	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	IBUPROFEN BP			
Specification No.	RMASI0015-01	**Revision No.**	01	**Item Code.:** RMAI0015
Supersedes	RMASI0015-00	**Effective Date**	17/02/2023	**Page No.:** 1 of 4

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	$C_{13}H_{18}O_2$
2	Molecular weight	206.3
3	Storage conditions	NA
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	6 g
6	Quantity of reserve sample	12 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	13/02/2023	14/02/2023	15/02/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	IBUPROFEN BP			
Specification No.	RMASI0015-01	**Revision No.**	01	**Item Code.:** RMAI0015
Supersedes	RMASI0015-00	**Effective Date**	17/02/2023	**Page No.:** 2 of 4

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or almost white, crystalline powder or colourless crystals.
2.	*Solubility	Practically insoluble in water, freely soluble in acetone, in methanol and in methylene chloride. It dissolves in dilute solutions of alkali hydroxides and carbonates.
3.	*Identification **A.** By Melting point **B.** By UV **C.** By IR **D.** By TLC	**A.** 75 °C to 78 °C. **B.** Absorption maxima At 264 nm and 272 nm. The ratio of the absorbance measured at the maximum at 264nm to that measured at the shoulder at 258nm is 1.20 to 1.30. The ratio of the absorbance measured at the maximum at 272nm to that measured at the shoulder at 258nm is 1.00 to 1.10. **C.** The Infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Ibuprofen RS. **D.** The principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with the reference solution.
4.	Appearance of solution	Solution S is clear and colourless

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Signature			
Date	13/02/2023	14/02/2023	15/02/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	IBUPROFEN BP			
Specification No.	RMASI0015-01	**Revision No.**	01	**Item Code.:** RMAI0015
Supersedes	RMASI0015-00	**Effective Date**	17/02/2023	**Page No.:** 3 of 4

S.NO	TEST (s)	SPECIFICATION (s)
5.	*Optical rotation	-0.05° to + 0.05°.
6.	*Related substances (By HPLC) (i) Impurity A (ii) Impurity J (iii) Impurity N (iv) Unspecified impurity (v) Total impurities	 Not more than 0.15% Not more than 0.15% Not more than 0.15% Not more than 0.05% Not more than 0.2%
7.	Impurity F (By GC)	Not more than 0.1%
8.	Sulphated Ash	Not more than 0.1% w/w
9.	*Loss on drying	Not more than 0.5% w/w
10.	*Assay By Titration (On dried basis)	Not less than 98.5% and not more than 101.0% w/w.

Remarks: The above * Marked tests are to be performed while retesting the material.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Date	13/02/2023	14/02/2023	15/02/2023

REVISION HISTORY:

Specification No.	Reason for Review	Change control No.	Effective Date
RMASI0015-01	Periodic review.	NA	17/02/2023

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	13/02/2023	14/02/2023	15/02/2023

1. DESCRIPTION: < REFER GAM 001>

White or almost white, crystalline powder or colourless crystals.

2. SOLUBILITY: < REFER GAM 002>

10mg of sample + 100mL of Water	Practically insoluble if the material not dissolves.
100mg of sample + 1mL of Acetone	Freely soluble if the material dissolves.
100mg of sample + 1mL of Methanol	Freely soluble if the material dissolves.
100mg of sample + 1mL of Methylene chloride	Freely soluble if the material dissolves.

It dissolves in dilute solutions of alkali hydroxides and carbonates.

3. IDENTIFICATION:

First identification: ***A,*** **C**

Second identification: ***A, B,*** **D**

A. By Melting point: < REFER GAM 028>

75 °C to 78 °C.

B. By UV:

Test solution:

Dissolve 50.0 mg in a 4 g/L solution of sodium hydroxide and dilute to 100.0 mL with the same alkaline solution.

Spectral range 240-300 nm, using a spectrophotometer with a band width of 1.0 nm and a scan speed of not more than 50 nm/min.

Absorption maxima At 264 nm and 272 nm.

Shoulder At 258 nm.

Absorbance ratio:

— A_{264} / A_{258} = 1.20 to 1.30;

— A_{272} / A_{258} = 1.00 to 1.10.

C. By IR: < REFER GAM 003>

The Infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Ibuprofen WS.

D. By Thin-layer chromatography:

Test solution:

Dissolve 50 mg of the substance to be examined in methylene chloride R and dilute to 10 mL with the same solvent.

Reference solution:

Dissolve 50 mg of ibuprofen WS in methylene chloride and dilute to 10 mL with the same solvent.

Plate TLC silica gel plate.

Mobile phase Anhydrous acetic acid, Ethyl acetate, Hexane (5:24:71 V/V/V).

Application 5 µL.

Development Over a path of 10 cm.

Drying At 120 °C for 30 min.

Detection Lightly spray with a 10 g/L solution of potassium permanganate in dilute sulfuric acid and heat at 120 °C for 20 min; examine in ultraviolet light at 365 nm.

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Results:

The principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with the reference solution.

4. APPEARANCE OF SOLUTION:

Solution S:

Dissolve 2.0 g in methanol and dilute to 20 mL with the same solvent.

Solution S is clear and colourless.

5. OPTICAL ROTATION:

-0.05° to + 0.05°.

Dissolve 0.50 g in methanol and dilute to 20.0 mL with the same solvent.

6. RELATED SUBSTANCES: (BY HPLC)

Chemicals/Reagents/Standards:

Ibuprofen	:	Working standard
Ibuprofen impurity B	:	Reference standard
Ibuprofen for peak identification	:	Reference standard
Phosphoric acid	:	AR grade
Acetonitrile	:	AR grade
Purified water	:	Milli-Q water (or) equivalent

Chromatographic Conditions:

Stationary phase	:	End-capped octadecylsilyl amorphous organosilica polymer 150mm x 4.6mm, (5µm) or equivalent
Flow Rate	:	2.0ml/min

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Wavelength : 214nm

Injection volume : 20µl

Retention time : Retention time of Ibuprofen peak is at about 21.0 minutes

Gradient Program:

Time (min)	Mobile phase A (per cent V/V)	Mobile phase B (per cent V/V)
0 - 25	100	0
25 - 55	100 → 15	0 → 85
55 - 70	15	85
70-75	100	0
75-80	STOP	

Mobile phase A:

Mix 0.5 volumes of phosphoric acid, 340 volumes of acetonitrile and 600 volumes of water to equilibrate and dilute to 1000 volumes with water.

Mobile phase B: Acetonitrile

Test solution:

Weigh accurately and dissolve about 20 mg of the substance to be examined in 2 mL of acetonitrile and dilute to 10.0 mL with mobile phase A.

Reference solution (a):

Dilute 1.0 mL of the test solution to 100.0 mL with mobile phase A. Dilute 1.0 mL of this solution to 10.0 mL with mobile phase A.

Reference solution (b):

Dilute 1.0 mL of ibuprofen impurity B RS to 10.0 mL with acetonitrile (solution A). Dissolve 20 mg of ibuprofen WS in 2 mL of acetonitrile, add 1.0 mL of solution A and dilute to 10.0 mL with mobile phase A.

Reference solution (c):

Dissolve the contents of a vial of ibuprofen for peak identification RS (mixture of impurities A, J and N) in 1 mL of acetonitrile and dilute to 5 mL with mobile phase A.

Identification of impurities:

Use the chromatogram supplied with ibuprofen for peak identification RS and the chromatogram obtained with reference solution (c) to identify the peaks due to impurities A, J and N.

Relative retention With reference to ibuprofen (retention time = about 21 min):
impurity J = about 0.2; impurity N = about 0.3; impurity A = about 0.9;
impurity B = about 1.1.

System suitability Reference solution (b):

— peak-to-valley ratio: minimum 1.5, where H_p = height above the baseline of the peak due to impurity B, and H_v = height above the baseline of the lowest point of the curve separating this peak from the peak due to ibuprofen. If necessary, adjust the concentration of acetonitrile in mobile phase A.

Limits:

— impurities A, J, N: for each impurity, not more than 1.5 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.15 per cent);

— unspecified impurities: for each impurity, not more than 0.5 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.05 per cent);

— total: not more than twice the area of the principal peak in the chromatogram obtained with reference solution (a) (0.2 per cent);

— disregard limit: 0.3 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.03 per cent).

Inject 20µl of the above solution as per following sequence.

Injection sequence:

S. No	Sample Name	No. of injections
1	Mobile phase (Blank)	1
2	Reference solution (a)	1
3	Reference solution (b)	1
4	Reference solution (c)	1
5	Blank	1
6	Test solution	1

Calculations:

Impurity A: (NMT 0.15%)

$$= \frac{AT_A}{AS} \times \frac{WT}{10} \times \frac{1}{100} \times \frac{1}{10} \times \frac{10}{WT} \times 100$$

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Where,

AT_A = Area of Impurity A peak in Test solution.

