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IDENTIFICATION OF DYESTUFFS IN I.G.

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IDENTIFICATION OF DYESTUFFS IN I.G.

This subject was discussed at Leverkusen with Dr. Thomae who is one of I.G.'s most experienced operators in this field with special reference to vat dyestuffs since he is responsible for all the testing arising from their "Indanthrene House" scheme.

The colour reactions and technique are for the most part generally known and it was stressed that success in this matter is essentially a matter of experience. Each system as far as it can be generally set out has defects.

In the I.G. system water soluble dyestuffs are identified in powder form or in solution; dyeings must be previously extracted.

The insoluble textile dyestuffs, vat dyestuffs, etc., are identified on the fibre; dyestuff powders are, therefore, dyed or printed first.

For the Determination of Single Dyestuffs (see the following are necessary:-

- (1) Reaction tables. These include the fundamental reactions, i.e., a number of colour reactions, which are given in a similar manner by each dyestuff. The fundamental reactions of water soluble dyestuffs are naturally different from, for instance, vat dyestuffs. Identically or similarly reacting dyestuffs must be further differentiated by additional reactions.
- (2) Authentic standards. Powder samples in the case of water soluble dyestuffs and dyeings in the case of vat dyestuffs, Naphthols, etc.

Mixtures where necessary, are identified spectroscopically or are separated by chromatographic absorbtion analysis (see Ruggli & Jensen Helv. Chem. Acta XVIII 8. 624).

The detailed scheme, as far as is possible, is set out below.

ROUGH GROUP-SEPARATION OF DYESTUFF IN SUBSTANCE

Heat.

Organic dyestuffs - char - or possibly sublime (Indigo-para red)

Inorganic dyestuffs - do not char.

H2SO4 Conc.

Inorganic dyestuffs - mostly insoluble, or possibly colourless solution.

Organic dyestuffs - mostly coloured solution
(Naphthol AS mostly very
strengly coloured sulphur
dyestuff mostly slightly
soluble and dull).

Solubility experiments.

Sprayed on wet filter paper, heat with water and pour on to filter paper. The same with alcohol.

Mostly water soluble basic also in alcohol Reactions with the sprayed-out dyestuff

Mostly insoluble in water and alcohol.

Insoluble

On filter paper H20 10% H2SO4 60 NaOH. NHz then heat.

Mostly changes colour with one or other reaction solubility increases.

Mostly fairly unaffected.

Insoluble

NaOH 60 + Hydro-

Dyeing on wool, tanned cotton & cotton Wool dyed, possibly also cotton slightly, also wool possibly tanned cotton dyed.

Tanned cotton dyed.

Cotton Vats dyed, possibly dyed, & mostly possibly weak & dull. wool most dved.

Vats mostly much stronger deep yellowish yellow or brownish coloured. (oxydisable)

sulphite

Dyestuff base extracted with ether.

Treat with NaOH, Base + ether shaken up. Ether + Acetic acid. Ether filtered.

Colourless

Dyed

Group of the Acid dyestuffs.

Group of the Basic dyestuffs.

Group Group of Group of the of the the Vat sulphur Direct dyestuffs dye-Cotton stuffs dyestuffs.

> Make Make dyeings. dyeings. Further tests follow on the fibre.

