

# Practical information for the Identification of Dyes on Historic Textile Materials



*Mediaeval Craftsmen. Wood-cut by Jost Amman (1539-91).*

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Sponsored by Conservation Analytical Laboratory

SMITHSONIAN INSTITUTION

Washington DC USA

1988

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identification of dyes on  
historic textile materials

Preliminary tests

(1) Extraction tests to identify the bleeding of dyeings

Boil an approx. 1-cm long thread of the dyeing to be investigated in a test tube with 5 ml of water, and the same amounts of ethanol, glacial acetic acid, and ammonia conc. ( $d = 0.91$ ) for about 1 minute, and filter the extract through a black ribbon into a second test tube. After boiling with glacial acetic acid, wash the sample thoroughly with water.

Then evaluate which of the extracts have been stained by extracted dye. Of the synthetic dyes, the acid and direct dyes bleed to some extent in water and more heavily in ammonia. The basic dyes, in contrast, stain the ethanol and glacial acetic acid extracts most heavily. The only natural dye that belongs to this dyeing class is berberine (C.I. Natural Yellow 18)\*.

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\*These generic names of the Color Index are a classification of natural and synthetic dyes in certain (dyeing) classes.

Synthetic metal complex dyes, including the chrome developing and mordant dyes, do not bleed in these extraction tests, or they only stain the ammonia extract lightly.

Most of the natural dyes treated in this manner stain the extract only lightly, or not at all. The reason for this is that the vast majority of the natural dyes that are suitable for dyeing textile materials belong to the class of mordant dyes and are present in the dyeings in the form of sparingly soluble lakes.

Exceptions are dyeings with safflower, the leaves of the dyer's thistle *Carthamus tinctorius* L. (C.I. Natural Red 26): The red dye carthamin goes into solution when boiled with water. When this solution is poured through a paper filter after it has cooled, the paper is stained red, while the filtrate is colorless. If the safflower has not been washed adequately with cold water prior to dyeing to remove the safflower yellow, this filtrate may be somewhat yellowish. When the red stained filter is then boiled with dilute ammonia, the latter becomes irreversibly colorless (clear identification of carthamin). Magenta (C.I. Basic Violet 14), which also becomes colorless with ammonia, shows its original shade, however, when acetic acid is poured over the sample of the dyeing.

Dyeings with indigocarmine (C.I. Natural Blue 2), which has been produced from indigo and sulfuric acid since 1740, also bleed with a blue shade when treated with ammonia. Dyeings with turmeric (C.I. Natural Yellow 3) bleed with a yellow-orange shade when boiled with ammonia, while dyeings with brazilin (C.I. Natural Red 24) bleed with a red color.

The glacial acetic acid extract may be stained blue by indigo (C.I. Natural Blue 1) and by Tyrian purple (C.I. Natural Violet 1). It is stained yellow by turmeric, kamala (C.I. Natural Yellow 25), unripe buckthorn berries (C.I. Natural Yellow 13), brazilin, logwood (C.I. Natural Black 1) and madder (C.I. Natural Red 8): It is stained orange by annatto (C.I. Natural Orange 4), cochineal (C.I. Natural Red 4), and sandelwood (C.I. Natural Red 22). \* It may be stained red by alkanet (C.I. Natural Red 20) and orchil (C.I. Natural Red 28).

Dyeings with sandelwood turn from orange to black when they are boiled with ammonia after the treatment with glacial acetic acid. This also applies to dyeings with barwood (*Pterocarpus soyanxii* TAUB.), which is listed in the Color Index under the same generic name as sandelwood.

(2) Dyeing tests to determine the class according to the dyeing behavior

When the dyeing to be investigated is boiled with water, ethanol, glacial acetic acid, and ammonia, and we have reason to assume that the dye has been produced with synthetic dyes, we can determine whether this is an acid or a basic dye by dyeing from acetic acid solution on wool and on cotton mordanted with tannic acid + potassium antimonyl tartrate. Acid dyes dye the wool in a deeper shade, while basic dyes stain the mordanted cotton more heavily. Direct dyes can be identified in a separate dyeing test; when applied from a neutral solution containing sodium sulfate, they stain unmordanted cotton on relatively deep shades.

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\*Sandelwood also bleeds when boiled with ethanol.

The dyeing tests are carried out in the following manner:

Evaporate the extracts from the extraction tests (1) that are relatively deeply stained on a steam bath until they are dry. The residue is dissolved in water and it is then used for the dyeing test.

