

on p. 468, is more convenient, and is more generally employed in volumetric work.

B.P.—The *B.P.* solution is on the lines indicated above. It is not employed volumetrically in the official volume.

U.S.P.—The *U.S.P.* alkaline Cupric Tartrate Solution is employed volumetrically. It consists of two solutions:—1. The Copper Solution, which is prepared by dissolving 34·67 (more correctly 34·6663) grammes of carefully selected un-effloresced pure Cupric Sulphate, free from adhering moisture, in a sufficiency of Distilled Water, and diluting to measure 500 c.c. at 25° C. (77° F.). The solution is directed to be kept in small, well-stoppered bottles. 2. The Alkaline Tartrate Solution, which is prepared by dissolving 173 grammes of crystallised Potassium Sodium Tartrate together with 75 grammes of Potassium Hydroxide in a sufficiency of Water to measure exactly 500 c.c. at 25° C. (77° F.). This solution is directed to be kept in small bottles fitted with rubber stoppers. When required for use the solutions are mixed in equal volumes.

P.G.—A weighed quantity of 3·5 grammes of Copper Sulphate is dissolved in 30 c.c. of Water, and the solution mixed with a solution of 17·5 grammes of Sodium Potassium Tartrate in 30 c.c. of Water, the latter solution having been previously mixed with 40 grammes of Sodium Hydroxide Solution (15 p.c.).

TENTH-NORMAL VOLUMETRIC AMMONIUM RHODANATE SOLUTION.

This solution is employed by the *P.G.* instead of Tenth-normal Volumetric Potassium Sulphocyanate Solution for the determination of the excess of Volumetric Silver Solution in the indirect Silver titration. Almost the only instance in which it is used in this volume is in the determination of Volatile Oil of Mustard.

B.P. }
U.S.P. } Not included. See Table.

P.G.—A solution containing 7·618 grammes of Ammonium Rhodanate in 1 litre. No method of preparation, standardisation, or preservation is indicated.

INDICATORS OF NEUTRALITY.

The following list shows at a glance the Solutions employed by the three Pharmacopœias as indicators in Volumetric Analysis.

<i>B.P.</i>	Cochineal Tincture.		Phenolphthalein T.S.
	Litmus.		Rosolic Acid T.S.
	Methyl Orange.		Starch T.S.
	Phenolphthalein.		Turmeric Tincture.
	Starch Mucilage.	<i>P.G.</i>	Hæmatoxylin.
	Turmeric Tincture.		Iodeosin Solution.
<i>U.S.P.</i>	Brazil Wood T.S.		Litmus paper.
	Cochineal T.S.		Phenolphthalein Solution.
	Hæmatoxylin T.S.		Rosolic Acid Solution.
	Iodeosin T.S.		Starch Solution.
	Litmus paper and T.S.		Turmeric paper.
	Methyl Orange T.S.		Turmeric Tincture.

BRAZIL WOOD SOLUTION.

Brazil Wood solution is coloured yellow by acids and crimson-red by alkalis. It is used chiefly as an indicator of neutrality in the titration of alkaloids.

B.P. }
P.G. } Not included. See Table.

U.S.P.—A solution obtained by boiling 50 grammes of finely cut Brazil Wood for 30 minutes with 100 c.c. of Water, the evaporated Water being replaced from time to time. The mixture is allowed to cool, strained, 100 c.c. of the strained liquid are mixed with 25 c.c. of Alcohol (94·9 p.c.), the solution filtered. It is required that the solution be excluded from contact with ammoniacal vapours.

COCHINEAL SOLUTION.

Cochineal solution assumes a yellow or yellowish-red coloration with acids, and a violet coloration with alkalis. As an indicator it is used chiefly in the titration of alkaloids.

It may be employed as an indicator of neutrality in the titration of solutions containing Ammonia, and affords a useful means of judging the neutrality of Ammonium Acetate and Ammonium Citrate Solutions, *see* Liquor Ammonii Acetatis and Liquor Ammonii Citratis. It may also be used for the titration of inorganic acids.

B.P.—The solution employed by the *B.P.* is the official Tincture of Cochineal described on p. 421.

U.S.P.—A filtered solution prepared by macerating 1 gramme of unbroken Cochineal for 4 days with a mixture of 20 c.c. of Alcohol (94.9 p.c.) and 60 c.c. of Water. The *U.S.P.* states that it is useless for titrating organic acids.

P.G.—Not included. *See* Table.

HÆMATOXYLIN SOLUTION.