		The dyestuffs by extraction (a) H2O (d) NH5 + H2O (e) NH5 + H2O alcohol	(b) Alcohol (c) Glacial		nd S	Treat fibre with cold stannous chloride (see over)	warmed with le	de . on Vatiling	tion then Oxi-	8. Pyridin
		210 01101	20 14	u 05000h.		(866 5/41)	test for	or autphite	Q 1 se	
l. Aci	id dyestuffs			Wool dyed, possibly also cotton, but less possibly tanued cotton						
11. Be	sic dyestuffs		27	Tanned ootton, possibly also wool						
111. 1	Not developed e.g. Sirius			Cotton dyed, and possibly						
IV.	developed e.g. Primulin	chiefly in e	(+)	wool.	,					
V. Mo	ordant dyeatuffs		Dyestuff often de-	Possibly wool, occasionally tanned cutton, not cotton.	sol	70 8- 70				
n.Indo	carbon CL dysstuf	°f's			y	ibres soon ellowish Oxidisable)	Rather strong.	Yellowish.	Colour	
V11. F	Hydron Blue dyestu	ffs					Clear			
V111.	Sulphur dyeatuffs blue, green, grey violet, olive bla	, and dyeing ok. basic and d	n extraction similar to irect But weaker).			•	Rather	Mostly yellowish		
ıx.	Sulphur dyestuffs yellow, orange brown.	· Ap		12 10 10	1	ostly ittle hange.	100	Mostly yellowish, brownish		
I.	Oxidation dyestuf aniline black, Di black, Faramine, 1 amine.	phenyl		Precipitates, but possibly contains basic by-products.	h	ibres ardly ltered.		Dull yellowish		Insolubl
n.	Vat dyestuffs (inc	digosols)	•	Precipitate		estroyed.	Some weakly positive.	Rapid change in colour of the fibre (Vata)		Soluble
X11, N	aphthol AS (Rapid Rapid Rapid	ogen,		Precipitate		ardly hanged		Fibres slowly go yellowish Very stable fibres fluorescent with U.V. lamp.	Does not return	Very soluble
X111.	Ice Colours, Para and other, naphthol combination	В		Precipitate				Fibres almost colourless. Fibres not fluorescent with U.V.		•

Stannous chloride Solution:

Ammonia:

I Part SnCl2

25% Aqueous ammonia

1 Part Conc. HCl

d = 0.910

1 Part Water.

SINGLE DYESTUFF IDENTIFICATION FOR ACID, BASIC & DIRECT DYESTUFFS SYSTEM FOR THE REACTION TABLES (FOR ALL THREE DYESTUFF GROUPS)

FUNDAMENTAL REACTIONS

andre a granten and control of the State Sta			filter pa	n to moist per, or so filter pa	lution	Reduction and amm	
Examples	H ₂ SO ₄ Conc.	Diluted with water.	+ N2SO4 10% sol- ution.	+ NaOH	+ NH ₃ 25%	filter paper.	
Direct Deep Black E Extra.	Violet blue,	Violet black.	Violet	Blue green	Blue	Violet blue	Greenish blue
Lirect Deep Black RW	H	H	•	# T	n		"
Artificial Silk Black G.		N	H	H	H	*	
Diamine Black BH	Blue	Reddish violet		Somewhat redder		Red Brown	Dull

ADDITIONAL REACTIONS

in the general Reactions as they are used for Vat dyestuffs and Naphthol AS series.

SEPARATION REACTIONS

chiefly chromatographic absorptionanalysis.

SINGLE DYESTUFF IDENTIFICATION FOR THE VAT DYESTUFFS. SCHEME OF FUNDAMENTAL REAJILONS

	Nitric Acid Spot NaOH & Hydrogulphi						sulphite	ACCOUNT OF THE PARTY OF THE PAR	
	Concentrated H ₂ SO _A	HNO3	Spot on filter paper	filter paper and SnCl2	Fibre in SnCla	Fibre	Solution	Acidified Vat	
Examples.									
Ind. Yellow GK	Tellow					Dull reddis violet (very soluble		Tellow	
* * 5GK								Red	
Algol Yellow GC	Lemon		Lemon	Red	Red	Violet	Violet		
Ind. Golden Yellow CK	Red-violet					Red	Red	Tellow	
" " " RK	Violet					Violet red	Violet red	Tellow	
n Red FBB	Almost colour	1005				Oliye	Greenish violet (red)	Wiolet red	
BE	Yallow brown		Rether yellower	Rather		Violet	Violet	Orange	
	Withou mad					Lemon	Lemon	Col outle	
* Brill.Pink R Extra	Fibre red, Solution green								
* Scarlet B	Green								
" Brill. Violet RR	Blue green					Blue	Blue	Red	
" Green B	Lagenta								
						Brown	Brown	Yellow	
* Olive R Indigo Pure K	Violet brown Yellow olive	Quickly	Yell ow	Lemon	Slowly	Lenon	Lemon	Almost Colourles	
MLB/6B	Blue green	Almost	or ango			Orange yellow	Orange yellow	Pale yellowish	
		unaltered				Blue	Blue	Blue	
Ind. Blue 5G	Green	Buller	Green	Blue green			Brown	Blue	
" " RK	Yellow olive	Yellow	Yellow	Blué		Dark			
e RS			Strong yellow	HNO3 Partibl	ue	B1 ue	Blue	Blue	
# # BC	•	Green yell ow	Clear	HNO3 partigi	reet				
For Comparison.									
Indocarbon CL	Blue + HNOs violet				Almost decolourised	Brown yellowish.	yellowish	yellowish	
Sulphur Black	Mostly sligh soluble & bl		Clear black- grey	Decolourised	1	Mostly brown yellowish	Mostly yellowish		
Hydron Blue	Slightly sol blue black + HNO ₃ = blue	uble	Pale blue			Yellowish	Yell owd, sh		
Alisarin Red	Brownish red	Yellower	Orange yellow + NaOH = violet	Unalter	Slowly yellower	Orange	Orange		