(3) Tests for identification of some synthetic dyes

There are two very simple reactions that can confirm the presence of synthetic dyes that have been identified by (1) and (2):

In the extraction tests with water, ethanol, glacial acetic acid, and ammonia, if the ammonia solution is heavily stained and becomes irreversibly colorless on addition of zinc dust even at room temperature, this indicates the presence of an azo dye.

The solution color in concentrated sulfuric acid can be an important indication for identifying synthetic dyes. In this test, a few drops of sulfuric acid ( $d = 1.84$ ) are dripped on a small sample of the dyeing, and the color of the sulfuric acid is observed after a few minutes. Intensive red-violet, violet, blue, and green solution colors indicate the presence of synthetic dyes. Exceptions are the solution colors of the natural dyes orchil (blue-violet) and alkanet (violet).

Slight modifications of this test with concentrated sulfuric acid also provide indications for the presence of the red insect dye cochineal (C.I. Natural Red 4), kermes (C.I. Natural Red 3), and lac dye (C.I. Natural Red 25). In this test, the solution colors in concentrated sulfuric acid are red in all three cases. If a few crystals of boric acid are then added

to the sulfuric acid, the solution color turns towards blue when cochineal or kermes is present; when lac dye is present, it turns to violet. After dilution with water and shaking-out the natural dyes with ethyl acetate and 3-methyl-butanol-1 (1:1), the dye solutions can be further used for TLC comparisons.

4. Behavior of natural mordant dyes when boiled with 10% sulfuric acid

Another method of obtaining an indication of the presence of certain types of natural dyes is to boil a sample of the dyeing for a short time with 10% sulfuric acid. This test is only carried out for dyeings that have shown little bleeding in the extraction tests (1).

In this test, the following reactions are typical:

- (a) Natural dyes belonging to the class of the hydroxy-flavones (e.g. C.I. Natural Yellow 1, 2, 4, 10, 11, 12, and 13) become almost colorless. On addition of ammonia, the original yellow shade returns. On subsequent boiling, after addition of a small amount of sodium dithionite, the yellow color remains. (Clear identification of hydroxyflavones and hydroxyisoflavones).
- (b) Iron tannate dyes become (almost) colorless when boiled with 10% sulfuric acid. Iron can be detected in the sulfuric acid solution.
- (c) Brazilwood and logwood dyeings bleed with an intensive red shade.

(d) Red and violet madder dyeings on alum or iron sulfate mordant turn (yellow)orange; the dye bleeds with a yellow shade, and after it has been shaken with ethyl acetate, it can be used for TLC comparisons.

(e) Dyeings with the red insect dyes cochineal, kermes, and lac dye bleed with an orange shade. After shaking with 3-methyl-butanol-1, the dyes can be used for a TLC comparison.

(5) Behavior of natural dyes to sodium dithionite and ammonia

An indication of the presence of natural vat dyes is obtained by treating a sample of the dyeing with sodium dithionite in ammoniacal solution at boiling temperature. Vat dyes are insoluble in water, ammonia, and dilute mineral acids. On reduction at alkaline pH, they go into solution, and this is usually accompanied by a change in shade. On reoxidation in the air, the original shade returns. The natural vat dyes indigo (C.I. Natural Blue 1) and Tyrian purple (C.I. Natural Violet 1) have a yellow vat, while Chinese green (C.I. Natural Green 1) has a magenta red vat.

Natural dyes from the class of hydroxynaphthoquinones, e.g. walnut shells (C.I. Natural Brown 7), henna (C.I. Natural Orange 6), and alkanet (C.I. Natural Red 20), can also be reduced, and the color of the vat is yellow. These natural dyes do not belong to the vat dyes; they should be included in the class of oxidation dyes, such as those used for dyeing human hair and furs.

Orchil (C.I. Natural Red 28), the natural dye obtained from lichens that was often used in the late Middle

Ages under the name "French purple", can also be vatted. In this case, we obtain a colorless vat and a colorless fiber. After reoxidation in the air, the fiber first turns violet, and after the ammonia has been washed out, the original magenta red shade returns.

When very old dyeings that have yellowed are treated with sodium dithionite and ammonia (sometimes known as "blank vat"), their shade becomes more brilliant. It may be assumed that the yellowing products of dyeings with moderate lightfastness are removed reductively by this treatment.