AS = Area of the principal peak in the Reference solution (a)

WT = Weight of the sample taken in mg.

Impurity J: (NMT 0.15%)

$$= \frac{AT_J}{AS} \times \frac{WT}{10} \times \frac{1}{100} \times \frac{1}{10} \times \frac{10}{WT} \times 100$$

Where,

AT_J = Area of Impurity J peak in Test solution.

AS = Area of the principal peak in the Reference solution (a)

WT = Weight of the sample taken in mg.

Impurity N: (NMT 0.15%)

$$= \frac{AT_N}{AS} \times \frac{WT}{10} \times \frac{1}{100} \times \frac{1}{10} \times \frac{10}{WT} \times 100$$

Where,

AT_N = Area of Impurity N peak in Test solution.

AS = Area of the principal peak in the Reference solution (a)

WT = Weight of the sample taken in mg.

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Unspecified impurity: (NMT 0.05%)

$$= \frac{ATU}{AS} \times \frac{WT}{10} \times \frac{1}{100} \times \frac{1}{10} \times \frac{10}{WT} \times 100$$

Where,

ATU = Area of unspecified impurity peak in Test solution.
AS = Area of the principal peak in the Reference solution (a)
WT = Weight of the sample taken in mg.

Total impurities: (NMT 0.2%)

$$= \frac{AT_T}{AS} \times \frac{WT}{10} \times \frac{1}{100} \times \frac{1}{10} \times \frac{10}{WT} \times 100$$

Where,

AT_T = Area of Total impurities peak in Test solution.
AS = Area of the principal peak in the Reference solution (a)
WT = Weight of the Test solution in mg.

Total impurities = Sum of known impurities + Unknown impurities

7. IMPURITY F: (BY GC)

Chromatographic Conditions:

Column	:	25m x 0.53mm, fused silica
Stationary phase	:	Macrogol 20 000 (film thickness 2 µm).
Flow Rate	:	5.0ml/min

Column temperature	:	150°C
Injection volume	:	1µl
Injection port	:	200°C
Detector temperature	:	250°C
Detection	:	Flame ionisation.

Methylating solution:

Dilute 1 mL of N,N-dimethylformamide dimethylacetal and 1 mL of pyridine to 10 mL with ethyl acetate.

Test solution:

Weigh accurately about 50.0 mg of the substance to be examined into a sealable vial, dissolve in 1.0 mL of ethyl acetate, add 1 mL of the methylating solution, seal and heat at 100 °C in a block heater for 20 min. Allow to cool. Remove the reagents under a stream of nitrogen at room temperature. Dissolve the residue in 5 mL of ethyl acetate.

Reference solution (a):

Dissolve 0.5 mg of ibuprofen impurity F RS in ethyl acetate and dilute to 10.0 mL with the same solvent.

Reference solution (b):

Weigh about 50.0 mg of ibuprofen WS into a sealable vial, dissolve in 1.0 mL of reference solution (a), add 1 mL of the methylating solution, seal and heat at 100 °C in a block heater for 20 min. Allow to cool. Remove the reagents under a stream of nitrogen at room temperature. Dissolve the residue in 5 mL of ethyl acetate.

Injection 1 µL of the test solution and reference solution (b).

Run time Twice the retention time of ibuprofen.

System suitability:

— relative retention with reference to ibuprofen (retention time = about 17 min): impurity F = about 1.5.

Limit:

— impurity F: Maximum 0.1 per cent.

8. SULPHATED ASH: < REFER GAM 032>

Maximum 0.1%. Determine on 1.0g of sample.

9. LOSS ON DRYING: < REFER GAM 026>

Maximum 0.5 per cent, determined on 1.000 g by drying in vacuo.

10. ASSAY: (By Titration)

Weigh accurately and dissolve about 0.450 g in 50 mL of methanol. Add 0.4 mL of phenolphthalein solution. Titrate with 0.1 M sodium hydroxide until a red colour is obtained. Carry out a blank titration.

1 mL of 0.1 M sodium hydroxide is equivalent to 20.63 mg of $C_{13}H_{18}O_2$.

Calculation:

$$= \frac{\text{Blank - Titer value x Molarity of 0.1 M sodium hydroxide x 0.02063 x 100 x100}}{\text{Sample weight in (g) X (100 – Sample LOD) x 0.1}}$$

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Date	13/02/2023	14/02/2023	15/02/2023

REVISION HISTORY:

STP No.	Reason for Review	Change control No.	Effective Date
RMATI0015-01	Periodic review.	NA	17/02/2023

****END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	13/02/2023	14/02/2023	15/02/2023

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	RAW MATERIAL SPECIFICATION			
Name of Product	MAGNESIUM STEARATE BP			
Specification No.	SPEC-RMEM0033-00	**Revision No.**	00	**Item Code.:** RMEM0033
Supersedes	RMESM0033-01	**Effective Date**	14/03/2024	**Page No.:** 1 of 4

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	$(C_{17}H_{35}CO_2)_2$ Mg
2	Molecular weight	591.27
3	Storage conditions	Store at ambient temperature.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	20 g
6	Quantity of reserve sample	40 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12/03/2024	13/03/2024	12/03/2024

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	RAW MATERIAL SPECIFICATION			
Name of Product	MAGNESIUM STEARATE BP			
Specification No.	SPEC-RMEM0033-00	**Revision No.**	00	**Item Code.:** RMEM0033
Supersedes	RMESM0033-01	**Effective Date**	14/03/2024	**Page No.:** 2 of 4

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	A white or almost white, very fine, light powder, greasy to the touch.
2.	*Solubility	Practically insoluble in water and in anhydrous ethanol.
3.	*Identification **A.** By Freezing point **B.** By Acid value **C.** By Fatty acid composition **D.** By Chemical test	 Not less than 53°. The acid value of the fatty acids is 195 to 210. The principle peaks in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution. A white crystalline precipitate is formed.
4.	Acidity or alkalinity	Not more than 0.05ml of 0.1M Hydrochloric acid or 0.1M Sodium hydroxide is required to change the colour of the indicator.
5.	Chlorides	Not more than 0.1%.
6.	Sulfates	Not more than 1.0%.
7.	Cadmium	Not more than 3 ppm

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12/03/2024	13/03/2024	13/03/2024

S.NO	TEST (s)	SPECIFICATION (s)
8.	Lead	Not more than 10 ppm
9.	Nickel	Not more than 5 ppm
10.	*Loss on drying	Not more than 6.0% w/w.
11.	*Assay for Magnesium (On dried basis)	Not less than 4.0% and not more than 5.0% w/w
12.	Stearic acid and Palmitic acid (i) Stearic acid (ii) Sum of Stearic acid and Palmitic acid	 Not less than 40.0% Not less than 90.0%
13.	*Microbial contamination (i) Total aerobic microbial count (ii) Total yeast and mold count iii) Escherichia coli iv) Salmonella species	 Not more than 1000 cfu/g Not more than 100 cfu/g Should be absent Should be absent

Remarks: The above * Marked tests are to be performed while retesting the material.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
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	RAW MATERIAL SPECIFICATION			
Name of Product	MAGNESIUM STEARATE BP			
Specification No.	SPEC-RMEM0033-00	**Revision No.**	00	**Item Code.:** RMEM0033
Supersedes	RMESM0033-01	**Effective Date**	14/03/2024	**Page No.:** 4 of 4

REVISION HISTORY:

Specification No.	Reason for Review	Change control No.	Effective Date
SPEC-RMEM0033-00	(i) Specification numbering procedure revised as per SOP No. ST/QC/058. (ii) There is no changes in specification as per current monograph.	ST/CC/23/063 ST/CC/24/067	14/03/2024

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12/03/2024	13/03/2024	13/03/2024

1. DESCRIPTION: < REFER GAM 001>

A white or almost white, very fine, light powder, greasy to the touch.

2. SOLUBILITY: < REFER GAM 002>

10mg of sample + 100mL of Water	Practically insoluble if the material does not dissolves.
10mg of sample + 100mL of Anhydrous ethanol	Practically insoluble if the material does not dissolves.

3. IDENTIFICATION: < REFER GAM 003>

First identification : C and D

Second identification: A, B and D

To 5.0g of sample add 50 ml of peroxide-free ether, 20ml of dilute nitric acid and 20ml of water and heat under a reflux condenser until dissolution is complete. Allow to cool. In a separating funnel, separate the aqueous layer and shake the ether layer with 2 quantities, each of 4ml of water. Combine the aqueous layers, wash with 15ml of peroxide-free ether and dilute to 50ml with water (Solution S). Evaporate the organic layer to dryness and dry the residue at 100-105°C. Keep the residue for identification tests A and B.

A. By Freezing point:

Determined on the residue obtained in the preparation of solution S has a freezing point not less than 53°C.