SINGLE DYESTUFF IDENTIFICATION OF THE VAT COLOURS

Additional reactions.

These supplement the fundamental reactions insofar as these are the same or similar for several dyestuffs, and also they characterise certain individual dyestuffs. They are also different from case to case and do not fall into a scheme. Below are some examples of their use and some of the other useful reagents or methods.

Differentiation of the Indigoid blues.

Glacial Acetic acid + 10% HNO3 Dyeing treated for 1 minute.

Alizarine Indigo

Indigo - not halogenated

Indigo halogenated

Indanthrene Printing Black B

Piperidine - cold. Indigo not halogenated.

> Indigo halogenated

Solution

rather stable blue.

quickly yellow

blue ---- yellow

blue --- orangebrown

blue-green solution.

orange-redorange (then 4G almost yellowish-green)

Conc. H2SO4, H2SO4/K1O3 H2SO4/HNO3) leave standing, differentiate the non halogenated Indigo.

H SO 580 Be, Fibre warmed in Stannous) Differentiation chloride Hoso4/HNO5, Fibre + HNO5

of the halogenated Indigo.

	Ind. Red FBB	Ind. Rubin B	Ind. Bordeaux B
Acid leuco compound HNO ₃ + little NaNO ₂	Red Red orange	Red Yellow	Green Green
		Indanthrene Golden Yellow RK	Indanthrene Printing Yellow GOW
H ₂ SO ₄ 58° Be	Orange	Yellow	Yellow
Spectrum in Pyridine	Good	· Displaced compa	
" " + " + " + " + " + " + " + " + " + "		Slight differen	ces.

Differentiation of the Indanthrene Blues (${\rm H_2SO_4}$ Conc. Yellow Olive, ${\rm HNO_5}$ spot yellow. Hydrosulphite blue).

			Fibre + HN03	HNO3 spot	Fibre in SnCl ₂ colour of the HNO ₃ part.
Ind.I	1/8	RS - Brill. Flue R 3G " 3G		Yellow	Full blue
14	. 19	GCD	и и	Н	Green blue
19	16	BC, BCS	Remains tr. greener Pure yellow	Pale yellow	Green

Spot with diluted HNO3 (50 cc. conc. HNO3 + 30 cc. H20) for the differentiation of RS and 36.

Spectrum of a rather conc. solution in conc. $\rm H_2SO_4; BC$, BCS good; CC good, but displaced slightly compared with BC.

Reagents. H₂SO₄/KlO₃ = 1,000 cc. conc. H₂SO₄ + 6 gm. KlO₃ H₂SO₄/HNO₃ = 400 cc. conc. H₂SO₄ + 15 cc. conc. HNO₃ H₂SO₄58° Be = 75% H₂SO₄ Starrous chloride = SnCl₂ crystals + conc. HCl + water (1:1:1) H₂SO₄/Boric acid for spectrum = 5% Boric acid otherwise = 10% "

Further Convenient Additional Reactions & Methods.

Pyridine + NaOH (4%). Distinguishes Ind. Yellow RK from Cibanone Yellow RR Indanthrene Brill. Violet RK, PBK = Green Indigracal Brill. Creen - I4G = Violet Indanthrene Olive Green B - dyestuff=Red

Pyridine + water + Hydrosulphite, then + NH3, then + NaOH Distinguishes Indanthrene Yellow CK and 5GK.

Conc. H2SO4 to which is added 1 drop HNO3, typical colour changes, e.g.

Indanthrene Red GG, Indanthrene Brilliant Green and Indocarbon CL dyestuffs, Hydron Blue.

