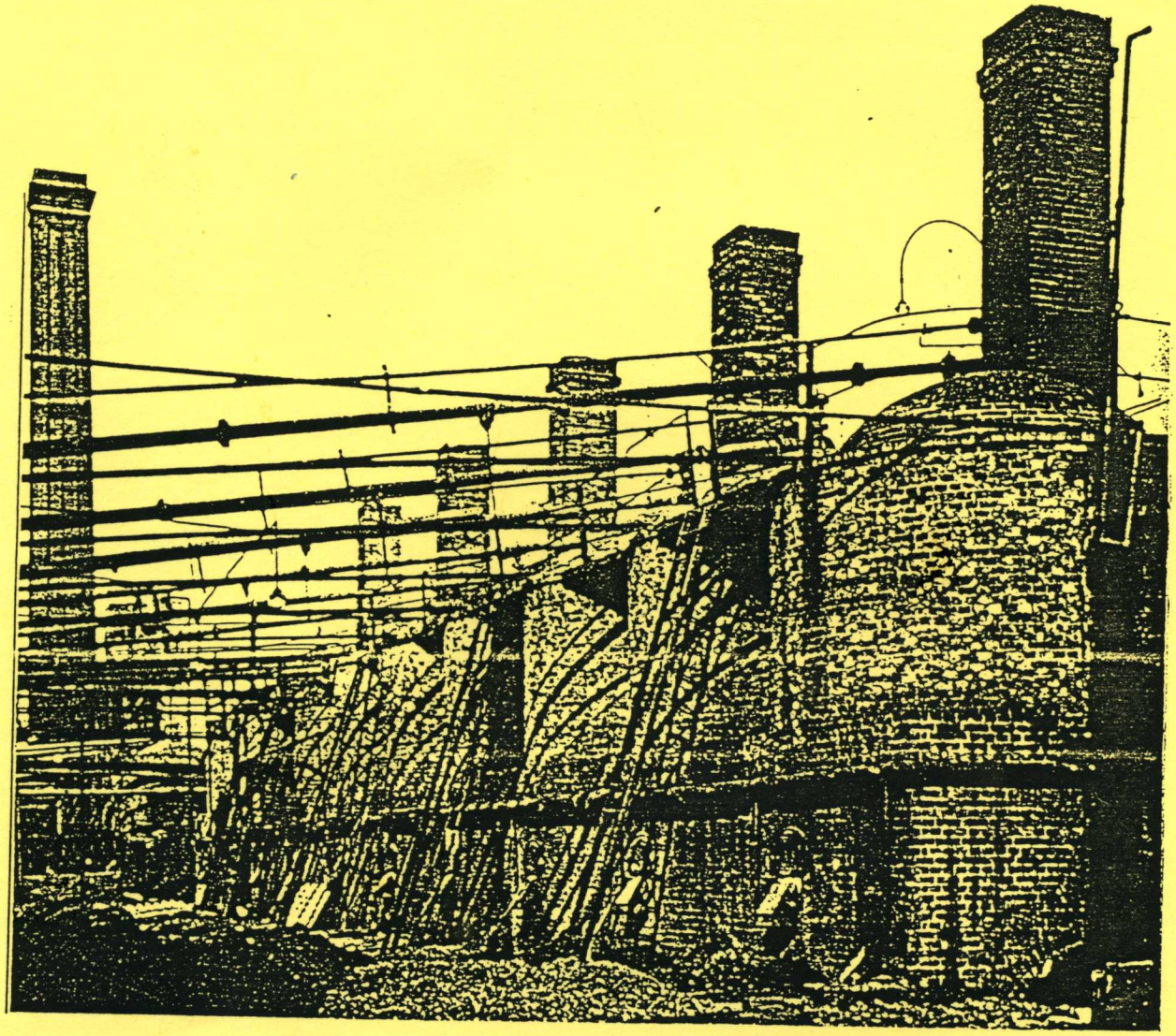
Practical information for the Identification of Early Synthetic Dyes
Practical hints on Dyeing with Early Synthetic Dyes



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Practical information for the identification of early synthetic 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 determine which of the extracts have been stained by the 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 of basic dyes is berberine (C.I. Natural Yellow 18)*.

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.

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

Dyeings with indigo (C.I. Vat Blue 1) bleed only with glacial acetic acid to any appreciable extent in these extraction tests.

(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 pronounced bleeding gives us reason to assume that the dyeing has been produced with early synthetic dyes, we can determine whether this is an acid or a basic dye by dyeing from an acetic acid solution on wool and on cotton mordanted with tannic acid + potassium antimony 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 relatively heavily.

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.

Table 1 indicates the experimental data used in these tests.

Table 1 Dyeing tests

Dyeing	Dye liquor	Material	Procedure
1.	2 ml dye solution + 0.5 ml 15% acetic acid	1-cm long threads of cotton and tannin-treated cotton	Boil for some minutes
2.	2 ml dye solution + 0.5 ml 5% sodium sulfate solution	Cotton fabric (calico), approx. 3 x 3 mm	Boil for some minutes, and give a short rinse with warm water

(3) Solution color in concentrated sulfuric acid

In some cases, the solution color of a dye in concentrated sulfuric acid can be a useful indication for identifying synthetic dyes. In this test, a few drops of sulfuric acid (d = 1.84) are dripped on to 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).

The solution color in concentrated sulfuric acid can sometimes also give an indication of the chemical nature of the dye. This does not apply, however, to the azo and the anthraquinone dyes, because their solution colors vary from yellow to green. The larger the number of azo groups contained in azo dyes, the more the solution color is shifted towards green. With adequate substitution, however, even monoazo dyes have a blue or even green solution color. The sulfuric acid reaction sometimes does, however, give some useful additional information (cf. Table 2).

Table 2 Sulfuric acid reaction of some dye groups

Dye group	Solution color in 98% sulfuric acid
Nitro dyes (Dyes 1 - 3)*	Yellow to orange
Xanthene dyes (Dyes 50 - 54)*	Yellow to red
Triphenylmethane dyes, basic, acid (Dyes 39 - 49)*	Yellow, orange, red-brown
Azine dyes (Dyes 56 - 58)*	Green, black
Thiazine dyes (Dye 59)*	Green
Azo dyes (Dyes 4 - 38)*	Yellow to green
Anthraquinone dyes Dyes 61 - 64)*	Yellow to green

* Numbers from the list of 66 "Important early synthetic dyes"

(4) Identification of synthetic dyes by their UV fluorescence

In some cases, fluorescence phenomena can be used for analyzing early synthetic dyes, because some dyes show direct fluorescence on the fiber in UV light (e.g. Rhodamine B (C.I. 45170) and 6G (C.I. 45160), Eosin A (C.I. 45380), and Auramine (C.I. 41000)). Other dyes fluoresce under the UV lamp only in certain solvents (e.g. in 98% sulfuric acid, water (Uranin A, C.I. 45350), ethanol).

(5) Investigating the ash to identify chrome-containing dyes

If a dyeing bleeds only lightly in the extraction tests (1), and it can therefore be assumed that it has been produced with a chrome developing or chrome mordant dye, further identification is carried out in the following manner:

Incinerate an approx. 1-cm long thread of the dyeing in a porcelain crucible, add 10-20 mg of a 1:1 mixture of sodium carbonate and sodium nitrate, and carry out an oxidation melt. A yellow melt on cooling indicates the presence of Cr, and this is confirmed if the melt dissolves in water with a yellow color and the addition of lead acetate causes a precipitation of yellow lead chromate.