B. By Acid value:

The acid value of the fatty acids is 195 to 210, dissolved on 0.2g of the residue obtained in the preparation of solution S in 25ml of the mixture of equal volumes of ethanol (96 per cent) and light petroleum, previously neutralised with 0.1M potassium hydroxide or 0.1M sodium hydroxide, unless otherwise specified, using 0.5mL of phenolphthalein solution as indicator. If necessary, heat to about 90°C to dissolve the substance to be examined. When the substance to be examined has dissolved, titrate with 0.1M potassium hydroxide or 0.1M sodium hydroxide until the pink colour persists for at least 15s (n mL of titrant). When heating has been applied to aid dissolution maintain the temperature at about 90°C during the titration.

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Signature			
Date	12/03/2024	13/03/2024	13/03/2024

$$I_A = \frac{5.61n}{m}$$

C. By Fatty acid composition:

Examine the chromatograms obtained in the assay of stearic acid and palmitic acid.

The two principle peaks in the chromatogram obtained with the test solution are similar in retention time to the two principal peaks in the chromatogram obtained with the reference solution.

D. By Chemical test:

Take 1ml of Solution S, add 1ml of dilute ammonia; a white precipitate is formed that dissolves on addition 1ml of ammonium chloride solution. Add 1ml of 120g/L solution of disodium hydrogen phosphate dodecahydrate; a white crystalline precipitate is formed.

4. ACIDITY OR ALKALINITY:

To 1.0g of sample, add 20ml of carbon dioxide free water and boil for 1 minute with continuous shaking. Cool and filter. To 10ml of filtrate add 0.05ml of bromothymol blue solution. Not more than 0.05ml of 0.1M hydrochloric acid or 0.1M sodium hydroxide is required to change the colour of the indicator.

5. CHLORIDES: < REFER GAM 008>

Not more than 0.1%.

Dilute 10.0mL of Solution S to 40mL with water. Neutralise if necessary with nitric acid using litmus as indicator. Add 1mL of nitric acid and 1mL of 0.1M silver nitrate and dilute to 50mL with water. Mix and allow to stand for 5 min protected from light. The turbidity, if any, is not greater than that produced in a solution containing 1.4mL of 0.02M hydrochloric acid.

6. SULFATES: < REFER GAM 009>

Not more than 1.0%.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Signature			
Date	12/03/2024	13/03/2024	13/03/2024

Dilute 6.0 ml of Solution S to 40.0 ml with water. Neutralise if necessary with hydrochloric acid using litmus as indicator. Add 1mL of 3M hydrochloric acid and 3mL of a 120g/L solution of barium chloride and dilute to 50mL with water. Mix and allow to stand for 10 min. The turbidity, if any, is not greater than that produced in a solution containing 3.0mL of 0.02M sulfuric acid.

7. CADMIUM: (ATOMIC ABSORPTION SPECTROMETRY)

Not more than 3ppm.

For the preparation of all aqueous solutions and for the rinsing of glassware before use, employ water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin before use. Select all reagents to have as low a content of cadmium, lead and nickel as practicable and store all reagent solutions in containers of borosilicate glass. Clean glassware before use by soaking in warm 8M nitric acid for 30 min and by rinsing with deionised water.

Blank solution:

Dilute 25mL of cadmium and lead-free nitric acid to 100.0 mL with water.

Modifier solution:

Dissolve 20g of ammonium dihydrogen phosphate and 1g of magnesium nitrate in water and dilute to 100mL with the same solvent. Alternatively, use an appropriate matrix modifier as recommended by the graphite furnace atomic absorption (GFAA) spectrometer manufacturer.

Test solution:

Place 0.100g of the substance to be examined in a polytetrafluoroethylene digestion bomb and add 2.5mL of cadmium- and lead-free nitric acid. Close and seal the bomb according to the manufacturer's operating instructions (when using a digestion bomb, be thoroughly familiar with the safety and operating instructions. Carefully follow the bomb manufacturer's instructions regarding care and maintenance of these digestion bombs. Do not use metal jacketed bombs or liners which have been used with hydrochloric acid due to contamination from corrosion of the metal jacket by hydrochloric acid). Heat the bomb in an oven at 170°C for 3 h. Cool the bomb slowly in air to room temperature according to the bomb manufacturer's instructions.

Place the bomb in a fume cupboard and open carefully as corrosive gases may be expelled. Dissolve the residue in water R and dilute to 10.0 mL with the same solvent.

Reference solution:

Prepare a solution of 0.0030 µg/mL of Cd by suitable dilutions of a 0.00825 µg/mL solution of cadmium nitrate tetrahydrate in the blank solution.

Dilute 1.0mL of the test solution to 10.0mL with the blank solution. Prepare mixtures of this solution, the reference solution and the blank solution in the following proportions: (1.0:0:1.0 V/V/V), (1.0:0.5:0.5 V/V/V), (1.0:1.0:0 V/V/V).

To each mixture add 50µL of modifier solution and mix. These solutions contain respectively 0µg, 0.00075µg and 0.0015µg of cadmium per millilitre from the reference solution (keep the remaining test solution for use in the test for lead and nickel).

Source: Cadmium hollow-cathode lamp.

Wavelength: 228.8 nm.

Atomisation device: Furnace.

Platform: Pyrolytically coated with integrated tube.

Operating conditions:

Use the temperature programme recommended for cadmium by the GFAA spectrometer manufacturer. An example of temperature parameters for GFAA analysis of cadmium is shown below.

Stage	Final temperature (°C)	Ramp time (s)	Hold time (s)
Drying	110	10	20
Ashing	600	10	30
Atomisation	1800	0	5

8. LEAD: (ATOMIC ABSORPTION SPECTROMETRY)

Not more than 10 ppm.

For the preparation of all aqueous solutions and for the rinsing of glassware before use, employ water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin before use.

Select all reagents to have as low a content of cadmium, lead and nickel as practicable and store all reagent solutions in containers of borosilicate glass. Clean glassware before use by soaking in warm 8M nitric acid for 30 min and by rinsing with deionised water.

Blank solution:

Use the solution described in the test for cadmium.

Modifier solution:

Use the solution described in the test for cadmium.

Test solution:

Use the solution described in the test for cadmium.

Reference solution:

Prepare a solution of 0.100 µg/mL of Pb by suitable dilutions of lead standard solution (100 ppm Pb) R with the blank solution.

Prepare mixtures of the test solution, the reference solution and the blank solution in the following proportions: (1.0:0:1.0 V/V/V), (1.0:0.5:0.5 V/V/V), (1.0:1.0:0 V/V/V). To each mixture add 50 µL of modifier solution and mix. These solutions contain respectively 0 µg, 0.025 µg and 0.05 µg of lead per millilitre from the reference solution.

Source: Lead hollow-cathode lamp.

Wavelength: 283.3 nm.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12\03\2024	13\03\2024	13\03\2024

Atomisation device: Furnace.

Platform: Pyrolytically coated with integrated tube.

Operating conditions:

Use the temperature programme recommended for lead by the GFAA spectrometer manufacturer. An example of temperature parameters for GFAA analysis of lead is shown below.

Stage	Final temperature (°C)	Ramp time (s)	Hold time (s)
Drying	110	10	20
Ashing	450	10	30
Atomisation	2000	0	5

9. NICKEL: (ATOMIC ABSORPTION SPECTROMETRY)

Not more than 5 ppm.

For the preparation of all aqueous solutions and for the rinsing of glassware before use, employ water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin before use. Select all reagents to have as low a content of cadmium, lead and nickel as practicable and store all reagent solutions in containers of borosilicate glass. Clean glassware before use by soaking in warm 8M nitric acid for 30 min and by rinsing with deionised water.

Blank solution:

Use the solution described in the test for cadmium.

Modifier solution:

Dissolve 20g of ammonium dihydrogen phosphate in water R and dilute to 100mL with the same solvent.

Alternatively, use an appropriate matrix modifier as recommended by the GFAA spectrometer manufacturer.

Test solution:

Use the solution described in the test for cadmium.

Reference solution:

Prepare a solution of 0.050 µg/mL of Ni by suitable dilutions of a 0.2477 µg/mL solution of nickel nitrate hexahydrate in the blank solution.

Prepare mixtures of the test solution, the reference solution and the blank solution in the following proportions: (1.0:0:1.0 V/V/V), (1.0:0.5:0.5 V/V/V), (1.0:1.0:0 V/V/V). To each mixture add 50 µL of matrix modifier solution and mix. These reference solutions contain respectively 0 µg, 0.0125 µg and 0.025 µg of nickel per millilitre from the reference solution.

Source: Nickel hollow-cathode lamp.

Wavelength: 232.0 nm.

Atomisation device: Furnace.

Platform: Pyrolytically coated with integrated tube.

