
**Rubber, raw — Determination of water
content by Karl Fischer method**

*Caoutchouc brut — Détermination de la teneur en eau par la
méthode de Karl Fischer*

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ISO 12492:2012

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12492 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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Introduction

The water content of raw rubber is one of the important characteristics to be determined as a quality control test. Different synthetic rubbers contain varying amounts of water. Water can affect product quality, texture, shelf life, chemical stability and reactivity. A high amount of water can cause processing difficulties.

Water contamination is a cause for major concern in a large number of applications. In the rubber industry, water is one of the major damaging contaminants and is often overlooked as a primary cause of component failure. For certain applications in the rubber industry, even a small amount of water may have damaging effects on production.

Several methods are available for the determination of water content. A Karl Fischer (KF) coulometric titrator is one of the most accurate methods. Unlike other techniques, it can trace low levels of free, emulsified and dissolved water (which cannot be detected with normal gravimetric methods). The test is capable of measuring water levels as low as 0,01 %.

Unlike gravimetric measurements, which are indirect methods that assume, all volatiles removed are water, Karl Fischer titration is a direct method that is almost specific for water. The method is especially useful for low moisture levels (<1 %).

The new test method will help to determine the water content of the raw rubber and rubber compounds in shortest possible time and will be helpful for quality control at the laboratories. Coulometric determination of water is an absolute method.

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Rubber, raw — Determination of water content by Karl Fischer method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard specifies a test method for the determination of water content of raw rubber and compounded rubber using a coulometric Karl Fischer titration method. It applies to the water content range between 0,01 % and 1 %. As this is a very sensitive method, contact of sample with any moisture, even from the surrounding environment, must be eliminated as much as possible.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

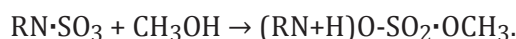
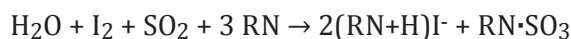
ISO 760, *Determination of water — Karl Fischer method (General method)*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedure*

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

3 Principle

The water determination test (Karl Fischer method) is designed to determine the water content in substances, utilizing the quantitative reaction of water with iodine and sulfur dioxide in the presence of a lower alcohol such as methanol and an organic base, as shown in the following formulae:

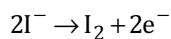


There are two determination methods which differ in the way the iodine is provided: the volumetric titration method and the coulometric titration method.

In the volumetric titration method, iodine required for reaction with water is previously dissolved and the water content is determined by measuring the amount of iodine consumed as a result of reaction with water present in a sample.

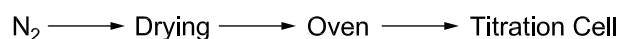
In the coulometric titration method, first, iodine is produced by electrolysis of the reagent containing iodide ion, then the water content in a sample is determined by measuring the quantity of electricity which is required for the electrolysis, i.e. for the production of iodine, based on the quantitative reaction of the generated iodine with water.

In coulometric Karl Fischer titration, iodine (I₂) is generated electrochemically from iodide (I⁻). When iodine (I₂) comes in contact with the water in the sample, water is titrated according to the above mentioned reaction scheme. Once all of the water available has reacted, the reaction is complete. The amount of water in the sample is calculated by measuring the current needed for the electrochemical generation of iodine (I₂) from iodide (I⁻) according to the following reaction:



According to Faraday's Law, the quantity of the iodine produced is proportional to the current generated. In the above equation, I₂ and H₂O react with each other in proportion of 1:1. Therefore, a mole of water (18 g) is equivalent to 2 × 96 500 coulombs, or 10,72 coulombs/1 mg of H₂O.

Conveying the water contained within the sample to the titration cell is an important part of the titration. The volumetric flow rate of the carrier (nitrogen) gas is precisely controlled by the flow controller. The sample remains in the sample boat. The carrier gas is dried by passing it through silica gel and zeolite type desiccants. The dry carrier gas enters the sample heating chamber and carries the total moisture into the titration cell. The carrier gas passes through the titration cell as long for as the sample is being heated. In this process, the major quantity of water is released at the beginning. The set-up and the connection of the oven are illustrated by the figure below:



Thus, the total amount of moisture can be determined by measuring the total consumption of electricity.