Conc. H2SO4/Boric acid possibly warmed slightly, e.g., Ind. Crange 6RTK and 7RK Violet (+ 1 drop HNO3 = Magenta Red)

Conc. H2SO4/HNO3 for example distinguishes Indanthrene Olive 3G from Indanthrene Khaki GG (compare also acid leuco compound) Indanthrene Brown NG fast blue.

Conc. HNO3 in test tube, e.g., Ind. Olive T.

Conc. HNO3 + H2O2 distinguishes Indanthrene Blue 5G, and

Indanthrene Green BB.

Conc. HNO3 + a little fuming nitric - Indanthrene Printing Blue R.

Conc. HCl and 20% HCl - Indanthrene Green 4G Conc. H2SO4 then 75% H2SO4 added. Typical colour change, Indanthrene Brown 3GT from blue to red violet.

Conc. H2SO4 + H20 100: 5 cc. Indanthrene Red Violet RH green solution (RRN red).

Conc. H2SO4 + H2O 100:75 cc. distinguishes Indanthrene Brilliant Violets RK and BBK.

H2SO4/K103 (see for Naphthol AS) distinguish between Navy Blue & Dark Blue. H2SO4 75%

Stannous Chloride Solution.

Treated with alcohol, then + some stannous chloride solution in U.V. (Hanover quartz-mercury lamps = U.V. lamp) Indanthrene Brilliant Pink R Extra yellow green fluorescence. Indanthrene Scarlet B - violet fluorescence.

Vat poured on paper under U.V. lamp, Indanthrene Brown RRB. Indanthrene Printing Brown R, yellow orange fluorescence.

Cold Piperidine solution: Indanthrene Brilliant Violets RK,
BBK blue green;

Indanthrene Printing Violet BBF - orange yellow.

Cold Piperidine solution under U.V. lamp. Indanthrene
Printing Violet Pre - orange yellow fluorescence.
Indigosol Printing Violet IRR green yellow.

Spectrum in Pyridine - distinguishes Indanthrene Brilliant Violet RR, 4R and 3B - Indanthrene Dark Blue BOA.

Spectrum in NaOH + Hydrosulphite solution - Indanthrene Navy Blue R.

Spectrum in H₂SO₄/Borie acia (5% Borie acia) - Distinguishes Indanthrene Blue 5G, and Indanthrene Green BB.

Spectrum in Conc. H₂SO₄ - Indanthrene Olive R. Spectrum in Conc. H₂SO₄ then + 1 drop HNO₃ - Indanthrene Olive R.

Vat shaken up with ether then ether in the U.V. lamp.

Indanthrene Red 5CK - crange fluorescence.

Cibanone Red B - no fluorescence.

Separation Methods for Vat Colour Mixtures

- Conc. HNO3. Dyestuffs which are soluble in conc. HNO3 are separated by the HNO3 spot (see fundamental reactions) from dyestuffs which are insoluble in HNO3. Therefore, further reactions can be done on the spot for identification. If several dyestuffs bleed off, then possibly a more dilute HNO3 or possibly also HCl can be used. By repeated extractions of the dyeing with HNO3 the dyestuffs insoluble in nitric acid are occasionally left on the fibre in a suitable condition for their isolation.
- Strong Soaping Test against white cotton removes individual dyestuffs, e.g., Ind. Yellow G, Indanthrene Red GG and in part Indigoid Reds, Indigoid Blues and Blacks. In a similar manner individual dyestuffs can be vatted off with a -

Weak Reducing Agent, e.g., Na & + possibly a little paridine.

- Hydrosulphite + 4% NaOH. By repeated treatment many
 dyestuffs may be completely removed, whereas
 others remain on the fibre. To vary this,
 others may be left in caustic and hydrosulphite
 at 80° for a longer period, or NaOH of other
 concentrations may be used.
- Hydrosulphite in Water, without alkali, possibly with or without pyridia.
- Strong Bleach. The fibres, possibly first moistened with a little pyridine, a little water added and warmed. Dyestuffs not fast to chlorine are decolourised in varying degree. Indanthrene Blue 5G, Indanthrene Brilliant Violets RK, BBK, Indigoid Blues and Blacks.
- Bleach + Acid destroys many dyestuffs, many are only changed to yellow.
- Permanganic Acid (KMnO4 + Acid) destroys for example,
 Indanthrene Brilliant Green, Ind.