A more sensitive method of identifying chrome is with diphenylcarbazide

Dissolve the oxidation melt in water, and then add a few drops of a 1% solution of diphenylcarbazide in ethanol. If chromate is present, the solution turns intensive red, and after acidification with 10% sulfuric acid it becomes blue-violet. If no chromate ions are present, the red shade of the alkaline solution disappears rapidly on addition of sulfuric acid. A blank test is necessary in identifying by this method. The detectable limit is between 0.5 and 2.5 micrograms, and the dilution limit is between 1:1 000 000 and 1:2 500 000.

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

(1) Tables with summaries of the preliminary tests described for 66 early synthetic dyes
Title of the tables: "Preliminary tests: Early synthetic dyes"

(2) Comparative dyeings with 66 early synthetic dyes for shade comparisons, and for studying the described preliminary tests on known dyeings

Spot reactions for the identification of early synthetic dyes

For the identification of synthetic dyes on the fiber, most of the dye identification tables indicate merely the class of a dye according to its dyeing behavior. For the identification of a dye, we require voluminous tables indicating for all dyes on the market, and for the shades dyed and printed with these dyes, the main basic reactions, arranged according to dyeing principles and dyed shades. Besides these tables, we must also always have for the final comparisons the corresponding, authentic commercial dye or its dyeing on the textile material in question.

The following basic reactions, which can be carried out as spot reactions on filter paper, have often proved to be very useful for identifying an early synthetic dye, in most cases in combination with a thin-layer chromatographical comparison:

(1) Spot reactions with the following reactants on the dye solution poured out on filter paper:

(1) 10% Sulfuric acid
(2) 4% Caustic soda solution
(3) Nitric acid (d 1.4)
(4) Tin(II) chloride solution (dissolve 100 g of $SnCl_2 \cdot 2H_2O$ in 100 ml of sulfuric acid conc. and dilute with 100 ml of water)
(5) Reaction to ammonia and zinc dust and in reoxidation on filter paper
(6) Change in shade when the edge of the dye solution (5) on filter paper is spotted with 10% sulfuric acid
(7) Addition of 1-naphthol to the reduction solution (5) (without addition, no typical reduction dyeing; with 1-naphthol violet to violet-blue : pyrazolone azo dyes)
Early synthetic dyes: Flavazine L (C.I. 18820) and Tartrazine (C.I. 19140)

These spot reactions can be carried out on the dye solutions obtained in the extraction tests of the preliminary tests. The most heavily stained extracts are evaporated to dryness, and the residues are taken up with a little water. The solution thus obtained is poured on to filter paper, and is then used for the spot reactions described above.

With the spot reactions described in (1), various early synthetic dyes can be identified easily and rapidly when they show typical and intensive shade changes.

Examples

(1) Only Metanil Yellow (C.I. 13065) and Orange IV (C.I. 13080) change in shade from yellow (orange) to intensive red-violet with 10% sulfuric acid.

(2) Monoazo dyes with 1-hydroxynaphthalene as coupling component, e.g. Orange I (C.I. 14600) and Naphthylamine Brown F (C.I. 14625), change from orange (brown) to intensive fuchsin red with 4% caustic soda solution.

(3) Congo Red (C.I. 22120) changes from red to blue with 10% sulfuric acid.

(4) Brilliant Yellow (C.I. 24890) changes from yellow to intensive fuchsin red with 4% caustic soda solution.

(5) Cotton Scarlet (C.I. 27290) changes to brown with 4% caustic soda solution, and to blue with concentrated nitric acid.

(6) Most basic triphenylmethane dyes, e.g. Diamond Green G (C.I. 42000), Diamond Green G (C.I. 42040), Fuchsin (C.I. 42510), Methyl Violet (C.I. 42535), Crystal Violet (C.I. 42555), and Victoria Blue R (C.I. 44040), change to yellow with 10% sulfuric acid.

(7) Most azo dyes are split reductively with ammonia and zinc dust, and the reduction solution becomes (almost) colorless. When the solution is poured out on filter paper and undergoes reoxidation in the air, the original shade does not return.

Exceptions

(a) Azo dyes belonging to the class of the 4-aminoazobenzene derivatives, e.g. Metanil Yellow (C.I. 13065), Orange IV (C.I. 13080), Cloth Scarlet (C.I. 26900), Wool Red B (C.I. 27200), and Cotton Scarlet (C.I. 27290), turn intensive yellow when reduced with ammonia and zinc dust. When the solution is poured out on filter paper, and the yellow edge of the stain is spotted with 10% sulfuric acid, the shade changes towards orange-red, fuchsin red, or red-violet.

(b) Basic azo dyes, e.g. Chrysoidine (C.I. 11270) and Vesuvine BA (C.I. 21000), turn blue-green or grey-blue in the reduction with ammonia and zinc dust. When the solution is poured out on filter paper and spotted with 10% sulfuric acid, the shade changes to red-violet.

(c) Bisazo dyes of the type A → Z ← A', which are produced by coupling two diazonium salts with H-acid or similar aminonaphtholsulfonic acids (C.I. 20320 - 20540), turn intensive blue in the reduction with ammonia and zinc dust; a typical example is Amido Black 10 B (C.I. 20470).

(8) Quinoline Yellow (C.I. 47005) and similar quinophthalone derivatives do not change their yellow color in the ammonia-zinc dust reduction, and then fluorescence under the UV lamp with an intensive greenish yellow color.

(9) Azine dyes, e.g. Safranine T (C.I. 50240), Mauvein (C.I. 50245 and Nigrosine, water-soluble (C.I. 50420), and thiazine dyes, e.g. Methylene Blue (C.I. 52015), are largely decolored by the ammonia-zinc dust reduction, but the original shade returns on oxidation in the air (i.e., they can be vatted).

(2) Specific identification of some early synthetic dyes

(a) Naphthol Yellow S (C.I. 10316)
Boil a small sample of the yellow dyeing to be tested with a few milliliters of tin(II) chloride solution (composition cf. (1) - (4) on p.8), wash the dyeing with water, and then pour dilute ammonia over it. The dyeing becomes orange-red.

(b) Flavazine L (C.I. 18820), Tartrazine (C.I. 19120) and other azo dyes that contain 1-phenyl-3-methyl-pyrazolone-5-derivatives as coupling component can be identified in the following manner:
When the dyeing to be tested is yellow and bleeds heavily in the extraction tests (cf. p.1) with ammonia, and the extract does not show any noticeable change in shade in the spot reactions (1) - (4) (cf. p.8), it can be assumed that this is a pyrazolone azo dye.
In order to prove this, add 10-20 mg of zinc dust to the ammonia extract, heat to the boil, and pour a part of the solution on to filter paper. Then add 2-3 mg of 1-naphthol to the rest of the reduction solution, heat to the boil, and pour the solution out again on to filter paper.
If the solution without any addition does not show any typical reduction dyeing, but turns violet to violet-blue with 1-naphthol, this is a pyrazolone azo dye (cf. also spot reaction (7) on p.8).

(c) Auramine (C.I. 41000)

If the dyeing to be tested has been identified in the extraction and the dyeing tests as a basic yellow that does not undergo any shade change in the spot reactions (1) - (4) (cf. p.8), this may be a dyeing with auramine or with the natural dye berberine.

In order to identify auramine, concentrate the extract obtained in the extraction tests with ethanol, add ammonia and zinc dust, and heat for about 1 minute. Then filter the reduction solution thus obtained into approx. 2 ml of hot glacial acetic acid. The latter turns blue if the dye in question is auramine. (This is a very sensitive reaction.)

For the section "Spot reactions for the identification of early synthetic dyes", the following materials are available for comparison purposes:

(1) Tables with summaries of the spot reactions described above for 66 early synthetic dyes

Heading of the tables:
Spot reactions: Early synthetic dyes

(2) Comparative dyeings with 66 early synthetic dyes for comparing the shades and for studying the spot reactions described on known dyeings

Identification of early synthetic dyes by thin-layer chromatography

In many cases, the amount of a dyeing available for investigating a historic textile material is inadequate to carry out all of the preliminary tests and spot reactions described in the first two sections.

