

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	1 of 9

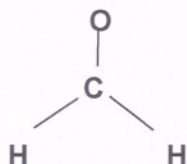
Test	Specification
1. Appearance	Clear, colorless liquid.
2. Solubility	Miscible with water and with ethanol (96 per cent). It may be cloudy after storage.
3. Identification	
A. Chemical test	A violet-blue or violet-red colour develops within 5 mins.
B. Chemical test	An intense red colour is formed.
C. Chemical test	A grey precipitate or a silver mirror is formed.
D. Assay	34.5% to 38.0% m/m of Formaldehyde.
4. Appearance of solution	Solution S is colourless.
5. Acidity	Not more than 0.4 ml of 0.1M sodium hydroxide is required to change the colour of the indicator to red.
6. Methanol (By GC)	Between 9.0% v/v to 15.0% v/v.
7. Sulphated ash	Maximum 0.1%
8. Assay	34.5% to 38.0% m/m of Formaldehyde.

	PREPARED BY	CHECKED BY	AUTHORIZED BY
	Q.C.	Q.C.	Q.A.
SIGNATURE	<u>Y Bhide</u>	<u>[Signature]</u>	<u>Pspandit</u>
DATE	25/06/18	26/06/18	26/06/18

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	2 of 9

GENERAL INFORMATION

Structure:



Molecular Formula: CH₂O

Molecular weight: 30.03

Desirable Pack:

To be supplied in HDPE container/ Glass bottle, properly identified with a label having name of the material, Name of the Manufacturer/Supplier, Quantity, Manufacturer's Batch Number, Manufacturing Date and Expiry Date.

Storage Condition:

Protected from light, at a temperature of 15°C to 25°C.

Handling precaution:

Use PPE (Personal Protective Equipments) during handling of the material.

Sampling SOP:

As per the current approved sampling procedure.

Quantity to be sampled:

Analysis Sample: 500 ml

Control Sample: 1000 ml

Shelf Life:

Three years from the date of manufacturing.

Note:

1. For Bullet Rinsing and Filter rinsing-
 - If previous product is Formaldehyde BP, Formaldehyde Ph.Eur, Formaldehyde USP or Formaldehyde IP or Formaldehyde BP/Ph.Eur/USP/IP, then perform Appearance, Solubility and Identification tests.
 - If previous product is different then check for its carry over on GC (NMT 0.2%) method; which is used to find out Assay of Raw material.
2. For Tanker Rinsing-
 - Perform Appearance, Solubility, Identification, Methanol (By GC) and Assay tests.

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	3 of 9

Document number	Supersedes	Changes made	Reason for change
QC/PH-FG/SPEC/27	Rev.0	Format change – 1. General Information added. 2. History page added.	As per requirement of Schedule M.
	Rev.1	1. Reference updated	Ph. Eur-7.0 (Refer Change control No.RCPL/CC/QC/001-14)
	Rev.2	1. Reference updated. 2. RUNA logo inserted along with the name of company	Ph. Eur-8.0 As per SOP of Document and Data control (Refer Change control No.RCPL/CC/QC/005-15)
	Rev. 3	Reference updated.	Ph. Eur-9.0 (Refer Change control No.RCPL/CC/QC/009-16)
	Rev. 4	1. Reference updated. 2. Mentioned tests to be perform for Bullet Rinsing, Filter Rinsing and Tanker Rinsing. 3. Shelf Life is added.	Ph. Eur -9.5 Supplement. Refer Change Control No. RCPL/CC/QC/008-18

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	4 of 9

1. Appearance**Procedure-**

Formaldehyde solution is a Clear, colorless liquid . Check by physical observation during sampling.

Report if any discrepancy with regard to appearance, nature, visible contaminants, damaged packaging etc.

2. Solubility**Procedure-**

Formaldehyde solution is miscible with water and with ethanol (96 per cent).

Mix a suitable quantity of substance to be examined with similar quantity of solvent. Examine the solution for clarity, black particles or fibers. It may be cloudy after storage.

3. Identification**A. Chemical test****Reagents required-**

- Solution S.
- Chromotropic acid sodium salt R.
- Sulfuric acid R.
- Water R.

Solution preparation-

- Solution S – Dilute 10mL, filtered if necessary, to 50mL with carbon dioxide-free water R.
- Chromotropic acid sodium salt R- 15 g/L solution.