 Brilliant Violet, while many yellow
 dyestuffs are more or less stable.
- Pyridine or Piperidine + H₂O + Hydrosulphite. Many
 dyestuffs remain on the fibre (Ind. Yellow
 3RT, Ind. Golden Orange 3G, Ind. Red FBB,
 Indanthrene Scarlet P, Aniline Black)
 Others are completely removed (Ind.
 Scarlet B, Indanthrene Blue 5G, Ind. Red
 Brown 5RF, etc.).
- Sparation with Orangic Solvents. Glacial acetic acid,

 Pyridine, then Glacial acetic acid;

 Alcohol then Glacial acetic acid, etc..
- Conc. HNO 3 + Hydrogen Peroxide destroys, for example, the Indanthrene Blue 8CK dye stuffs.

The choice of method of separation depends on the dyestuffs present and emphasises the importance of experience in the use of the fundamental reactions.

Identification of Single Dyestuffs of the Naphthol AS Group

The dyestuffs of the yellow and brown naphthol series AS-SR, AS-SG, AS-GR are not yet included analytically in a system. They are identified individually (Reagents as for the rest of the Naphthol AS series). By spottic with HNO3 many AS-G dyestuffs become greenish, and dyestuffs of the other yellow naphthols become orange. On shade grounds these Naphthol AS dyestuffs are mostly well distinguished from the following.

The other, approximately 700, dyestuffs from the Naphthols AS, AS-BS, BO, BR, BG, TR, D, RL, SW, OL, E, LT, ITR, LC, S, can be fitted in to a system. Dye stuffs prepared from one and the same base (independently of which Naphthol, the base is coupled with) give with certain reagents almost the same colour change. These colour changes are different from base to base, and the base is first determined by means of these "Base Reactions" (see Tables on pages 13 and 14). This summary is subdivided into a "Red Group" (Table on page 13) and into a "Special Group" (Table on page 14). In the use of Absorption spectra these must be accurately compared. Then follows the identification of the Naphthols. There are few Naphthol reactions, and the colour changes obtained with them are sometimes also dependant on the base. not possible, therefore, to set down a system, but the reactions and technique used are set out on page 15. Careful comparisons are necessary especially with the H SO,/Kloz reagent since the colour changes are transient. The approximately 15 dyestuffs of one base with different Naphthols can, however, be distinguished in part by the shade.

MAPHTHOL AS - RED GROUP BASE IDENTIFICATION

Red Group comprises the oranges, reds to bordeaux dyessuffs insofar as they became red, red violet, violet or blue with concentrated HgSO4 in the reaction of Past Bordeaux GP.

		Cono. H2904	Pyridine +	Pibres treated but with cond NaOH, RaoS about 2 min. Some times moistened with pyridin Pibros washed off,	10.		
Past	Bordeaux BO Red RC Red FR Red FG	d RC Pure Blue d FR Violet blue		Almost unaltered	H ₂ SO ₄ /Borio Acid - green blue Quickly gold yellow H ₂ SO ₄ /HNO ₅ red, allowly yellow Differen H ₂ SO ₄ /HNO ₅ " spectrum Conc.H ₂ S		
:	Red GTR Soarlet LG }	Dark violet Going redder		Mestly little bluer Almost unaltered	Scarlet RC is similar HNO3 bleed typical with AS.ITR		
*	Red ITR).	Violet magenta Going redder	Wach bluer (violet)	COLUMN TRICKY	H2SO4/HNO3 in basin, lemon yellow H2SO4/HNO3- soon yellower		
n 10	Red TR Red RBE Red KB	(Violet)magenta	All less typical	Mostly little bluer Almost unaltsred			
	Orange RD Scarlet TR	Magenta Going redder	(Somewhat bluer, (somewhat				
	Orange GC	a a	redder)	Mostly somewhat bluer	}		
#.	Gold Orange GR	Red violet			Warm with & NaOH - red violet Hydrosulphite + WaOH - Fibre at first redder.		
* * *	Scarlet RC Orange LG Scarlet G	Blue violet Red violet		Much bluer	Bewars Past Scarlet LC (see above). Different spectra in Geno. H ₂ SO ₄		
**	Orange R Red RL Red GG	Magenta red	Blue } a group		Different H2SO4 66/HNO3 Solution A		
	Me or a	Violet blue Magenta red	Clear blue	Decolourised })Different - trace redder		
9	Rad GL Red 5GL Bordeaux GP	(b1)Violet Blue (b1)Violet	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	group)H2304/HB03 - trace bluer Hydrosulphite + NaOH = Orange-		

BASES PROM FAST RED GTR TO FAST OWANGE GGU

In addition to the resotions given some measure of differentiation is given by the Spectra which are placed one after another and should be compared:

(1) Concentrated $\rm H_2SO_4$ then some $\rm H_2SO_4/HBO_5$ added to it. (2) To (2) add some $\rm H_2SO_4/HNO_5$ (4) Concentrated $\rm H_2SO_4$, then add some $\rm H_2SO_4/HNO_5$.