In investigating yellow dyeings with natural dyes from the class of the hydroxyflavones, which have usually yellowed heavily owing to their moderate lightfastness, this treatment with sodium dithionite should be carried out prior to the formation of the color lakes with tin, aluminium, iron, copper, and uranyl salts on the fiber. (See following section: Identification of natural mordant dyes.) After this treatment with dithionite, these color lakes are also obtained in more brilliant shades, which can then be compared more easily with known dyeings.

For the section "Preliminary tests", the following materials are available for comparison:

- (1) Tables with summaries of the preliminary tests described
- (2) Comparative dyeings with natural dyes and with "early" synthetic dyes for comparing the shades and for studying the described reactions on known dyeings

- (3) Identification of natural and synthetic dyes by treatment with 10% sulfuric acid and sodium dithionite
  - (a) Coloring before treatment
  - (b) Coloring after treatment with 10% sulfuric acid
  - (c) Coloring after treatment with ammonia
  - (d) Coloring after treatment with sodium dithionite
  
- (4) Samples of historic textile materials for comparison purposes

## Identification of natural mordant dyes

In dyeing with natural mordant dyes, we usually obtain widely varying shades, depending on the mordant that has been used to pretreat the textile material. Madder dyeings on an alum mordant, for instance, are red, while those on an iron sulfate mordant are violet. In old dyeing recipes, it is sometimes recommended, for instance, to convert the aluminium lake of a mordant dye on the fiber into the iron lake by aftertreating a mordant dyeing with inorganic salts. This conversion can also be utilized for identifying natural mordant dyes: This is done by boiling a small sample of the dyeing (threads with a length of approx. 5 mm) with dilute solutions of tin-II-chloride, aluminium sulfate, iron-II-sulfate, copper sulfate, and uranyl acetate, respectively, and leaving the solutions standing for 2-3 minutes. As indicated above in connection with the preliminary trials (Page 7), it is advisable to boil the dyeing with ammoniacal sodium dithionite solution prior to the formation of the color lake, and then wash it thoroughly with water. During the treatment with the indicated salt solutions, the corresponding color lakes form on the fiber; the shades of these color lakes depend not only on the salt used for the aftertreatment, but also on the mordant dye present.

After this treatment, the samples are boiled several times with distilled water, then rinsed once with methanol, and dried after printing between two sheets of filter paper. For comparison purposes, the dyeings are then bonded with transparent pressure-sensitive adhesive film on paper and marked accordingly.

For the section "Identification of natural mordant dyes",  
the following materials are available for comparison purposes:

Reference colorings:

Identification of natural mordant dyes by formation of  
the tin, aluminium, iron, copper, and uranium lakes

- (1) Original coloring
- (2) Coloring after treatment with tin salt
- (3) Coloring after treatment with aluminium salt
- (4) Coloring after treatment with iron salt
- (5) Coloring after treatment with copper salt
- (6) Coloring after treatment with uranium salt

Identification of natural dyes  
by thin-layer chromatography

It is often possible to distinguish between very similar natural dyes by thin-layer chromatography on polyamide layer material. When the dye consists of several different components, it is in many cases even possible to identify a definite dyer's plant that has been used to produce the dyeing. Thus, madder of the type of *Rubia tinctorum* L. can be distinguished from other similar plants, e.g. hedge bedstraw, reibun root, and mang-kouda. The yellow natural dyes belonging to the flavone class, buckthorn berries and old fustic, can also be clearly identified in this manner, and the three red insect dyes cochineal, kermes, and lac dye can be clearly distinguished from one another.

The table below lists some solvents that are particularly suitable for separating natural dyes belonging to different classes on Micropolyamide F 1700 (Schleicher & Schüll) as layer material.

<u>Solvents:</u>	1	Toluene-acetic acid (9:1)
	2	Butanone-formic acid (95:5)
	3	Butanone-formic acid (7:3)
	4	Chloroform-methanol (95:5)
	5	Chloroform-methanol-butanone-formic acid (6:2:1:1)
	6	Butanone-methanol-formic acid (65:30:5)

<u>Class of natural dye</u>	<u>Suitable solvents</u>
Madder dyes	1, 2, 4
Red insect dyes	3, 6
Hydroxyflavone dyes	5
Vegetable tannins	3, 6
Santalin A, B, C from sandelwood	2, 4
Curcuma dyes	4 (layer material: silica gel)

For the identification of early synthetic dyes on silica gel as layer material, the following solvents are suitable:

- (1) n-Butanol-acetic acid-water (5:1:2)
- (2) Butanone-methanol-formic acid (65:30:5)
- (3) n-Butyl acetate-pyridine-water (4:4:2)

Preparing the samples for a TLC comparison of natural dyes and early synthetic dyes

Madder dyes and red insect dyes can be dissolved by boiling a dyeing with 10% sulfuric acid (see "Preliminary trials" 4(d) and 4(e)). The sulfuric acid is shaken with ethyl acetate. Madder dyes go into the upper layer of the ethyl acetate with a yellow shade, and kermessic acid from kermes does the same with a yellow-orange shade. If the lower layer of sulfuric acid is not colorless but orange after shaking with ethyl acetate, the same volume of isoamyl alcohol (= 3-methyl-butanol-1) is added to shake the dyes out. In this manner, the dyes from cochineal and lac dye can also be shaken out. The layer of sulfuric acid is separated in the separating funnel, and the layer of ethyl acetate or ethyl acetate-isoamyl alcohol is shaken several times with water to remove the sulfuric acid. The solution of the dyes is then evaporated to dryness in a porcelain dish on the steam bath, and the residue is taken up with

a little methanol. This solution can then be used for thin-layer chromatography.

Yellow natural dyes from the class of the hydroxyflavones remain (mostly) on the fiber when treated with 10% sulfuric acid (see preliminary trials 4(a)). In this case, the dyes are dissolved with a small amount of concentrated sulfuric acid at room temperature (and left standing for 15-20 minutes). The solution is then diluted with roughly 20 times the volume of water, and the hydroxyflavones are shaken out with ethyl acetate. After neutralization of the ethyl acetate layer with water, the process is continued as described for the madder dyes.

Early synthetic dyes go (mostly) into solution in the extraction tests (preliminary tests (1)) with water and ammonia (acid and direct dyes) or with ethanol and glacial acetic acid (basic dyes). The most heavily stained solutions are evaporated to dryness in porcelain dishes on the steam bath, the residues are taken up with a little methanol or methanol-water (1:1), and these solutions are used for TLC comparisons.

#### How thin-layer chromatography is carried out

On a thin-layer plate, mark a series of dots starting about 2 cm away from one side of the plate and at distances of roughly 1 cm between the dots with a very soft pencil to position the dye solutions. With a micropipette, apply solutions of the dye to be investigated and of the corresponding comparison dyes one after the other on these dots. The dye spots should have a diameter of 2-3 mm. After drying in the air, place the chromatogram in a separating chamber, in which the solvent has been filled to a level of about 1 cm at least 2 hours earlier. When the solvent rises to a level of 8-10 cm on the thin-layer

plate, mark the level to which the solvent has risen with a pencil, and dry the chromatogram in the air.

Then immerse the chromatogram in a solution of 0.5% of uranyl acetate in methanol-water (1:1), press it between several layers of filter paper, and dry it in the air.

For the section "Identification of natural dyes by thin-layer chromatography", the following materials are available for comparison purposes:

A ring book: Thin-layer chromatograms of natural dyes and early synthetic dyes

#### Testing of yellow natural dyeings

The process of analysis can be accelerated by including the shade of the dyeing in the analytical scheme; in testing red dyeings, for instance, a different procedure is used than for testing blue dyeings, and certain reactions that are not typical for testing red dyeings can be disregarded.

When a yellow dyeing is boiled with glacial acetic acid (see preliminary tests (1)), berberine (C.I. Natural Yellow 18) is the only natural dye that is dissolved. It can be identified by TLC comparison on silica gel with the solvent n-butanol-acetic acid-water (5:1:2).

The most important natural dyes used for dyeing textile materials belong to the class of the hydroxyflavones. Dyeings of this type become (almost) colorless when boiled with 10% sulfuric acid. If, after the sulfuric acid has

been washed out, ammonia is added to the dyeing, the original yellow returns, and it remains unchanged on subsequent addition of sodium dithionite and boiling.

When the dyeing treated in this manner is heated with tin, aluminium, iron, copper, and uranyl salts, we can identify flavone dyes that contain luteolin as main component fairly easily from the color of the lakes. In such cases, we can assume that dyer's rocket or dyer's broom (both are C.I. Natural Yellow 2) has been used.