A solution containing 0.2 p.c. w/v of Hæmatoxylin prepared by dissolving 0.2 of a gramme of Hæmatoxylin in sufficient Alcohol (90 p.c.) to produce 100 c.c. The solution is used as an indicator of neutrality in the titration of alkaloids. It assumes a yellow or orange colour in acid solutions and a violet to purple colour in alkaline solutions. It is advisable in working with this indicator to experiment side by side with an equal quantity of a neutral liquid containing exactly the same amount of the solution as has been added to the liquid under titration, as very different tints may be assumed according to the volume of the solution used.

B.P.—Not included. *See* Table.

U.S.P.—A 0.2 p.c. w/v solution of Hæmatoxylin prepared by dissolving 0.2 of a gramme of crystalline Hæmatoxylin in 100 c.c. of Alcohol (94.9 p.c.). The *U.S.P.* specifies about 5 drops are to be used for each titration. The titration is to be considered complete when the change in colour remains permanent upon the addition of 1 drop of the volumetric solution after stirring the liquid.

P.G.—A solution prepared by dissolving a crystal of Hæmatoxylin in 1 c.c. of Alcohol (90 p.c.). The instructions are to use a solution of this composition as an indicator for each titration.

IODEOSIN.

A solution containing 0.1 p.c. w/v of Iodeosin [(Tetraiodfluorescein) $C_{20}H_4I_4O_3$, eq. 829.20] prepared by dissolving 0.1 of a gramme of Tetraiodfluorescein in 100 c.c. of Alcohol (90 p.c.). The solution is colourless in the presence of acids, but assumes a pink coloration in the presence of alkalis. It is an extremely sensitive indicator, and is suitable for the titration of minute quantities of alkaloids. The Alcohol used in its preparation should be absolutely neutral.

B.P.—Not included.

U.S.P.—A 0.1 p.c. w/v solution of Iodeosin in 100 c.c. of Alcohol (94.9 p.c.) prepared by dissolving 0.1 of a gramme of Iodeosin in 100 c.c. of Alcohol (94.9 p.c.). The *U.S.P.* recommends the volume of the solution titrated should be about 100 c.c. 20 c.c. of Ether should be added and 5 drops of the Iodeosin T.S., the solution being well shaken after each addition of Volumetric Alkali solution. The titration is continued until a faint pink colour remains after vigorous shaking.

P.G.—A solution containing 0.2 p.c. w/w of Iodeosin prepared by dissolving 1 part by weight of Iodeosin in 500 parts by weight of Alcohol (90 p.c.). The directions given in the *P.G.* for the verification of the indicator are as follows: To 100 c.c. of Water contained in a flask of white glass is added sufficient Ether to form a layer 1 cm. in depth, 1 drop of Hundredth-normal Volumetric Hydrochloric Acid Solution and 5 drops of Iodeosin Solution are added, after vigorous shaking the lower aqueous layer should remain uncoloured, but if 2 drops of Hundredth-normal Volumetric Potassium Hydroxide Solution be added, after vigorous shaking the lower aqueous layer should be coloured pale rose.

LITMUS.

Litmus is the blue colouring matter prepared from various species of *Roccella*. It occurs commercially in small, dark blue, rectangular cubes, possessing a characteristic floral odour. It is employed in the form of a solution, or as **Litmus paper**. It is unaffected by alkalis, but changes to a red colour on the addition of acid, and this red colour is restored to blue when the solution is again rendered alkaline. It is employed for the titration of acids and alkalis, acid salts, and occasionally in the titration of alkaloids and alkaloidal acid salts, *e.g.*, Quinine Bihydrochloride, but its use in the latter instance is not altogether satisfactory. It yields no satisfactory end reaction with Boric Acid. The titration of Carbonates with this indicator is a tedious process, owing to the necessity of boiling off Carbonic Anhydride before a definite end reaction can be obtained. **Solution of red Litmus** is prepared by the cautious addition of a very dilute Hydrochloric Acid Solution, only just sufficient to ensure a faint red colour being added. Red Litmus paper is prepared by impregnating white bibulous paper with the above solution, and drying.

B.P.—The Litmus of the *B.P.* is obtained from various species of *Roccella*.

U.S.P.—The *U.S.P.* does not mention the source of the Litmus.

P.G.—Not described.

LITMUS SOLUTION.

A solution of Litmus may be prepared by repeatedly exhausting the cubes with Water until all soluble matters have been extracted, evaporating the mixed extracts to a small bulk, adding sufficient Acetic Acid to decompose Carbonates, evaporating to a thick extract and adding a large quantity of Alcohol (90 p.c.) which precipitates the blue colour, the latter is washed with hot Alcohol (90 p.c.) and dissolved in Water. **Blue or red Litmus paper** may be prepared by dipping strips of calendered or unsized paper in either the blue or red solution, and drying.