In such cases, it is advisable to carry out thin-layer chromatographic comparisons between the dye solutions obtained in the extraction trials and the most commonly occurring early synthetic dyes. These dyes, arranged in order of their shades, are as follows:

Yellow: Naphthol Yellow S (C.I. 10316), Metanil Yellow (C.I. 13065), Tartrazine (C.I. 19140)

Orange: Orange II (C.I. 15510), Orange GG (C.I. 16230)

Red: Fast Red AV (C.I. 15620), Ponceau RR (C.I. 16150), Crystal Ponceau 6R (C.I. 16250), Cochineal Red A (C.I. 16255), Congo Red (C.I. 22120), Cotton Scarlet (C.I. 27290), Eosin A (C.I. 45380), and Rhodamine 6G (C.I. 45160)

Violet: Fuchsin (C.I. 42510), Methyl Violet (C.I. 42535), and Rhodamine B (C.I. 45170)

Blue: Water Blue IN (C.I. 42780), Alkali Blue (C.I. 42750), Victoria Blue B (C.I. 44045)

Green: Diamond Green B (C.I. 42000), Diamond Green G (C.I. 42040)

Black: Amido Black 10B (C.I. 20470)

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-formic acid-water (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

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 silica gel 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 thin-layer plate in a separating chamber, in which the solvent has been filled at least 2 hours earlier to a level of about 1 cm. 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 under an exhaust hood.

Evaluation of the thin-layer chromatograms

When we have a comparison chromatogram with the most important early synthetic dyes, we must first of all see whether the dye to be identified corresponds to one of the comparison dyes in its R_f value and in the shade of the stain.

If this is not the case, we must try to find other dyes for chromatographic comparisons by comparing our chromatogram with the thin-layer chromatograms in the ring book entitled "Thin-layer chromatograms of natural dyes and early synthetic dyes". Thin-layer chromatograms of the unknown dye are then prepared with these comparison dyes. The important point is that thin-layer chromatograms are always prepared with the solvents (1) - (3) (cf. p.14), so that we can see whether the R_f values of the unknown and the known dyes correspond to each other under three different chromatographic conditions. When the R_f values of two different samples correspond to each other, this does not necessarily mean that the two samples are identical. On the other hand, if the R_f values of two samples differ from each other, it is certain that the two samples are not identical.

When several comparison dyes correspond with the unknown dyes in the R_f values and in the shades of the stains, we can try to identify them by means of the spot reactions (1) - (4) described on p.8.

The spot reactions are carried out on the thin-layer chromatogram direct; this is done by applying one drop of each reagent with a micropipette to the dye stains. This method of identification by spotting with 10% sulfuric acid can be demonstrated on Metanil Yellow (C.I. 13065), Orange IV (C.I. 13080), and Azoflavine 3R (C.I. 13090), three dyes whose R_f values do not differ to any significant extent in the three solvents. The shades of the Metanil Yellow and Orange IV stains change to intensive red-violet, while that of Azoflavine 3R becomes grey-olive. The first two of these three dyes differ from each other in the shades of their dyeings and can, therefore, be easily distinguished from each other.

For the section "Identification of early synthetic dyes by thin-layer chromatography", the following material is available for comparison purposes:

A ring book: Thin-Layer Chromatograms of Natural Dyes and Early Synthetic Dyes

Identification of early synthetic dyes by comparison of the IR spectra

An unambiguous identification of dyes is carried out today in the analytical laboratories of the leading dye manufacturers by IR spectra search with the aid of electronic data processing. Essential conditions for this method are storage of the data in the computer and a suitable search system. If we succeed in obtaining an IR spectrum for a dye sample that has been suitably precleansed, an IR spectrum that does not contain too many foreign bands from secondary components, it is not difficult to retrieve this spectrum in an IR data bank, even when a very large number of IR spectra have been stored.

The following problem is involved, however, in using this outstanding method for the identification of early synthetic dyes on historic textile materials:

A relatively large amount of material is required, in order to remove all disturbing components from the dyed sample by extraction with organic solvents, before the dyes are dissolved by systematic extraction. Examples of disturbing secondary components are lanolin and finishes such as starch and dextrin, and also residues of print thickenings, e.g. locust bean flour. The dye that is then extracted must be further purified, e.g. by preparative paper chromatography, and then drawn off the paper again. Each of these cleaning steps involves loss of substance.

The following technique is recommended for the production of a potassium bromide tablet for producing an IR spectrum for water-soluble dyes:

Precipitate the dye to be identified from an aqueous solution with a saturated, aqueous solution of potassium bromide. Separate the precipitate from the solution by filtration or by centrifuging, and dry it. After addition of further potassium bromide it is then used to produce a potassium bromide tablet for the purpose.

For this method of preparing the samples, we require milligram quantities, not microgram quantities, of the dye.

For the section "Identification of early synthetic dyes by comparison of their IR spectra", the following literature is available:
Reprint of the publication: "Eine IR-Datenbank im täglichen Einsatz", B. Franke, H. Pekar, H. Schweppe, and H. Wagner: Fresenius Zeitschrift für Analytische Chemie 303, pp.349-359 (1980).

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Helmut Schweppe
(8/1987)

Preliminary Tests: Early Synthetic Dyes (Yellow Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Picric Acid (Acid Dye, 10305)	yellow +	yellow (+)	------	yellow +++	deepest shade on wool	nearly c.	yellow
Martius Yellow (CI Acid Yellow 24,10315)	yellow +	yellow (+)	------	yellow +++	deepest shade on wool	yellow	yellow p.
Naphthol Yellow S (CI Acid Yellow 1, 10316)	yellow +	-------	-------	yellow +++	deepest shade on wool	yellow	yellow (no p.)
Metanil Yellow (CI Acid Yellow 36,13065)	yellow +	-------	-------	yellow +++	deepest shade on wool	violet	s and p: magenta red
Flavazine L (CI Acid Yellow 11,18820)	yellow +	------	-------	yellow +++	deepest shade on wool	yellow	yellow
Tartrazine (CI Acid Yellow 23,19140)	yellow +	-------	-------	yellow +++	deepest shade on wool	yellow	yellow
Uranine A (CI Acid Yellow 73,45350)	yellow + UV = green	yellow (+) UV = green	-------	yellow +++ UV = green	deepest shade on wool	yellow	yellow p

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Yellow Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Quinoline Yellow S (CI Acid Yellow 3,47005)	yellow +	------	-------	yellow +++	deepest shade on wool	orange yellow	yellow

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence ;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Yellow Basic Dyes)

(Yellow Direct Dyes) (Yellow Mordant Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Auramine (CI Basic Yellow 2, 41000)	------	yellow ++	yellow +++	f = c	deepest shade on mordanted cotton	c	pale yellow
Brilliant Yellow (CI Direct Yellow 4,24890)	yellow +	------	------	yellow +++	deepest shade on cotton	red violet	violet p
Alizarin Yellow GGN (CI Mordant Yellow 1, 14025)	yellow +	------	------	yellow +++	deepest shade on wool	orange	pale greenish yellow

