International Standard 5381

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Starch hydrolysis products — Determination of water content — Modified Karl Fischer method

Produits d'hydrolyse de l'amidon ou de la fécule — Dosage de l'eau — Méthode Karl Fisher modifiée

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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 5381 was developed by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, and was circulated to the member bodies in April 1982.

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

Canada	Germany, F.R.	South Africa, Rep. of
Egypt, Arab Rep. of	Netherlands	USA
France	Poland	USSR

No member body expressed disapproval of the document.

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0 Introduction

This International Standard is based on the method described in ISO 760. However, it has been improved by determining the content directly using the methanol/formamide solvent.

1 Scope and field of application

This International Standard specifies a method for the determination of the water content of starch hydrolysis products.

2 Reference

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

3 Principle

Reaction of a solution of iodine, sulphur dioxide, pyridine and 2-methoxyethanol (stabilized Karl Fischer reagent) with the water contained in the product dispersed previously in a mixture of methanol and formamide.

4 Reactions

$$H_2O + I_2 + SO_2 + 3C_5H_5N \rightarrow 2C_5H_5N.HI + C_5H_5N.SO_3$$

$$C_5H_5N.SO_3 + ROH \rightarrow C_5H_5NH.OSO_2OR$$

where R is the 2-methoxyethyl radical.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity; all solvents shall have a water content of less than 0,1 % (*m/m*).

5.1 "Stabilized" Karl Fischer reagent.

The stabilized reagent is commercially available in the form of a prepared solution. It is also possible to prepare the reagent in the laboratory (see ISO 760).

5.2 Methanol/formamide solvent.

Mix 700 ml of anhydrous methanol with 300 ml of anhydrous formamide.

This reagent shall be handled with care.

5.3 Sodium tartrate, crystalline ($Na_2C_4H_4O_6 \cdot 2H_2O$).

This product is commercially available in the form "special quality for Karl Fischer". If this quality cannot be obtained, wash the tartrate with 10 ml of the methanol/formamide solvent (5.2), and carry out an appropriate blank test.

Crush the product so that it passes completely through a sieve of nominal aperture size 250 µm, complying with the requirements of ISO 565. The water content of this hydrate is approximately 15,66 % (*m/m*) and shall be checked by vacuum drying at 150 °C until constant mass is obtained.

6 Apparatus

Ordinary laboratory apparatus, in particular

6.1 One-mark pipette, of capacity 20 ml.

6.2 Devices, into which the sample to be analysed can be introduced.

6.2.1 Weighing tube (for solid products), consisting of a test tube of suitable diameter so as to allow the introduction of samples, and fitted with a stopper.

6.2.2 Syringe (for viscous liquids), of capacity 10 ml, for example in accordance with annex A.