If very brilliant orange-colored tin lakes are obtained, this is an indication for quercetin and its glycosides, hydroxyflavones that often occur in the vegetable kingdom. Tin lakes with this color are obtained, for instance, when the dyeing has been produced with quercitron (C.I. Natural Yellow 10), Chinese yellow pods (*Sophora japonica* L.), or unripe buckthorn berries (C.I. Natural Yellow 13). More precise information regarding the dyer's plants that have been used is obtained by means of a thin-layer chromatographic comparison, when other hydroxyflavones as well as quercetin are present, e.g. in dyeings with unripe buckthorn berries.

#### Testing of red natural dyeings

Red dyeings with safflower (C.I. Natural Red 26) can be identified in the extraction tests (preliminary tests (1)) with water and with ammonia (= colorless). Sandelwood (C.I. Natural Red 22) can also be identified in this manner. In the extraction tests (preliminary tests (1)), aftertreatment with glacial acetic acid, these dyeings turn black with ammonia. After previous treatment with

10% sulfuric acid, they turn deep violet with ammonia. On washing with water, the original red shade returns. When boiled with 10% sulfuric acid, the solution obtained from madder dyeings (C.I. Natural Red 8) turns yellow (fibers = orange), while solutions from dyeings with cochineal, lac dye, and kermes turn orange (fibers = orange-red to red), and solutions from Brazilwood dyeings (C.I. Natural Red 24) turn red (fibers = yellow-orange).

When vatted with sodium dithionite and ammonia, orchil dyeings (C.I. Natural Red 28) become colorless and they turn violet when reoxidized in the air, and magenta red after washing out the ammonia. Early synthetic dyes belonging to the azo series become irreversibly colorless when vatted.

#### Testing of blue natural dyeings

Prussian blue (C.I. Pigment Blue 27) and indigocarmine (C.I. Natural Blue 2) were produced as early as the 18th century on a (semi-)synthetic basis. They are often to be found on old dyeings. Both of them undergo changes in the extraction test with ammonia.

Prussian blue, which was used mainly for dyeing silk and for printing on cotton, turns brown-yellow to colorless during this treatment. A few drops of 4% caustic soda are then added, and the solution is poured through a paper filter. After the filtration, iron can be identified on the filter, and ferrocyanide in the filtrate.

Indigocarmine goes mostly into solution when boiled with ammonia, and it can then be identified by thin-layer chromatographic comparison on silica gel with the solvent butyl acetate-pyridine-water (4:4:2).

Indigo (C.I. Natural Blue 1) shows a yellow vat with sodium dithionite and ammonia. When the vat is shaken with n-butanol, the upper n-butanol layer turns blue.

#### Testing of black natural dyeings

A sample of the black dyeing is first boiled with tin-II-chloride solution. During this treatment, dyeings with iron tannate (C.I. Natural Brown 6) and with tanner's sumach (Rhus coriaria L., herba) (C.I. Natural Brown 6) turn yellow-orange owing to their quercetin content.

If the dyeing has been produced with logwood black (C.I. Natural Black 1), the tin-II-chloride solution turns magenta red, and the dyeing becomes violet.

If the black dyeing remains unchanged when treated with tin-II-chloride solution, it is washed out with water and then boiled with dimethyl formamide; during this treatment, indigo dissolves. This decoction is repeated until the dimethyl formamide remains colorless. If the remaining fibres are then brown, they are boiled with sodium dithionite and ammonia. If the fibers undergo no change, this proves that the material is naturally brown wool. The black has, therefore, been produced by crossdyeing naturally brown wool with indigo.

## Testing of violet natural dyeings

Violet dyeings, are first boiled with 10% sulfuric acid. If the dye dissolves with a yellow color, this indicates a madder dyeing on iron mordant (identification by thin-layer chromatography). If the sulfuric acid turns orange, and a blue remains on the fiber, the dyeing is a mixture of cochineal and indigo, which can be identified separately by the methods described above.

If the violet dyeing becomes only somewhat paler when treated with 10% sulfuric acid, the specimen is washed out with water and vatted with sodium dithionite and ammonia. If the dyeing then turns yellow, and is re-oxidized in the air to a blue, this is a dyeing with alkanet (C.I. Natural Red 20) on alum mordant.

Dyeings with Tyrian purple (C.I. Natural Violet 1) turn yellow when vatted with sodium dithionite and ammonia, and the original violet shade returns on reoxidation. When the UV lamp is used for reoxidation, the two bromium atoms of 6,6'-dibromindigo are split off, and the blue indigo is obtained.

A sample of the dyeing is tested separately for the presence of logwood, with which violet shades can be dyed on alum and tin mordants. The dyeing is boiled with a solution of tin-II-chloride. If the solution turns magenta red, and the dyeing remains violet, this is a dyeing with logwood.

