

# **RESOLUTION OENO 19/2003**

# REAGENTS AND TITRATED SOLUTIONS USED IN THE TEST TRIALS OF PRODUCTS USED IN OENOLOGY (Oenological Codex)

THE GENERAL ASSEMBLY,

CONSIDERING Article 5, paragraph 4 of the International Convention for the Unification of Methods of Analysis and Appraisal of Wine of 13 October 1954,

UPON PROPOSAL of the Sub-commission of the Methods of Analysis and Appraisal of Wine,

DECIDES:

TO REPLACE and complete Chapter III of the International Oenological Codex by the following reagents and titrated solutions:

# List of reagents and titrated solutions<sup>[1]</sup>

Acetic	crystallisable acid 98-100%
	diluted acid (10% m/m)
	neutral lead acetate (see Lead)
	potassium acetate (see Potassium)
	sodium acetate (see Sodium)
	uranyl and magnesium acetate
Starch	Paste (aqueous solution at 5 g/l)

# Mention (R)<sup>[2]</sup>



Ammonium	concentrated hydroxide solution (20% NH <sub>3</sub> , $d(20/4)=0.92$
	diluted hydroxide solution (10 g concentrated solution/100 g)
	Aqueous hydroxide solution about 5 M
	chloride in solution at 20% (m/m)
	citrate in solution
	oxalate in solution at 4% (m/m)
	persulphate in solution at 15% (m/m)
Aniline	reagent
Silver	Nitrate (99.5%)
	nitrate solutions at 5% (m/m) (R1)
	Nitrate solution at 1% (m/m) (R2)
	ammonia nitrate solution
Barium	$BaCl_2.2H_2O$ solution at 10% (m/m) chloride
Bore	boric acid, $H_3BO_3$ 99% concentrated boric acid solution at 4% (m/v)
Bromine	Br <sub>2</sub> (d(20/4)=3.12) bromine water
Bromophenol	tetrabromophenolsulfonephtaleine (blue) alcoholic solution (blue)
Bromothymol	dibromothymolsulfonephtaleine (blue) alcoholic solution (blue)
Bromocresol	tetrabromo-m-cresol-sulfonephtaleine (green) (green) alcoholic solution
	methyl red and (green) in solution (mixed indicator)



Calcium	acetate aqueous solution at 25% (m/v)
	chloride saturated solution
	chloride solution at 20% (m/v)
	hydroxide (milk of lime)
	saturated sulphate solution
Mineralisation catalyser	
Chloramine T	solution at 1% (m/v)
Chlorine	concentrated hydrochloric acid at 35% (d( $20/4$ )=1.19) hydrochloric acid diluted at 30% (v/v) hydrochloric acid diluted at 10% (m/m) hydrochloric acid diluted at 10% (v/v) potassium dichromate (see Potassium)
Chrome	potassium dichromate (see Potassium)
Chromotropic acid	sodium salt sodium salt solution
Citric acid	Monohydrated 99% aqueous solution at 21% (m/m) aqueous solution at 20% (m/v) aqueous solution at 10% (m/v) aqueous solution at 5% (m/v) aqueous solution 0.003 M
	hydrochloric solution solution adjusted to pH 3
Cobalt	chloride $CoCl_2.6H_20$ aqueous solution at 5% (m/m)
Copper	sulphate $CuSO_4.5H_2O$ sulphate solution at 1 g of copper per litre
	sulphate solution at 0.01 g of copper per litre copper sulphate ammonia solution (II)
	alkaline copper reagent





Dichlorophenolindophenol	sodium salt of 2,6-dichlooro-N-(4- hydroxyphenyl)-1,4-benzoquinone monoimine dihydrate aqueous solution at 0.5 g per litre
Diphenylcarbazide	1,5-diphenylcarbonodihydrazide at 0.5 g per litre of alcoholic solution at 95% vol.
Dithizone	1,5-diphenylthiocarbazone solution at 0.5 g/l in chloroform extemporaneous preparation
Iron	iron sulphate (II) FeSO <sub>4</sub> .7H <sub>2</sub> O 99%
	iron sulphate (II) solution at 5% (m/m)
	iron sulphate (II) and ammonium Fe(NH <sub>4</sub> ) <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub> .6H <sub>2</sub> O 98.5% iron sulphate (II) solution and ammonium at 10% (m/m) iron sulphate (III) Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .7H <sub>2</sub> O solution at 0.01 g of iron (III) per litre
Formaldehyde	aqueous solution at 35% (m/m)
Basic fuchsine	Mixture of rosaniline hydrochloride and pararosaniline hydrochloride Solution bleached by sulphur dioxide
Hydrazine	dihydrochloride aqueous solution
Hydrogen peroxide	concerntrated solution at 30% (m/m) (=110 volumes) diluted solution 3% (m/m) (10 vol.)
Iodine	99.5% iodine solution
Sodium indigo-sulphonate	(see sodium)
Mixed indicator	(see methyl red)
Magnesium	chloride MgCl <sub>2</sub> .6H <sub>2</sub> O 99%
	magnesian mixture



mercury oxide (II), yellow mercuric oxide 99% mercury sulphate (II) solution
(see m-phenylenediamine)
(see red methyl)
sodium 4(dimethylamino)azobenzene-4-sulfonate. Methyl orange alcoholic solution at 1% (m/v)
reagent (see Nitric)
□-naphthol (2-naphtol)
solution at 5% (m/m)
concentrated acid 63%
acid diluted at 10% (m/m)
nitromolybdic reagent
nitro-vanadomolybdic reagent
lead nitrate (see Lead)
Biting black 11 solution at 0.2% (m/v) in triethanolamine
acid $C_2O_4H_2$ .2 $H_2O$ 99% aqueous solution at 5% (m/m)
dihydrochloride C <sub>6</sub> H <sub>8</sub> N <sub>2</sub> .3 HCl 99%
(see phenol red)
phenolphtalein solution at 1% in alcohol (m/v)
concentrated phosphoric acid (orthophosporic acid) 85% d(20/4)=1.7
Diluted solution of phosphoric acid at 50% (m/m)
Diluted solution of phosphoric acid at 25% (m/v) Dihydrogenophosphate (see Potassium)



