

European Pharmacopoeia, Fourth Edition (2002)

4 Reagents

Ammonia, concentrated. 1004700.
See *concentrated ammonia solution* (0877).

Ammonia. 1004701. X
Contains not less than 170 g/l and not more than 180 g/l of NH_3 (M_r 17.03).

Dilute 67 g of *concentrated ammonia* R to 100 ml with *water* R.
 $d_{20}/20$: 0.931 to 0.934.

When used in the limit test for iron, *ammonia* R complies with the following additional requirement. Evaporate 5 ml of ammonia to dryness on a water-bath, add 10 ml of *water* R, 2 ml of a 200 g/l solution of *citric acid* R and 0.1 ml of *thioglycollic acid* R. Make alkaline by adding *ammonia* R and dilute to 20 ml with *water* R. No pink colour develops. Store protected from atmospheric carbon dioxide, at a temperature below 20 °C.

Ammonia, dilute R1. 1004702.
Contains not less than 100 g/l and not more than 104 g/l of NH_3 (M_r 17.03).
Dilute 41 g of *concentrated ammonia* R to 100 ml with *water* R.

Ammonia, dilute R2. 1004703.
Contains not less than 33 g/l and not more than 35 g/l of NH_3 (M_r 17.03).
Dilute 14 g of *concentrated ammonia* R to 100 ml with *water* R.

Ammonia, dilute R3. 1004704.
Contains not less than 1.6 g/l and not more than 1.8 g/l of NH_3 (M_r 17.03).
Dilute 0.7 g of *concentrated ammonia* R to 100 ml with *water* R.

Lead-free ammonia. 1004705.
Complies with the requirements prescribed for *dilute ammonia R1* and with the following additional test: to 20 ml of lead-free ammonia, add 1 ml of *lead-free potassium cyanide solution* R, dilute to 50 ml with *water* R and add 0.10 ml of *sodium sulphide solution* R. The solution is not more intensely coloured than a reference solution prepared without sodium sulphide.

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Bromophenol blue. $\text{C}_{19}\text{H}_{10}\text{Br}_4\text{O}_5\text{S}$. (M_r 670). 1012800. [115-39-9]. 3',3'',5',5''-Tetrabromophenolsulfonphthalein 4,4-(β H-2, 1-Benzoxathiol-3-ylidene)bis(2,6-dibromophenol) S,S-dioxide.

A light orange-yellow powder, very slightly soluble in water slightly soluble in alcohol, freely soluble in solutions of all hydroxides.

Bromophenol blue solution. 1012801.
Dissolve 0.1 g of bromophenol blue R in 1.5 ml of 0.1 N sodium hydroxide and 20 ml of alcohol R and dilute with 100 ml with *water* R.

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Hydrochloric acid. 1043500. [7647-01-0].
See *Concentrated hydrochloric acid* (0002).

Hydrochloric acid R1. 1043501.
Contains 250 g/l of HCl.
Dilute 70 g of *hydrochloric acid* R to 100 ml with *water* R.
Hydrochloric acid, brominated. 1043507.
To 1 ml of *bromine solution* R add 100 ml of *hydrochloric acid* R.

Hydrochloric acid, dilute. 1043503.
Contains 73 g/l of HCl.
Dilute 20 g of *hydrochloric acid* R to 100 ml with *water* R.
Hydrochloric acid, dilute R1. 1043504.
Contains 0.37 g/l of HCl.]
Dilute 1.0 ml of *dilute hydrochloric acid* R to 200.0 ml with *water* R.
Hydrochloric acid, dilute R2. 1043505.
Dilute 30 ml of 1 M *hydrochloric acid* to 1000 ml with *water* R; adjust to pH 1.6 +/- 0.1.