Operating conditions:

Use the temperature programme recommended for nickel by the GFAA spectrometer manufacturer. An example of temperature parameters for GFAA analysis of nickel is shown below.

Stage	Final temperature (°C)	Ramp time (s)	Hold time (s)
Drying	110	10	20
Ashing	1000	20	30
Atomisation	2300	0	5

10. LOSS ON DRYING: < REFER GAM 026>

Not more than 6.0 per cent, determined on 1.0g by drying in an oven at 105°C.

11. ASSAY:

Magnesium:

Weigh 0.5g of sample in a 250ml conical flask, add 50ml of a mixture of equal volumes of anhydrous ethanol and butanol, 5ml of concentrated ammonia, 3ml of ammonium chloride buffer solution pH 10.0, 30.0ml of 0.1M sodium edetate and 15mg of mordant black II triturate. Heat at 45°C to 50°C until the solution is clear and titrate with 0.1M zinc sulphate until the colour changes from blue to violet. Carry out a blank titration.

1ml of 0.1 M sodium edetate is equivalent to 2.431 g of Mg.

Calculation:

$$= \frac{\text{Titer value-Blank value} \times \text{Molarity of 0.1M disodium edetate} \times 2.431 \times 100 \times 100}{0.1 \times \text{Sample weight in mg} \times (100 - \text{Sample LOD})}$$

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12/03/2024	13/03/2024	13/03/2024

12. STEARIC ACID AND PALMITIC ACID:

Determine by gas chromatography:

Test solution:

In a conical flask fitted with a reflux condenser, dissolve 0.10g of the substance to be examined in 5mL of boron trifluoride-methanol solution. Boil under a reflux condenser for 10 min. Add 4 mL of heptane through the condenser and boil again under a reflux condenser for 10 min. Allow to cool. Add 20 mL of saturated sodium chloride solution. Shake and allow the layers to separate. Dry the organic layer over 0.1g of anhydrous sodium sulfate (previously washed with heptane). Dilute 1.0mL of the solution to 10.0mL with heptane.

Reference solution:

Prepare the reference solution in the same manner as the test solution using 50.0 mg of palmitic acid CRS and 50.0mg of stearic acid CRS instead of the substance to be examined.

Chromatographic conditions:

Material	:	Fused silica;
Size	:	l = 30 m, Ø = 0.32 mm;
Stationary phase	:	Macrogol 20,000 (film thickness 0.5 µm).
Carrier gas	:	Helium for chromatography.
Flow rate	:	2.4 mL/min.
Detection	:	Flame ionisation.
Injection	:	1 µL.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 2	70
	2 - 36	70 → 240
	36 - 41	240
Injection port		220
Detector		260

Relative retention:

With reference to methyl stearate: methyl palmitate = about 0.9.

System suitability: Reference solution.

Resolution: Minimum 5.0 between the peaks due to methyl palmitate and methyl stearate;

Relative standard deviation:

Maximum 3.0 per cent for the areas of the peaks due to methyl palmitate and methyl stearate, determined on 6 injections; maximum 1.0 per cent for the ratio of the areas of the peaks due to methyl palmitate to the areas of the peaks due to methyl stearate, determined on 6 injections.

13. MICROBIAL CONTAMINATION:

Total Viable aerobic count and Pathogen test refer as per the current SOP No: ST/MB/011.

REVISION HISTORY:

STP No.	Reason for Review	Change control No.	Effective Date
STP-RMEM0033-00	(i) STP numbering procedure revised as per SOP No. ST/QC/058. (ii) Testing procedure for acid value has been incorporated in the identification test.	ST/CC/23/063 ST/CC/24/067	14/03/2024

**** END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Dy. Manager-QC	GM-QC	AGM-QA
Signature			
Date	12/03/2024	13/03/2024	13/03/2024

	Safetab Life Science Puducherry			
	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	MAIZE STARCH BP			
Specification No.	RMESM0034-01	**Revision No.**	01	**Item Code.:** RMEM0034
Supersedes	RMESM0034-00	**Effective Date**	11/01/2023	**Page No.:** 1 of 4

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	NA
2	Molecular weight	NA
3	Storage conditions	Store protected from moisture.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	50 g
6	Quantity of reserve sample	100 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	MAIZE STARCH BP			
Specification No.	RMESM0034-01	**Revision No.**	01	**Item Code.:** RMEM0034
Supersedes	RMESM0034-00	**Effective Date**	11/01/2023	**Page No.:** 2 of 4

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White to slightly yellowish, very fine powder that creaks when pressed between the fingers.
2.	*Solubility	Practically insoluble in cold water and in ethanol (96%).
3.	*Identification **A.** Microscopic **B.** By Chemical test **C.** By Chemical test	 **A.** The starch granules show a distinct black cross intersecting at hilum. **B.** A Thin, cloudy mucilage is formed. **C.** An Orange-red to dark blue color is produced, which disappears on heating.
4.	*pH	Between 4.0 and 7.0
5.	Foreign matters	No starch grains of any other origin are present.
6.	Oxidising substances	Not more than 20ppm.
7.	Sulphur dioxide	Not more than 50ppm
8.	Iron	Not more than 10ppm

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	MAIZE STARCH BP			
Specification No.	RMESM0034-01	**Revision No.**	01	**Item Code.:** RMEM0034
Supersedes	RMESM0034-00	**Effective Date**	11/01/2023	**Page No.:** 3 of 4

S.NO	TEST (s)	SPECIFICATION (s)
9.	Sulphated ash	Not more than 0.6% w/w.
10.	*Loss on drying	Not more than 15.0% w/w.
11.	*Microbial contamination (i) Total aerobic microbial count (ii) Total yeast and mold count iii) Escherichia coli iv) Salmonella species	 Not more than 1000 cfu/g Not more than 100 cfu/g Should be absent Should be absent

Remarks: The above * Marked tests are to be performed while retesting the material.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

REVISION HISTORY:

Specification No.	Effective Date	Reason for Review
RMESM0034-00	11-01-2020	New specification prepared
RMESM0034-01	11/01/2023	Periodic Review.

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

	Safetab Life Science Puducherry			
	STANDARD TESTING PROCEDURE			MASTER COPY
Name of Product	MAIZE STARCH BP			
STP No.	RMETM0034-01	**Revision No.**	01	**Item Code.:** RMEM0034
Supersedes	RMETM0034-00	**Effective Date**	11/01/2023	**Page No.:** 1 of 4

1. DESCRIPTION: < REFER GAM 001>

White to slightly yellowish, very fine powder that creaks when pressed between the fingers.

2. SOLUBILITY: < REFER GAM 002>

1mg of sample + 10mL of Cold water	Practically insoluble if the material does not dissolve.
1mg of sample + 10mL of ethanol (96%)	Practically insoluble if the material does not dissolve.

3. IDENTIFICATION: < REFER GAM 003>

A. Microscopic:

In a mixture of equal volumes of glycerol and water (1:1), It appears as either angular polyhedral granules of irregular sizes with diameters ranging from about 2 µm to about 23 µm or as rounded or spheroidal granules of irregular sizes with diameters ranging from about 25 µm to about 35 µm. The central hilum consists of a distinct cavity or 2- to 5-rayed cleft and there are no concentric striations. Between orthogonally orientated polarising plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.

B. By Chemical test:

Disperse 1.0g of sample dissolved in 50ml of water boil for 1 minutes and cool. A thin, cloudy mucilage is formed.

C. By Chemical test:

To 1 mL of the mucilage obtained in identification test B add 0.05 mL of iodine solution. An orange-red to dark blue colour is produced, which disappears on heating.

4. pH: < REFER GAM 030>

Weigh about 5.0g of sample add 25ml water. Agitate continuously at a moderate rate for 60 s. Stop the agitation and allow to stand for 15 min.

Particulars	**PREPARED BY**	**REVIEWED BY**	**APPROVED BY**
Name	**K.SARAVANAN**	**M.VIJAYAKUMAR**	**A.G.KANNAN**
Designation	**Asst. Manager-QC**	**AGM-QC**	**GM-QA**
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

Rinse the electrodes with distilled water and wipe dry with tissue paper. Set the instrument using buffer solution pH 6.87 by following instrument Operating Procedure. Clean the electrode. Immerse the electrode in the solution being examined and measure the pH.

5. FOREIGN MATTER:

Examined under a microscope using a mixture of equal volumes of glycerol and water (1:1), not more than traces of matter other than starch granules are present. No starch grains of any other origin are present.