Lead	neutral lead acetate $C_4H_6O_4Pb.3H_2O$
	aqueous solution at 10% (m/m) (in water free from carbon dioxide) nitrate Pb(NO <sub>3</sub> ) <sub>2</sub> 99% lead nitrate aqueous solution at 1 g of lead per litre lead nitrate aqueous solution at 0.01 g of lead per litre





Potassium	acetate $C_2H_3KO_2$ 99% aqueous solution at 5% (m/m)
	anhydrosulphite $K_2S_2O_5$ (disulphite) 94% free from selenium
	potassium anhydrosulfite aqueous solution at 2% (m/m) cyanide KCN 98% aqueous solution at 10 g per 100 ml
	potassium cyanide aqueous solution at 1 mg of hydrocyanic acid per litre
	dichromate $K_2Cr_2O_799\%$ aqueous solution at 10% (m/m) aqueous solution at 1 g of chromium per litre aqueous solution at 0.01 g of chromium per litre
	dihydrogenophosphate $H_2KPO_4$ 99% aqueous solution at 0.05 g of phosphorous per litre hexacyanoferrate (II) $K_4Fe(CN)_6.3 H_2O$ 98%
	aqueous solution at 5% (m/m) hydroxide KOH 85%
	aqueous solution at 40% (m/m); d(20/4) = 1.38 iodide KI 99%
	iodine potassium iodide solution permanganate KMn0 <sub>4</sub> 99% aqueous solution at 5% (m/m) aqueous solution at 3% (m/m) aqueous solution at 2% (m/m) aqueous solution at 1% (m/m) aqueous solution at 0.5% (m/m) aqueous solution at 0.2% (m/m)
	potassium permanganate phosphoric solution saturated aqueous solution
	thiocyanate KSCN 99% aqueous solution at 5% (m/m)
Pyridine-pyrazolone	reagent



Quinine	sulphate $C_{40}H_{48}N_4O_4$ . $H_2SO_4$ .2 $H_2O$ 99% quinine sulphate sulphuric solution at 0.1 mg per litre of sulphuric acid 0.05 M
Rosaniline	hydrochloride (see fuchsine) aqueous solution at 0.1 g per 100 ml
Methyl red	acid 4-dimethylamino-2-phenylazobenzoic red methyl alcoholic solution methyl red mixed indicator
Phenol red	phenolsulfonephtaleine 98%

	phenol red solution
Selenium	dioxide $S_eO_2$ 99% aqueous solution at 100 mg of selenium per litre



Sodium	acetate C <sub>2</sub> H <sub>3</sub> NaO <sub>2</sub> .3 H <sub>2</sub> O
	aqueous solution at 10% (m/m)
	borate (tetraborate) $Na_2B_4O_7.10 H_2O 99\%$ saturated aqueous solution
	decahydrate carbonate $Na_2CO_3$ ,10 H <sub>2</sub> O 99% aqueous solution at 25% (m/m)
	diethyldithiocarbamate $C_5H_{10}NS_2Na.3 H_2O$ 99% alcoholic solution at 1% (m/v)
	ethylenediaminetetracetate (disodic edetate) $C_{10}H_{14}N_2O_8Na_2.2 H_2O 98.5\%$ aqueous solution 0.01 M
	fluoride NaF 98.5 % aqueous solution at 4% (m/m)
	concentrated hydroxide solution (caustic soda) at 30% (m/m); d(20/4)=1.33 diluted aqueous solution of sodium hydroxide at 10% (m/m)
	hydrogenophosphate (disodic dihydrate phosphate) HNa $_2PO_4.2 H_2O 99.5\%$ aqueous solution at 10% (m/m)
	pyrophosphate $Na_4P_2O_7$ .10 $H_2O$ (diphosphate decahydrate tetrasodium) 98%
	aqueous solution at 1% (m/m) thiosulphate $Na_2S_2O_3.5 H_2O$ 99% aqueous solution at 25% (m/v)
	disodic indigo-sulphonate (see indigo carmine) indigo carmine solution
Hydrogen sulphide	saturated aqueous solution acid aqueous acid solution at 1 g of sulphur per litre aqueous acid solution at 0.01 g of sulphur per litre
Sulforesorcinic	reagent



Sulphuric	concentrated acid 95% d(20/4)=1.83 concentrated acid 97% (m/m)
	aqueous solution at 25% (m/m) aqueous solution diluted at 10% (m/m) aqueous solution diluted at 5% (m/m) acid free from nitrogen
Buffers	purified acetate (search for zinc)
	ammoniac pH 7.5
Tannin	definition
	aqueous solution at 2% (m/m) aqueous solution at 4% (m/v) aqueous solution at 10% (m/m)
Thioacetamide	reagent
Uranyl	nitrate UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> .6 H <sub>2</sub> O 99%
	aqueous solution at 4% (m/m) uranyl acetate UO <sub>2</sub> (CH <sub>3</sub> COO) <sub>2</sub> .2 H <sub>2</sub> O 99% magnesium and uranyl acetate and acetate solution
Bromocresol green	(see Bromocresol)
Bromocresol green and methyl red	(see Bromocresol)
Zinc	Solution 1 mg per litre

# **REAGENTS AND TITRATED SOLUTIONS**

# Crystallisable acetic (acid)

 $\Box 20=1.051$ ; contains as a minimum 98.0% (m/m) of  $C_2H_4O_2$ .

## **Diluted acetic (acid)**

Aqueous solution containing about 10 g of acetic acid in 100 g of reagent.  $\Box 20=1.0125$  approximately.