Procedure-

Dilute 1 ml of solution S to 10 ml with water R. To 0.05 ml of the solution add 1 ml of a 15 g/L solution of Chromotropic acid sodium salt R., 2 ml of water R and 8ml of sulfuric acid R.

Interpretation-

A violet-blue or violet-red colour develops with 5 min.

B. Chemical test**Reagents required-**

- Solution S
- Water R.
- Phenylhydrazine hydrochloride R.
- Potassium ferricyanide solution R.
- Hydrochloric acid R.

Solution preparation-

- Solution S – Dilute 10mL, filtered if necessary, to 50mL with carbon dioxide-free water R.

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	5 of 9

B. Chemical test continued ...

- Phenylhydrazine hydrochloride R - 10 g/L solution. Prepare immediately before use.
- Potassium ferricyanide solution R- Wash 5 g of potassium hexacyanoferrate(III) crystals with a little water and dissolve in sufficient water to produce 100 mL. Prepare immediately before use.

Procedure-

To 0.1 ml of solution S add 10 ml of water R. Add 2 ml of a 10 g/L solution of phenylhydrazine hydrochloride R, prepared immediately before use, 1 ml of potassium ferricyanide solution R and 5 ml of hydrochloric acid R.

Interpretation-

An intense red colour is formed.

C. Chemical test**Reagents required-**

- Water R.
- Silver nitrate solution R₂.
- Dilute ammonia R₂.

Solution preparation-

- Silver nitrate solution R₂ - Dissolve 1.7 gm of silver nitrate into water and dilute up to 100 ml with water.
- Dilute ammonia R₂- Dilute 14 g of concentrated ammonia to 100 mL with water.

Procedure-

Mix 0.5 ml with 2 ml of water R and 2 ml of Silver nitrate solution R₂ in a test tube. Add dilute ammonia R₂ until slightly alkaline. Heat on a water-bath.

Interpretation-

A grey precipitate or a silver mirror is formed.

D. Assay**Reagents required-**

- Water R.
- Dilute sodium hydroxide solution R.
- 0.05M Iodine.
- Dilute sulfuric acid R
- Starch solution R
- 0.1M Sodium thiosulfate.

Solution preparation-

- Dilute sodium hydroxide solution R - Dissolve 8.5 g of sodium hydroxide in water and dilute to 100 mL with the same solvent.

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	6 of 9

D. Assay continued...

- Dilute Sulfuric acid R- Dilute Sulfuric Acid is prepared by adding 104 g of Sulfuric Acid to 896 g of Purified Water with constant stirring and cooling.
- Starch solution R - 0.5 g of soluble starch in 35 mL of water. Boil for 2 minutes and allow to cool.

Procedure-

Refer Assay test.

Interpretation-

It complies with the limits of the assay.

4. Appearance of solution**Reagents required-**

- Solution S

Solution preparation-

- Solution S – Dilute 10mL, filtered if necessary, to 50mL with carbon dioxide-free water R..

Procedure-

Using identical tubes of colourless, transparent, neutral glass with a flat base and an internal diameter of 15 mm to 25 mm, compare the liquid to be examined with water R or the solvent or the reference solution (see Tables of reference solutions) prescribed in the monograph, the depth of the layer being 40 mm. Compare the colours in diffused daylight, viewing vertically against a white background.

Interpretation-

Solution S is colourless.

5. Acidity**Reagents required-**

- Solution S .
- Phenolphthalein solution R.
- 0.1M Sodium hydroxide

Solution preparation-

- Solution S – Dilute 10mL, filtered if necessary, to 50mL with carbon dioxide-free water R.
- Phenolphthalein solution R - A 1.0% w/v solution of phenolphthalein in ethanol (96%).

Procedure-

To 10 ml of solution S, add 1 ml of Phenolphthalein solution R.

Interpretation-

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	7 of 9

5. Acidity continued...

Not more than 0.4 ml of 0.1 M sodium hydroxide is required to change the colour of the indicator to red.