Hydrochloric acid, ethanolic. 1 043506.
Dilute 5.0 ml of 1 M *hydrochloric acid* to 500.0 ml with *alcohol* R

Hydrochloric acid, lead-free. 1043508.
Complies with the requirements prescribed for S *hydrochloric acid* R and with the following additional r test

Lead. Not more than 20 ppm of Pb determined by atomic (emission spectrometry (*Method J*); 2.2.22).

Test solution. In a quartz crucible evaporate 200 g of n the acid to be examined almost to dryness. Take up the residue in 5 ml of nitric acid prepared by sub-boiling distillation of *nitric acid* R and evaporate to dryness. Take up the residue in 5 ml of nitric acid prepared by sub-boiling distillation of *nitric acid* R.

Reference solutions. Prepare the reference solutions using *lead standard solution* (0.1 ppm Ph) R diluted with nitric acid prepared by sub-boiling distillation of *nitric acid* R. Measure the emission intensity at 220.35 nm.

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Iodine. 1045800. [7553-56-2].
See *Iodine* (0031).

Iodine solution R1. 1045801.
To 10.0 ml of 0.05 M *iodine* add 0.6 g of *potassium iodide* R and dilute to 100.0 ml with *water* R. Prepare immediately before use.

Iodine solution R2. 1045802.

To 10.0 ml of 0.05 M iodine add 0.6 g of potassium iodide R and dilute to 1000.0 ml with water R. Prepare immediately before use.

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Sodium hydroxide. 1081400. [1310-73-2].

See *Sodium hydroxide* (0677).

Sodium hydroxide solution. 1081401.

Dissolve 20.0 g of sodium hydroxide R in water R and dilute to 100.0 ml with the same solvent. Verify the concentration by titration with 1 M hydrochloric acid, using methyl orange solution R as indicator, and adjust if necessary to 200 g/l.

Sodium hydroxide solution, dilute. 1081402.

Dissolve 8.5 g of sodium hydroxide R in water R and dilute to 100 ml with the same solvent.

Sodium hydroxide solution, methanolic. 1081403.

Dissolve 40 mg of sodium hydroxide R in 50 ml of water R. Cool and add 50 ml of methanol R.

Sodium hydroxide solution, methanolic R1. 1081405.

Dissolve 200 mg of sodium hydroxide R in 50 ml of water R. Cool and add 50 ml of methanol R.

Sodium hydroxide solution, strong. 1081404.

Dissolve 42 g of sodium hydroxide R in water R and dilute to 100 ml with the same solvent.

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Thioacetamide. C₂H₅NS. (M, 75.1). 1089600. [62-55-5].

A crystalline powder or colourless crystals, freely soluble in water and in alcohol.

mp: about 113 °C.

Thioacetamide reagent. 1089601.

To 0.2 ml of thioacetamide solution R add 1 ml of a mixture of 5 ml of water R, 15 ml of 1 M sodium hydroxide and 20 ml of glycerol (85 per cent) R. Heat in a water-bath for 20 s. Prepare immediately before use.

Thioacetamide solution. 1089602.

A 40 g/l solution.

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Starch, soluble. 1085100. [9005-84-9].

A white powder.

Prepare a 20 g/l solution in hot water R. The solution is at most slightly opalescent and remains fluid on cooling.

Starch iodate paper. 1085101.

Immerse strips of filter paper in 100 ml of iodide-free starch solution R containing 0.1 g of potassium iodate R. Drain and allow to dry protected from light.

Starch iodide paper. 1085106.

Immerse strips of filter paper in 100 ml of starch

solution R containing 0.5 g of potassium iodide R. Drain and allow to dry protected from light.

Test for sensitivity. Mix 0.05 ml of 0.1 M sodium nitrite with 4 ml of hydrochloric acid R and dilute to 100 ml with water R. Apply one drop of the solution to starch iodide paper; a blue spot appears.

Starch solution. 1085103.

Triturate 1.0 g of soluble starch R with 5 ml of water R and whilst stirring pour the mixture into 100 ml of boiling water R containing 10 mg of mercuric iodide R.