B.P.—A filtered solution of Litmus prepared by exhausting 10 grammes of Litmus for 1 hour with 3 successive portions of Alcohol (90 p.c.), using first 40, 30, and finally 30 c.c. The Litmus (from which all matter soluble in Alcohol of this strength has been removed) is digested in 100 c.c. of Distilled Water.

U.S.P.—A filtered solution of Litmus prepared by exhausting powdered Litmus for 1 hour with 3 separate quantities consisting of about 4 times its weight of boiling Alcohol (94.9 p.c.) in order to remove Erythrolitmin; the superfluous Alcohol is allowed to drain off and the residue digested with an equal weight of cold Water and filtered. This solution after being acidulated may be used to prepare **red Litmus paper**. The residue is extracted with about 5 times its weight of boiling Water, and after it has been thoroughly cooled, filtered, the filtrate is preserved in wide-mouthed bottles closed with a plug of Cotton-Wool. The *U.S.P.* states that an addition of 1 drop of Tenth-normal Volumetric Acid or Alkali to 50 c.c. of Water containing 5 drops of the indicator should produce a distinct change in colour. The **blue and red Litmus paper** of the *U.S.P.* is prepared by impregnating strips of white unsized paper, free from wood pulp, with either blue or red Litmus Solution as described above, and drying. Litmus papers should be kept in well-stoppered bottles.

P.G.—A solution prepared by digesting 10 parts by weight of Litmus for 24 hours at a temperature of 15° to 20° C. (59° to 68° F.) with 100 parts by weight of Water, the mixture being repeatedly shaken. The mixture after being allowed to deposit is filtered. It is used for the preparation of **blue Litmus paper**, the following method of procedure being adopted:—Sufficient diluted Sulphuric Acid is added to the above solution, brought to a boiling temperature to cause it to assume only a violet-blue coloration when diluted with about 100 parts by weight of Water. Strips of the best unglazed paper are coloured with this 10 p.c. w/w Litmus Solution (neutralised as described above and dried) and are then kept from the light. It is required that blue Litmus paper shall be immediately coloured red by 1 drop of a mixture of 1 c.c. of Tenth-normal Volumetric Hydrochloric Acid Solution and 100 c.c. of Water. In the preparation of **red Litmus paper** the above-mentioned Litmus Solution is mixed with sufficient diluted Sulphuric Acid until a test portion diluted with about 100 parts by volume of Water is of a pale red colour, and strips of the best unglazed paper are coloured

with this 10 p.c. w/w acid Litmus Solution, dried and protected from the light. It is required that red Litmus paper should be immediately coloured blue by the addition of a single drop of a mixture of 1 c.c. of Tenth-normal Volumetric Potassium Hydroxide Solution and 100 c.c. of Water. Both blue and red Litmus paper are directed to be preserved in well-closed vessels protected from the light.

METHYL ORANGE. Poirrier's Orange III.

Methyl Orange, Helianthin $\text{NaC}_{11}\text{H}_{14}\text{N}_2\text{SO}_3$. Poirrier's Tropæolin D.

An orange-yellow powder readily soluble in Water, sparingly soluble in Alcohol (90 p.c.). Commercially it is the Ammonium or Sodium salt of Dimethyl-amido-azobenzenesulphonic Acid or Para-sulphobenzene-azodimethylaniline, a body produced by the action of Dimethylaniline on Diazobenzene-sulphonic Acid. The *B.P.* and the *U.S.P.* employ the Sodium salt. The addition of Hydrochloric Acid to a hot concentrated aqueous solution precipitates the free Sulphonic Acid, basic Lead Acetate Solution throws down an orange-yellow precipitate. It should yield no precipitate with Barium Chloride or Calcium Chloride Solution, nor on the addition of an alkali Hydroxide Solution.

METHYL ORANGE SOLUTION.

A solution containing 0.1 p.c. w/v of Methyl Orange. It may be prepared by dissolving 0.1 of a gramme of Methyl Orange in 50 c.c. of Distilled Water, adding 10 c.c. of Alcohol (90 p.c.), mixing thoroughly and diluting with sufficient Distilled Water to measure 100 c.c. and filtering. It is used as an indicator of neutrality for the titration of acids and alkalis, and is of special service in the titration of Carbonates, as the latter do not effect the end reaction, and the necessity for boiling off the Carbonic Anhydride is obviated. It is not a satisfactory indicator for use in titration of organic acids, such as Oxalic, Acetic, Citric or Tartaric, as the end reaction is indefinite. It is employed in the titration of alkaloids and alkaloidal salts. Care should be taken that only a minimum quantity of the solution should be used in the titration, the end reaction being more delicate the smaller the quantity of solution employed compatible with the observance of a true end reaction. It affords a yellow coloration with alkalis, and a pink or reddish coloration with acids.