# Starch (paste) at 0.5% (m/v)

In a mortar, grind 2.5 g of soluble starch and 10 mg mercury iodide (II) with the necessary amount of water in order to obtain a fluid slurry. Introduce this in 500 ml of boiling water that is maintained 10 minutes. The liquid obtained is clear. Filter if necessary.

#### **Concentrated ammonium hydroxide solution**

□20= 0.922.

Concentrated aqueous solution of ammonia gas containing about 20 g of ammonia ( $NH_3$ ) in 100 g of reagent.

#### Diluted ammonium hydroxide solution

Aqueous solution of ammonia gas containing about 10 g of ammonia (NH3) in 100 g of reagent.

□20= 0.959 approximately.

#### Ammonium (chloride) in solution

Aqueous solution containing 20 g of ammonium chloride in 100 g of reagent.

#### Ammonium (citrate) in solution

Slowly pour 500 ml of concentrated ammonium hydroxide solution (R) in 400 g of citric acid in a 1000 ml graduated flask. The mass is heated and the dissolution is carried out. After cooling, complete the volume of 1000 ml with concentrated ammonium hydroxide (R).

#### Ammonium (hydroxide) in solution about 5 M

Dilute 460 ml of concentrated ammonium hydroxide ( $\Box 20=0.922$ ) with a sufficient amount of water to obtain 1 l.

#### Ammonium (oxalate) in solution at 4% (m/m)

Aqueous solution containing 4 g of diammonium oxalate in 100 g of solution.

#### Ammonium (persulphate) in solution at 15% (m/m)

Aqueous solution containing 15 g of ammonium persulphate for 100 g of solution.





#### Aniline

 $C_6H_5NH_2 = 93.1.$ The product used as a reagent must be clear and barely yellow.  $\Box 20= 1.020$  to 1.023. During distillation, 95% as a minimum must pass between 183°C and 185°C.

# Silver (nitrate) in solution at 5% (m/m)

Aqueous solution containing 5 g of desiccated silver nitrate for 100 g of reagent.

# Silver (nitrate) in solution at 1% (m/m)

Aqueous solution containing 1 g of desiccated silver nitrate for 100 g of reagent.

# Silver (nitrate) in ammonia solution

Ammonia solution prepared with 10 g of desiccated silver nitrate for about 100 g of reagent.

In 30 g of distilled water, dissolve 5 g of desiccated silver nitrate. Pour into this solution, drop by drop with caution, the diluted ammonium hydroxide solution (R) until nearly total redissolution of the precipitated silver oxide. Complete to 50 ml, filter and store the reagent away from light in a flask with a glass stopper.

# Barium (chloride) in solution at 10% (m/m)

Aqueous solution containing 10 g of  $BaCl_2.2H_2O$ , for 100 g of reagent.

# Concentrated boric (acid) in solution at 40 g per litre

This acid must be pure, entirely soluble in water (insoluble residue below 50 mg for 1 kg) and must not turn brown during incineration (absence of organic matters).

The aqueous solution at 40 g for 1 l of solution must be neutral to methyl orange. The orange coloration of this indicator must be obtained with less than 3 ml of hydrochloric acid solution 0.1 M for 1 l of this solution at 40 g per litre.

Boric acid that does not respond to these test trials can be purified by hot filtration of a boiling, saturated boric acid solution (at about 350 g per litre of water) and crystallisation by cooling.

Prepare a solution of 40 g of this concentrated acid for 11 of solution.



# **Bromine (water)**

Bromine saturated aqueous solution containing about 3.5 g of bromine for 100 ml at 20°C.

#### Bromophenol blue in solution

Alcohol solution at 95% vol. containing 0.04 g of bromophenol blue in 100 ml in total.

#### Bromothymol blue in solution

Alcohol solution at 95% vol. containing 0.04 g of bromothymol blue in 100 ml in total.

# Calcium (acetate) at 25% (m/v)

Calcium acetate aqueous solution at 25 g for 100 ml.

#### Calcium (acetate) in solution pH 6

In a cylindrical vase place:

- calcium carbonate 10 g
- acetic acid 12 g
- water 100 ml

Heat until dissolution, adjust the pH to 6 and adjust to 1 l.

#### Calcium (chloride) in saturated solution

It contains about 80 g of  $CaCl_2.6H_2O$  for 100 g of solution.

#### Calcium (chloride) in solution at 20% (m/v)

Aqueous solution containing 20 g of crystallised calcium chloride  $CaCl_2.6H_2O$  in 100 ml of reagent.

# Calcium hydroxide (milk of lime) at 10% (m/m)

The calcium hydroxide suspension (milk of lime) is obtained by treating 10 g of calcium oxide (quicklime) with 90 g approximately of boiling water.

# Calcium (sulphate) in saturated solution

Saturated aqueous solution; it contains about 0.2 g of  $CaSO_4$  for 100 g.





#### **Mineralisation catalyser**

Pulverise and mix:

- selenium 2.5 g
- copper sulphate (II) 5 g
- dipotassic sulphate 100 g

#### Chloramine T solution at 1% (m/v)

Aqueous solution containing 1 g of chloramine T (sodium salt of p-toluene N-chlorosulphanomide) for 100 ml of reagent.

#### **Concentrated hydrochloric (acid)**

Aqueous solution of hydrochloric acid ( $\square 20=1.18$  to 1.19) containing 35.5 to 37.25 g of hydrochloric acid (HCl) in 100 g or 100 ml.

#### Hydrochloric (acid) diluted at 30% (v/v)

Dilute 300 ml of concentrated hydrochloric acid ( $\square 20=1.19$ ) with a sufficient amount of water to obtain 1 l.

This solution contains about 13 g of HCl for 100 ml.

