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**Graphic technology — Ink, paper and
labels — Requirements on hot alkali
penetration and resistance**

*Technologie graphique — Encres, papier et étiquettes — Exigences de
pénétrabilité et résistance aux alcalins chauds*



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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 130, *Graphic technology*.

Introduction

Bottles or other containers for beverages and soft drinks may be labeled with wet glued printed labels of various substrates. If the bottles and containers are reused, they need to be cleaned thoroughly. Often, bottles and containers are used for different types of drinks. This requires the removal of the labels from the bottles and containers during the cleaning process. Usually, hot alkaline solution is used within the cleaning facilities. To ensure the stability of the process, the penetration of the cleaning solution through the label needs to occur rapidly. In addition, the labels need to stay intact to ease the process of removing them from the cleaning agent. Also, no dyeing of the solution must occur so as to minimize costs for the waste treatment of the cleaning solutions after neutralization. The test methods given in this International Standard have been successfully applied in the European beverage industry for decades and are also standardized as DIN Standards.

Graphic technology — Ink, paper and labels — Requirements on hot alkali penetration and resistance

1 Scope

This International Standard specifies test methods for

- penetrability and removal times of labels that have been wet glued on bottles intended to be refilled and reused, and
- resistance of printed labels against hot alkaline solution.

Test results always deal with systems comprising of substrates, ink films and varnish films if present. This International Standard is valid for label substrates and printed labels. Label substrates include metalized papers. The test procedures are also valid for the evaluation of inks and papers to be used in printing labels.

This International Standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1

penetration time

time of travel for a liquid through a substrate

2.2

removal time

time of separation of a glued label from a glass substrate

2.3

thymolphthaleine

organic chemical compound that is employed as an acid-base indicator, changing in appearance from red to yellow to blue as the pH changes from acidic (low pH) to neutral (pH = 7) to basic (high pH)

2.4

wire bar

coating device consisting of a metal rod covered with a tightly wound wire, in which the applied coating thickness is a function of the diameter of the wire (also known as a wire-wound rod)

3 Principle

Labels of bottles intended to be refilled and reused are removed from the bottles by applying hot alkaline solution.

3.1 Test method for the penetration time of hot alkaline solution on labels

To test if the hot alkaline solution is able to penetrate printed labels or label substrates from bottles within a required time, a glue, containing thymolphthalein solution is applied on the glue side of the label, the label is then glued onto the bottom of a petri dish and the dish is brought into contact with the hot alkaline solution. Appearance of a blue colour indicates the penetration of the hot alkaline solution

through the label. The experimenter records the time elapsed between adding the alkaline solution and the appearance of the blue colour.

3.2 Test method for the removal time of labels by hot alkaline solution

To test the time required to remove glued labels or label substrates from bottles, the test of [3.1](#) is continued until the labels are separated from the petri dish.

3.3 Test method for the resistance of labels against hot alkaline solution

To test the resistance of labels against hot alkaline solution, a specified area of printed labels will be placed in contact with a specified solution of NaOH at a defined temperature and for a specified period of time. The evaluation includes inspection of the surface of the labels [layer separation (ink and paper), colour changes] and the colour of the solution. To evaluate the usability of inks, either test prints on label stock or printed labels shall be used. The ink film thickness of test prints shall be comparable to that of printed labels.

4 Apparatus and reagents

4.1 Apparatus

4.1.1 **Beaker**, diameter ≥ 140 mm, volume about 2 l.

4.1.2 **Drying cabinet**.

4.1.3 **Filter hopper**.

4.1.4 **Glass plates**, 2, 60 mm \times 90 mm \times 2 mm.

4.1.5 **Hot plate**, equipped with thermostat and magnetic stirrer.

4.1.6 **Beaker insert for the beaker**, to avoid direct contact between labels and the magnetic stirrer.

4.1.7 **Laboratory filter paper**, for qualitative chemical analysis, white, neutral.

4.1.8 **Laboratory funnel**.

4.1.9 **Petri dish**, 90 mm to 100 mm in diameter.

4.1.10 **Safety glasses and protective gloves**.

4.1.11 **Stopwatch**.

4.1.12 **Tweezers**.

4.1.13 **Wire bar**, capable of applying wet films of a thickness of 50 μm .

4.2 Reagents

4.2.1 **1 % sodium hydroxide solution (by mass)**, for testing the penetrability and the removal time of labels, prepared from demineralized water. This solution may be used for up to 20 tests.

