Textile Chemistry

# identification of fibres

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# Qualitative fiber analyses

**Necessary is deep knowledge of fiber properties**

**-Chemical composition**
**-Melting point**
**-Density**
**-Color**
**- ...**

**We don't have method for fiber identification, which is universal, cheap and robust.**

# Overview of basic properties of fibers

| fiber | Melting point °C | Density g.cm$^{-3}$ |
|---|---|---|
| polyethylene | 115-130 | 0,90 |
| polypropylene | 170 | 0,91 |
| polyamide 6 | 215 | 1,14 |
| polyamide 6.6 | 255 | 1,14 |
| acrylic | Decomposition at 300°C | 1,19 |
| polyurethane | 180-260 | 1,21 |
| polyvinylalkohol | 235 | 1,28 |
| wool | decomposition | 1,32 |
| semidiacetate | decomposition | 1,32 |
| Triacetate | decomposition | 1,32 |
| Silk | decomposition | 1,34 |
| Polyester | 256 | 1,38 |
| Polyvinylchloride | 170 | 1,38 |
| Kevlar (aramide) | Decomposition at 450°C | 1,44 |
| Flax | decomposition | 1,48 |
| Hemp | decomposition | 1,48 |
| Viscose | decomposition | 1,49 |
| Cotton | decomposition | 1,52 |
| teflon | 400 | 2,20 |

# Burn test

**To determine if a fiber is natural or synthetic we perform the burn test.**

Obtain a piece of the textile. Hold the tuft with a tweezers so as to not burn your fingers. Apply flame to the fiber and observe the following.

1. Does it melt or not?
2. How it burns.
   a. Rapidly or slowly?
   b. Does it go out when the flame is removed or continue to burn.
   c. Does it smolder?
3. How it smells
   a. Odor after burning
4. The ash
   a. Color
   b. Hardness
5. The molten bead, if it melts.
   a. Color
   b. Harness

# Burn test

**The test is done as follows:**

**Hold a small piece of yearn near the flame and observe whether the yarn melts as you bring the flame close.**

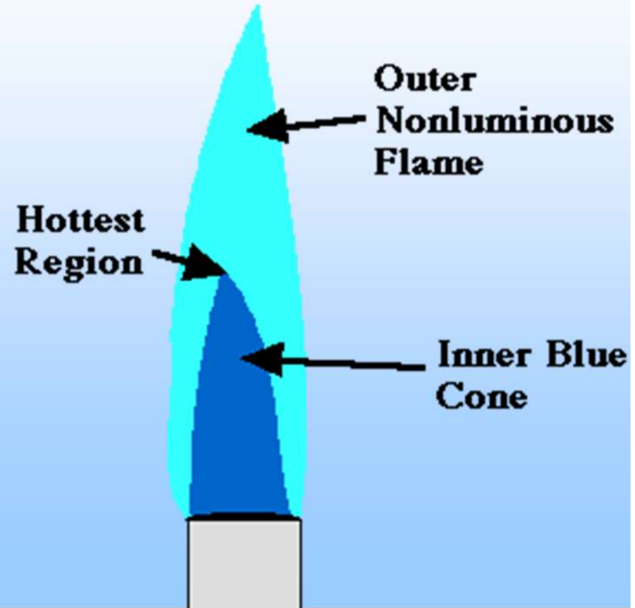
**Hold the yarn in the flame and note how fast it burns.**

**Withdraw the flame and note if the yarn continues to burn or goes out.**

**Smell the odor of the burnt yarn.**

**Note the color of the ash and whether it is hard and brittle by pinching between fingers.**

**If a molten bead forms, note the color and hardness.**



# Burn test - NATURAL FIBERS (ANIMAL)

| Fiber | Smell | Ember and Flame |
|---|---|---|
| Wool | Burning Hair | Small flickering flame, brittle ash, no smoldering (will not burn after flame is removed). |
| Silk | Burning Feathers | Calm flame, no smoldering. (Will not burn after flame is removed). Black beads, crushable. |

**Silk Is a protein fiber which burns slowly and curls away from the flame. It leaves dark bead which can be easily crushed. It is self-extinguishing and leaves ash that is dark, gritty, fine powder. It smells like burned hair or charred meat. It gives out a little or no smoke and the fume has no hazard.**

**Wool Is a protein fiber which burns slowly. It sizzles and curls away from flame and may curl back onto fingernail. It leaves beads that are brittle, dark, and easily crushed. It is self-extinguishing and leaves harsh ash from crushed bead. It gives out a strong odor of burning hair or feathers. It gives out dark smoke and moderate fume.**

# Burn test - NATURAL FIBERS (VEGETABLES)

| Fiber | Smell | Ember and Flame |
|---|---|---|
| Cotton | Burning Paper | Flame amber or yellow, slow burning; fluffy grayish ash. |

## Cotton

**It burns and may flare up when lit. No melted bead is left by it. After burning, it continues to glow. It gives out smell like that of a burning paper. The smoke is gray or white. The ash is fine, soft that can be easily crumbled.**

## Hemp

**burns quickly with bright flame. It leaves no melted bead and after burning no sign of flame is seen but it does not melts. It smells like burning leaves or wood. The ash is gray and smoke has no fume hazard.**

# Burn test – man made fiber from natural polymers

| Fiber | Smell | Ember and Flame |
| --- | --- | --- |
| Rayon (Synthetic) | Burning Wood | Rapid burning flame, slow burning embers, no ash, no bead. |

## Rayon

**Is a manufactured cellulose fiber. It burns without flame or melting and may flare up. Unless there is a fabric finish, it doesn't leave any bead. After the flame is removed, it may glow a bit longer than cotton. It smells like burning paper and leaves soft, gray ash. It's smoke is a little hazardous.**