#### Hydrochloric (acid) diluted at 10% (m/m) ( $\Box$ 20= 1.0489)

Aqueous solution containing 10 g of hydrochloric gas (HCl) in 100 g.

#### Hydrochloric (acid) diluted at 10% (v/v)

Aqueous solution of hydrochloric acid containing about 10 ml of concentrated hydrochloric acid (R) in 100 ml, i.e. about 3.6 g HCl for 100 ml.

#### **Chromotropic (acid)**

1.8-dihydroxy-3.6-naphtalene-1.6-disulphonic acid ( $C_{10}H_8O_8S_2.2H_2O = 356.3$ ).

White powder that turns brown in light, soluble in water. The disodium salt of this acid is generally used which is a yellow or light brown product and very soluble in water.

Chromotropic acid solution (sodium salt) at 0.05% (m/v)





Dissolve 60 mg of of sodium salt of chromotropic acid in about 80 ml of water, complete to 100 ml with water. To be used within 24 hours.

# Citric (acid) in solution at 21% (m/m)

Aqueous solution at 21 g for 100 g.

# Citric (acid) in solution at 20% (m/v)

Aqueous solution of citric acid at 20 g for 100 ml.

# Citric (acid) in solution at 10% (m/v)

Aqueous solution of citric acid at 10 g for 100 ml.

# Citric (acid) in solution at 5% (m/v)

Aqueous solution of citric acid at 5 g for 100 ml.

# Citric (acid) in solution 0.033 M

Solution containing exactly one tenth of the equivalent of a gramme of monohydrated citric acid per litre (i.e. 7.003 g per litre).

# Citric (acid) in hydrochloric solution

Dissolve 150 g of concentrated monohydrated citric acid in 800 ml of water; add 100 ml of concentrated hydrochloric acid and add volume to 1 l.

# Citric (acid) in solution to 5 g per litre adjusted to pH 3

Dissolve 5 g of citric acid in 900 ml of water. Add 8 ml of the sodium hydroxide solution 1 M and adjust to 1 l.

# Cobalt (chloride) in solution at 5% (m/m)

Solution containing 5 g of cobalt chloride  $CoCl_2.6H_2O$  in 100 g of reagent.

# Copper (II) (sulphate) solution at 1 g and 0.01 g per litre

The aqueous solution at 1 g of copper per litre contains 3.9295 g of copper sulphate (CuSO<sub>4.</sub>5H<sub>2</sub>O) and 1 ml of concentrated sulphuric acid per litre. This solution is diluted to a hundredth to obtain the solution at 0.01 g of copper per litre.





## Copper (sulphate) in ammonia solution

Copper sulphate Cu SO<sub>4</sub>.5H<sub>2</sub>O 5 g

Water 500 ml

Concentrated ammonium hydroxide (R) 300 ml

Dissolve the copper sulphate in water. Add the ammonium hydroxide and homogenise.

# Alkaline copper (reagent)

The titrated alkaline copper reagent contains for 1000 ml:

Copper, Cu 4.454 g

It is obtained by mixing the two solutions:

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a) Copper solution (II), (C)
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Weigh exactly 35 g of copper sulphate (R) and introduce into a 1000 ml graduated flask with about 500 ml of distilled water and 5 ml of concentrated sulphuric acid (R). Shake to dissolve and complete to 20°C with distilled water until the graduated line. Mix.

b) Alkaline tartaric solution, (T)

Weigh 150 g of sodium and potassium L-tartrate (R) and introduce in a 1000 ml graduated flask containing about 500 ml of hot distilled water. Shake to dissolve. Allow to cool and add 300 ml of concentrated sodium hydroxide solution (R) non carbonated.

Complete to 20°C with distilled water the volume of 1000 ml of solution. Mix.

10 ml of the solution C with 10 ml of solution T are brought to the boil with 0.05 g of inverted sugar, 0.048 g of pure glucose and 0.0695 g of anhydrous lactose or 0.073 g of hydrated lactose.

#### 2.6-dichlorophenolindophenol in solution

Dissolve 0.50 g of 2.6-dichlorophenolindophenol in 200 ml of water heated at 90°C. Allow to cool and complete to 1000 ml with water. Filter.

#### Diphenylcarbazide in solution

Solution of 0.50 g of diphenylcarbazide in 11 of alcohol at 95% vol.

#### Peroxide in diluted solution

See Hydrogen (peroxide).





# Iron (II) (sulphate) in solution at 5% (m/m)

Solution prepared extemporaneously with boiled distilled water containing 5 g of iron sulphate (II)  $FeSO_4.7H_2O$  in 100 g of reagent (air oxidises it quickly).

## Iron (III) (sulphate) in saturated solution

Prepare a saturated solution of iron sulphate (III)  $Fe_2(SO_4)_37H_2O$ .

## Iron (II) (and ammonium sulphate) in solution at 10% (m/m)

Aqueous solution containing 10 g of ammonium and iron sulphate (II) in 100 g of reagent.

#### Iron (III) (salt) in solution at 0.010 g of iron per litre

Dissolve 0.1 g of pure iron in 20 ml of water and 5 ml of concentrated  $H_2SO_4$  (R). Heat, add 10 drops of concentrated  $HNO_3$  (R) and bring to the boil for 10 minutes to peroxidise the iron. Adjust the volume to 11. Dilute 1/10.

#### Formaldehyde in solution

Aqueous solution with 35% (m/m) of formaldehyde.

#### Fuchsine bleached by sulphurous acid

8 g of potassium anhydrosulphite are dissolved in 150 ml of distilled water; add 30 ml of basic fuchsine solution at 1 per 1000 (m/v) in alcohol at 95% vol. and 55 ml of hydrochloric acid 3 M. Complete to 250 ml with distilled water. Store in a yellow flask with an emery stopper.

