NOOTAN PHARMACEUTICALS, BAROTIWALA, BADDI

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STANDARD TEST SPECIFICATION QUALITY CONTROL DEPARTMENT			
RAW MATERIAL SPECIFICATION			
Name of Material.	SODIUM METHYL PARABEN		
Reference	IP	**Spec/STP No.**	RMS/043
Supersedes	Nil	**Revision No.**	00
Effective Date	06/01/2025	**Review Date**	As & when required

Sr. No.	Test	Specification
1.	Description	A white, crystalline powder; hygroscopic.
2.	Solubility	Freely soluble in water; sparingly, soluble in ethanol (95per ent), practically insoluble in fixed oils.
3.	**Identification**	
	A. By IR	The infrared absorption spectrum of the sample is concordant obtained from sodium methylparaben RS or with the reference spectrum of sodium methylparaben.
	B. By Chemically	A white precipitates are produced
4.	pH	9.5 to 10.5
5.	Related Substances (By liquid Chromatography)	
	A. Methyl paraben Impurity A	Not More Than 0.5%
	B. Individual Impurity	Not More Than 0.1%
	C. Total Impurity	Not More Than 1.0%
6.	Chlorides	Not more than 330 ppm
7.	Sulphates	Not more than 0.12 per cent
8.	Water	Not more than 5.0 %
9.	Assay(On anhydrous basis)	Not less than 95.0 % and not more than 102.0 %

	Prepared by	Checked by	Reviewed by	Approved by
Department	QC	QC	Q.A.	QA
Sign.		Akumar	Kalpana	
Date	06/01/2025	06-01-2025	06/01/2025	06/01/2025
Name	Ram Swrat	Awadhesh kumar	Kalpana Sharma	Dr. H.S. Yadav

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QUALITY CONTROL DEPARTMENT			
RAW MATERIAL STANDARD TEST PROCEDURE			
Name of Material	SODIUM METHYL PARABEN		
Reference	IP	**Spec/STP No.**	RMT/043
Supersedes	Nil	**Revision No.**	00
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1.0 DESCRIPTION:

Procedure:

Spread appropriately 1.0 g of sample over the Petri- dish and examine visually. Check the appearance of colour, nature and visible foreign particles.

2.0 SOLUBILITY:

Weigh accurately 1.0 g of sample in a suitable clean and dry conical flask and observe for solubility by adding Solvent.

The descriptive term explaining solubility is given as below:

Descriptive Term	Volume of solvent in ml/gm of sample
Very Soluble	Less than 1.0 ml
Freely Soluble	From 01 to 10 ml
Soluble	From 10 to 30 ml
Sparingly Soluble	From 30 to 100 ml
Slightly Soluble	From 100 to 1000 ml
Very Slightly Soluble	From 1000 to 10,000 ml
Practically Insoluble or Insoluble	10,000 and over.

3.0 IDENTIFICATION:A. Dissolve 0.5 g in 5 ml of water and acidify to litmus paper with hydrochloric acid, a white precipitate is produced. Wash the precipitate with water and dry.

Determine by infrared absorption spectrophotometry. Compare the spectrum with that obtained with methylparaben IPRS.

B. A 5 per cent w/v solution gives the reactions of sodium salts.

4.0 APPEARNCE OF SOLUTION: 10.0 per cent w/v solution in water is clear.

5.0 pH :- Prepare a slurry of 0.1 per cent w/v solution of sample shake for 15 minutes and check pH using a calibrated pH meter.

6.0 Related substances.

Determine by liquid chromatography

Test Solution : Dissolve 50.0 mg of the substance under examination in 2.5 ml of methanol and dilute to 50.0 ml with the mobile phase. Dilute 10.0 ml of the solution to 100.0 ml with the mobile phase.

Reference solution (a). Dissolve 5 mg of 4-hydroxybenzoic acid and 5 mg of the substance under examination in the mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

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Department	QC	QC	QA	QA
Sign.		AKumar	Kalpana Sharma	
Date	06/01/2025	06-01-2025	06/01/2025	06/01/2025
Name	Ram Swrat	Awadhesh Kumar	Kalpana Sharma	Ajeet Kumar Yadav

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Reference solution (b). Dissolve 50 mg of methyl paraben IPRS in 2.5 ml of methanol and. dilute to 50.0 ml with the mobile phase. Dilute 10 ml of the solution to 100.0 ml with the mobile phase.

Reference solution (c). Dilute 1.0 ml of the test solution to 20.0 ml with the mobile phase. Dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

Chromatographic system . - a. stainless steel column 15 cm x 4.6 mm, packed-with octadecylsilane bonded to porous silica (5 µm)

- **Mobile phase:** a mixture of 35 volumes of 0.68 per cent w/v solution of potassium dihydrogen phosphate, and 65 volumes of methanol,
- **Flow rate:** 1:3 ml per minute,
- **Spectrophotometerset** at`272 nm,
- **injection volume:** 10 µl.

The relative retention time with reference to methy paraben for 4-hydroxybénzoic acid is about 0.6.
Inject reference solution (a). The test is not. valid unless the resolution between the peaks due to methyl paraben and 4-hydroxybenzoic acid is not less than 2.0. inject reference solution (c) and the test solution. Run the chromatogram five times the retention time of the principal. In the chromatogram obtained with the test solution the area of any peak corresponding to 4-hydroxybenzoic acid , multiply by 1,4 is not more than the six times the area of the Principal peak in the chromatogram obtained with reference solution (c) (3.0 per cent), the area of any other secondary peak is not more than the area of the principal peak in the chromatogram obtained with reference solution (c) (0.5 per cent) and the sum of areas of all the secondary peaks other than 4-hydroxybenzoic acid is not more than twice the area of the principal peak in the chromatogram obtained with reference solution (c) (1.0 per cent). Ignore the peak with an area less than 0.2 times the area of the principal peak in the chromatogram obtained with reference solution (c) (0.1 per cent).

7.0 Chlorides: Dissolve 1.0 g in 20 ml of water, add 0.4 ml of nitric acid and filter. 15 ml of the filtrate complies with the limittest for chlorides (330 ppm).

8.0 Sulphates: Dissolve 0.5 g in 40 ml of water, add 3.5 ml of 2 M hydrochloric acid, dilute to 60 ml with water and filter. 15 ml of the filtrate complies with the limit test for sulphates (0.12 per cent).

9.0 Water: Determined on 1.0 g.
Transfer 35 to 45 ml of methanol into titration vessel and titrate with KF reagent, which is standardized earlier, to the electrometric end point to consume any moisture present & refill the burette 0 mark again Quickly , add 0.50 g of the substance, mix and titrate with the reagent to electrometric end point the % of water content with display on the screen of the instrument.
Calculate the percentage of Water content by following formula:-

$$\% \text{ Water Content} = \frac{V \times F \times 100}{A}$$

V = Volume of KF Reagent consumed in ml.

F = Water equivalent factor of KF Reagent mg/ml.
A = Weight of sample in mg.

10.0 Assay. Determine by liquid chromatography:
As described under Related substances. Inject reference solution (b) and the test solution. Calculate the content of $C_8H_7NaO_3$, multiplying the content of methyl paraben by a correction factor of 1.145.