## Acetate, Triacetate

**burns quickly and can flare even after flame is removed. The bead is hard, brittle, and can't be crushed. It melts into very hot bead and drips very dangerously. No ash is left by it and the smell is like hot vinegar or burning pepper. It gives out black smoke and the fume is hazardous.**

# Burn test - SYNTHETIC FIBERS I

| Fiber | Smell | Ember and Flame |
|---|---|---|
| Acrylic | Sharp, pungent, unpleasant odor | Hard, black residue. Burns quickly. |
| Polyester | Sweetish | Burns rapidly; produces a black, hard, rounded ash. |

## Polyester

**Is a polymer produced from coal, air, water, and petroleum products It burns quickly and shrinks away from flame, may also flare up. It leaves hard, dark, and round beads. After the flame, it burns slowly and is not always self-extinguishing. It has a slightly sweet chemical odor. It leaves no ash but its black smoke and fume are hazardous.**

## Acrylic, Modacrylic, Polyacrylic

**They flare up at match-touch, shrink from flame, burn rapidly with hot sputtering flame and drip dangerously. Beads are hard, dark, and with irregular shapes. They continue melting after flame is**

# Burn test - SYNTHETIC FIBERS II

| Fiber | Smell | Ember and Flame |
|---|---|---|
| Nylon | Boiling green vegetables (string beans or celery) | Dissolves and forms an effervescent flame; produces a hard, amber-beaded ash. |
| Olefin | Asphalt | Melts and produces a scorching flame; forms a hard tan bead. |

## Nylon, Polymide

**Are made from petroleum. Due to their fabric finish, they quickly burn and shrink to flame. The beads are hard, grayish and uncrushable. After flame, they burn slowly and melt. They are self-extinguishing but drip dangerously. Their odor is like celery and they leave no ash but the fume is very hazardous.**

# Burn test

**results of this test are used only as a method to classify fibers into broad classes !!!**

**If some type of flame retardant or other finish has been applied to fibers, it may not respond naturally to the burn test.**

**A textile may be make by using one or more fibers. This fact should be taken into consideration when you attempt to determine what fiber or fibers have been used. When a yarn is made up of a blend of two or more fibers, it may be impossible to detect any or all of them. Mixture of fibers burns typically easily then single compounds – melted fibers wick on the solid fibers.**

# Solubility test

Different liquors are solvents only for selected fibers

- Based on density and kind of intermolecular forces between molecules of solvent and polymer macromolecules

- Some solvents decomposed fibers to smaller molecules and decomposition products (typically strong acids)

- Fibers from regenerated cellulose (viscose fibers) are sensitive to strong alkali - 6.5% solution NaOH at room temperature dissolve viscose fibers, but cotton is stable (insoluble)

# Solubility test

| Solvent | Fiber | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | acetate | triacetate | polyester | polyamide 6.6 | polyamide 6 | polyurethane | polyvinylchloride | acrylic | polyethylene | polypropylene |
| acetone | S | VP | | | | | | | | |
| cyklohexanon | V | V | | | | V | S | | V | V |
| Formic acid | S | S | | S | S | V | | | | |
| toluenee | | | | | | | | | V | V |
| xylene | | | | | | | | | V | V |
| Dimethylformamide DMF | S | V | V | | V | V | S | V | | |
| Monochlorbenzene | | | | | | | V | | V | V |
| nitrometan | S | V | | | | | V | | | |
| nitrobenzene | V | V | V | VP | VP | V | S | | V | V |