B.P.—A solution containing 0.2 p.c. w/v of Methyl Orange in a mixture of Alcohol (90 p.c.) and Distilled Water, prepared by dissolving 0.2 of a gramme of Methyl Orange in Distilled Water, adding 25 c.c. of Alcohol (90 p.c.) and sufficient Distilled Water to produce 100 c.c.

U.S.P.—A solution containing 0.1 p.c. w/v of Methyl Orange prepared by dissolving 1 gramme of Methyl Orange in sufficient Water to produce 1000 c.c. It is mixed with just sufficient Tenth-normal Volumetric Sulphuric Acid Solution to colour the liquid red, and until it just ceases to be transparent and then filtered. The *U.S.P.* directions for carrying out the titrations with this indicator are that 1 to 3 drops are sufficient for the volume of from 50 to 100 c.c. It is not to be employed in alcoholic or boiling solutions.

P.G.—Not included.

PHENOLPHTHALEIN.

Phenolphthalein is described on p. 881.

PHENOLPHTHALEIN SOLUTION.

A solution containing 0.5 p.c. w/v of Phenolphthalein prepared by dissolving 0.5 of a gramme of Phenolphthalein in 50 c.c. of Alcohol (90 p.c.), and diluting with sufficient Distilled Water to produce 100 c.c. This affords a convenient strength for use as an indicator of neutrality. It is colourless in acid or strictly neutral solutions, but assumes a fine pink coloration in the presence of even a minute quantity of alkali. It is employed in titrating acids and alkalis. It is the most convenient indicator of neutrality for organic acids, *e.g.*, Acetic, Tartaric, Citric, Oxalic and Valerianic Acids, etc. It is, however, useless for the titration of free Ammonia or Ammonium salts or other fixed alkalis when Ammonium salts are present. It may be used in the titration of Carbonates or

Bicarbonates if the precaution of boiling off Carbonic Anhydride is observed. Its utility in this respect, however, is inferior to Methyl Orange Solution. The great advantage is that it may be used in alcoholic solutions or mixtures of Alcohol and Ether, and many organic acids insoluble in Water may thus be titrated. It may also be used for determining the proportion of acid radical present in alkaloidal salts, the salt may be dissolved in a sufficiency of Distilled Water, sufficient Ether to form a separate layer and to hold the liberated alkaloid in solution is added and titration carried out.

B.P.—A solution containing 0.2 p.c. w/v of Phenolphthalein prepared by dissolving 0.2 of a gramme of Phenolphthalein in 60 c.c. of Alcohol (90 p.c.), and adding sufficient Distilled Water to measure 100 c.c.

U.S.P.—A 1 p.c. w/v solution of Phenolphthalein prepared by dissolving 1 gramme of Phenolphthalein in 50 c.c. of Alcohol (94.9 p.c.) and diluting with sufficient Water to produce 100 c.c. The *U.S.P.* employs **Phenolphthalein Paper** which is prepared by soaking white unsized paper in the solution, and drying. The *U.S.P.* recommends 3 drops of the solution as a sufficient quantity for use with 50 c.c. of solution to be titrated.

P.G.—A solution containing 1 p.c. w/v of Phenolphthalein obtained by dissolving 1 part by weight of Phenolphthalein in 99 parts by weight of Alcohol (68 to 69 p.c.).

ROSOLIC ACID SOLUTION.

A solution containing 0.2 p.c. w/v of Rosolic Acid, Methylaurin or Corallin, $C_{20}H_{16}O_3$, eq. 301.84.

Rosolic Acid is obtained by the action of Nitrous Acid on Pararosaniline or Rosaniline, or by treating a dilute solution of Aniline Hydrochloride with Sodium Nitrite, which results in the formation of Diazorosaniline Hydrochloride, which when boiled with the addition of Sulphuric Acid affords Rosolic Acid. The solution is prepared by dissolving 0.2 of a gramme in sufficient Alcohol (60 p.c.) to measure 100 c.c. The colour of the solution, which is pale yellow, is unaffected by acids, but in the presence of alkalis a violet-red colour is assumed. It is employed chiefly in the titration of Ammonia and Ammonium salts, and in the titrations involving the use of Sodium or Potassium Sulphites, such as the determination of Citral in Lemon Oil. It is also employed in the determination of Formaldehyde. It is not suitable for the titration of Carbonates, nor, with the exception of Oxalic Acid, for organic acids.