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence;

w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Orange Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Orange IV (CI Acid Orange 5, 13080)	orange yellow +	orange yellow (+)	------	orange yellow +++	deepest shade on wool	blue violet	violet p
Azo Flavine 3R (CI Acid Orange 1, 13090)	yellow +	yellow (+)	------	yellow +++	deepest shade on wool	red violet	brownish yellow p
Chrysoin (CI Acid Orange 6, 14270)	orange yellow +	orange yellow (+)	------	orange yellow +++	deepest shade on wool	yellow	orange yellow
Orange I (CI Acid Orange 20, 14600)	red orange +	orange (+)	------	orange red +++	deepest shade on wool	red violet	red brown p
Orange II (CI Acid Orange 7, 15510)	orange +	orange (+)	------	orange +++	deepest shade on wool	magenta red	orange
Croceine Orange G (CI Acid Orange 12,15970)	orange +	orange (+)	------	orange +++	deepest shade on wool	golden orange	golden orange
Ponceau G (CI Acid Orange 14, 16100)	orange +	orange (+)	------	orange +++	deepest shade on wool	cherry red	orange

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence ;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Orange Acid Dyes) (Orange Basic Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Orange GG (CI Acid Orange 10, 16230)	orange +	orange (+)	-----	orange +++	deepest shade on wool	yellowish orange	orange yellow
Chrysoidine (CI Basic Orange 2, 11270)	-------	yellow orange ++	yellow orange +++	------	deepest shade on mordanted cotton	yellow	red

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Red Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Archil Red (CI Acid Red 74, 13355)	reddish brown +	brown (+)	------	brown red +++	deepest shade on wool	magenta red	brown red p
Fast Red AV (CI Acid Red 88, 15620)	red +	red (+)	-------	red +++	deepest shade on wool	blue violet	red
Scarlet N for Silk (CI Acid Red 9, 15635)	orange red +	orange red (+)	-------	brownish red +++	deepest shade on wool	magenta red	brown p
Fast Red E (CI Acid Red 13, 16045)	red +	red (+)	-------	red +++	deepest shade on wool	violet	cherry red (orange p)
Ponceau 3RO (CI Acid Red 25, 16050)	orange red +	------	-------	orange red +++	deepest shade on wool	red violet	orange red
Ponceau RR (CI Acid Red 26, 16150)	orange red +	-------	-------	orange red +++	deepest shade on wool	cherry red	orange red
Fast Red B (CI Acid Red 17, 16180)	magenta red +	magenta red (+)	-------	brown red +++	deepest shade on wool	deep blue	magenta red

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Red Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Amaranth (CI Acid Red 27, 16185)	magenta red +	--------	--------	brown red +++	deepest shade on wool	violet	magenta red
Kristallponceau 6R (CI Acid Red 44, 16250)	red +	orange (+)	--------	brown red +++	deepest shade on wool	violet	scarlet
Cochineal Red A (CI Acid Red 18, 16255)	red +	------	-------	yellowish brown +++	deepest shade on wool	violet	reddish orange
Ponceau 6R (CI Acid Red 41, 16290)	magenta red +	-------	--------	yellowish brown +++	deepest shade on wool	violet	cherry red
Fast Acid Magenta B (CI Acid Red 33, 17200)	magenta red +	magenta red (+)	--------	brown red +++	deepest shade on wool	magenta red	orange red
Amido Naphthol Red G (CI Acid Red 1, 18050)	red +	-------	-------	brown yellow +++	deepest shade on wool	red	red
Cloth Scarlet (CI Acid Red 151, 26900)	red +	orange red (+)	-------	brown red +++	deepest shade on wool	green blue	blue → red

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Red Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc	Ammonia			
Wool Red B (CI Acid Red 115, 27200)	magenta red +	------	------	bordeaux +++	deepest shade on wool	deep blue	violet red
Cotton Scarlet (CI Acid Red 73, 27290)	red +	red (+)	-------	brown red +++	deepest shade on wool	reddish violet	blue → brown p
Eosine A (CI Acid Red 87, 45380)	red (UV= yellowish green) +	red (UV=yellowish green) (+)	-------	red +++	deepest shade on wool	yellow	yellowish red p
Erythrosine (CI Acid Red 51, 45430)	cherry red (UV = no) +	red (+)	-------	red +++	deepest shade on wool	brown yellow	brown yellow p

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Red Basic Dyes)
(Red direct Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc	Ammonia			
Rhodamine 6G (CI Basic Red 1, 45160)	scarlet red (UV = green yellow) (+)	yellowish red (UV = green yellow) ++	red +++	-------	deepest shade on mordanted cotton	yellow	red
Safranine T (CI Basic Red 2, 50240)	red (+)	red (UV = yellowish red) ++	red +++	-------	deepest shade on mordanted cotton	green	blue, then red
Congo Red (CI Direct Red 28, 22120)	red brown (+)	-------	--------	red +++	deepest shade on cotton	blue	blue p

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence;
w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Red mordant Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Alizarin (Alum mordant) (CI Mordant Red 11, 58000)	-------	--------	f orange s yellow ++	--------	-------	brown red	yellow
Alizarin Red S* (CI Mordant Red 3, 58005) * Alum mordant	-------	-------	f orange s yellow +	red (+)	-------	orange	yellow
Alizarin Red PS* (Mordant Dye, CI 58210) * Alum mordant	-------	--------	f red s yellow +	red (+)	--------	reddish orange	unaltered
Alizarin Red SS * (CI Mordant Red 2, 58260) * Alum mordant	-------	--------	f orange s yellow +	red (+)	-------	orange red	yellow

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Acid Violet Dyes) (Basic Violet Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc	Ammonia			
Azo Fuchsine 6B (CI Acid Violet 7, 18055)	magenta (+)	------	-------	red +++	deepest shade on wool	red	red
Magenta (CI Basic Violet 14,42510)	magenta red +	red +	red +++	------- fiber nearly c	deepest shade on mordanted cotton	yellow brown	nearly c
Methyl Violet (CI Basic Violet 1, 42535)	violet ++	violet ++	violet +++	------- fiber nearly c	deepest shade on mordanted cotton	orange yellow	olive
Crystal Violet (CI Basic Violet 3, 42555)	violet ++	violet ++	violet +++	------- fiber nearly c	deepest shade on mordanted cotton	orange yellow	olive
Rhodamine B (CI Basic Violet 10, 45170)	red +	red (+)	red +++	red +	deepest shade on mordanted cotton	yellowish brown (UV= green yellow)	scarlet, then bluish red and orange
Mauveine (Basic Dye, CI 50245)	magenta red +	magenta red +	magenta red ++	-------- fiber nearly c	deepest shade on mordanted cotton	olive green	green, then blue, then red violet

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Blue Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Patent Blue V (CI Acid Blue 3, 42051)	blue +	blue (+)	--------	blue +++	deepest shade on wool	brown-olive yellow	yellow, then green
Water Blue IN New (CI Acid Blue 93, 42780)	--------	blue +	--------	f = c, with acetic acid = blue / s = c, with acetic acid = blue	deepest shade on wool	red brown	blue
Alkali Blue (CI Acid Blue 110, 42750)	--------	blue +	--------	f = c, with acetic acid = blue / s = c, with acetic acid = blue	deepest shade on wool	red brown	blue
Indigo Carmine (CI Acid Blue 74, 73015)	blue +	--------	--------	blue +++ f = nearly c	deepest shade on wool	violet blue	blue

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Blue Basic Dyes) (Blue Pigments)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Victoria Blue R (CI Basic Blue 11, 44040)	blue +	blue ++	blue +++	-------	deepest shade on mordanted cotton	brownish yellow	light green, then blue
Victoria Blue B (CI Basic Blue 26, 44045)	blue +	blue ++	blue +++	--------	deepest shade on mordanted cotton	reddish brown	yellow, then green, then blue
Methylene Blue (CI Basic Blue 9, 52015)	blue ++	blue +	blue +++	blue +	deepest shade on mordanted cotton	yellowish green	blue
Prussian Blue (CI Pigment Blue 27, 77510)	---------	-------	--------	yellower	-------	-------	------

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Green Acid Dyes) (Green Basic Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Light Green SF Bluish (CI Acid Green 6, 42075)	green +	green (+)	-------	green +++	deepest shade on wool	golden yellow	green
Diamond Green B (CI Basic Green 4,42000)	green +	green ++	green +++	------- f = c	deepest shade on mordanted cotton	yellow	orange
Diamond Green G (CI Basic Green 1,42040)	green +	green ++	green +++	-------	deepest shade on mordanted cotton	yellow	green

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence; w = weak; p = precipitate.