6. Methanol (By GC)**Chromatographic condition-**

Carrier Gas	: Nitrogen
Column	: Glass column of 1.5 - 2.0m x 2-4mm containing ethylvinylbenzene-divinylbenzen copolymer R (150-180µm).
Column Temperature (Initial)	: 120°C
Detector	: FID
Detector Port Temperature	: 150°C
Injector Port Temperature	: 150°C
Injection Volume	: 1 µl
Flow rate	: 30-40 ml/min.

Reagents required-

- Ethanol R1
- Methanol R.

Solution preparation-

- Internal standard solution – Dilute 10 ml of ethanol R1 with water to 100 ml.
- Test solution – To 10.0 ml of the solution to be examined add 10.0 ml of the Internal standard solution, and dilute with water to 100.0 ml.
- Reference solution – To 1.0 ml of methanol R add 10.0 ml of the Internal standard solution, and dilute with water to 100.0 ml.

System Suitability- (Reference solution)

- Resolution – Minimum 2.0 between the peaks due to methanol and ethanol.

Limit-

- Methanol 9.0 % v/v to 15.0 % v/v.

Procedure-

Inject 1 µl of blank, internal standard solution, Reference solution and test solution.

Calculation-

$$\% \text{ v/v of methanol} = 100 \times (V_M / V) (R_U / R_S)$$

Where,

V_M is the volume in ml, of methanol taken to prepare the reference solution.

V is the volume in ml, of solution taken to prepare the test solution.

R_U is the peak response ratio of methanol to that of the internal standard obtained from the test solution.

R_S is the peak response ratio of methanol to that of the internal standard obtained from the reference solution.

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	8 of 9

7. Sulfated ash**Procedure-**

Heat a platinum dish to redness for 10 minutes, allow to cool in a desiccator and weigh. Place 1 g of the substance being examined in the dish, moisten with sulfuric acid, ignite gently, again moisten with sulfuric acid and ignite at about 800°. Cool, weigh again, ignite for 15 minutes and repeat this procedure until two successive weighings do not differ by more than 0.5 mg.

Calculation-

$$\text{Sulfated ash} = \frac{\text{Wt.of residue} \times 100}{\text{Wt.of sample}}$$

Where,

W1= Weight of the empty platinum dish in gm

W2= Weight of the platinum dish + sample in gm

W3= Weight of sample in gm

W4= Weight of the platinum dish + sample dried till const. wt. in gm

W5= Weight of residue in gm

8. Assay**Reagents required-**

- Dilute Sodium hydroxide solution R
- 0.05M Iodine
- Dilute Sulfuric acid R
- Starch solution R.
- 0.1 N Sodium thiosulphate
- Water R

Solution preparation-

- Dilute Sodium hydroxide R
- Dilute Sulfuric acid- (10 percent)—Cautiously add 57 mL of sulfuric acid to about 100 mL of water, cool to room temperature, and dilute with water to 1000 mL.
- Starch TS - Mix 1 g of soluble starch with 10 mg of red mercuric iodide and sufficient cold water to make a thin paste. Add 200 mL of boiling water, and boil for 1 minute with continuous stirring. Cool, and use only the clear solution.

Procedure-

Into a 100 ml volumetric flask containing 2.5 ml of water R and 1 ml of dilute sodium hydroxide solution R, introduce 1.0 g of the solution to be examined, shake, and dilute with water R to 100.0 ml. To 10.0 ml of this solution add 30.0 ml of 0.05M iodine. Mix, and add 10 ml of dilute sodium hydroxide solution R. After 15 minutes, add 25 ml of diluted sulfuric acid R and 2 ml of starch solution R. Titrate with 0.1N sodium thiosulphate.

1 ml of 0.05M iodine is equivalent to 1.501 mg of CH₂O.

Rev.5	FORMALDEHYDE SOLUTION Ph.Eur. FG SPECIFICATION WITH METHOD OF ANALYSIS		Reference
			Ph.Eur-9.5 Supplement
FG Specification No	Supersedes	Effective date	Page No
QC/PH-FG/SPEC/27	Rev.4	01/07/2018	9 of 9

8. Assay continued...

Calculation-
$$\frac{(\text{B.R. for blank} - \text{B.R. for sample}) \times \text{Molarity Factor} \times 0.001501 \times 100 \times 100}{\text{Wt. of sample} \times 10}$$

Where, B.R. – Burette reading

Interpretation-

34.5% to 38.0% m/m