</u>)	<p>Combustion manometric (low-pressure) method</p> <p>Combustion of a test portion at a high temperature (1 200 to 1 350°C, depending on the type of material) in a current of pure oxygen, converting the carbon to carbon dioxide. Entrainment of the carbon dioxide by the current of oxygen, elimination of oxides of sulphur and of water vapour, and separation of the carbon dioxide by solidification in a freezing trap immersed in liquid oxygen.</p> <p>After elimination of oxygen from the measuring system, vaporization of the carbon dioxide, by raising the temperature, into a calibrated volume which has been evacuated. Determination of the carbon content of the test portion by measuring the pressure in the volume which has previously been calibrated using known volumes of carbon dioxide.</p>

(1) CARBONE (total)

Référence	Domaine d'application		Méthode de dosage (Principe)
		Gamme de concentration	
ISO 437-1982	Aciers et fontes	égales ou supérieures à 0,1 % (<u>m/m</u>)	<p>Méthode gravimétrique après combustion</p> <p>Combustion de la prise d'essai à une température élevée (1 200 à 1 350°C) dans un courant d'oxygène pur, si nécessaire, en présence d'un fondant et transformation du carbone en dioxyde de carbone.</p> <p>Absorption par de l'amiante sodée contenue dans des blocs absorbants tarés, du dioxyde de carbone entraîné par un courant d'oxygène, et détermination de l'augmentation de masse.</p>
ISO/TR 4830/1-1978	Acier	0,002 à 0,1 % (<u>m/m</u>)	<p>Méthode manométrique (basse pression) après combustion</p> <p>Combustion d'une prise d'essai à température élevée (1 200 à 1 350°C, selon la qualité du matériau) dans un courant d'oxygène pur et transformation du carbone en dioxyde de carbone. Entrainement du dioxyde de carbone par le courant d'oxygène, élimination des oxydes de soufre et de la vapeur d'eau, puis séparation du dioxyde de carbone par solidification dans une trappe réfrigérée par immersion dans de l'oxygène liquide.</p> <p>Vaporisation du dioxyde de carbone, après élimination de l'oxygène du système de mesure par élévation de la température, dans une capacité de volume connu dans laquelle on a préalablement fait le vide. Déduction de la teneur en carbone de la prise d'essai de la mesure de la pression dans la capacité étalonée préalablement à l'aide de volumes connus de dioxyde de carbone.</p>