#### Hydrazine (dichlorhydrate) in solution

Hydrazine dichlorhydrate 500 mg

Water q.s.f. 100 ml

Dissolve the hydrazine dichlorhydrate in about 80 ml of water, then adjust the volume to 100 ml.

Reagent to be prepared extemporaneously.

#### Hydrogen (peroxide) in solution to 3 volumes

This solution contains 9.1 g of  $H_2O_2$  per litre; it liberates 3 times its volume of oxygen





by catalytic decomposition by  $MnO_2$  in an alkaline medium.

# lodine (solution)

Aqueous solution saturated with iodine.

# Sodium indigo-sulphonate

Indigo-disulphonate sodium salt (improperly called indigo carmine): C<sub>16</sub>H<sub>8</sub>O<sub>8</sub>S<sub>2</sub>N<sub>2</sub>Na<sub>2</sub>

This product in solution at 10% (m/v) should turn yellow when oxidised by the potassium permanganate in a sulphuric environment; 50 ml of this solution requires 14 ml to 17 ml of potassium permanganate solution 0.02 M.

If, by permanganic oxidation, this solution does not turn yellow, it is advisable to purify the sodium indigo-sulphonate by the following process:

Put 10 g of sodium indigo-sulphonate in contact with 50 ml of concentrated sulphuric acid (R). After two days, add 100 ml of water; filter the day after. Reject the rusty-coloured filtrate. Take up the residue with 100 ml of water, reject again the filtrate. Dissolve the residue with 800 to 1000 ml of acidulated water and 5 ml of concentrated sulphuric acid (R).

Indigo carmine solution: dissolve 0.2 of indigo carmine in a mixture of 10 ml of hydrochloric acid (R) and 990 ml of sulphuric acid solution free of nitrogen (R) to 200 g per litre.

# Magnesium (chloride) in solution 0.01 M

Dissolve 0.45 g of pure magnesium oxide  $MnO_2$  in the necessary quantity of diluted hydrochloric acid (R). Bring to a litre. Titrate this solution using a sodium ethylenediaminetetraacetate solution 0.01 M in the presence of eriochrome black T.

# Magnesian (mixture)

Dissolve 82 g of magnesium chloride (MgCl<sub>2</sub>.6H<sub>2</sub>O) and 100 g of ammonium chloride in 800 ml of water. Add 400 ml concentrated ammonium hydroxide ( $\Box 20 = 0.92$ ) (R). Mix.

# Mercury (II) (sulphate) in acid solution

Aqueous solution and mercury sulphate acid (II)  $HgSO_4$ . In a 200 ml graduated flask, introduce 10 g of yellow mercuric oxide, 120 ml of water and 75 g of concentrated sulphuric acid (R) (40 ml). After cooling, adjust the volume to 200 ml.





## Metaphenilene-diamine (hydrochloride)

Grey-mauve amorphous powder: C<sub>6</sub>H<sub>8</sub>N<sub>2</sub>.2 HCl.

#### Methyl orange in solution

Solution prepared with alcohol at 90% vol. containing 1 g of methyl orange in 100 ml of reagent.

#### □-naphthol in solution at 5% (m/m)

Dissolve 5 g of  $\square$ -naphthol in 40 ml of concentrated ammonium hydroxide solution (R) and adjust the volume to 100 ml with distilled water. Prepare extemporaneously.

# **Concentrated nitric (acid)**

 $\Box 20/4 = 1.39$ Concentrated nitric acid contains about 63% of nitric acid (HNO<sub>3</sub>).

## **Diluted nitric (acid)**

 $\Box 20/4 = 1.056$ 

Solution containing about 10 g of nitric acid ( $HNO_3$ ) in 100 g of reagent prepared with 15.8 g of nitric acid (11.35 ml) ( $\Box 20 = 1.39$ ) at 63 g for 100 g and 84.2 g of water.

# Nitromolybdic (reagent)

Dissolve 60 g of ammonium molybdate in 200 g of warm water. Filter if necessary. Slowly pour this solution in 720 g of diluted nitric acid while constantly shaking the latter. This diluted acid is obtained by mixing 370 g of concentrated nitric acid (R) with 350 g of water. Allow to stand 8 days. Adjust the volume to 1000 ml with distilled water. Filter or decanter.

This reagent, heated at 40°C, should not leave a deposit of precipitate.

Sensitivity: 25 µg of phosphorus for 5 ml.

#### Nitro-vanado-molybdic (reagent)

Prepare the following solutions A) Solution of ammonium molybdate Ammonium molybdate 100 g Concentrated ammonium hydroxide (R) 10 ml





Distilled water q.s.f. 1000 ml B) Solution of ammonium vanadate Ammonium metavanadate 2.35 g Distilled water 500 ml Slightly heat to dissolve. After complete dissolution, cool and gradually add while shaking the following mixture: Concentrated nitric acid (R) 7 ml Distilled water 13 ml Complete the volume to 1000 ml with distilled water. Mix.

To obtain the nitro-vanado-molybdic reagent, mix in a 500 ml graduated flask 67 ml of concentrated nitric acid (R), 100 ml of molybdic solution (A), 100 ml of nitro-vanadic solution (B) and adjust the volume to 500 ml. Mix.

# Eriochrome black T in solution

Solution containing 0.2 g eriochrome black T in 100 ml of triethanolamine.

# Oxalic (acid) in solution

Aqueous solution containing 5 g of crystallised acid oxalic  $C_2O_4H_2.2H_2O$  in 100 g of reagent.

#### Phenolphtalein in solution

Solution prepared with alcohol at 90% vol. containing 1 g de phenolphthalein in 100 ml of reagent.

#### Phosphoric (acid) solution at 85% (m/m)

Aqueous solution containing 85 g of orthophosphoric acid ( $H_3PO_4$ ),  $\Box 20 = 1.70$ , for 100 g.