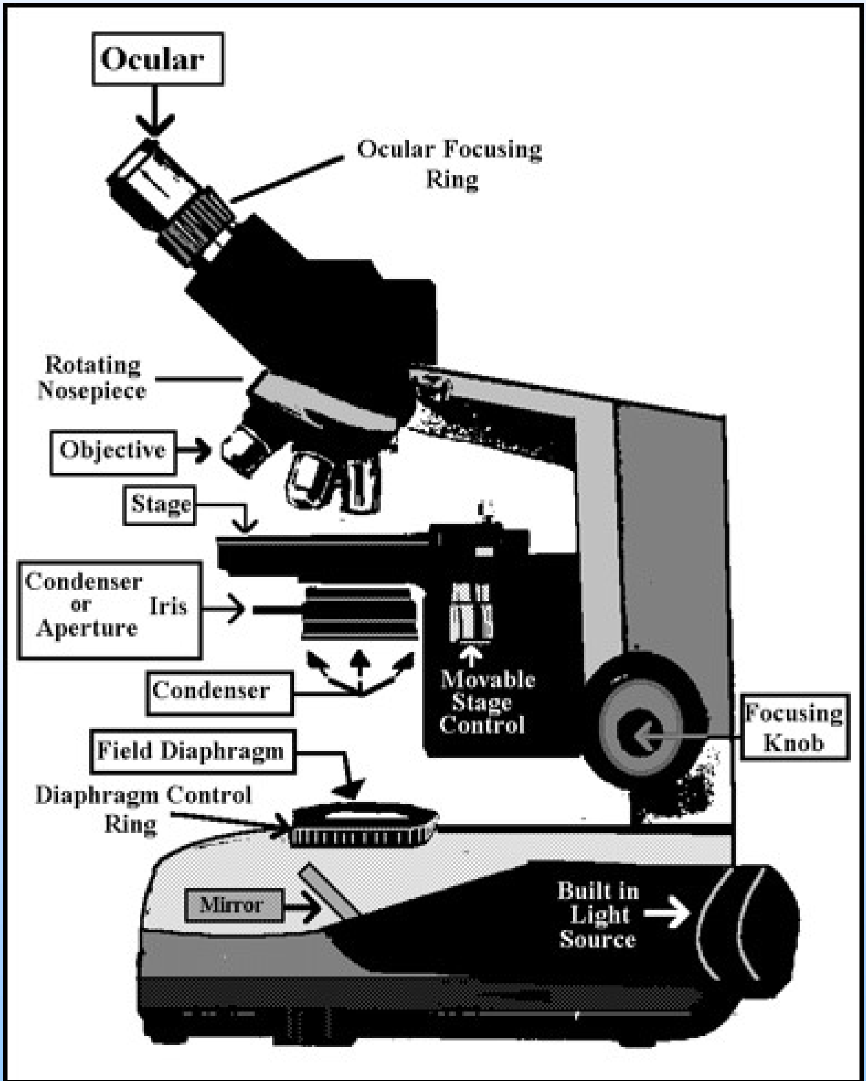
P ... Partial solution

S ... Soluble at room temperature

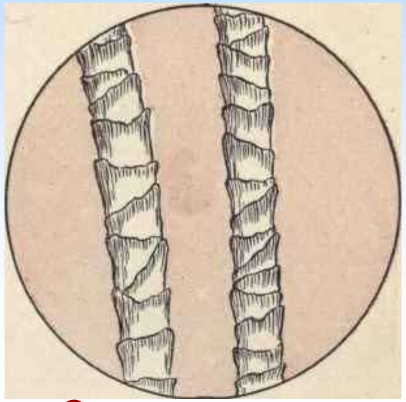
V ... Soluble at boiling temperature

# Microscopy

## Optical or electron microscopy

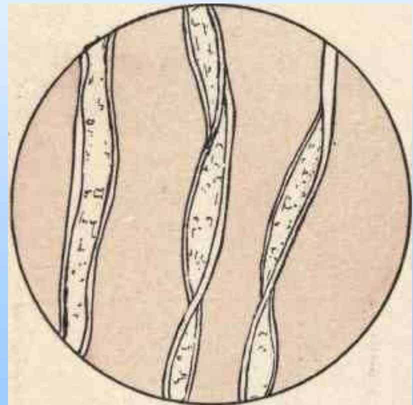



Fibre cross section



Surface roughness of fibre (wool scales)

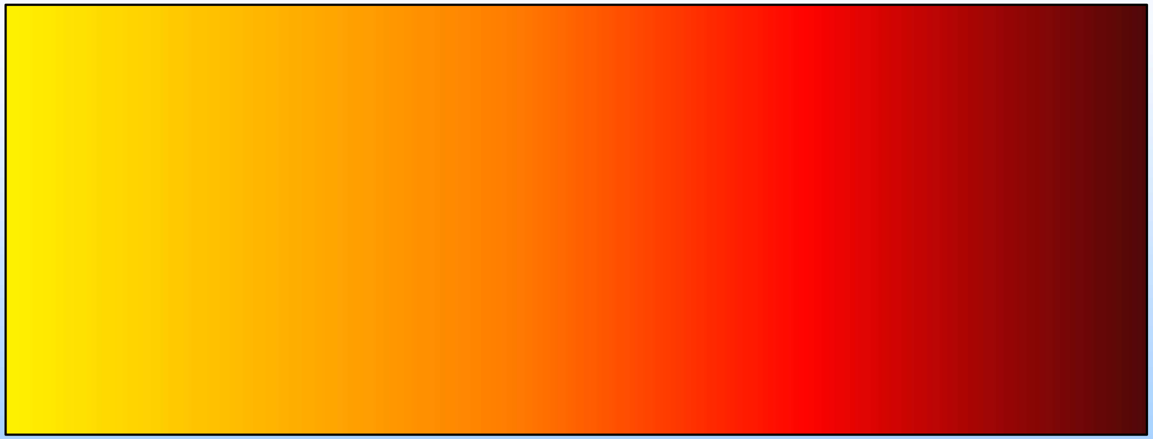
Delustrants



# Melting point

**up to 300°C**
**- testing of single fiber**

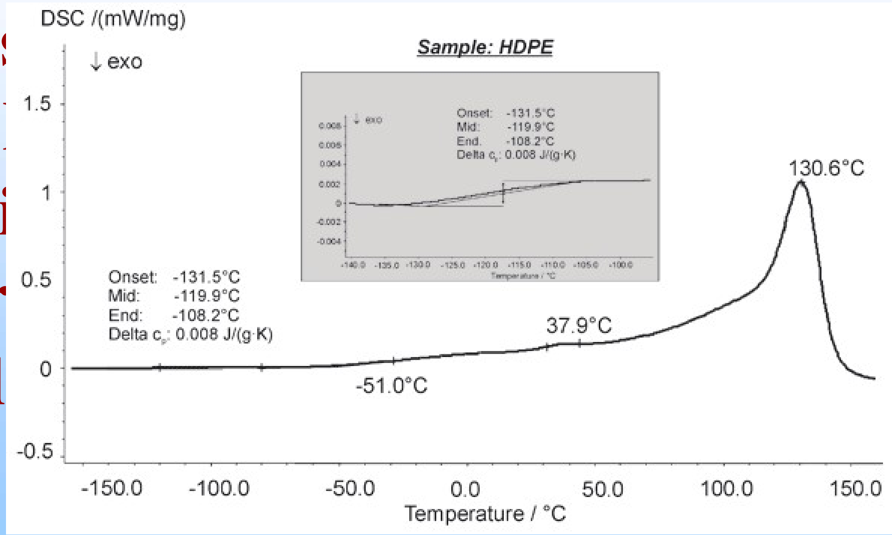
**Melting point on special meting microscope or meting table (quickly orientation test)**