REACENTS.

H_9504/HNO3 solution A = Concentrated H_9504 400 cc. + concentrated HNO3 15 cc. H_9504/Horiz Acid for Spectra = Concentrated H_9504 > 5% Boric Acid.
H_9504/horiz Acid otherwise = Concentrated H_9504 + 10% Boric Acid.
H_9504/horiz Acid otherwise = Concentrated H_9504 + 10% Boric Acid.

Gencentrated NaOH + Hag5 = 100 gm. NaOH 55° Tw.

+ 62 gm. Hag5 Cryat.

+ 16 cc. H_90

The Special Group includes:

Of Red Tones - no pure red, but only bouldeaux reds and those which in conc. H2SO4 give green-blue or green. The Corinth violets, blue or dark blue shades of these dyestuffs, which give blue, green blue, or green in conc. H2SO4. Also Fast Violet B, Fast Corinth B (Conc. H2SO4 Violet Blue) and the Variamine Blue group (Conc. H2SO4 - red or violet) Pyridine + NaCH ---> red. Also the Brown Bases, which react typically in conc. H2SO4 and Pyridine + caustic.

Variamine Blue B Red " RT " Past Brown V Olive brown RR " RR " RR " RR Intense magenta	" Black G Slowly blackish blue " Dark Blue R Bright green " Black K (blue) green " LB (blue) green " Blue BB " "	Fast Corinth V Bright green " Carnet GO " " " Blue B " " " Corinth B Green blue	Concenti
uble	h ITR = wiolet black SW, BG, LC, slowly wiolet black SW soon violet black BG slowly wiolet black LC slowly almost blue	SW - blue> violet blue Vi BG blue> violet blue Vi BG slowly bluer BG slowly bluer BG slowly bluer	
Much redder(scarlet)) Different with H2SO4/HNO3 (400:15) Little bluer and HNO3 bleed typical duller Intense red Perric Sulphate (500 cc. + 0.5gm) HNO3 bleed typical ** HNO3 bleed typical Strong clive green HNO3 bleed typical Luch yellower red (almost wine red)	**************************************	Violet Intense blue Magenta Intense violet blue Magenta Intense blue Mag	Furidine> when + 1/2 NaOH

IDENTIFICATION OF THE NAPHTHOL COMPONENTS FOR THE RED AND SPECIAL GROUPS

This follows, after the base is determined, by comparison with the dyestuffs of this base in general with the following reagents:-

- (1) Conc. H₂SO₄ + KlO₃ 1,000 cc. + 6 gm. KlO₃ (Stable) Reagent for AS-BR, AS, AS-LC, AS-BG, AS-S.
- (2) Conc. H₂SO₄ + H₂O₂ 30 cc. + 3.5 cc. H₂O₂(15%)
- (3) Fibre spetted with cone. HNO3, and pressed on filter paper, AS. ITR, often distinguishable.
- (4) Conc. H₂SO₄ + H₃PO₃ 30 cc. conc. H₂SO₄ + 10 cc. phosphoric acid (23%).
- (5) H₂SO₄ + KlO₃ + fumaric acid 15 cc. H₂SO₄/KlO₃ + fumaric acid.
- (6) H2SO4 + K1O3 + Maleic acid 15 cc. H2SO4/K1O3 + maleic acid.
- (7) H₂SO₄ + Boric acid + KlO₃ 15 cc. conc. H₂SO₄/KlO₃ + about 15 cc. H₂SO₄ + 10% boric acid.
- (8) H₂SO₄ 75%
- (9) Treat with glacial acetic acid, then add a little stannous chloride.
- (10) Crystallisation (usually glacial acetic acid) (occasionally also pyridine).

Reagents 2, 4, 5 and 6 are frequently freshly made up since they are of limited stability.