6. OXIDISING SUBSTANCES:

Weigh accurately about 4.0g of sample to a glass-stoppered, 125ml conical flask and add 50.0ml of water. Insert the stopper and swirl for 5 minutes. Transfer to a glass-stoppered 50ml centrifuge tube and centrifuge to clarify. Transfer 30.0ml of the clear supernatant liquid to a glass-stoppered 125-ml conical flask. Add 1ml of glacial acetic acid and 0.5g to 1.0g of potassium iodide. Insert the stopper, swirl, and allow to stand for 30 minutes in the dark. Add 1ml of starch solution and titrate with 0.002M sodium thiosulphate until the starch-iodine colour disappears. Carry out a blank titration. Not more than 1.4ml of 0. 002M sodium thiosulphate is required (0.002 per cent, calculated as H_2O_2).

1 ml of 0.002M sodium thiosulphate is equivalent to 0.034mg of oxidising substances, calculated as H_2O_2.

Calculation:

$$= \frac{\text{Titer value} \times \text{Molarity of 0.002M sodium thiosulphate} \times 0.034 \times 100}{\text{Sample weight in mg} \times 0.002}$$

7. SULPHUR DIOXIDE:

Add 150 mL of water to the boiling flask. Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide at a rate of 100±5 mL per minute through the apparatus. Start the condenser coolant flow. Add 10mL of hydrogen peroxide solution to a receiving test tube. After 15 minutes without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask. Weigh 25.0g of sample into the boiling flask add 100mL of water. Apply stopcock greased to the outer joint of the separatory funnel.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

And replace the separatory funnel in the boiling flask.

Weigh 25.0g of sample into the boiling flask add 100mL of water. Apply stopcock greased to the outer joint of the separatory funnel. And replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80mL of dilute hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 hour. Remove the receiving test tube, and transfer to a 200mL wide necked conical flask. Rinse the receiving tube with small portion of water, and transfer the rinsing to the 200 mL conical flask, and mix. Heat on a water bath for 15mins and cool. Add 0.1 mL of bromophenol blue indicator solution. Titrate the contents with 0.1N sodium hydroxide VS until the color changes from yellow to violet blue, with the color change lasting for atleast 20 seconds. Perform a blank determination and make any necessary correction.

1 ml of 0.1N sodium hydroxide is equivalent to 32.03 mg of SO_2.

8. IRON: < REFER GAM 007>

Dissolve 1.5g of sample in 15ml of dilute hydrochloric acid, filter. This solution complies with the limit test for iron (NMT 10ppm).

9. SULPHATED ASH: < REFER GAM 032>

Not more than 0.6% w/w, Determined on 1.0g of sample.

10. LOSS ON DRYING: < REFER GAM 026>

Not more than 15.0% w/w, Determined on 1.0g of sample by drying in an oven at 130°C for 90 minutes.

11. MICROBIAL CONTAMINATION:

Use 11.0g of sample for Total Microbial count and pathogen test.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

Total Aerobic microbial count:

Procedure: Proceed as per the current general testing procedure GAM-035.

Total Yeast and mold count:

Procedure: Proceed as per the current general testing procedure GAM-036.

Escherichia Coli:

Procedure: Proceed as per the current general testing procedure GAM-037.

Salmonella species:

Procedure: Proceed as per the current general testing procedure GAM-038.

REVISION HISTORY:

STP No.	Effective Date	Reason for Review
RMETM0034-00	11-01-2020	New STP Prepared.
RMETM0034-00	11/01/2023	Periodic review.

****END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	07/01/2023	09/01/2023	10/01/2023

	Safetab Life Science Puducherry			
	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	PARACETAMOL BP			
Specification No.	SPEC-RMAP0030-02	**Revision No.**	02	**Item Code.:** RMAP0030
Supersedes	RMASP0030-01	**Effective Date**	08/06/2023	**Page No.:** 1 of 3

S.NO	RAW MATERIAL GENERAL SPECIFICATION (S)	
1	Molecular formula	$C_8H_9NO_2$
2	Molecular weight	151.2
3	Storage conditions	Protected from light.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	3 g
6	Quantity of reserve sample	6 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			QUALITY ASSURANCE
Date	06/06/2023	07/06/2023	07/06/2023

	Safetab Life Science Puducherry			
	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	PARACETAMOL BP			
Specification No.	SPEC-RMAP0030-02	**Revision No.**	02	**Item Code.:** RMAP0030
Supersedes	RMASP0030-01	**Effective Date**	08/06/2023	**Page No.:** 2 of 3

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or almost white, crystalline powder.
2.	*Solubility	Sparingly soluble in water, freely soluble in ethanol (96 per cent), very slightly soluble in methylene chloride.
3.	*Identification **A.** By Melting point **B.** By IR	 **Result A:** 168°C to 172°C. **Result B:** Not greater than 2°C The Infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Paracetamol RS.
4.	*Related substances (By HPLC) (i) Impurity K (ii) Impurity J (iii) Unspecified impurity (iv) Total impurities	 Not more than 50 ppm Not more than 10 ppm Not more than 0.05% Not more than 0.2%
5.	Sulphated Ash	Not more than 0.1% w/w
6.	*Loss on drying	Not more than 0.5% w/w

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	06/06/2023	07/06/2023	07/06/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	PARACETAMOL BP			
Specification No.	SPEC-RMAP0030-02	**Revision No.**	02	**Item Code.:** RMAP0030
Supersedes	RMASP0030-01	**Effective Date**	08/06/2023	**Page No.:** 3 of 3

S.NO	TEST (s)	SPECIFICATION (s)
7.	*Assay By Titration (On dried basis)	Not less than 99.0% and not more than 101.0% w/w.

Remarks: The above * Marked tests are to be performed while retesting the material.

REVISION HISTORY:

Specification No.	Reason for Review	Change control No.	Effective Date
SPEC-RMAP0030-02	(i) There is no changes in Specification. (ii) Specification number has been changed as per ERP. This changes captured as per change control number.	ST/CC/23/063	08/06/2023

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	06/06/2023	07/06/2023	07/06/2023

Safetab Life Science Puducherry-107 QUALITY ASSURANCE

UNCONTROLLED COPY

1. DESCRIPTION: < REFER GAM 001>

White or almost white, crystalline powder.

2. SOLUBILITY: < REFER GAM 002>

100mg of sample + 10mL of Water	Sparingly soluble if the material dissolves.
100mg of sample + 1mL of Ethanol (96%)	Freely soluble if the material dissolves.
10mg of sample + 100mL of methylene chloride	Very slightly soluble if the material dissolves.

3. IDENTIFICATION: < REFER GAM 003>

First identification: B.

Second identification: A.

A. By Melting point:

Determination A:

Determine the melting point of the substance to be examined.

Result A: 168 °C to 172 °C.

Determination B:

Mix equal parts of the substance to be examined and Paracetamol RS and determine the melting point of the mixture.

Result B: The absolute difference between the melting point of the mixture and the value obtained in determination A is not greater than 2°C.

B. By IR:

The Infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Paracetamol RS.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	06/06/2023	07/06/2023	07/06/2023

4. RELATED SUBSTANCES: (BY HPLC)

Chemicals/Reagents/Standards:

Paracetamol Impurity K	:	Reference standard
Paracetamol Impurity J	:	Reference standard
Potassium dihydrogen phosphate	:	AR grade
Dipotassium hydrogen phosphate	:	AR grade
Purified Water	:	Milli-Q water (or) equivalent
Methanol	:	HPLC grade

Chromatographic Conditions:

Column	:	150mm x 4.6mm, end-capped solid core Octadecylsilyl silica, (5µm).
Column Temperature	:	30°C
Auto sampler temperature	:	5°C
Flow Rate	:	1.5ml/min
Wavelength	:	254nm
Injection volume	:	50µl
Retention time	:	Retention time of Paracetamol peak is at about 4.0 minutes

Mobile phase A:

Dissolve 1.7g of Potassium dihydrogen phosphate and 1.8 g of Dipotassium hydrogen phosphate in water for chromatography and dilute to 1000 mL with the same solvent.

Mobile phase B: Methanol

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	K.JAYARAJ
Designation	Asst. Manager-QC	AGM-QC	Asst. Manager-QA
Signature			
Date	06/06/2023	07/06/2023	07/06/2023

Safetab Life Science QUALITY ASSURANCE Puducherry-607

Gradient program:

Time (min)	Mobile phase A (per cent V/V)	Mobile phase B (per cent V/V)
0 – 1.5	95	5
1.5 – 14.4	95 → 90	5 → 10
14.4 – 28.8	90	10
28.8 – 57.6	90 → 66	10 → 34
57.6 - 60	66	34

Solvent mixture:

A mixture of 15 volumes of Methanol and 85 volumes of water.

Test solution:

Weigh accurately and dissolve about 50.0mg of the substance to be examined in 0.75ml of methanol and dilute to 5.0ml with water.

Reference solution (a):

Dilute 1.0 mL of the test solution to 100.0 mL with the solvent mixture. Dilute 1.0 mL of this solution to 20.0 mL with the solvent mixture.