Preliminary Tests: Early Synthetic Dyes (Brown Acid Dyes) (Brown Basic Dyes) (Black Acid Dyes)

Dye	Extraction Tests				Dyeing Test	Sulfuric Acid, conc.	+ Water
	Water	Ethanol	Acetic Acid, conc.	Ammonia			
Naphthylamin Brown F (CI Acid Brown 6, 14625)	orange (+)	orange (+)	--------	magenta red +++	deepest shade on wool	violet blue	brown red p
Vesuvine BA (CI Basic Brown 1,21000)	yellow (+)	yellow +	orange +++	--------	deepest shade on mordanted cotton	brown	reddish brown
Amido Black 10B (CI Acid Black 1, 20470)	blue +	-------	---------	blue +++	deepest shade on wool	bluish green	greenish blue p
Nigrosine water-soluble (CI Acid Black 2, 50420)	yellow +	-------	---------	brown +++	deepest shade on wool	black blue	violet

Abbreviations: f = fiber; s = solution; c = colorless; UV = UV fluorescence ; w = weak; p = precipitate.

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Picric Acid (Acid Dye, 10305)	nearly c	-------	c	c	int.yellow brown	int. grey blue	
Martius Yellow (CI Acid Yellow 24,10315)	c + ether = ether yellow	--------	c	c	int. red orange	int.orange red	
Naphthol Yellow S (Ci Acid Yellow 1, 10316)	c + ether = ether c	-------	c	c	int. red orange	int.orange red	See specific test 2a
Metanil Yellow (CI Acid Yellow 36,13065)	int. red violet	--------	int. red violet	int. red violet	int.greenish yellow	int. red violet	
Flavazine L (CI Acid Yellow 11,18820)	--------	-------- ww	-------	slowly c	a little yellow	stronger yellow	See specific test 2b
Tartrazine (CI Acid Yellow 23,19140)	--------	ww	-------	slowly c	a little yellow	stronger yellow	See specific test 2 b
Uranine A (CI Acid Yellow 73,45350)	---------	-------	--------	Dyeing in $SnCl_2$: — + NH_4OH: int. yellow orange	c	a little yellow	Sol.in water: yellow, with int. green fluorescence

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Yellow Acid Dyes)

Spot Tests

Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Quinoline Yellow S (CI Acid Yellow 3,47005)	———	brown, then nearly c	nearly c		Int.greenish yellow with int.UV-fluores.		Reduction with ammonia and zinc dust

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Yellow Basic Dyes) (Yellow Direct Dyes) (Yellow Mordant Dyes)
Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Auramine (CI Basic Yellow 2,41000)	a little ww	c	c	c	c	-	See specific test 2c
Brilliant Yellow (CI Direct Yellow 4,24890)	int. green olive, then black brown	int.magenta red	violet	violet, then grey	int. orange yellow	int.greenish yellow	
Alizarin Yellow GGN (CI Mordant Yellow 1, 14025)	nearly c	int. orange yellow	c	nearly c	yellow	nearly c + $FeCl_3$: int. brown violet	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Orange Acid Dyes)

Spot Tests

Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Orange IV (CI Acid Orange 5,13080)	int. red violet	———	int. red violet	int. red violet	int.greenish yellow	int. red violet	
Azo Flavine 3R (CI Acid Orange 1,13090)	grey olive	——— a little greyer	magenta red	brown (border:red violet)	int. brown orange	int. red violet	
Chrysoin (CI Acid Orange 6,14270)	———	int. brown orange	yellow orange border	———	int. dark bluish grey	lighter	
Orange I (CI Acid Orange 20,14600)	———	int. magenta red	int, red violet,then c	int. red violet,then c	weak greenish yellow		
Orange II (CI Acid Orange 7,15510)	———	red brown	int. magenta red	slowly c	weak greenish yellow		
Croceine Orange G (CI Acid Orange 12,15970)	———	a little red brown	int. brown red	c	weak greenish yellow	a little reddish	
Ponceau G (CI Acid Orange 14,16100)	———	int. red brown	——— a little bluish	int. magenta red	weak greenish yellow	nearly c	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Orange Acid Dyes) (Orange Basic Dyes)

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Orange GG (CI Acid Orange 1o,16230)	———	brown red	———	c	weak greenish yellow	———	
Chrysoidine (CI Basic Orange 2,11270)	orange	greenish yellow	———	———	nearly c,then slowly int. blue green	int. red violet	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt. = precipitate

(Red Acid Dyes)
Spot Tests

Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Archil Red (CI Acid Red 74, 13355)	int.brown red violet	———	int. red violet, then c	c	a little violet	a little reddish	
Fast Red AV (CI Acid Red 88, 15620)	———	int. red brown	int. brownish red violet	int. brown yellow, then c	weak greenish yellow	weak brown red	
Scarlet N for Silk (CI Acid Red 9, 15635)	———	distinct brown orange	weak red violet, then c	weak red violet, then slowly c	weak brown yellow	weak brown red	
Fast Red E (CI Acid Red 13,16045)	———	int. red brown	a little bluer, the c	slowly c	weak greenish yellow	nearly c	
Ponceau 3RO (CI Acid Red 25,16050)	weak magenta red, then c	int. reddish brown	weak magenta red	weak magenta red, then c	weak greenish yellow	a little brown red	
Ponceau RR (CI Acid Red 26,16150)	———	int. brown yellow	———	———	weak greenish yellow	nearly c	
Fast Red B (CI Acid Red 17, 16180)	———	int. red brown	a little brown	slowly c	weak greenish yellow	weak brown red	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Amaranth (CI Acid Red 27,16185)	———	int. red brown	a little bluish	slowly c	weak greenish yellow	weak brown red	
Kristallponceau 6R (CI Acid Red 44, 16250)	——— a little bluish	brownish yellow orange	——— a little bluish	a little bluish. then c	weak greenish yellow	weak brownish red	
Cochineal Red A (CI Acid Red 18, 16255)	weak magenta red, nearly c	int. yellowish brown	weak magenta red	weak magenta red, then c	weak greenish yellow	weak brown red	
Ponceau 6R (CI Acid Red 41, 16290)	———	int. yellow olive	a little more bluish	slowly c	weak greenish yellow	nearly c	
Fast Acid Magenta B (CI Acid Red 33,17200)	int. brown red	int. red brown	int. bluish grey	c	int. red orange, the brown	int. blue violet, then red violet	
Amido Naphthol Red G (CI Acid Red 1, 18050)	———	int. brownish yellow	———	c	weak yellow	weak red	
Cloth Scarlet (CI Acid Red 151, 26900)	———	brownish violet	brown, then blue violet	———	int. yellow	orange red	4-Aminoazobenzene-class according to the behaviour at reduction

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

S p o t T e s t s — E a r l y S y n t h e t i c D y e s

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Wool Red B (CI Acid Red 115, 27200)	———	int.brownish violet	int. violet blue	———	int.greenish yellow	int. orange red	4-Aminoazo-benzene-class according to the brhaviour at reduction.
Cotton Scarlet (CI Acid Red 73, 27290)	———	int. brownish grey olive	int. violet blue	———	int.greenish yellow	int. orange red	4-Aminoazo-benzene-class according to the behaviour at reduction.
Eosine A (CI Acid Red 87, 45380)	weak yellow orange	———	int. greenish yellow	———	c	c	
Erythrosine (CI Acid Red 51, 45430)	int. yellow orange	———	int. yellow orange	———	weak yellow	ww	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Red Basic Dyes)(Red Direct Dyes)