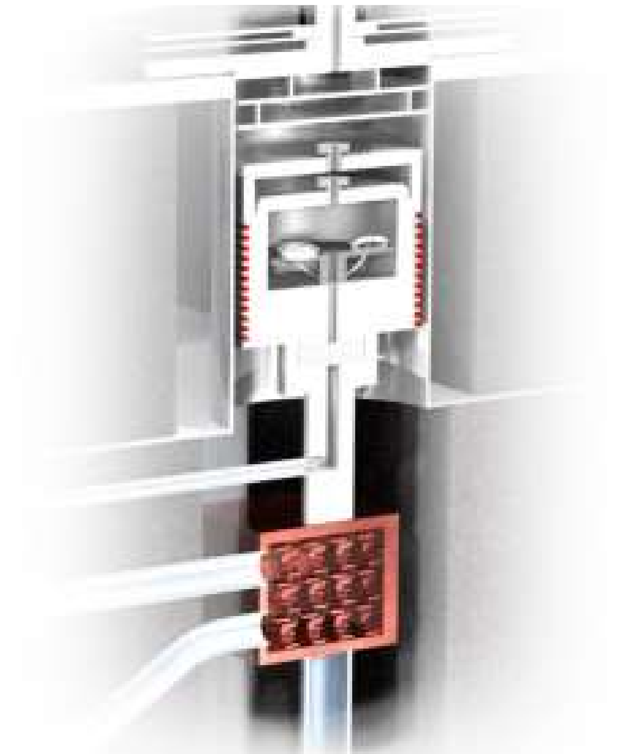
**100°C** **200°C** **300°C**

# Melting point

**DSC (differential scanning calorimetry) instruments work according to the heat flow principle and are characterized by a three-dimensional symmetrical construction with homogeneous heating. Sensors with high calorimetric sensitivity, short time constants and a condensation-free sample chamber in the DSC cell guarantee high detection sensitivity and s**
**over the entire**
**ideal qualificati**
**application in r**
**materials devel**





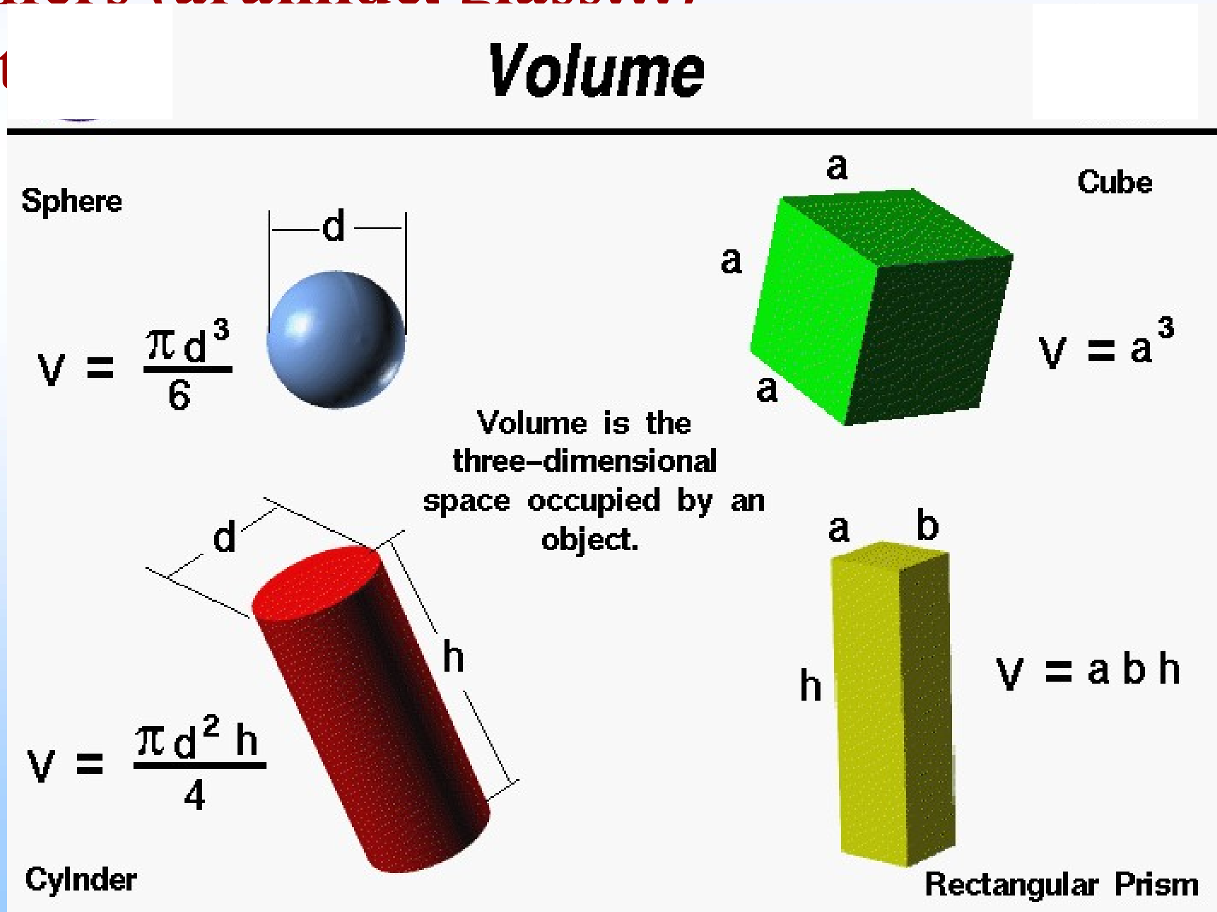


# Density of fibers

**Quickly analyses of fibers with different density (PPxPLxPA…)**

**Identification of special fifers (aramide, glass…)**

**Basic methods: pycnomet**

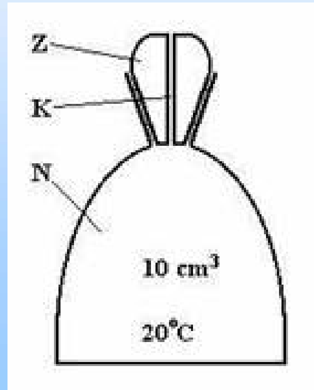


# Pycnometer metod

Pycnometer is a glass bowl with calibrated volume

The mass of an solid is determined by weighing. When the solid is placed in a pycnometer filled with a liquid of known density, the volume of the liquid which will overflow is equal to the volume of the solid.