### Testing of green natural dyeings

Green shades dyed with natural dyes have in most cases been produced with mixtures of indigo and dyer's rocket or unripe buckthorn berries. After identification of the indigo by vatting with sodium dithionite and ammonia, and shaking with n-butanol (see "Testing of blue natural dyeings"), the indigo is removed by repeated boiling with dimethyl formamide, and the yellow remaining on the fiber is identified as a hydroxyflavone dye by its behavior towards 10% sulfuric acid, ammonia, and sodium dithionite (see preliminary tests (4) and (5)). We can distinguish between dyer's rocket and unripe buckthorn berries by the formation of the various lakes on the fiber, as described above.

### Testing of orange natural dyeings

Orange natural dyeings have in most cases been produced with madder and yellow hydroxyflavone dyes. When these dyeings are boiled with 10% sulfuric acid, the madder goes into solution with a yellow color, and after shaking with ethyl acetate, it can be identified by thin-layer chromatographic comparison. The yellow hydroxyflavones, most of which have remained on the fibre, are then dissolved with concentrated sulfuric acid at room temperature, and after dilution with water and shaking with ethyl acetate, this solution can be used for a thin-layer chromatographic comparison.

### Testing of brown natural dyeings

When brown dyeings produced with natural dyes become much lighter in shade on boiling with 10% sulfuric acid, this usually indicates the presence of iron tannate. In such cases, iron can be identified in the sulfuric acid solution. When ammonia in excess is added to the sulfuric acid decoction, the fiber regains its original brown color.

When a separate sample of the dyeing is boiled with tin-II-chloride solution, the color turns yellow to orange, if the dyeing has been produced with dyer's plants that contain tannin and at the same time hydroxyflavones. There are many dyer's plants of this kind in nature.

When a brown natural dyeing turns yellow on vatting with sodium dithionite and ammonia, and the original shade returns on reoxidation, the dyeing has probably been produced with walnut shells (C.I. Natural Brown 7) or with some other hydroxynaphthoquinone.

If the dyeing remains unchanged on vatting, this indicates that the material is naturally brown wool or some other animal fiber with a brown inherent color.

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Sept. 1985

Preliminary Tests: Yellow Dyes

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc	Ammonia			
<u>Dyer's Rocket</u> (CI Natural Yellow 2)	-----	-----	f nearly c	f:yellow	-----	w yellow UV: int. green-yellow	yellow, then c.
<u>Turmeric</u> (CI Natural Yellow 3)	-----	yellow	yellow	f:orange s:yellow orange	-----	red brown	c
<u>Safflower Yellow</u> (CI Natural Yellow 5)	-----	-----	-----	w yellow	-----	orange brown	nearly c
<u>Saffron</u> (CI Natural Yellow 6)	-----	-----	-----	-----	-----	brown violet	c
<u>Quercetin</u> (CI Natural Yellow 10)	-----	-----	f: nearly c	f: yellow	-----	yellow, UV:green yellow	nearly c
<u>Chinese Yellow Berries</u> (CI Natural Yellow 10)	-----	-----	f:nearly c	f:yellow	-----	yellow, UV:green yellow	nearly c
<u>Old Fustic</u> (CI Natural Yellow 11)	-----	-----	f: w low	f: int. yellow	-----	yellow, UV:yellow green	nearly c

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; dl = dithionite(Na); w = weak;

Preliminary Tests: Yellow Dyes

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Dye	Extraction Tests		Acetic Acid, conc.		Ammonia	Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	f: nearly c s: yellow	f: int. orange				
<u>Unripe Buckthorn Berries</u> (CI Natural Yellow 13)	-----	-----	f: nearly c s: yellow	yellow	-----	yellow UV: yellow	nearly c	
<u>Kamala</u> (CI Natural Yellow 25)	-----	-----	f and s: yellow	f: int. orange	-----	orange yellow	orange	
<u>Naphthol Yellow S</u> ® (CI Acid Yellow 1)	yellow	w yellow	-----	yellow	dyes wool	yellow	yellow	
<u>Tartrazin</u> (CI Acid Yellow 23)	yellow	w yellow	-----	yellow	dyes wool	yellow	yellow	
<u>Metanil Yellow</u> (CI Acid Yellow 36)	yellow	w yellow	-----	yellow	dyes wool	blue violet	red violet, then magenta	

