# INTERNATIONAL STANDARD

# ISO 9184-4

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## Paper, board and pulps — Fibre furnish analysis —

## Part 4:
Graff "C" staining test

*Papier, carton et pâtes — Détermination de la composition fibreuse — Partie 4: Coloration de Graff "C"*

Reference number
ISO 9184-4:1990(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 9184-4 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps.*

ISO 9184 consists of the following parts, under the general title *Paper, board and pulps — Fibre furnish analysis*:

— *Part 1: General method*

— *Part 2: Staining guide*

— *Part 3: Herzberg staining test*

— *Part 4: Graff "C" staining test*

— *Part 5: Lofton-Merritt staining test (modification of Wisbar)*

— *Part 6: Weight factors by fibre coarseness method*

— *Part 7: Weight factors by comparison method*

Part 1 gives general instructions for the performance of fibre furnish analysis. It should be used in conjunction with the staining guide (see part 2) and the staining tests (see parts 3 to 5).

Additional parts of this International Standard will be published if required by the development of new kinds of fibres or new staining tests.

Annex A of this part of ISO 9184 is for information only.



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# Paper, board and pulps — Fibre furnish analysis —

# Part 4:
Graff "C" staining test



## 1 Scope

This part of ISO 9184 specifies the preparation, use and colour reactions of Graff "C" stain in fibre furnish analysis. It should be used in conjunction with ISO 9184-1 and, if necessary, with other staining tests defined in ISO 9184-2.

The field of application of Graff "C" stain is very large; it allows the identification of almost all the common papermaking fibres. However, this is based upon very minute differences in the shade and strength of the colours, and accordingly calls for a great deal of training and experience. In practice, the main applications of the Graff "C" stain are as follows:

a) Differentiation of chemical, semi-chemical and mechanical pulps.

b) Differentiation of bleached kraft and sulfite in softwood pulps. Generally, the colour difference is sufficient for counting the fibres.

c) Differentiation of kraft and sulfite in hardwood pulps. The difference in colour is more obvious in unbleached than in bleached pulps.

d) Differentiation of softwood and hardwood pulps (except dissolving grade pulps). In kraft pulps, the colour difference is sufficient; in sulfite pulps it is somewhat weaker, although in most cases sufficient for counting.

e) Differentiation of bleached straw and esparto pulps in softwood pulps. The colour difference is distinct.

## 2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 9184. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9184 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 9184-1:1990, *Paper, board and pulps — Fibre furnish analysis — Part 1: General method.*

## 3 Principle

Fibres are stained with Graff "C" stain and examined under the microscope.

## 4 Reagents

**CAUTION — Some of the components used in preparing this stain are toxic. The stain should be prepared and handled in keeping with safe laboratory practice.**

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

**4.1 Aluminium chloride**, solution ($\rho$ = 1,16 g/ml at 20 °C).

Dissolve about 40 g of aluminium chloride hexahydrate ($AlCl_3.6H_2O$) in 100 ml of water.

**4.2 Calcium chloride**, solution ($\rho$ = 1,37 g/ml at 20 °C).

Dissolve about 100 g of calcium chloride ($CaCl_2$) in 150 ml of water.

**4.3 Zinc chloride**, solution ($\rho$ = 1,82 g/ml at 20 °C).

Add about 100 g of dry zinc chloride ($ZnCl_2$) to about 50 ml of warm water until an undissolved residue remains. Allow to cool to room temperature and check that some zinc chloride crystallizes.

**4.4 Iodine**, solution.

Mix 0,90 g of potassium iodide (KI) and 0,65 g of iodine ($I_2$). Add 50 ml of water dropwise to the mixture by means of a pipette, with constant stirring. If some iodine remains undissolved — probably because the water was added too rapidly — discard the solution.

Store the solutions (4.1 to 4.4) in brown reagent bottles. Solutions (4.1), (4.2) and (4.3) are stable. The iodine solution (4.4) should be prepared every 2 or 3 months.