The mass of the liquid which will overflow is determined as the difference between the sum of the mass of the pycnometer filled with liquid plus the mass of the solid and the mass of the pycnometer filled with liquid after the solid has been placed inside.



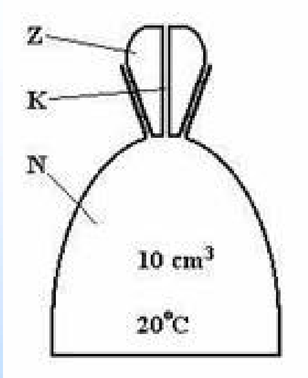

The volume occupied by this mass is determined from the known density of the liquid. It is necessary that the solid be insoluble in the liquid used. The density of the solid is determined from these measurements of mass and volume.

# Pycnometer metod

**Principle of fiber density (<u>dF</u>) is based in know weigh of empty pycnometer (mE), dry fibers (<u>mF</u>) and pycnometer with fibers and liquid together (<u>mFL</u>) and the density of used liquid (<u>dL</u>)**

**Calculation:**

**volume of pycnomether: vP= mL/xL**



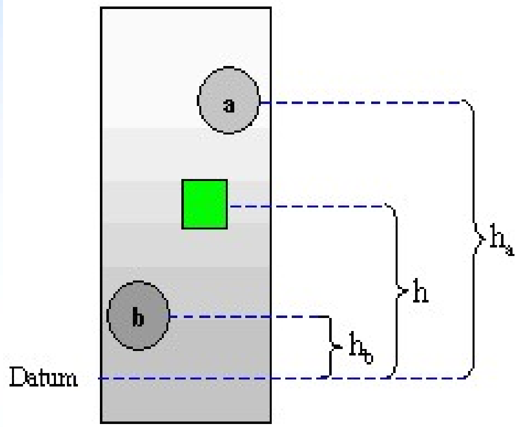

**Weight equation: mFL – mE= mF+ (vP-vF).dL**

**mF=dF.vF**

**Result: mFL – mE = mF + (vP-(mF/dF)).dL**

# Density gradient column

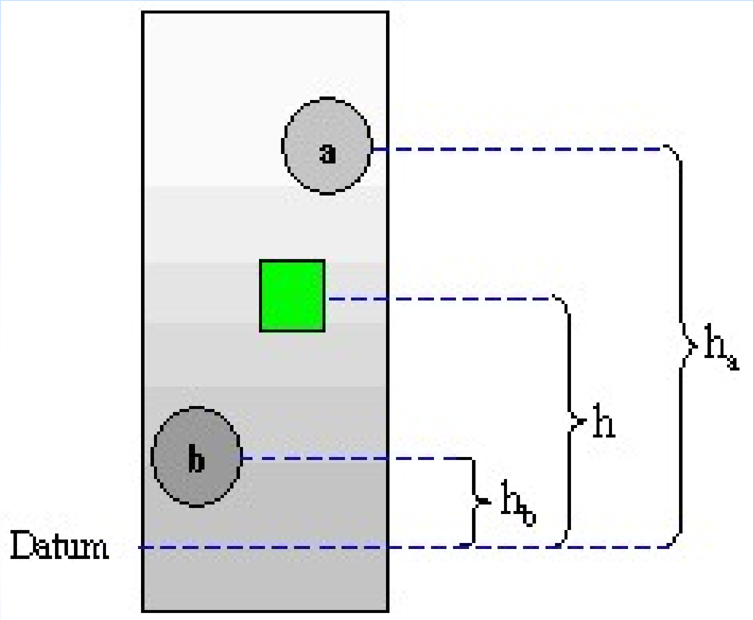
**Other principle of density measurement**




**Fiber is putted into a column with mixture of two liquids – nonhomogenic mixture (density of liquor and ratio between components depend on position in the column)**
**Liquors: octane + perchlorethylene (two liquor with different density**

**In this method the density of a material is determined by the density-gradient technique. A material is placed in a liquid column of variable density with standard floats (glass beads of known density). The material must float between a pair of the floats. The density of the material is then calculated based on its position in the column and the densities of the glass beads**

# Density gradient column




**Density gradient column**

**Graduated column with working length of 70cms**

**Out side acrylic jacket covered on to the column with temperature controlling facility**

**Calibrated glass floats with mark identity**

**Magnetic stirrer with hot plate provision**

# Dyeing test

**Quickly process**
**Different fiber = different color**

**Evaluation should by observed by optical microscopy (necessary to observe mixture of fibers)**

**Useful dyes:**

1) **strong acid: wool, silk, PA**
2) **cationic: acrylics (above 90°C), anion-modified polyester (above 90°C), wool (above 50°C)**
3) **disperse: wool and polyamide above 50°C, acrylics acetate and polyester above 90°C**

# Dyeing test

***comertial mixures of dyes for fiber identification***

**T.I.S. Stain no. 1 is recommended for use with natural fibers. T.I.S. Stain no. 3A is recommended for synthetic fibers. Using both stain solutions provides a better match asyou will have two colors to use for fiber identification.**

**T.I.S. Stain no. 1:**
**To identify fibers or cloth samples, prepare a 1% solution (*w/v) of T.I.S. Identification* Stain No. 1.**
**Heat the solution to boiling. Maintain a hot, but not actively boiling solution.**
**Wet the fiber or cloth, along with a strip of multifiber fabric, with distilled or deionied water.**
**Squeeze out the excess liquid and place the samples in the hot dye bath for 3 to 5 minutes.**
**Remove the samples and wash out any excess dye.**