#### Phosphoric (acid) solution at 25% (m/v)

Aqueous solution containing 25 g of phosphoric acid ( $H_3PO_4$ ),  $\Box 20 = 1.70$ , in 100 ml.

# Phosphoric (acid) solution at 50% (m/m)

Aqueous solution containing 50 g of orthophosphoric acid ( $H_3PO_4$ ),  $\Box 20 = 1.70$  in 100 g.





# Phosphate (solution at 0.05 g of phosphorus per litre)

## Potassium dihydrogenophosphate

Dissolve 4.392 g of monopotassium phosphate ( $KH_2PO4$ ) in a sufficient quantity of water to obtain 1 l. This solution contains 1 g of phosphorus per litre. Dilute to the twentieth to obtain the solution at 0.05 g per litre.

# Lead (nitrate) in solution at 1 g and 0.01 g of lead per litre

Dissolve 1.60 g of lead nitrate  $Pb(NO_3)_2$  in a sufficient quantity of water to obtain 1 l of solution at 1 g of lead per litre. This solution is diluted to the hundredth to obtain the solution at 0.01 g of lead per litre.

# Lead (neutral acetate) in solution at 10% (m/m)

Aqueous solution containing 10 g of lead acetate (II)  $Pb(C_4H_6O_4).3H_2O$  in 100 g of reagent.

# **Potassium sulfite**

# Potassium (acetate) in solution at 5% (m/m)

Aqueous solution containing 5 g of crystallised potassium  $KC_2H_3O$  acetate in 100 g of reagent  $CH_3CO_2K$ .

# Potassium (anhydrosulphite) $K_2S_2O_5$ (formerly potassium disulphite) free from selenium.

To search for selenium in sulphur dioxide, potassium anhydrosulphite free from selenium should be used. To check the absence of selenium, proceed with the following test trial:

Weigh 2.55 g of the potassium anhydrosulphite sample, dissolve with heat in 7 ml of distilled water and 2 ml of concentrated hydrochloric acid (R). Allow to cool and add 3 ml of formaldehyde solute (R). Allow to stand 10 minutes. Place the tube in a water bath at 100°C and add 50 mg of the sample of pulverised potassium anhydrosulphite.

The total sample is 2.60 g of potassium anhydrosulphite corresponding to 1.50 g of sulphur dioxide. A pink coloration should not develop.



# Potassium (anhydrosulphite) in solution at 2% (m/m)

Aqueous solution containing 2 g of crystallised potassium anhydrosulphite in 100 g of reagent.

# Potassium (cyanide) in solution at 1 mg of hydrocyanic acid per litre

Prepare an aqueous solution containing 2.44 g of KCN per litre, dilute to 1/100 to obtain the titrating solution of 1 mg of hydrocyanic acid per litre.

# Potassium (dichromate) at 1 g and 0.01 g of chrome per litre

Dissolve 2.8283 g of potassium dichromate  $K_2Cr_2O_7$  in a sufficient quantity of water to obtain 1 l of solution at 1 g of chrome per litre. This solution is diluted to the hundredth to obtain the solution at 0.01 g of chrome per litre.

# Potassium (dichromate) in solution at 10% (m/m)

Aqueous solution containing 10 g of potassium dichromate in 100 g of reagent.

# Potassium (hexacyanoferrate (II))/ potassium (ferrocyanide) in solution at 5% (m/m)

Aqueous solution containing 5 g of crystallised potassium  $K_4Fe(CN)_6.3H_2O$  hexacyanoferrate in 100 g of reagent.

# Potassium (hydroxide) at 40%

Dissolve 40 g of potassium hydroxide (KOH) in a sufficient quantity of water to obtain 100 ml.

#### Potassium (iodide) in iodine solution

Iodine-iodide solution - aqueous iodine solution  $(I_2)$  in potassium iodide (KI).

In a tared flask with a glass stopper, introduce 2 g of iodine, 4 g of potassium iodide and about 10 g of water. Allow the dissolution to operate, then complete with water, the weight of 100 g.

# Potassium (permanganate) in solution at 5% (m/m)

Aqueous solution containing 5 g of potassium permanganate (KMnO4) in 100 g of reagent.



# Potassium (permanganate) in solution at 3% (m/m)

Aqueous solution containing 3 g of potassium permanganate in 100 g of reagent.

# Potassium (permanganate) in solution at 2% (m/m)

Aqueous solution containing 2 g of potassium permanganate in 100 g of reagent.

# Potassium (permanganate) in solution at 1% (m/m)

Aqueous solution containing 1 g of potassium permanganate in 100 g of reagent.

# Potassium (permanganate) in solution at 0.2% (m/m)

Aqueous solution containing 0.2 g of potassium permanganate in 100 g of reagent.

# Potassium (permanganate) in saturated solution

Saturated aqueous solution containing about 6 g of potassium permanganate in 100 g of reagent.

# Potassium (permanganate) at 5 per 1000 (m/m)

Aqueous solution containing 5 g of potassium permanganate in 1000 g of reagent.

# Potassium (permanganate) phosphoric solution

Dissolve 3 g of potassium permanganate (R) in a mixture of 15 ml of phosphoric acid (R) and 70 ml of water; complete to 100 ml with water.

# Potassium (thiocyanate) in solution at 5% (m/m)

Aqueous solution containing 5 g of potassium thiocyanate KSCN in 100 g of reagent.

# Pyridine-pyrazolone (reagent)

<u>Bis(1-phenyl-3-methyl-5-pyrazolone</u>). (F. 320°C) - Dissolve 17.4 g of 1-phenyl-3-methyl-5-pyrazolone in 100 ml of alcohol at 95% vol., add 25 g of freshly distilled phenylhydrazine, bring to the boil under reflux for 4 hours. The mixture is filtered hot and the precipitate washed several times with alcohol at 95% vol.

The boiling under reflux could be prolonged beyond 4 hours if the occurrence of yellow crystals is not very abundant after this time.