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; dl = dithionite(Na); w = weak;

Preliminary Tests: Orange Dyes

Dye	Extraction Tests		Acetic Acid, conc.		Ammonia	Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol						
<u>Orleans, Bixin</u> (CI Natural Orange 4)	-----	-----	s: int. orange f: w orange	f: w orange	-----	-----	gray blue	c
<u>Orange II</u> (CI Acid Orange 7)	orange	orange	-----	orange	dyes wool	-----	magenta	yellow orange
<u>Madder and Dyer's Rocket</u> (CI Natural Red 8 + CI Natural Yellow 2)	-----	-----	yellow, + water + ether: ether yel low, ether-lay er + NaOH violet (madder)	+ di: f yel low (=Dyer's Rocket) Detection of this dye by the Sn-, Al-, Fe-, Cu- and U- lakes.	-----	-----	brown red	yellow

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive;  
di = dithionite(Na); w = weak;

Preliminary Tests: Red Dyes

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
<u>Cochineal (Alum)</u> (CI Natural Red 4)	-----	-----	s: orange f: red	-----	-----	red, + boric acid: blue	red
<u>Madder (Alum)</u> (CI Natural Red 8)	-----	-----	s: yellow f: red	-----	-----	brown red	orange, then yellow
<u>Sanderswood</u> (CI Natural Red 22)	-----	orange	orange	f: black	-----	orange brown	dull red
<u>Brazilin</u> (CI Natural Red 24)	yellow	-----	yellow, + ether: yellow, + NaOH: magenta	red	-----	yellow, UV: int. yellow	yellow
<u>Lac Dye (Alum)</u> (CI Natural Red 25)	-----	-----	-----	-----	-----	red, + boric acid: blue	red
<u>Safflower (on silk)</u>	red, filter pa- per: red	-----	-----	f: c	-----	brown red	c
<u>Orchil (Alum)</u> (CI Natural Red 28)	-----	-----	s and f: red	f: violet, f + di: c, after re- oxidation: violet	-----	blue violet	magenta

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive;  
di = dithionite(Na); w = weak;

Preliminary Tests: Red dyes

® = Registered Trade Mark

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
<u>Fast Red AV</u> ® (CI Acid Red 88)	red	w red	-----	red	dyes wool	blue violet	red
<u>Ponceau RR</u> (CI Acid Red 26)	red	w red	-----	red	dyes wool	red	yellow red
<u>Magenta</u> (CI Basic Violet 14)	w magenta	magenta	magenta	f: c, + acetic acid: magenta	dyes mordanted cotton	yellow brown	nearly c

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; di = dithionite(Na); w = weak;

Preliminary Tests: Violet Dyes

Dye	Extraction Tests			Acetic Acid, conc.	Ammonia	Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol						
<u>Alkanet (Alum)</u> (CI Natural Red 20)	-----	-----		red, + ether + water: ether red	-----	-----	violet	dull red violet
<u>Madder (Iron-mordant)</u> (CI Natural Red 8)	-----	-----		s: yellow f: brown yellow	-----	-----	brown red	orange, then yellow
<u>6,6'-Dibromoindigo</u> (Main component of Tyrian Purple; CI Natural Violet 1)	-----	-----		s: blue, + ether + water: ether blue	----- + di: yellow vat	dyes cotton after vatting.	violet	violet
<u>Logwood (Alum)</u> (CI Natural Black 1)	-----	-----		f: red brown, s: yellow, ether: yglw	f: violet	-----	yellow brown	yellow
<u>Indigo + Cochineal</u> (CI Natural Blue 1 + CI Natural Red 4)	-----	-----		s: brown + ether + water: ether blue water orange	-----	dyes cotton blue after vatting	DMF: * Indigo solved, red f (cochineal): + H2SO4 conc.: red + boric acid: blue	
<u>Methyl Violet</u> (CI Basic Violet 1)	w violet	violet		violet, + ether + water: water: violet	w violet + agetic int. violet	dyes mordanted cotton	yellow	yellow green

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive;  
di = dithionite(Na); w = weak; \*DMF = Dimethyl formamide