**T.I.S. Stain no. 3A:**
**To identify fibers known to be synthetic, prepare a 0.05% solution of T.I.S. Identification Stain No. 3A. (0.05 g for each 100 mL water) Heat the solution to boiling and add 2 mL 5% acetic acid solution for each 100 mL of solution. Maintain a hot, but not actively boiling solution.**
**Wet the fiber or cloth, along with a strip of multifiber fabric, with distilled or deionied water.**
**Squeeze out the excess liquid and place the samples in the hot dye bath for 5 minutes.**
**Remove the samples and wash out any excess dye.**

# Dyeing test

T. I. S. IDENTIFICATION STAIN No. 1
PREPARATION:
Sample to be tested must be boiled off, or in case of a dyed or printed sample, stripped, and all finishes removed.
Prepare 1% solution. Immerse sample for 3-5 minutes at boiling temperature. Then rinse thoroughly in cold water and dry.
Testfabrics, Inc.,
P.O. Box 26, West Pittston, PA 18643
570-603-0432 www.testfabric.com
Spun Diacetate
SEF (Modacrylic)
Filament Triacetate
Bleached Cotton
Creslan 61 (Acrylic)
Dacron 54 (Polyester)
Dacron 64 (Polyester)
Nylon 66 (Polyamide)
Orlon 75 (Acrylic)
Spun Silk
Polypropylene (Polyolefin)
Viscose (Rayon)
Wool (Worsted)
TESTFABRICS IDENTIFICATION STAIN No. 3A
Dyed or finished fabrics must be stripped completely. Dissolve 50 mg. of Fiber Indicator No. 3A in 100 cc. hot water. Bring to a boil. Add .5 cc. - 1 cc. of a 10% solution Acetic Acid 56%. Enter material, boil 5 minutes. Rinse at 120° F. Extract - Dry.
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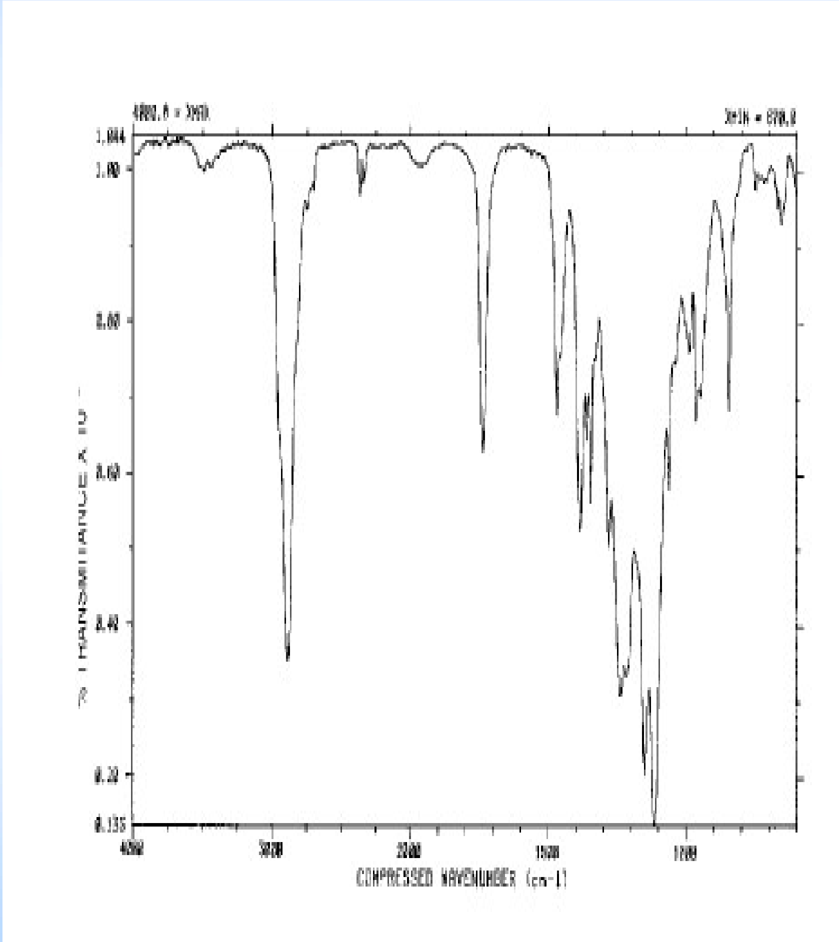
Viscose (Rayon)
Wool (Worsted)

# IR spectrophotometry

| [cm$^{-1}$] | group |
|---|---|
| 3400–3200 | alcohol, O−H |
| 3500–3300 | amin, N−H |
| 3350–3260 | alkin, ≡C−H |
| 3080–3020 | alken, =C−H |
| 3400–2400 | Carboxyl acid, −OH |
| 2820–2800 a 2720–2700 | H−C v H−C=O |
| 2250–2100 | alkine, −C≡C |
| 2260–2200 | nitrile, −C≡N |
| 1750–1730 | ester, C=O |
| 1730–1720 | aldehyde, C=O |
| 1720–1680 | Carboxyl acid, C=O |
| 1715–1700 | keton, C=O |
| 1670–1645 | alkene, C=C |
| 1250–1050 | ether C−O−C |
| 1300–1050 | ester C−O−C |



# Specific tests – wool and silk

Difference between wool and silk

both: natural fibers, protein structure

differences: wool contains sulphur, natural silk no!