4.2.2 2,5 % sodium hydroxide solution (by mass), for testing the resistance of labels against hot alkaline solution, prepared from demineralized water. This solution shall contain commercial additives used in industrial bottle cleaning facilities in typical concentrations (e.g. 0,2 %).

4.2.3 Thymolphthaleine, ground into powder form.

4.2.4 Ethanol.

4.2.5 pH-neutral casein glue.

4.2.6 Demineralized water.

4.3 Preparation of the thymolphthaleine containing glue

Two spatula tips of thymolphthaleine are dissolved in 50 ml ethanol and filtered. Five parts of this solution are mixed with 95 parts of pH-neutral casein glue. Both the thymolphthaleine solution and the thymolphthaleine containing glue should not be older than one week. The pH neutral casein glue should not be older than one year.

5 Specimens

Inks shall be completely dried/cured, e.g. oxidative drying inks should be stored for 48 h prior to testing.

5.1 Penetration and removal time

At least three samples shall be tested. Circular specimens with a preferred diameter of 80 mm are cut out of larger sheets. Smaller die-cut labels may be used.

5.2 Resistance against hot alkaline solution

Multiple specimens shall be tested. They shall have an area of (500 ± 50) cm².

6 Test procedure

Hot alkaline solution might cause severe etching of the human skin. Hot alkaline solution may irreversibly damage the eye if droplets are spilled into the eye. Safety glasses and protective gloves shall be used.

6.1 Penetrability time of hot sodium hydroxide solution through labels

The glue is coated onto a glass plate using the wire bar. The size of the coated area shall be larger in all dimensions than the label specimen to be tested. The specimen is placed onto the coated area by means of tweezers and uniformly pressed onto the glass plate by a rubber roller. The label is removed from the glass plate using tweezers at an angle of 90°. The label is glued onto the outer bottom of the petri dish without bubbles and dried for 24 h at room temperature.

The petri dish with the specimen facing downwards is submerged into 1 000 ml hot 1 % sodium hydroxide solution in the beaker and placed on the beaker insert. The temperature of the sodium hydroxide solution shall be (75 ± 2) °C if labels for glass bottles are to be tested and shall be adapted to the real temperature of the cleaning equipment if labels for plastic bottles are to be tested. The hot sodium hydroxide solution shall be stirred magnetically with (250 ± 50) revolutions per minute. The time needed for the penetration of the sodium hydroxide solution through the label is measured. The endpoint is reached if three quarters of the specimen area have turned blue.

6.2 Removal time of labels

The test is performed according to [6.1](#). The time is measured from the start of the test until the label separates from the petri dish.

6.3 Resistance against hot alkaline solution

Place 1 000 ml 2,5 % sodium hydroxide solution in the beaker together with the beaker insert and a magnetic stirring bar. The solution is stirred by a magnetic stirrer at a speed of (250 ± 50) revolutions per minute and heated to 85 °C. Specimens are placed into the label insert and held for a period of 20 min. Multiple specimens shall be placed into the solution one by one using tweezers. The specimens are removed from the solution one by one after shaking individually in order to separate labels and to ensure the surface of the label is in contact with the alkaline solution. The specimens are dried on laboratory filter paper. The alkaline solution is filtered using laboratory filter paper and a laboratory funnel.

NOTE 1 The filtered alkaline solution may be used for the determination of the chemical oxygen demand (COD) and/or the absorbable organic halogenated compounds (AOX). The amount of the filter residue may be weighed and expressed in g per 500 cm².

Resistance against hot alkaline solution requires

- integrity of the substrate (no physical separation of the substrate fibers),
- integrity of the printed image (discolouration is possible for non-alkali resistant inks) (samples on metalized substrates may show larger ink removal), and
- no colouration of the alkaline solution (after filtration).

NOTE 2 There can be very fine solid ink particles present, erroneously simulating a coloration of the solution, which might not be able to be filtered off. This can be identified by leaving the solution for a long time without agitation. Alternatively, the solution might be centrifuged.

7 Test report

The test report shall include the following information:

- a reference to this International Standard, i.e. ISO 12632:2015;
- identification of the material tested (print process, substrate, ink, finishing, size, etc.);
- deviation from this procedure.

In addition for tests according to [6.1](#) and [6.2](#):

- glue used;
- penetration time;
- removal time.

In addition for tests according to [6.3](#):

- statement on damage of the printed sujet;
- statement on integrity of the substrate;
- statement on colour of the filtered alkaline solution;
- additives in the alkaline solution.