Reference solution (b):

Dissolve 5.0mg of Paracetamol impurity J RS in 25ml of methanol and dilute to 250.0 mL with the solvent mixture. Dilute 1.0 mL of the solution to 200.0 mL with the solvent mixture.

Reference solution (c):

Weigh accurately about 5.0mg of Paracetamol impurity K RS in the solvent mixture and dilute to 100.0 mL with the solvent mixture. Dilute 1.0 mL of the solution to 10.0 mL with the solvent mixture.

Reference solution (d):

Dilute 1.0 mL of the reference solution (c) to 10.0 mL with the solvent mixture.

Reference solution (e):

Mix 1.0 mL of the reference solution (a) and 1ml of reference solution (c) and dilute to 10.0 mL with the solvent mixture.

Procedure:

Identification of impurities Use the chromatogram obtained with reference solution (b) to identify the peak due to impurity J; use the chromatogram obtained with reference solution (d) to identify the peak due to impurity K.

Relative retention With reference to Paracetamol (retention time = about 4 min): impurity K = about 0.4; impurity J = about 10.1.

System suitability Reference solution (e):

— **Resolution:** minimum 5.0 between the peaks due to impurity K and Paracetamol.

Calculation of percentage contents:

— for impurity J, use the concentration of impurity J in reference solution (b);

— for impurity K, use the concentration of impurity K in reference solution (d);

— for impurities other than J and K, use the concentration of Paracetamol in reference solution (a).

Limits:

— impurity K: maximum 50 ppm;

— impurity J: maximum 10 ppm;

— unspecified impurities: for each impurity, maximum 0.05 per cent;

— total: maximum 0.2 per cent;

— reporting threshold: 0.03 per cent, except for impurities J and K.

Inject 50µl of the above solution as per following sequence.

Injection sequence:

S. No	Sample Name	No. of injections
1	Solvent mixture (Blank)	1
2	System suitability (Reference solution (e))	1
3	Reference solution (b)	1
4	Reference solution (d)	1
5	Reference solution (a)	1
6	Blank	1
7	Test solution	1

Calculations:

Impurity K : (NMT 50ppm)

$$= \frac{AT_K}{AS_K} \times \frac{RS}{100} \times \frac{1}{10} \times \frac{1}{10} \times \frac{5}{WT} \times \frac{P_K}{100} \times 1000 \times 1000$$

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Where,

AT_K = Area of Impurity K peak in Test solution.

AS_K = Area of the Impurity K peak in the Reference solution (d)

RS = Weight of the Impurity K Reference standard in mg.

WT = Weight of the sample taken in mg.

P = Potency of Impurity K Reference standard in % (as such basis).

Impurity J : (NMT 10ppm)

$$= \frac{AT_J}{AS_J} \times \frac{RS}{250} \times \frac{1}{200} \times \frac{5}{WT} \times \frac{P_J}{100} \times 1000 \times 1000$$

Where,

AT_J = Area of Impurity J peak in Test solution.

AS_J = Area of the Impurity J peak in the Reference solution (b)

RS = Weight of the Impurity J Reference standard in mg.

WT = Weight of the sample taken in mg.

P = Potency of Impurity J Reference standard in % (as such basis).

Unspecified impurity: (NMT 0.05%)

$$= \frac{AT_I}{AS} \times \frac{WT}{5} \times \frac{1}{100} \times \frac{1}{20} \times \frac{5}{WT} \times 100$$

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Where,

AT_I = Area of Unspecified impurity peak in Test solution.

AS = Area of the principal peak in the Reference solution (a)

WT = Weight of the sample taken in mg.

Total impurities: (NMT 0.2%)

$$= \frac{AT_T}{AS} \times \frac{WT}{5} \times \frac{1}{100} \times \frac{1}{20} \times \frac{5}{WT} \times 100$$

Where,

AT_T = Area of Total impurities peak in Test solution.

AS = Area of the principal peak in the Reference solution (a)

WT = Weight of the Test solution in mg.

5. SULPHATED ASH: < REFER GAM 032>

Not more than 0.1% w/w. Determine on 1.0g of sample.

6. LOSS ON DRYING: < REFER GAM 026>

Not more than 0.5% w/w, determined on 1.0g of sample by drying in an oven at 105°C.

7. ASSAY: (By Titration)

Weigh accurately about 0.300g of sample in a mixture of 10mL of water and 30mL of dilute sulfuric acid. Boil under a reflux condenser for 1 h, cool and dilute to 100.0mL with water. To 20.0mL of the solution add 40mL of water, 40g of ice, 15mL of dilute hydrochloric acid and 0.1mL of ferroin. Titrate with 0.1M cerium sulfate until a greenish-yellow colour is obtained. Carry out a blank titration

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Safetab Life Science QUALITY ASSURANCE Puducherry-107

1ml of 0.1M Cerium sulphate is equivalent to 0.00756g of Paracetamol.

Calculation:

$$= \frac{\text{Blank-Titer value} \times \text{Molarity of 0.1M Cerium sulphate} \times 0.00756 \times 100 \times 100 \times 100}{20 \times \text{Sample weight in (g)} \times (100 - \text{Sample LOD}) \times 0.1}$$

REVISION HISTORY:

STP No.	Reason for Review	Change control No.	Effective Date
STP-RMAP0030-02	(i) Related substance test procedure has been changed as per BP 2023 (ii) STP numbering procedure revised as per SOP No. ST/QC/058.	ST/CC/23/084 ST/CC/23/063	08/06/2023

****END OF THE DOCUMENT****

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	RAW MATERIAL SPECIFICATION MASTER COPY			
Name of Product	POVIDONE K 90 BP (BOVIDONE K 90)			
Specification No.	SPEC-RMEP0045-00	**Revision No.**	00	**Item Code.:** RMEP0045
Supersedes	RMESP0045-00	**Effective Date**	25/08/2023	**Page No.:** 1 of 4

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	$(C_6H_9NO)_n$
2	Molecular weight	NA
3	Storage conditions	Store in well-closed, air-tight containers.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	25 g
6	Quantity of reserve sample	50 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

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	RAW MATERIAL SPECIFICATION			
Name of Product	POVIDONE K 90 BP (BOVIDONE K 90)			
Specification No.	SPEC-RMEP0045-00	**Revision No.**	00	**Item Code.:** RMEP0045
Supersedes	RMESP0045-00	**Effective Date**	25/08/2023	**Page No.:** 2 of 4

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or yellowish-white, hygroscopic powder or flakes.
2.	*Solubility	Freely soluble in water, in ethanol (96%) and in methanol, very slightly soluble in acetone.
3.	*Identification **A.** By IR **B.** By Chemical test **C.** By Chemical test **D.** By Chemical test	 The infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Povidone K 25. A pink colour is produced. A red colour is produced. The substance dissolves.
4.	Appearance of solution	Solution S is clear, and not more intensely coloured than reference solution B_6, BY_6 or R_6.
5.	*pH	Between 4.0 to 7.0
6.	Viscosity (Expressed as K- value)	Between 81.0 to 96.3
7.	Aldehydes (Expressed as Acetaldehyde)	Not more than 500ppm

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	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	POVIDONE K 90 BP (BOVIDONE K 90)			
Specification No.	SPEC-RMEP0045-00	**Revision No.**	00	**Item Code.:** RMEP0045
Supersedes	RMESP0045-00	**Effective Date**	25/08/2023	**Page No.:** 3 of 4

S.NO	TEST (s)	SPECIFICATION (s)
8.	Peroxides	Not more than 400 ppm
9.	Formic Acid	Not more than 0.5%
10.	Limit of Hydrazine	Not more than 1ppm.
11.	Impurity A	Not more than 10ppm.
12.	Impurity B	Not more than 3.0%
13.	*Water content (By KFR)	Not more than 5.0%
14.	Sulphated ash	Not more than 0.1%
15.	*Assay on anhydrous basis	Not less than 11.5% and not more than 12.8% of Nitrogen content.

Remarks: The above * Marked tests are to be performed while retesting the material.

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REVISION HISTORY:

Specification No.	Reason for Review	Change control No.	Effective Date
SPEC-RMEP0045-00	(i) Periodic review. (ii) Specification numbering procedure revised as per SOP No. ST/QC/058.	ST/CC/23/016	25/08/2023

**** END OF THE DOCUMENT ****

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	STANDARD TESTING PROCEDURE			
Name of Product	POVIDONE K 90 BP (BOVIDONE K 90)			
STP No.	STP-RMEP0045-00	**Revision No.**	00	**Item Code.:** RMEP0045
Supersedes	RMETP0045-00	**Effective Date**	25/08/2023	**Page No.:** 1 of 12

1. DESCRIPTION: < REFER GAM 001>

White or yellowish-white, hygroscopic powder or flakes.