*Preparation of reagent* <u>pyridine-pyrazolone</u>. – In a 100 ml graduated flask, introduce 0.150 g of 1-phenyl-3-methyl-5-pyrazolone and dissolve in 50 ml of alcohol at 95% vol.





distilled on potassium hydroxide; complete to 100 ml with distilled water.

On the other hand, weigh 20 mg of bis(1-phenyl-3-methyl-5-pyrazolone), and dissolve by prolonged shaking in 20 ml of pyridine.

Mix the two resulting solutions by pouring them in a yellow glass flask wrapped in black paper. Store in the refrigerator.

# Quinine (sulphate) in solution at 0.1 mg per litre of sulphuric acid 0.05 M

Dissolve 0.100 g of quinine sulphate in a sufficient quantity of sulphuric acid 0.05 M to obtain 1 l. Dilute three times 1/10 this solution with a sulphuric acid solution 0.05 M to obtain the solution at 0.1 mg of quinine sulphate per litre.

# Rosaniline (hydrochloride) in solution discoloured by sulphurous acid

In a mortar, pulverise 30 mg of pure rosaniline hydrochloride, then add 30 ml of alcohol at 95% vol. The dissolution is rapid and complete. On the other hand, in a 250 ml graduated flask, dissolve 8 g of potassium anhydrosulphite in about 150 ml of distilled water. Add the alcoholic solution of hydrochloride rosaniline, then 55 ml of hydrochloric acid solution 3 M and bring to the graduation line with water. The reagent must be completely discoloured in less than an hour. It is stable for several months.

# Methyl red in solution

Alcohol solution at 90% vol. containing 0.10 g of methyl red in 50 ml of reagent.

Methyl red mixed indicator:

Solution in alcohol at 90% vol. containing 0.10 g of red methyl and 0.05 g of blue methylene in 10 ml of reagent.

#### Phenol red in solution

Heat 0.05 g of phenol red with 2.85 ml of sodium hydroxide solution 0.05 M and 5 ml of alcohol at 90% vol. To the solution obtained, add a sufficient quantity of alcohol at 20% vol. to obtain 250 ml.

# Selenium (dioxide) in solution at 100 mg of selenium per litre

Grind 2 g of pure selenium dioxide  $(SeO_2)$  and allow to stand for 24 hours in a desiccator for sulphuric acid. Weigh 1.4553 g of this dry dioxide and dissolve in a sufficient quantity of water to obtain 1 l of solution.

This solution contains 1 g of selenium per litre. Dilute 1/10 with distilled water to





obtain the solution at 100 mg of selenium per litre.

Sodium (acetate) -  $NaC_2H_3O_2.3H_2O = 136.1$ .

The salt used as a reagent must be neutral.

## Sodium (acetate) in solution at 10% (m/m)

Aqueous solution containing 10 g of sodium acetate  $\rm NaC_2H_3O2.3H_2O$  in 100 g of reagent.

# Sodium (borate) in saturated solution

Saturated aqueous solution containing about 4 g of crystallised sodium borate for 100 g of solution. Sodium tertraborate  $Na_2B_4O_7$ .

# Sodium (neutral carbonate) in solution at 25% (m/m)

Aqueous solution containing 25 g of crystallised disodic carbonate at 10  $H_2O$  in 100 g of reagent  $Na_2CO_3.10H_2O$ .

# Sodium (diethyldithiocarbamate) in solution at 1% (m/v)

Dissolve 1 g of sodium diethyldithiocarbamate in a sufficient quantity of alcohol at 40% vol. to obtain 100 ml of solution  $(C_2H_5)_2NCS_2Na.3H_2O$ .

#### Sodium (ethylenediaminetetraacetate) in solution 0.01 M

Sodium ethylenediaminetetraacetate 4.0 g

Magnesium chloride, MgCI<sub>2</sub>.6H<sub>2</sub>O 0.1 g

Water q.s.f. 1000 ml

The titre of this solution must be checked and adjusted after titration by a calcium chloride solution 0.01 M obtained by dissolving 1 g of pure calcium carbonate in 25 g of concentrated hydrochloric acid (R) with 20 ml of water and by adjusting the volume to 1000 ml with distilled water.

#### Sodium (fluoride) in solution at 4% (m/m)

Aqueous solution containing 4 g of sodium fluoride (NaF) in 100 g of reagent. This solution is nearly saturated.

# Sodium (hydroxide) in concentrated solution (caustic soda)

Aqueous solution with density 1.330 containing 30 g of sodium hydroxide (NaoH) in





100 g of solution.

# Sodium (hydroxide) in diluted solution at 10% (m/m)

Aqueous solution containing 10 g of sodium hydroxide (NaoH) in 100 g of reagent.

# Sodium (phosphate) in solution at 10% (m/m)

Aqueous solution containing 10 g of crystallised disodic phosphate in 100 g of reagent.

# Sodium (pyrophosphate) at 1% (m/m)

Aqueous solution containing 1 g of crystallised tetrasodic pyrophosphate,  $Na_4P_2O_7.10H_2O$ , in 100 g of reagent.

# Sodium (thiosulphate) in solution at 25% (m/v)

Aqueous solution containing 25 g of sodium thiosulphate ( $Na_2S_2O3$ ) for 100 ml.

# Hydrogen sulphide (acid) in saturated solution

Aqueous solution of saturated hydrogen sulphide acid. It contains about 3.8 g of  $H_2S$  per litre. It is alterable in air.

# Hydrogen sulphide (acid) solution at 1 g of sulphur per litre and at 0.01 g per litre

Dissolve 7.5 g of  $Na_2S.9H_2O$  in a sufficient quantity of water to obtain 1 l. This solution is diluted to a hundredth to obtain the solution at 0.01 g per litre (solutions rapidly oxidised by air).