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4 Reagents

- 4.1 **Water standard for coulometric Karl Fischer titration**, 0,1 % (NIST Traceable).
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- 4.2 **Anode solution** (for use when a titration cell with a diaphragm is being used).
- 4.3 **Cathode solution** (for use when a titration cell with a diaphragm is being used).
- 4.4 **Universal reagent** (for use when a titration cell without a diaphragm is being used).
- 4.5 **Dry N₂ gas of instrument grade**.
- 4.6 **Aluminium oxide**.
- 4.7 **Ethanol**.
- 4.8 **Concentrated HNO₃**.
- 4.9 **Hexane**.

5 Apparatus

5.1 **Coulometric Karl Fischer titrator with evaporator**. Different components of coulometric Karl Fischer titrator are shown in Figure 1.

The water evaporator consists of an oven capable of heating the test portion to 300 °C, a heating tube, a temperature control unit, a carrier-gas flow meter and carrier-gas drying tubes containing desiccant.

5.2 **Analytical balance**, capable of weighing to the nearest 0,0000 1 g.

5.3 **Micro-syringe**, calibrated.

5.4 **Sample holder**.

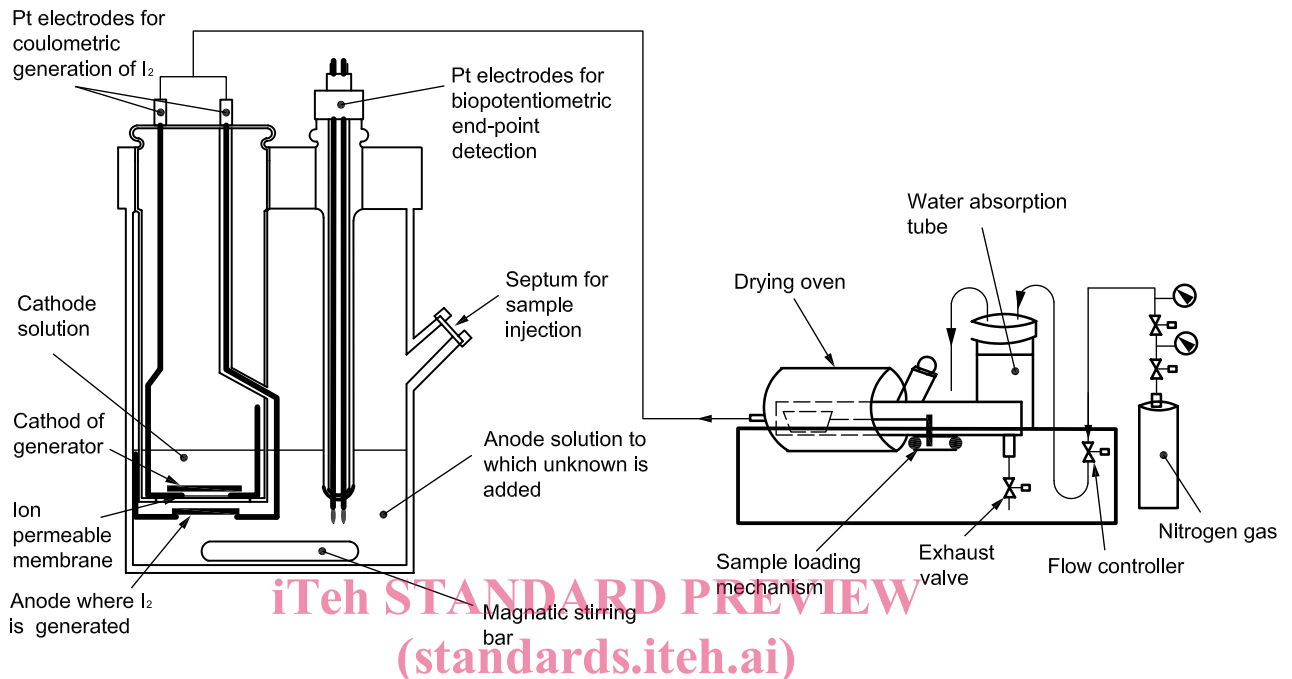


Figure 1 — Different components of coulometric Karl Fischer titration equipment

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6 Calibration

6.1 Standardize the instrument using a traceable water standard (4.1) and determine the % water content to check the recovery.

6.2 The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

7 Sampling and preparation of test piece

7.1 Take the laboratory sample in accordance with the method specified in ISO 1795 and prepare the test piece of 0,5 g to 1 g from the laboratory sample. Cut the test piece into small thin pieces using suitable tools such as scissors or a knife.

7.2 The test piece size should be small so that as many tests as possible can be carried out using the same electrolyte solution and the titration time can be kept short.

8 Procedure

8.1 Power on

Turn on the power to the instrument as per the instructions of the instrument manufacturer.