Spot Tests

Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Rhodamine 6G (CI Basic Red 1, 45160)	———	———	red violet (yellow orange border)	———	c	red violet	In water: green yellow fluorescence. In ethanol: yellow fluorescence.
Safranine T (CI Basic Red 2, 50240)	———	———	int. violet blue	int. violet blue	int. reddish orange	int. violet red, then magenta red	Behaviour like a vat dye at reduction
Congo Red (CI Direct Red 28, 22120)	int. violet blue	———	int. blue, the red violet	int. violet blue	nearly c	———	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Red Mordant Dyes)

Spot Tests

Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
	(Spot tests made with a solution in ethanol)						
Alizarin (Alum mordant) (CI Mordant Red 11,58000)	greenish yellow	blue violet	greenish yellow	greenish yellow			
Alizarin Red S (Alum mordant) (CI Mordant Red 3,58005)	int. greenish yellow	red violet	greenish yellow	greenish yellow	red violet; later destroyed	———	
Alizarin Red PS (Alum mordant) (Mordant Dye, 58210)	yellow orange	int. red violet	orange yellow	orange yellow	red violet; later destroyed	———	
Alizarin Red SS (Alum mordant) (CI Mordant Red 2,58260)	int. greenish yellow	int. red violet	greenish yellow	greenish yellow			

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Azo Fuchsine 6B (CI Acid Violet 7, 18055)	———	yellow brown	nearly c	c	greenish yellow, then green olive	nealy c	
Magenta (CI Basic Violet 14,42510)	yellow olive	——— (ww)	int. greenish yellow	int. greenish yellow	c	int. magenta red	
Methyl Violet (CI Basic Violet 1,42535)	yellow	——— (a little browner)	yellow	yellow, then c	c	a little blue	
Crystal Violet (CI Basic Violet 3,42555)	yellow	———	yellow	yellow, then c	c	a little blue	
Rhodamine B (CI Basic Violet 1o,45170)	———	———	nearly c, yellow border	———	c	weak red violet	In ethanol: int. yellow orange fluorescenz.
Mauveine (Basic Dye, CI 50245)	———	int. grey violet	int. violet blue	weak violet blue	int. red violet (Reaction more intensive in ethanol).	distinctly bluer	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Patent Blue V (CI Acid Blue 3, 42051)	int. greenish yellow	——— (a little ww)	int. greenish yellow (border yellowish green	int. greenish yellow	c	c	
Water Blue IN New (CI Acid Blue 93, 42780)	int. bluer	int. red brown	bluish grey (border: int. blue)	red violet	c (very slowly blue)	int. violet blue	
Alkali Blue (CI Acid Blue 110, 42750)	int. bluer	ww and redder, later c	grey (border:blue)	——— a little ww	c (very slowly blue)	int. violet blue	
Indigo Carmine (CI Acid Blue 74, 73015)	———	int. greenish yellow	c	c	yellow vat, on filter paper blue.	———	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

(Blue Basic Dyes)(Blue Pigments)

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Victoria Blue R (CI Basic Blue 11,44040)	int. greenish yellow	int. dirty brown	int. yellowish green	int. greenish yellow	c	c	
Victoria Blue B (CI Basic Blue 26,44045)	———	int. violet brown	brown yellow (border: brown)	grey brown	c	c	
Methylene Blue (CI Basic Blue 9,52015)	———	——— (slowly int. red violet)	int. blue green	c	int. violet blue	unchanged	
Prussian Blue (CI Pigment Blue 27,77510)	Insoluble in water.		Detection of Prussian Blue (Ferric ferrocyanide): A sample of the unknown printing or dyeing is heated with 3-5 milliliters of 4% NaOH for one minute and the solution is then filtered. a) The residue (= $Fe(OH)_3$) is detected by potassium ferrocyanide and 10% sulfuric acid as 'Prussian Blue' b) The ferrocyanide ions in the solution are detected by $FeCl_3$ and 10% sulfuric acid as 'Prussian Blue'.				Iron in the ash.

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Light Green SF Bluish (CI Acid Green 6, 42075)	———	c	int. orange yellow	int. orange yellow	c, very slowly int. green	int. green	
Diamond Green B (CI Basic Green 4, 42000)	yellow olive	www, later nearly c	yellow	yellow	c	c, very slowly green	
Diamond Green G (CI Basic Green 1, 42040)	oliv	ww, nearly c	yellow	yellow	c	c, very slowly green	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Spot Tests — Early Synthetic Dyes

Dye	Spot Tests				Reduction with ammonia + zinc dust poured on on filter paper	Spot test of the reduction solution poured on filter paper, with 10% H_2SO_4	Specific test for the dye
	10% H_2SO_4	4% NaOH	HNO_3 (conc)	$SnCl_2$			
Naphthylamin Brown F (CI Acid Brown 6, 14625)	yellow brown	int. magenta red	grey olive	grey olive	red brown	———	
Vesuvine BA (CI Basic Brown 1, 21000)	———	yellow orange	nearly c	c	greyish blue	brownish red violet	
Amido Black 10B (CI Acid Black 1, 20470)	———	——— (ww)	weak brown red, olive green border	int. magenta red, then c	int. blue	ww	
Nigrosine water-soluble (CI Acid Bkack 2, 50420)	int.greyish blue	a little ww	violet	violet	int. red violet	int. blue	

Abbreviations : f = fiber; c = colorless; UV = UV fluorescence; w = weak; ww = weaker; int. = intensive; ppt.= precipitate

Practical hints on dyeing with early synthetic dyes

(Production of comparative dyeings for the identification of dyes on historic textile materials)

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General

Wool was in the past the textile material most commonly used, and for this reason the following practical hints for the production of comparative dyeings refer mainly to wool dyeing.

For the production of dyeings that are intended for use as comparative materials for analytical purposes, small wool hanks weighing 5 grams are adequate. When commercial hank wool is used for dyeing, a thorough wash prior to mordanting or dyeing is not necessary. A rinse in lukewarm water with an addition of a wool detergent is adequate to remove residues of spinning oils.

All dyeing recipes given in the following refer to the dyeing of 100 grams of hank wool.

Prior to dyeing or mordanting, the wool hanks are tied loosely with wool threads at two or more parts to prevent the individual yarns in the hank from becoming entangled during dyeing. The beginning and the end of the hank is tied into one of the tying threads.

When several hanks are to be dyed at the same time, a fairly long stable cord is drawn through all hanks, and the ends of the cord are then tied together. The hanks can then be moved in the dyebath, or lifted out in

washing or rinsing, by gripping this loop. The cord can also be used for securing information regarding the dyeing. For instance, if wool is to be dyed with various mordants in the same dyebath, the hanks can be distinguished by one, two, or three knots tied in the cord, and a note is made in the record book to explain what these knots mean.

It is best to use soft water for dyeing. In most cases, roughly 3.5 liters of water is used for dyeing 100 grams of hank wool. In the dyer's language, this corresponds to a liquor ratio of 35:1. For mordanting wool to be dyed with synthetic mordant dyes, a liquor ratio of 20:1 to 22:1 is adequate. Data on additions of acids and salts to the dyebath in per cent always refer to the weight of the fibers to be dyed. An addition of 5 % of sulfuric acid thus means an addition of 5 grams of sulfuric acid in the dyeing of 100 grams of wool.

The wool is always entered in the wet state into the mordanting or dyeing bath to ensure that the liquor is taken up evenly. Wool yarn should never be subjected to sudden changes in temperature. For this reason, the yarn is first placed in a dish with cold water, and warm water is then run slowly into the dish from the water pipe until the water has a temperature of about 40 °C. The yarn can then be entered into the dyebath at roughly the same temperature.

