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International Standard



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Tobacco and tobacco products — Determination of maleic hydrazide residues

*Tabac et produits du tabac — Détermination des résidus d'hydrazide maléique*

First edition — 1980-04-01

Sample Document

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UDC 663.97 : 543.8

Ref. No. ISO 4876-1980 (E)

**Descriptors :** tobacco, chemical analysis, determination of content, residues, spectrophotometric analysis, test equipment.

## 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 4876 was developed by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, and was circulated to the member bodies in November 1978.

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

Australia	Ireland	Sweden
Belgium	Italy	Switzerland
Brazil	Korea, Rep. of	Thailand
Bulgaria	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	Romania	USSR
Greece	South Africa, Rep. of	Yugoslavia
India	Spain	

The member body of the following country expressed disapproval of the document on technical grounds :

Germany, F. R.

# Tobacco and tobacco products — Determination of maleic hydrazide residues

## 1 Scope and field of application

This International Standard specifies a method for the determination of maleic hydrazide residues in tobacco and tobacco products.

The method is applicable to tobacco and tobacco products and to the residues from maleic hydrazide sucker control agents used on tobacco crops.

## 2 References

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

ISO 1770, *Solid-stem general purpose thermometers*.<sup>1)</sup>

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*.<sup>2)</sup>

ISO 4874, *Tobacco and tobacco products — General conditions of sampling*.<sup>2)</sup>

## 3 Principle

Boiling of the test portion with sodium hydroxide solution to remove volatile basic compounds. Addition of granulated zinc and reduction by the nascent hydrogen of the maleic hydrazide to succinic hydrazide which is then hydrolyzed. Distillation of the liberated hydrazine and spectrophotometric determination of its yellow complex with 4-dimethylaminobenzaldehyde.

If necessary, an acid pre-digestion of the test portion and a carbon clean-up of the distillate may be included.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

**4.1 4-dimethylaminobenzaldehyde**, 20 g/l solution in 0,5 M sulphuric acid solution.

### 4.1.1 Purification of reagent

Dissolve 20 g of 4-dimethylaminobenzaldehyde in 150 ml of absolute ethanol. Add 5 g of powdered activated charcoal and stir mechanically for 5 min. Filter through a Buchner funnel and add, slowly and with constant stirring, 200 ml of water at 0 °C to the filtrate. Filter the white or pale yellow crystals on a Buchner funnel and wash the crystals with 50 ml of cold water. Dry in a vacuum desiccator over phosphorus(V) oxide and store in a dark bottle.

### 4.1.2 Preparation of reagent solution

Dissolve 2 g of the purified crystals in 100 ml of the 0,5 M sulphuric acid solution (4.6); filter through a sintered glass filter funnel, if necessary.

The reagent is stable for up to 1 month if stored in the dark in a refrigerator; otherwise fresh reagent should be prepared daily.

**4.2 Maleic hydrazide**, standard solution corresponding to 10 µg/ml.

Weigh, to the nearest 0,1 mg, 10 mg of pure maleic hydrazide, dissolve it in 100 ml of 0,1 M sodium hydroxide solution (4.5) and dilute to 1 000 ml with water.

**4.3 Granulated zinc**, of particle size 500 µm, having a bulk density of not more than 1,70 g/cm<sup>3</sup>.

It has been found that the grade of zinc used is of prime importance. It is recommended that the zinc to be used should be checked by comparing the colour produced by standard solutions of hydrazine sulphate and 4-dimethylaminobenzaldehyde with that from maleic hydrazide after reduction and distillation.

**4.4 Sodium hydroxide**, 12,5 M solution.

**4.5 Sodium hydroxide**, 0,1 M solution.

**4.6 Sulphuric acid**, 0,5 M solution.

1) At present at the stage of draft. (Revision of ISO/R 1770-1970).

2) At present at the stage of draft.