Preliminary Tests: Blue Dyes

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc	Ammonia			
<u>I n d i g o</u> (CI Natural Blue 1)	-----	-----	blue, + ether + Water ether: blue	----- + di : yellow vat + butanol: blue	dyes cotton after vatting	yellow green	blue
<u>I n d i g o C a r m i n e (Alum)</u> (Indigo disulfonic acid) (CI Natural Blue 2)	-----	-----	-----	blue, + di : yellow	dyes wool	blue violet	blue
<u>P r u s s i a n B l u e</u> (CI Pigment Blue 27)	-----	-----	-----	f and s nearly c	-----	w brown	nearly c
<u>P a l a t i n e F a s t B l u e G G N</u> (CI Acid Blue 158)	-----	-----	-----	very little blue	-----	gray green	blue Ash: Cr (chromium)
<u>W a t e r B l u e I N</u> (CI Acid Blue 93)	-----	blue	-----	f and s : colorless, s + acetic acid: blue	dyes wool	red brown	blue violet

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive;  
di = dithionite(Na); w = weak;

Preliminary Tests: Green Dyes

Dye	Extraction Tests						Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia					
Indigo and a Flavone-Dye (CI Natural Blue and CI Natural Yellow 2,13 ..)	-----	-----	blue, + ether + water; ether: blue	f: int. yellow	-----	yellow green	blue		
Chinese Green (CI Natural Green 1)	-----	-----	-----	-----	Detection of the flavone by the Sn-, Al+, Fe-, Cu-, U-Lakes.	dyes cotton after vatting	brown red		
								Extract all indigo by DMF. Fiber nearly c or w yellow	
Diamond Green B (CI Basic Green 4)	w green	green	green, + ether + water; green	f: c + acetic acid: green	dyes mordanted cotton	yellow	nearly c		

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; di = dithionite(Na); w = weak; \* DMF = Dimethyl formamide

Preliminary Tests: Brown Dyes

Dye	Extraction Tests			Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.			
Young Fustic (Alum) (CI Natural Brown 1)	-----	-----	f: w yellow	f: orange	orange, UV: yellow	nearly c
Walnut shells (CI Natural brown 7)	-----	-----	-----	+ di: ellow vat	dark brown	brown
Henna (CI Natural Orange 6)	-----	-----	-----	+ di: yellowish	brown	yellow
Smyrna Galls (CI Natural brown 6) (Quercus pseudocerris BOISS)	-----	-----	-----	-----	ellow	nearly c

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; di = dithionite(Na); w = weak;

Preliminary Tests: Black Dyes

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Dye	Extraction Tests			Dyeing Test	Sulfuric Acid, conc.	+ Water	
	Water	Ethanol	Acetic Acid, conc.				Ammonia
<u>Logwood (Cr)</u> (CI Natural Black 1)	-----	-----	f: red brown s: yellow ether: yellow	f: black	-----	yellow brown	yellow
<u>Palatine Fast Black WAN</u> ® (CI Acid Black 52)	-----	-----	-----	little violet	-----	violet blue	gray Ash: Cr (Chromium)
<u>Diamond Black PV</u> (CI Mordant Black 9 )	-----	-----	-----	-----	-----	black	black Ash: Cr (Chromium)
<u>Indigo on natural brown wool:</u>	-----	-----	s: blue + ether + water ether: blue	-----	-----	-----	After DMF* extraction f: brown di: no change

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; int. = intensive; di = dithionite(Na); w = weak; \* DMF = Dimethyl formamide

**Additions & Revisions by Dr. H. Schweppe to**

**Practical information for the Identification of Dyes on Historic Textile  
Materials by Helmut Schweppe (1988)**

p.5a Behavior of natural mordant dyes when boiled with 10% sulfuric acid

4. Behaviour of natural mordant dyes when boiled with 10% sulfuric acid

Corrected method for the detection of madder dyes:

Pseudopurpurin (1,2,4-Trihydroxy-anthraquinone-3-carboxylic acid) is the main dye of some madder types, for instance of the galium and the relbunium plants. By the treatment with 10% sulfuric acid most of the pseudopurpurin will be destroyed by decarboxylation and changed to purpurin (1,2,4-Trihydroxyanthraquinone).

To avoid this change if possible, dyeings with madder should be treated with a mixture of 10% sulfuric acid and butyl acetate (1:1) for 15-20 minutes in a water bath. By this treatment the dissolved pseudopurpurin at once is in the butyl acetate layer, before it can be destroyed by the sulfuric acid.

The butyl acetate layer then will be separated in a separatory funnel and shaken with water, until it is neutral.

Then the butyl acetate solution is evaporated to dryness in a porcelain dish on the steam bath, and the residue is taken up with a little methanol. This solution can be used for Thin-layer chromatography.