The dyebath must never be heated to a bubbling boil; at most, it should be simmering. Silk can only withstand dyeing temperatures up to 70 °C. In any case, the most suitable temperature indicated in the dyeing recipe should be used.

Dyeing processes for the early synthetic dyes

(1) Dyeing process for wool with acid dyes*

Acid dyes are sodium salts of dye acids (nitrophenols, sulfonic acids, or carboxylic acids), mainly azo dyes, and also nitro dyes, members of the triphenylmethane group, the xanthene dyes, and the anthraquinone series.

The large class of acid dyes is of outstanding importance for wool dyeing. Their main field of application is the dyeing of wool yarns and fabrics, for which an even uptake of the dyes (good levelling) and good penetration of the material are essential.

In this respect, the acid dyes vary widely from one another, and to cope with the difficulties involved, different dyeing processes have been developed for the individual dyes.

For dyes that are readily soluble in water and in dilute acids, and consequently have good levelling and even migrate from the dyed parts of the material to the undyed parts, an excess of sulfuric acid and rapid boiling cause no trouble at all. But if the dye is sparingly soluble and has poor penetrating and levelling properties, it goes rapidly and unevenly on to the fiber when sulfuric acid is added in large amounts and the temperature is raised rapidly to the boil; in this case, care must be taken in metering the addition of sulfuric acid and in heating. In dyeing with sparingly soluble dyes, therefore, the amount of Glauber's salt is increased and the addition of sulfuric acid is cut

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* Dye tables by G. Schultz, Vol. 2, p. 238

down or replaced partly or wholly by weaker acids, e.g. acetic and formic acid; dyeing is commenced at a lower temperature, and the bath is heated slowly to simmering temperature. For instance, 5-10 % of acetic acid is added slowly during the process of dyeing, and if necessary, a small amount of sulfuric acid is added at the end of the process. For some of these dyes, it is advisable to use, not acetic acid, but its ammonium salt, from which acetic acid is liberated in boiling, with evaporation of ammonia.

For the individual types of acid dyes, the following processes are recommended:

For dyes with very good levelling

(Dyeing process S: Dyeing in sulfuric acid bath)

Set the dyebath with 10-15 % (usually 10 %, seldom 20 %) of Glauber's salt (cryst.) and 3-5 % (usually 4 %) of sulfuric acid (d = 1.84) or 10 % of Glauber's salt and 10 % of sodium hydrogen sulfate ($NaHSO_4$). Add the dye solution and heat the dyebath to 60-80 °C or to the boil. Enter the wool hanks, preheated in warm water, to the dyeing liquor cooled to 60 °C, raise the temperature within 15-30 minutes to a weak boil, and continue boiling for 45-90 minutes, until only a small amount of dye remains in the bath. In dyeing relatively deep shades and blacks, add $NaHSO_4$ or sulfuric acid to ensure good exhaustion of the bath.

When the liquor has cooled, wash the wool thoroughly with tap water and dry it in the air.

The following early synthetic dyes can be dyed by this process:

(1)	Picric Acid	CI 10305)
(2)	Martius Yellow	CI 10315)
(3)	Naphthol Yellow S	CI 10316)
(4)	Metanil Yellow	CI 13065)
(5)	Orange IV	CI 13080)
(6)	Azoflavine 3R	CI 13090)
(7)	Archil Red	CI 13355)
(8)	Chrysoin	CI 14270)
(9)	Orange I	CI 14600)
(10)	Naphthylamine Brown F	CI 14625)
(11)	Orange II	CI 15510)
(12)	Fast Red AV	CI 15620)
(13)	Scarlet N for Silk	CI 15635)
(14)	Crocein Orange G	CI 15970)
(15)	Fast Red E	CI 16045)
(16)	Ponceau 3RO	CI 16050)
(17)	Ponceau G	CI 16100)
(18)	Ponceau RR	CI 16150)
(19)	Fast Red B	CI 16180)
(20)	Amaranth	CI 16185)
(21)	Orange GG	CI 16230)
(22)	Crystal Ponceau 6R	CI 16259)
(23)	Cochineal Red A	CI 16255)
(24)	Ponceau 6R	CI 16290)
(25)	Fast Acid Magenta B	CI 17200)
(26)	Amido Naphthol Red G	CI 18050)
(27)	Azo Fuchsin 6B	CI 18055)
(28)	Flavazine L	CI 18820)
(29)	Tartrazine	CI 19140)
(30)	Amido Black 10B	CI 20470)
(31)	Cloth Scarlet	CI 26900)

(32) Wool Red B	CI 27200)
(33) Cotton Scarlet	CI 27290)
(34) Patent Blue V	CI 42051)
(35) Light Green SF Bluish	CI 42075)
(36) Water Blue IN New	CI 42780)
(37) Uranine A	CI 45530)
(38) Quinoline Yellow	CI 47005)
(39) Nigrosine, water-soluble	CI 50240)
(40) Indigo Carmine	CI 73015)

For dyes that are applied from a weakly acid bath and have less good levelling

(Dyeing process ES: Start dyeing in a weakly acetic bath and complete dyeing with sulfuric acid)

Set the dyebath with the requisite amount of dye, 10 % of Glauber's salt, and 2-4 % of acetic acid (30 %) or the corresponding amount of formic acid (90 %). Enter the wool at approx. 50 °C into the dyebath, heat within 30 minutes to the boil, and continue boiling for 45-90 minutes. In order to exhaust the bath, add 2-5 % of $NaHSO_4$ or the corresponding amount of acetic acid (approx. 2 %) or formic acid (0.7 %) and, if necessary a small amount of sulfuric acid, and boil for another 30 minutes.

The following early synthetic dyes can be dyed by this method:

For dyes with moderate levelling

Dyeing process WA: Dyeing in weakly acetic acid bath

Set the bath with 10-20 % of Glauber's salt (cryst.), 2-5 % of acetic acid (30 %) or 2-5 % of ammonium acetate at 10 °Bé (= 70 %). Enter the wool at 30-40 °C, heat within 30-45 minutes to the boil, with gradual addition of 2-5 % of acetic acid (30 %), and continue boiling for another 30 minutes.

The following early synthetic dyes can be dyed by this process:

For resorcin dyes

Dyeing process A: Dyeing in acetic acid bath

Set the dyebath with 10 % of acetic acid, 10 % of sodium acetate, and the dye solution, enter the wool hanks at room temperature, heat slowly to 80 °C and complete dyeing within one hour.

The following early synthetic dyes can be dyed by this process:

(1) Methyl Violet (CI 42535)	(2) Crystal Violet (CI 42555)
(3) Victoria Blue R (CI 44040)	(4) Victoria Blue B (CI 44045)
(5) Eosin A (CI 45380)	(6) Erythrosine (CI 45430)

For alkali blue etc.

Dyeing process Alk: Dyeing in alkaline bath followed by development with sulfuric acid

Set the bath with 2-5 % of borax or 1-3 % of soda ash or 5 % of waterglass and the dye solution, and enter the wool hanks at 50 °C. Heat the bath slowly to 95 °C and dye for 30-60 minutes at this temperature. Then give a good rinse and develop in a fresh bath at 60-70 °C with 2-5 % of sulfuric acid conc. for about 20 minutes, and finally give another rinse.

The following early synthetic dye can be dyed by this process:

(1) Alkali Blue (CI 42750)

(2) Processes for dyeing wool with mordant dyes

The mordant dyes for wool can be classified in two main groups:

(a) Mordant dyes for which the wool has to be provided with the mordant in a separate bath. The dyes of the alizarin series and chemically allied products belong to this group.

(b) Mordant dyes that are applied from an acid bath and fixed by subsequent or simultaneous addition of the mordant. The mordant-dyeing azo dyes belong to this group.