B.P.—Not included. See Table.

U.S.P.—A solution containing 1 p.c. w/v of commercial Rosolic Acid prepared by dissolving 1 gramme of the commercial acid in 10 c.c. of Alcohol (48.9 p.c.), and adding sufficient Water to measure 100 c.c. The quantity recommended by the *U.S.P.* for each 100 c.c. of solution to be titrated is 0.5 c.c. The use of Pæonin (Aurin, R.) is permitted in the place of Rosolic Acid. When used for the titration of ammoniacal solutions the *U.S.P.* requires that they should be highly diluted.

P.G.—A solution containing 1 p.c. w/v of Rosolic Acid prepared by dissolving 1 part by weight of Rosolic Acid in 100 parts by weight of Alcohol (90 p.c.).

STARCH TEST SOLUTION.

A solution containing 1 p.c. w/v of Starch prepared by triturating 1 gramme of pure Potato Starch with 10 c.c. of Water, mixing with 30 c.c. of boiling Water, boiling for a few minutes, cooling, diluting with sufficient Distilled Water to produce 50 c.c. and mixing with an equal volume of Glycerin. A solution of this strength, after filtration or decantation from insoluble cell envelopes, will keep bright for years. It is employed as a sensitive reagent for Iodine.

B.P.—A recently prepared solution containing approximately 2 p.c. of Starch prepared by rubbing 1 gramme of Starch with sufficient Distilled Water to produce a smooth paste, and adding a further sufficient quantity of Distilled Water to produce 50 c.c., boiling for a few minutes, and cooling.

U.S.P.—A freshly prepared and filtered solution containing approximately 0.5 p.c. w/v of Starch prepared by triturating 0.5 of a gramme of Starch with

5 c.c. of Water and adding sufficient boiling Water with constant stirring to measure 100 c.c.

P.G.—A filtered solution of indefinite strength prepared by shaking a small piece of white Starch with hot Water and filtering the solution.

TURMERIC.

Turmeric as an indicator is seldom used in the three Pharmacopœias; when required the Tincture is usually employed. Its chief use is for the detection of Boric Acid. Strips of Turmeric paper so immersed in a solution of free Boric Acid or in an acidified solution of a Borate that only one half the paper is coloured, yield when dried a brown coloration if Boric Acid is present, changing to a dark green or greenish-black on the addition of a fixed alkali solution, or on the addition of Ammonia Solution.

B.P.—A Tincture of Turmeric prepared by macerating 1 of bruised rhizome to 6 of Alcohol (90 p.c.). Turmeric paper is unglazed white paper which has been soaked in this Tincture and dried.

U.S.P.—A filtered Tincture prepared by first exhausting a convenient quantity of brown Turmeric root with repeated small quantities of Water, and digesting the dried residue with 6 times its weight of Alcohol (94.9 p.c.) for several days. **Turmeric paper** is white unsized paper impregnated with the Tincture and dried.

P.G.—A Tincture prepared by digesting at a gentle heat 10 parts of coarsely powdered Turmeric root with 75 parts by weight of Alcohol (90 p.c.) with frequent shaking. After being allowed to deposit, the liquid is filtered. It is used for the preparation of Turmeric paper, and for this purpose 10 parts by weight of the above Tincture are diluted with 30 parts by weight of Alcohol (90 p.c.) and 40 parts by weight of Water and strips of the best unglazed paper are impregnated with this mixture, and whilst protected from the light are dried at the ordinary temperature of the air. **Turmeric paper** should assume a brown coloration on the addition of a single drop of a mixture prepared by mixing 1 c.c. of Tenth-normal Volumetric Potassium Hydroxide Solution and 25 c.c. of Water.

It should be kept in well-closed vessels and protected from the light.

STARCH TEST SOLUTION.

A solution containing 1 p.c. w/w of Starch prepared by triturating 1 gramme of pure Potato Starch with 10 c.c. of Water, mixing with 50 c.c. of boiling Water, boiling for a few minutes, diluting with sufficient Distilled Water to produce 50 c.c., and mixing with an equal volume of the same. A solution of the same strength after filtration or decantation from the starch will not interfere with the reaction of the starch solution. It is employed as a sensitive reagent for iodine. *A.S.P.*—A quantity of starch solution containing approximately 2 p.c. of Starch prepared by triturating 2 grammes of starch with 10 c.c. of Water, boiling for a few minutes, diluting with sufficient Distilled Water to produce 50 c.c., and mixing with an equal volume of the same. *U.S.P.*—A starch solution prepared and filtered solution containing approximately 1 p.c. of starch prepared by triturating 1 g. of a genuine starch with