Carry out the test for sensitivity each time the reagent is used.

Test for sensitivity. To a mixture of 1 ml of the starch solution and 20 ml of water R, add about 50 mg of potassium iodide R and 0.05 ml of iodine solution R1. Starch solution, iodide-free. 1085104.

Prepare the solution as prescribed for starch solution R omitting the mercuric iodide. Prepare immediately before use.

Starch solution R1. 1085105.

Mix 1 g of soluble starch R and a small amount of cold water R. Add this mixture, while stirring, to 200 ml of boiling water R. Add 250 mg of salicylic acid R and boil for 3 min. Immediately remove from the heat and cool.

If long storage is required, the solution shall be 4 °C to 10 °C. A fresh starch solution shall be prepared when the end-point of the titration from blue to colourless fails to be sharp. If stored under refrigeration, the solution is stable for about 2 to 3 weeks.

Test for sensitivity. A mixture of 2 ml of starch solution R, 20 ml of water R, about 50 mg of potassium iodide R and 0.05 ml of iodine solution R1 is blue.

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Sulphuric acid. H₂SO₄. (Mr 98.1). 1086800. [7664-93-9].

Contains not less than 95.0 per cent m/m and not more than 97.0 per cent m/m of H₂SO₄.

A colourless, caustic liquid with an oily consistency, highly hygroscopic, miscible with water and with alcohol producing intense heat.

*d*₂₀²⁰: 1.834 to 1.837.

A 10 g/l solution is strongly acid and gives the reactions of sulphates (2.3.1).

Appearance. It is clear (2.2.1) and colourless (Method II, 2.2.2).

Oxidisable substances. Pour 20 g cautiously, with cooling, into 40 ml of water R. Add 0.5 ml of 0.002 M potassium permanganate. The violet colour persists for at least 5 min.

Chlorides. Pour 10 g, carefully and while cooling, into 10 ml of *water R* and after cooling dilute to 20 ml with the same solvent. Add 0.5 ml of *silver nitrate solution R2*. Allow to stand for 2 min protected from bright light. The solution is not more opalescent than a standard prepared at the same time using a mixture of 1 ml of *chloride standard solution (5 ppm Cl) R*, 19 ml of *water R* and 0.5 ml of *silver nitrate solution R2* (0.5 ppm).

Nitrates. Pour 50 g or 27.2 ml, carefully and while cooling, into 15 ml of *water R*. Add 0.2 ml of a freshly prepared 50 g/l solution of *brucine R* in *glacial acetic acid R*. After 5 min any colour is less intense than that of a reference mixture prepared in the same manner and containing 12.5 ml of *water R*, 50 g of *nitrogen-free sulphuric acid R*, 2.5 ml of nitrate standard solution (10 ppm NO₃) R and 0.2 ml of a 50 g/l solution of *brucine R* in *glacial acetic acid R* (0.5 ppm).

Ammonium. Pour 2.5 g, carefully and while cooling, into *water R* and dilute to 20 ml with the same solvent. Cool, and add dropwise 10 ml of a 200 g/l solution of *sodium hydroxide R*, followed by 1 ml of *alkaline potassium tetraiodomercurate solution R*. The colour of the solution is less intense than that of a mixture of 5 ml of ammonium standard solution (1 ppm NH₄) R, 15 ml of *water R*, 10 ml of a 200 g/l solution of *sodium hydroxide R* and 1 ml of *alkaline potassium tetraiodomercurate solution R* (2 ppm).

Arsenic (2.4.2). To 50 g add 3 ml of *nitric acid R* and evaporate carefully until the volume is reduced to about 10 ml. Cool, add to the residue 20 ml of *water R* and concentrate to 5 ml. The solution complies with limit test A for arsenic (0.02 ppm). Prepare the standard using 1.0 ml of *arsenic standard solution (1 ppm As) R*.