2. SOLUBILITY: < REFER GAM 002>

100mg of sample + 1ml of Water	Freely soluble if the material dissolves.
100mg of sample + 1ml of Ethanol (96%)	Freely soluble if the material dissolves.
100mg of sample + 1ml of Methanol	Freely soluble if the material dissolves.
10mg of sample + 100ml of Acetone	Very slightly soluble if the material dissolves.

3. IDENTIFICATION: < REFER GAM 003>

First identification test: A and D.

Second identification test: B,C and D

Solution S1: Dissolve 2.5g of sample in carbon dioxide-free water and dilute to 25ml with the same solvent. Add the substance to be examined to the water in small portions, stirring using a magnetic stirrer. **Note:** Solution S1 will be used in Identification B & C tests.

A. By IR:

Previously dried at 105°C for 6 hour.

The infrared absorption spectrum of sample should be concordant with that of reference spectrum or with the spectrum obtained from Povidone K 25 WS.

B. By Chemical test:

To 1ml of Solution S1, add 0.2ml of dimethylaminobenzaldehyde solution and 0.1ml of sulfuric acid. A pink colour is produced.

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C. By Chemical test:

To 0.1ml of Solution S1, add 5ml of water and 0.2ml of 0.05M iodine. A red colour is produced.

D. By Chemical test:

Weigh about 0.5g of sample dissolved in 10ml of water and shake. The substances dissolves.

SOLUTION S:

Dissolve 1.0g of sample in carbon dioxide free water and dilute to 20ml with the same solvent. Add the substance to be examined to the water in small portions, stirring using a magnetic stirrer.

4. APPEARANCE OF SOLUTION:

Solution S is clear and not more intensely coloured than reference solution B_6, BY_6 or R_6.

5. pH: < REFER GAM 030>

Between 4.0 to 7.0 for Solution S.

6. VISCOSITY (EXPRESSED AS K-VALUE):

Procedure:

Dissolve a 1.0 g of substance in 100 ml of water. Allow to stand for 1 hr. and determine the viscosity of the solution at 25°C, using viscometer no.1 with a minimum flow time of 100 s.

The time required for the level of the liquid to drop from one mark to the other is measured with a stop-watch to the nearest one-fifth of a second. The result is valid only if two consecutive readings do not differ by more than 1 per cent. The average of not fewer than three readings gives the flow time of the liquid to be examined.

Calculate the K-value using the following expression:

$$1.5\log v_{rel} - 10.15 + 0.003c + 300c\log v_{rel} + (c + 1.5c\log v_{rel})2\sqrt{0.15c + 0.003c^2}$$

Where,

c = concentration of the substance to be examined (anhydrous substance, in grams per 100 ml.

v_{rel} = kinematic viscosity of the solution relative to that of water.

Acceptance criteria:

Between 81.0 to 96.3.

7. ALDEHYDES:

Test solution:

Dissolve a quantity of the substance to be examined equivalent to 1.0g of the anhydrous substance to be examined in phosphate buffer solution pH 9.0 and dilute to 100.0ml with the same solvent. Stopper the flask tightly and heat at 60°C for 1 hrs. Allow to cool to room temperature.

Reference solution:

Dissolve 0.140g of acetaldehyde ammonia trimer trihydrate in water and dilute to 200.0ml with the same solvent. Dilute 1.0ml of this solution to 100.0ml with phosphate buffer solution pH 9.0.

Into 3 identical spectrophotometric cells with a path length of 1 cm, introduce separately 0.5 ml of the test solution, 0.5 ml of the reference solution and 0.5 ml of water (blank). To each cell, add 2.5 ml of phosphate buffer solution pH 9.0 and 0.2 ml of nicotinamide-adenine dinucleotide solution. Mix and stopper tightly. Allow to stand at 22 ± 2°C for 5 min. measure the absorbance of each solution at 340 nm using water as the compensation liquid. To each cell add 0.05 mL of aldehyde dehydrogenase solution, mix and stopper tightly. Allow to stand at 22 ± 2 °C for 5 min. Measure the absorbance of each solution at 340 nm using water as the compensation liquid.

Calculate the content of using the following expression:

$$= \frac{(A_{t2} - A_{t1}) - (A_{b2} - A_{b1})}{(A_{s2} - A_{s1}) - (A_{b2} - A_{b1})} \times 100000 \times Cm$$

Where,

A_{t1} = absorbance of the test solution before the addition of aldehyde dehydrogenase.
A_{t2} = absorbance of the test solution after the addition of aldehyde dehydrogenase.
A_{s1} = absorbance of the reference solution before the addition of aldehyde dehydrogenase.
A_{s2} = absorbance of the reference solution after the addition of aldehyde dehydrogenase.
A_{b1} = absorbance of the blank solution before the addition of aldehyde dehydrogenase.
A_{b2} = absorbance of the blank solution after the addition of aldehyde dehydrogenase.
m = mass of the substance to be examined (anhydrous substance) in the test solution, in grams
C = concentration of acetaldehyde in the reference solution, calculated from the weight of the acetaldehyde ammonia trimer trihydrate with the factor 0.72, in milligrams per millimeter.

Acceptance criteria: Not more than 500ppm, expressed as acetaldehyde.

8. PEROXIDES:

Dissolve a quantity of the substance to be examined equivalent to 4.0g of the anhydrous substance in 100ml of water. To 25ml of this solution, add 2ml of titanium trichloride-sulfuric acid reagent. Allow to stand for 30 min. The absorbance of the solution, measured at 405 nm using a mixture of 25ml of the stock solution and 2ml of a 13% V/V solution of sulfuric acid as the compensation liquid, is not greater than 0.35.

9. FORMIC ACID: (By HPLC)

Chromatographic Condition:

Columns : 30-cm x 7.8-mm; 9-µm

Detector	:	UV 210 nm
Columns temperature	:	35°C
Flow rate	:	1.0 ml/min
Injection Volume	:	50µl
Retention time	:	About 8 minutes

Mobile phase:

Dilute 1.0ml of perchloric acid to 700ml with water.

Test solution:

Dissolve a quantity of the substance to examined equivalent to 2.0g of the anhydrous substance in water and dilute to 100.0ml with the same solvent (Test stock solution). Transfer a suspension of strongly acidic ion exchange resin for column chromatography in water to a to a column of about 0.8cm in internal diameter to give a packing of about 20 mm in length and keep the strongly acidic ion exchange resin layer constantly immersed in water. Pour 5ml of water and adjust the flow rate to about 1ml/min. When the level of the water comes down to near the top of the strongly acidic ion exchange resin layer, introduce the stock solution into the column.

Discard the first 2ml of the eluate, then collect 1.5ml of the solution and use this solution as the test solution.

Reference solution:

Dissolve 0.100g of anhydrous formic acid in water and dilute to 100ml with same solvent. Dilute 1.0ml of this solution to 100ml with water.

Suitability requirements:

Repeatability: maximum relative standard deviation of 2.0 per cent determined on 6 injections.

Column efficiency:

NLT 1000 theoretical plates for the formic acid peak

Symmetry factor:

0.5-1.5 for the formic acid for six injections

Calculate the percentage of formic acid using the following expression:

$$\text{Result} = A_1A_2 \times Mm$$

A_1 = Area of the peak due to formic acid in the chromatogram obtained with the Test solution.

A_2 = Area of the peak due to formic acid in the chromatogram obtained with the Reference solution.

m = Mass of the substance to be examined (anhydrous substances) in the test solution, in grams.

M = Mass of anhydrous formic acid in the reference solution, in grams.

Acceptance criteria:

Not more than 0.5%

10. HYDRAZINE: (By Thin-layer chromatography)

Chromatographic conditions:

Plate	:	TLC silanised silica gel plate. F_{254}.
Mobile phase	:	Water and methanol (1:2 V/V).
Application	:	10µl
Development	:	Over 3/4 of the plate.
Drying	:	In air

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	Detection	: Examine in ultraviolet light at 365 nm.
	Retardation factor	: salicylaldehyde azine = about 0.3

Note: Use freshly prepared solutions.

Test solution:

Dissolve a quantity of the substance to examined equivalent 2.5 g of the anhydrous substance in 25 ml of water. Add 0.5 ml of a 50g /L solution of salicylaldehyde in methanol, mix and heat in a water-bath at 60 °C for 15 min. Allow to cool, add 2.0 ml of toluene, shake for 2 min. and centrifuge. Use the upper layer of the mixture.

Reference solution:

Dissolve 90 mg of salicylaldehyde azine in toluene and dilute to 100 ml with the same solvent. Dilute 1 ml of the solution to 100 ml with toluene.

Limit:

Any spot corresponding to salicylaldehyde azine obtained with the test solution is not more intense than the spot obtained with the reference solution (Not more than 1ppm).