**1) reaction fiber solution in NaOH with Pb or Sn or Ag ions (dark color)**

**2) morphology of surface (microscopy)**

# Specific tests – Flax and hemp

Same: properties, cellulose polymer, chemical and mechanical characteristics, morphological structures, similar processing

Different: price, contain (?) of THC (hallucinogen chemical in hemp)

| property | Flax fiber | Hemp fiber |
|---|---|---|
| Cellulose contain | 65-87%<br>(bleached up to 98%) | About 80% |
| Lignin contain | low | higher |
| density | 1460 – 1500 $kg.m^{-3}$ | 1480-1500$kg.m^{-3}$ |
| Length of elemental fibers | 3 – 60 mm | 4 – 55 mm |
| Fiber cross section shape | polygonal | polygonal |
| Relative humidity | 12 % | 13 % |
| fineness | 0.25 – 0.33 tex | 0.25 – 0.38 tex |
| breaking length | 52 km | 30 – 50 km |
| elongation to fracture | 1 – 2.5 % dry<br>2 – 4 % wet | 2 % dry<br>4 % wet |
| flexibility | low | low |
| lumen | small | broad |

# Specific tests – Flax and hemp

The flax (at fiber form) is practically undifferentiated from the hemp and it threatens maybe exchange with the hemp, which is considerably price different. The flax and the hemp are the cellulose fibers produced from the stocks. Their properties are similar and they are hardly differentiated at the fiber form. An analytical differentiation is complicated by strong interventions into the fibers during the textile treatment, which is the similar at the flax and the hemp: fibers are separated, blanched, undesirable additions are removed. These operations are connected with the change of average chemical composition of fiber material – e.g. the concentration of lignin decreases, the portion of low molecular celluloses decreases and the macromolecules of cellulose are abbreviated. With this thing bear also a big variance of fiber characteristics at wide interval e.g. specific strength of fibers, length of fibers fluctuates.

# Specific tests – Flax and hemp

## Microscopic differentiation

Morphological characteristics can be used for microscopic differentiation of the flax and the hemp. The observation is mostly oriented onto the observing of the shapes of the fiber cross-sections and fiber ends at the longitudinal view. This method is timely exacting (preparation of preparations), appreciation of observed characteristics is rather subjective and it requires considerable experiences. Advantage is the fact that the shape of elementary fibers does not change during the processing.

## Swelling test

Various morphological structures of the flax and the hemp are exhibited by various extent of the swelling property of the fibers in the cuoxam. The flax swells uniformly and relatively rapidly, resists to the solvent. The hemp swells slowly; during this process the tube in the raw fiber often obtains the typical harmonica-shape.

# Specific tests – Flax and hemp

**<u>Dyeing tests</u>**

**The hemp contents more lignin and non-cellulose portions than the flax. On this base the group of tests is founded, when the dyestuff at the agent sorbs only e.g. on the lignin part of the fiber or when the agent reacts with the non-cellulose parts of fiber within colour compounds origin. The dyeing tests are applicable especially for raw fibers before elimination non-cellulose substances from fibers (preliminary finish or otherwise), after their elimination the fibers wil not coloured. The methods are easily executed and their results are apparent by visual evaluation even without microscopic equipment.**

**<u>Summary of standard methods</u>**

**<u>Standard methods</u> (microscopic, swelling and dyeing) of distinguishing of the flax and hemp are little robust for routine differentiation of the flax and the hemp and they are rather subjective because they are based on the observing of the characteristics only little varying at the flax and the hemp.**

# Specific tests – Flax and hemp

**Twist test**

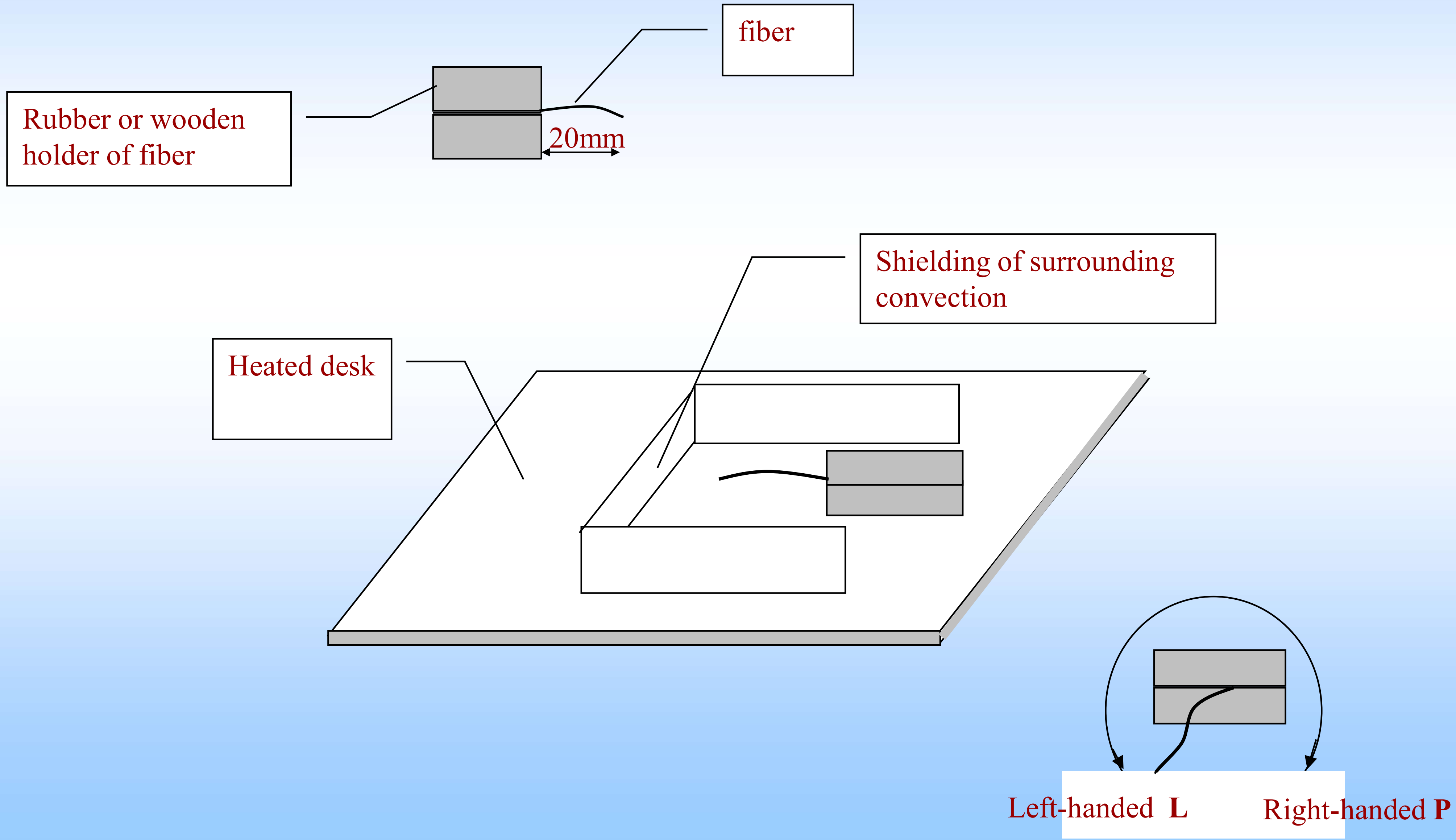
Indirect method of determination of fibril slope in the flax and the hemp