Certain members of these two groups can be applied by both methods.

(a) Dyeing on premordant

On prolonged boiling of the wool in the mordant baths, the fiber takes up the metal oxide from the mordant salts, viz. alumina from alum in the presence and under the influence of organic acids and acid salts (tartaric acid, oxalic acid) or chromium oxide from bichromate. In mordanting with bichromate, the chromic acid is reduced to chromium oxide by the use of organic acid or acid salts (tartar, formic acid, lactic acid).

Dyeing on alum mordant

Dyeing process Al P = dyeing on alum premordant

Mordanting bath: Set the bath with 10 % of alum, 3 % of tartar, and 2 % of oxalic acid, then enter the wool, heat slowly to the boil and continue boiling for 90 minutes. Finally give a good rinse.

Dyebath: Add the dye, 2.5-7.5 % of calcium acetate, and 2 % of tannin to the cold dyebath, enter the wool into the liquor, heat slowly to the boil and continue boiling for 90 minutes. Finally, give a good rinse.

This process is suitable for the following dyes:

(1) Alizarin (CI 58000) (2) Alizarin Red S (CI 58005)
(3) Alizarin Red PS (CI 58210) (4) Alizarin Red SS (CI 58260)

Dyeing on chrome mordant

Dyeing process CrP: Dyeing on chrome premordant

Mordanting bath: For medium shades, set the mordanting bath with 3 % of bichromate and 2.5 % of tartar, enter the wool, heat slowly to the boil and continue boiling for 90-120 minutes. Finally, give a good rinse.

For paler shades, the amount of mordant can be cut down to 1 % of bichromate and 1 % of tartar, while for very deep shades 4 % of bichromate and 3 % of tartar must be used. The expensive tartar can be replaced by oxalic acid, formic acid, or lactic acid, with or without sulfuric acid or acetic acid, e.g. 2 % of bichromate, 3 % of lactic acid, and 1 % of sulfuric acid.

Dyebath: Set the cold dyebath with 2-3 % of acetic acid, enter the wool and heat slowly to the boil. For complete fixation of the dyes, continue boiling for 90-120 minutes. After dyeing, give a good rinse.

This process is used for all alizarin dyes and for some azo dyes.

The following dyes can be applied by this process:

(1) Alizarin (CI 58000) (2) Alizarin Red S (CI 58005)
(3) Alizarin Red PS (CI 58210) (4) Alizarin Red SS (CI 58260)

(b) Dyeing in acid bath and aftertreatment with metal salts

The most suitable aftertreating agent is bichromate (chroming process); alum and copper sulfate can also be used for the aftertreatment of various dyes.

Dyeing process Cr: Chroming process

Set the bath with the dye, 10 % of Glauber's salt, and 3 % of sulfuric acid, enter the wool, and heat within 30 minutes to the boil. After boiling for 30 minutes, add another 1-2 % of sulfuric acid and heat for further 60 minutes. Then run cold water into the bath to cool it slightly, and add 1-3 % of bichromate; then heat the bath again to the boil and let it simmer for 30-45 minutes; finally, give a good rinse.

Certain dyes are best dyed in more weakly acid baths. For these dyes, use 10-20 % of Glauber's salt, and replace the sulfuric acid by 3-10 % of acetic acid or 0.5-2.5 % of formic acid. For dyes that are affected by lime, it is advisable to add 2 % of ammonium oxalate to make the lime contained in hard water harmless.

This process is generally used for the chromable azo dyes and it is also suitable for individual alizarin dyes.

Process Cr is suitable for the following dyes:

(1) Alizarin Yellow GGW (CI 14025) (2) Alizarin (CI 58000)
(3) Alizarin Red S (CI 58005) (4) Alizarin Red PS (CI 58210)
(5) Alizarin Red SS (CI 58260)

(3) Dyeing processes for wool with basic dyes

Dyeing process N: Dyeing in neutral bath or in a bath slightly acidified with acetic acid

Basic dyes are used for dyeing wool only on a limited scale, and in cases where exceptionally lively, fiery or pale shades that cannot be achieved with other synthetic dyes are required.

Add 2-4 % of acetic acid (30%) to the bath, then add the dye and heat to 50 °C. Enter the hank wool and dye for about 90 minutes at 80-95 °C.

This process is suitable for the following dyes:

(1) Chrysoidine (CI 11270)
(2) Vesuvin BA (CI 21000)
(3) Auramine (CI 41000)

(4) Dyeing process for silk

Early synthetic dyes used for silk dyeing include the following:

Acid dyes

Yellow:	Quinoline yellow, Naphthol Yellow S, tartrazine, Azoflavine 3R
Orange and red:	Orange II, Orange GG, Ponceau RR, Crystal Ponceau 6R, eosine
Violet and blue:	Alkali Blue (CI 42750), formyl violet, acid violet, Patent Blue V

Basic dyes

Yellow, orange, brown:	Auramine, chrysoidine, vesuvin
Red and pink:	Rhodamine B, Rhodamine 6G, fuchsin, Safranine T
Blue and violet:	Methyl violet, crystal violet, mauvein, methylene blue, Victoria Blue B, R
Green:	Diamont Green B, G

Generally speaking, the acid dyes are applied with an addition of sulfuric acid, and the basic dyes with an addition of acetic acid, by the processes described above for wool dyeing. In silk dyeing, however, the dyeing temperature must not be higher than 70 °C.

5) Dyeing processes for cotton

(a) Dyeing processes for cotton with basic dyes

Dyeing process TP: Tannin premordant

Basic dyes are particularly suitable for dyeing lively, pure shades. They do not dye cotton direct; the material must therefore be premordanted with tannin and tartar emetic.

Mordant: Set the bath at 80-90 °C with 1-6 % of tannin (on the weight of cotton) depending on the depth of shade to be dyed, enter the cotton, give a number of turns, and leave lying in the mordant for several hours, or preferably overnight. Then wring out the cotton thoroughly and, without washing it, enter it into a cold bath containing 0.3-3 % of tartar emetic (antimony potassium tartrate) and treat in this bath for 20 minutes. Finally, give a good rinse.

Dyeing: Set the cold bath with 1-5 % of acetic acid or 2-10 % of alum and the dye, which has been previously dissolved in water. (The dye may be added all at once or in several portions.) Then enter the cotton, give a few turns, and heat the bath to 50-70 °C. Leave the cotton in the bath for 30-45 minutes, and then give a good rinse and dry.

The following early synthetic dyes can be applied by this process:

(1) Auramine (CI 41000)	(2) Diamond Green B (CI 42000)
(3) Diamond Green G (CI 42040)	(4) Magenta (CI 42510)
(5) Methyl Violet (CI 42535)	(6) Crystal Violet (CI 42555)
(7) Victoria Blue R (CI 44040)	(8) Victoria Blue B (CI 44045)
(9) Methylene Blue (CI 52015)	

(b) Dyeing process for cotton with direct dyes

Dyeing process D: Direct dyeing

Direct dyes are the dyes most commonly used for dyeing cotton. They are cheap in price and can be applied by a very simple method, and some of them have excellent fastness properties. Not only direct dyed shades, but also dyeings produced by various types of aftertreatment, e.g. with metal salts, are extremely popular.

Dyeing is carried out in a neutral Glauber's salt bath at salt concentrations of 5-30 % on the weight of the cotton, depending on the depth of shade to be dyed. Pale shades require the smallest amounts of Glauber's salt, and dark shades the largest amounts. Dyeing is carried out for 45-60 minutes at the boil. After dyeing, the cotton is rinsed and dried.

This process is suitable for dyeing the following early synthetic dye:

Congo Red (CI 22100)

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Methylene Blue	TP	16
Methyl Violet	A	8
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