# Sulforesorcinic (reagent)

Dissolve 2 g of pure resorcinol in 100 ml of water and add 0.5 ml of concentrated sulphuric acid (R).

#### Sulphuric (acid) concentrated at 95% minimum

 $\Box 20/4 = 1.83$  to 1.84. (H<sub>2</sub>SO<sub>4</sub>)

# Sulphuric (acid) at 97% (m/m)

This absolutely colourless acid should not be able to be differentiated after heating at 120°C from an unheated control. It should be stored in flasks with emery stoppers. Its titre should be  $97 \pm 1\%$ .



# Sulphuric (acid) at 25% (m/m)

□20/4 = 1.1808 approximately.

Aqueous solution of sulphuric acid containing about 25 g of acid  $\rm H_2SO_4$  in 100 g of reagent.

## Sulphuric (acid) diluted at 10% (m/m)

 $\Box 20/4 = 1.0682$  approximately.

Aqueous solution of sulphuric acid containing about 10 g of acid  $\rm H_2SO_4$  in 100 g of reagent.

# Sulphuric (acid) diluted at 5% (m/m)

Aqueous solution of sulphuric acid containing about 5 g of acid  $H_2SO_4$  in 100 g of reagent.

**Sulphuric (acid) free from nitrogen** must satisfy the following test trial: nitrate. To 5 ml of water, carefully add 45 ml of sulphuric acid free from nitrogen, allow to cool to 40°C and add 8 mg of diphenylbenzidine. The solution is barely pink or pale blue.

#### Acetate buffer, purified, for search for zinc

Dissolve 136 g of sodium acetate in 440 ml of water, add 58 ml of concentrated acetic acid. Purify this solution by shaking with a dithizone solution at 125 mg per litre of chloroform.

#### Ammoniac buffer

Concentrated ammonium hydroxide 350 ml Ammonium chloride 54 g Distilled water q.s.f. 1000 ml

# Buffer pH 7.5

Monopotassium phosphate 94 g Sodium hydroxide in molar solution 565 ml Distilled water q.s.f. 1 000 ml

#### **Pure tannin**

Tannin, called ether tannin or officinal tannin is extracted from the Aleppo gall.





It is in the form of a light mass, yellowish white, very soluble in water and alcohol at 90% vol. It is insoluble in ethylic ether. It must comply with the following test trials:

1. The aqueous tannin solution at 10% must be clear and have a very light yellow colour like white wine. The tannin solution at 10% in alcohol at 90% vol. must also be clear with hardly any colour.

A solution at 1 g of tannin in 5 g of water with its volume of alcohol at 90% vol. and half of its volume of ethylic ether, should give a clear solution (aqueous extract or alcoholic extract).

2. The officinal tannin must be combustible without leaving residue more than 0.05% (set mineral matters).

3. Desiccated at 100°C, the officinal tannin must not loose more than 12% in its weight (excess water). The anhydrous tannin content is calculated from this test trial. Its knowledge is necessary for the preparation of the solution at 4 per 1000.

# Tannin in solution at 2% (m/m)

Aqueous solution containing 2 g of tannin in 100 g of reagent. It must be prepared extemporaneously.

# Tannin in solution at 4% (m/v)

Dissolve a quantity of pure tannin containing 1 g of anhydrous tannin in a sufficient quantity of water to obtain 250 ml.

# Tannin in solution at 10% (m/m)

Aqueous solution containing 10 g of tannin in 100 g of reagent.

# Thioacetamide (reagent)

F 🛛 113°С

To 0.2 ml of aqueous thioacetamide solution at 40 g/l, add 1 ml of a mixture of 5 ml of water, 15 ml of sodium hydroxide 1 M and 20 ml of glycerol at 85% (m/m). Heat in a water bath at 100°C for 20 seconds. Prepare extemporaneously.

# Uranyl (nitrate) in solution at 4% (m/m)

Solution containing 4 g of uranyl nitrate  $UO_2(NO_3)_2.6H_2O$  in 100 g of reagent.

# Uranyl and magnesium (acetates) in hydro-alcoholic and acetic solution

Dissolve 32 g of crystallised uranyl acetate and 100 g of magnesium acetate in 300 ml





of water, 20 ml of acetic acid and 500 ml of alcohol at 95% vol. by heating in a water bath at 100°C and by shaking; adjust the volume to 1 litre with water (distilled) and allow to stand 48 hours; decant or filter.

This reagent must be stored away from light. 2.5 ml of reagent must be used per milligramme of sodium to be precipitated and per millilitre of solution to be treated.

Phosphates, arseniates and fluorides must be absent from this solution. Heavy metals, iron (II) and alkaline-earth are not bothersome.

## Bromocresol green in solution

Alcohol solution at 95% vol. containing 0.04 g of bromocresol green (3',3'',5',5'') tetrabromo-*m*-cresolsulfonephtaleine) for 100 ml pf reagent.

# Bromocresol green and methyl red in solution (mixed indicator)

Dissolve Bromocresol green 0.04 g Methyl red 0.06 g in alcohol at 95% vol. 100 ml Add 2.5 ml of sodium hydroxide solution 0.1 M. This indicator from red (pH 4.6) turns blue-green with pH 4.9. It is violet with pH 4.75.

# Zinc in solution at 1 mg per litre

Dissolve 1 g of pure zinc in the minimum concentrated hydrochloric acid (R) by gently heating. Dilute the solution to 500 ml and neutralise by adding sodium carbonate until a light precipitate appears which disappears when a few drops of hydrochloric acid are added.

Dilute successively three times 1/10 when using.

<sup>&</sup>lt;sup>[1]</sup> this list does not contain the titrated acid solutions, sodium hydroxide, iodine, silver nitrate, etc.

<sup>&</sup>lt;sup>[2]</sup> The composition of reagents "(R As)" is indicated for determining arsenic.