**Flax and hemp have different orientation of the fibril bundles in the fiber. Indirectly this fact is verified by opposing behavior of the flax and the hemp in the polarized light (as it assigned above) and from possibility to distinguish the fibers by X-ray diffraction.**

**From the analytical aspect, the orientation of the fibrils at the hydration and dehydration of lamellas is important. At these processes the changes of geometry characteristics of fibril bundles occur. These changes are macroscopically expressed by fiber effort to turn and so eliminate the internal stress at the sorption (or desorption of water).**

**On this base the method for differentiation of the flax and the hemp is founded – i.e. "Twist test", which merit is observing of spontaneous twisting of fiber during its drying.**

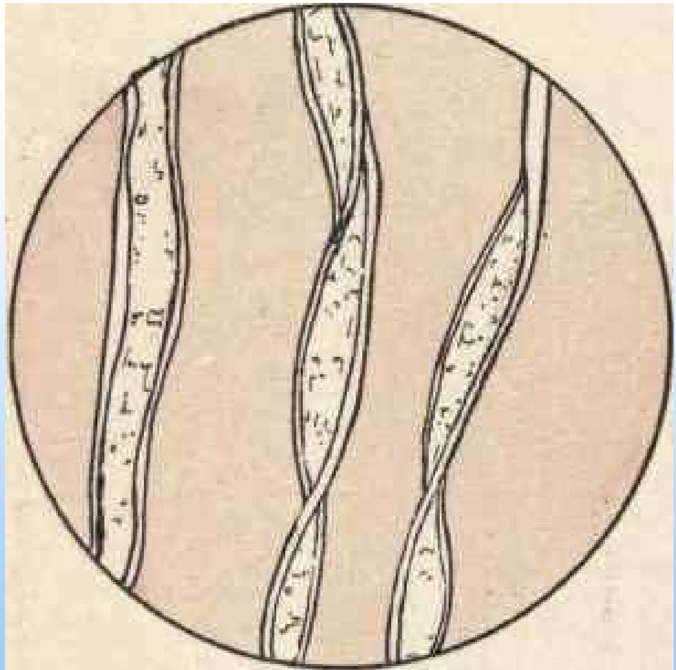
# Specific tests – Flax and hemp



# Unique fiber properties

**For estimation of fibers is interesting know a characteristic (unique) property, which is characteristic for this fiber**

**If you will not know this unique property, you have to use many standard tests**



# Unique fiber properties

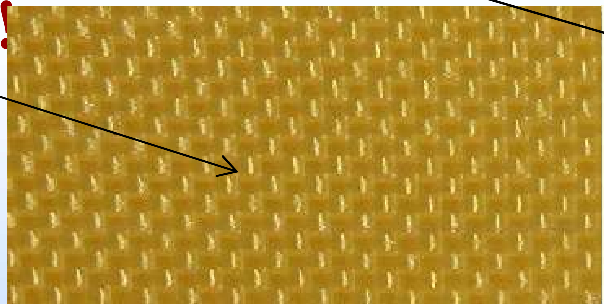
| Fiber (or group of fibers) | Unique property | Distinguish between fibers in group |
|---|---|---|
| cotton | morphology | |
| Flax, hemp | morphology | above |
| Polyester PL | *????* | |
| Polyamide PA6, PA6.6 | Solubility in HCl or in formic acid at room temperature | Dimetyformamide DMF at boiling temperature dissolve PA 6 |
| Polypropylene, polyethylene - PP PE | Low density, lower then water | Melting point of PE is 130°C (PP 170°C) |
| Polyvinyl chloride | Soluble in cyclohexanone at room temperature | |
| wool | Morphology – scales on the surface | |
| Acetate CA | Soluble on acetone | |
| Acrylic PAN | Soluble in saturated solution of ZnCl2 | |
| viscose | Solubility in NaOH (10%) at room temperature, morphology – longitudinal lines in surface | |

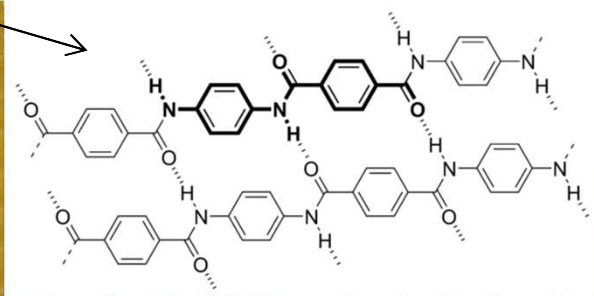
# Special fibers

Such as aramides (Nomex, Kevlar), Teflon, inorganic and mineral fibers (glass, asbestos, basalt...)

Extreme properties – chemical and mechanical rigidity (stability), extreme price

rigid macromolecular chains with extreme high Tg and Tm - High Tg = low dyeability !!!





Glass: soluble in HF

Inorganic and mineral fibers: high thermal stability (stable in