**4.5 Graff "C" stain.**

Mix

20 ml of aluminium chloride solution (4.1);

10 ml of calcium chloride solution (4.2);

10 ml of zinc chloride solution (4.3);

12,5 ml of iodine solution (4.4).

Pipette required volumes of solutions (4.1), (4.2) and (4.3) into a measuring cylinder and mix. Add the required volume of stock solution (4.4), mix again and place in the dark. After 12 h to 24 h, when any precipitate formed has settled, decant the clear solution into a brown dropper bottle, and add a flake of iodine. Keep the stain in the dark when not in use. Make fresh stain about every 2 or 3 months.

Before using a fresh stain, check with a sample containing bleached softwood kraft and sulfite pulp. If the colours obtained do not coincide with those given in table 1, add some iodine ($I_2$) and check again. If the colours are not satisfactory after this, make a new mixture.

## 5 Procedure

### 5.1 Staining

Stain the fibres by applying 2 or 3 drops of the Graff "C" stain (4.5) on the fibre slide prepared in accordance with ISO 9184-1.

### 5.2 Determination

Place the stained fibre slide under the microscope and examine using a magnification of × 40 to × 120. Identify and count the fibres in accordance with ISO 9184-1 on the basis of the colours developed by the Graff "C" stain (see table 1).

## 6 Expression of results and test report

Express and report the results in accordance with ISO 9184-1.

**Table 1 — Colour chart for Graff "C" stain**

| Type of pulp | Colour[1] |
|---|---|
| **Softwood chemical pulp** | |
| Unbleached kraft | Shades of yellow and brown |
| Bleached kraft | Light bluish-grey or grey |
| Dissolving grade sulfate | Brownish-purple |
| Unbleached sulfite | Shades of yellow[2] |
| Bleached sulfite | Light brownish[2] |
| Dissolving grade sulfite | Light brownish or purple[3] |
| **Hardwood chemical pulp** | |
| Unbleached kraft | Bluish-green — dark blue |
| Bleached kraft | Intense blue |
| Dissolving grade sulfate | Blue-purple |
| Unbleached sulfite | Yellowish-greyish |
| Bleached sulfite | Light blue or bluish-grey |
| Dissolving grade sulfite | Light brownish |
| **Semi-chemical pulp** | |
| Softwood | Vivid yellow |
| Unbleached hardwood | Greenish (different shades) |
| Bleached hardwood | Intense blue (like bleached hardwood sulfate) |
| **Mechanical pulp** | Vivid yellow[4] |
| **Straw and esparto chemical pulp** | |
| Unbleached | Greenish-blue (many-coloured) |
| Bleached | Grey-blue, violet-blue, intense blue (like bleached hardwood sulfate) |
| **Rag** (cotton, linen, hemp, ramie, etc.) | Wine or brown-red |

1) The colours obtained by Graff "C" stain are of many shades and consequently, different analysts may express dissenting opinions of them. Accordingly, the colours indicated in the colour chart are not necessarily analogous to the colours described in the literature. The analysis of some fibre furnishes cannot be made on the basis of colour developed by the stain. Some knowledge of fibre morphology is also required.

2) The pitch content of ray cells is stained yellow by Graff "C" stain, which often is an indication of softwood sulfite pulp.

3) Different types of dissolving grade pulps are not always distinguishable from each other.

4) If the specimen is boiled in sodium hydroxide solution during the pretreatment or before staining (see ISO 9184-1), hardwood and softwood mechanical pulps are distinguishable from each other as follows:

— softwood mechanical pulp: vivid yellow,

— hardwood mechanical pulp: greenish.

## Annex A
(informative)

## Bibliography

[1] *Fiber analysis of paper and paperboard.* TAPPI Test Method T 401, om-88.

[2] GRAFF, J.H.: New stains and their use for fiber identification. *Paper Trade J.* **100** (1935): 16, 45-50 (Ts. 203-208).

[3] GRAFF, J.H.: *A Color Atlas for Fiber Identification.* The Institute of Paper Chemistry, Appleton, WI, 1940, Plate III.