11. IMPURITY A: (By HPLC)

Chromatographic conditions:

Precolumn	:	Size: l = 10mm, Ø = 4.0 mm
Stationary phase	:	Base deactivated end capped octadecylsilyl silica gel for chromatography (5 µm).
Column	:	Size: l = 150mm, Ø = 4.6 mm
Stationary phase	:	Base deactivated end capped octadecylsilyl silica gel for chromatography (5 µm).
Temperature	:	40°C.
Detection	:	UV at 235 nm.

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Injection : 20µl.

Flow rate : 1.0ml /min.

Mobile phase : Acetonitrile, Water (10:90 V/V).

Test solution:

Dissolve a quantity of the substance to be examined equivalent to 0.250g of the anhydrous substance in the mobile phase and dilute to 10.0ml with the mobile phase.

Reference solution (a):

Dissolve 50mg of 1-vinylpyrrolidin-2-one in mobile phase and dilute to 100.0ml with the mobile phase. Dilute 1.0ml of the solution to 100.0ml with mobile phase. Dilute 5.0ml of this solution to 100.0ml with mobile phase.

Reference solution (b):

Dissolve 10mg of 1-vinylpyrrolidin-2-one and 0.5g of vinyl acetate in methanol and dilute to 100.0ml with the same solvent. Dilute 1.0ml of the solution to 100.0ml with mobile phase.

* **Note:** After injection of the test solution, wait for about 2 min and wash the precolumn by passing the mobile phase backwards, at the same flow rate applied in the test, for 30 min.

Relative retention time with reference to vinyl acetate (retention time = about 14 min.) impurity A = about 0.6 min.

System suitability:

In the chromatogram obtained with reference solution (b), Resolution: minimum 2.0 between the peaks due to impurity A and to vinyl acetate.

In the chromatogram obtained with reference solution (a), Repeatability: maximum relative standard deviation of 2.0 %.

In the chromatogram obtained with reference solution (a), the symmetry factor for peak due to 1-vinylpyrrolidin-2-one (impurity A) is between 0.8 to 1.5.

Calculate the percentage of content of impurity A in parts per million using the following expression:

$$\text{Result} = A_1A_2 \times 2.5m$$

Where,

A_1 = area of the peak due to impurity A in the chromatogram obtained with the test solution.

A_2 = area of the peak due to impurity A in the chromatogram obtained with the reference solution (a).

m = mass of the substance to be examined (anhydrous substance) in the test solution in grams.

Acceptance criteria: Not more than 10ppm.

12. IMPURITY B: (By HPLC)

Chromatographic conditions:

Precolumn	:	Size: l = 10mm, Ø = 3 mm
Stationary phase	:	Base deactivated end capped octadecylsilyl silica gel for chromatography (5 µm).
Column	:	Size: l = 150mm, Ø = 4.6 mm
Stationary phase	:	Base deactivated end capped octadecylsilyl silica gel for chromatography (5 µm).
Temperature	:	40°C.
Detection	:	UV Spectrophotometer at 205 nm.
Flow rate	:	0.8 ml / min.
Injection	:	50 µl.
Retention time	:	Impurity B about 7 minutes.
Mobile phase	:	Methanol and water in the ratio (5:95 V/V)

Test solution:

Dissolve a quantity of the substance to be examined equivalent to 0.500 g of the anhydrous substance in the mobile phase and dilute to 100.0 ml with the mobile phases.

Reference solution:

Dissolve 0.150 g of 2-pyrrolidone in mobile phase and dilute to 100.0 ml with the mobile phase. Dilute 2.0 ml of the solution to 100.0 ml with the mobile phase.

* **Note:** After each injection of the test solution, wait for about 2 min. and wash the precolumn by passing the mobile phase through the column backward for about 30 min. at the same flow rate as applied in the test.

System suitability: Reference solution:

Repeatability: maximum relative standard deviation of 2.0%.

The symmetry factor for peak due to 2-pyrrolidone (impurity B) is between 0.8 to 1.5.

Calculate the percentage of content of impurity B using the following expression:

$$\text{Result} = A_1A_2 \times 0.3m$$

Where,

A_1 = area of the peak due to impurity B in the chromatogram obtained with the test solution

A_2 = area of the peak due to impurity B in the chromatogram obtained with the reference solution

m = mass of the substance to be examined (anhydrous substance) in the test solution in grams

Acceptance criteria: Not more than 3.0%.

13. WATER: < REFER GAM 010>

Not more than 5.0%, determined on 0.5g of sample.

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14. SULFATED ASH: < REFER GAM 032>

Not more than 0.1% w/w, determined on 1.0g of sample.

15. ASSAY: (Nitrogen Determination)

Place 100.0mg of the substance to be examined (m mg) in a combustion flask, add 5g of a mixture of 1g of copper sulfate pentahydrate, 1g of titanium dioxide and 33g of dipotassium sulfate, and 3 glass beads. Wash any adhering particles from the neck into the flask with a small quantity of water. Add 7ml of sulfuric acid, allowing it to run down the insides of the flask. Heat the flask gradually until the solution has a clear, yellowish-green colour, and the insides wall of the flask is free from a carbonized material and then heat for a further 45 min. after cooling, and cautiously 20ml of water, and connects the flask to the distillation apparatus previously washed by passing steam through it. To the absorption flask add 30ml of a 40 g / L solution of boric acid, 3 drops of bromocresol green-methyl red solution and sufficient water to immerse the lower end of the condenser tube. Add 30ml of strong sodium hydroxide solution through the lower the funnel, rinse the funnel cautiously with 10 ml of water, immediately close the clamp on the rubber tube, and then start distillation with steam to obtain 80 to 100ml of distillate. Remove the absorption flask from the lower end of the condenser tube, rinsing the end part with a small quantity of water and titrate the distillate with 0.025M Sulfuric acid until the colour of the solution changes from green through pale greyish blue to pale greyish reddish-purple. Carry out a blank determination.

1 ml of 0.025 M Sulfuric acid is equivalent to 0.700 mg of Nitrogen.

Calculation:

$$= \frac{\text{Titer value} \times \text{Molarity of 1ml 0.025N sulphuric acid} \times 0.700 \times 100 \times 100}{\text{Sample weight in mg} \times 0.025 \times (100 - \%\ \text{of LOD})}$$

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Asst. Manager-QC	AGM-QC	AGM-QA
Signature			
Date	21/08/2023	22/08/2023	22/08/23

REVISION HISTORY:

STP No.	Reason for Review	Change control No.	Effective Date
STP-RMEP0045-00	(i) Periodic review. (ii) STP numbering procedure revised as per SOP No. ST/QC/058.	ST/CC/23/016	25/08/2023

****END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	S.MARAN
Designation	Asst. Manager-QC	AGM-QC	AGM-QA
Signature			
Date	21/08/2023	22/08/2023	22/08/23

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	PREGELATINISED STARCH BP (STARCH 1500)			
Specification No.	RMESP0046-01	**Revision No.**	01	**Item Code.:** RMEP0046
Supersedes	RMESP0046-00	**Effective Date**	14/02/2023	**Page No.:** 1 of 3

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	NA
2	Molecular weight	NA
3	Storage conditions	Store protected from moisture.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	50 g
6	Quantity of reserve sample	100 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	PREGELATINISED STARCH BP (STARCH 1500)			
Specification No.	RMESP0046-01	**Revision No.**	01	**Item Code.:** RMEP0046
Supersedes	RMESP0046-00	**Effective Date**	14/02/2023	**Page No.:** 2 of 3

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or yellowish-white powder. It swells in cold water.
2.	*Identification **A.** Microscopic **B.** By Chemical test	 **A.** The starch granules with a distinct black cross intersecting at the hilum may be seen. **B.** A reddish-violet or blue colour is produced.
3.	*pH	Between 4.5 to 7.0
4.	Oxidising substances	Not more than 0.002%
5.	Sulphur dioxide	Not more than 50ppm
6.	Iron	Not more than 20ppm
7.	Foreign matter	Not more than traces of matter other than starch granules are present.
8.	*Loss on drying	Not more than 15.0% w/w.
9.	Sulfated ash	Not more than 0.6% w/w.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/08/2023	10/08/2023	11/02/2023

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	PREGELATINISED STARCH BP (STARCH 1500)			
Specification No.	RMESP0046-01	**Revision No.**	01	**Item Code.:** RMEP0046
Supersedes	RMESP0046-00	**Effective Date**	14/02/2023	**Page No.:** 3 of 3

S.NO	TEST (s)	SPECIFICATION (s)
10.	*Microbial contamination	
	(i) Total aerobic microbial count	Not more than 1000 cfu/g
	(ii) Total yeast and mold count	Not more than 100 cfu/g
	iii) Escherichia coli	Should be absent/g
	iv) Salmonella species	Should be absent/10g

Remarks: The above * Marked tests are to be performed while retesting the material.

REVISION HISTORY:

Specification No.	Effective Date	Reason for Review
RMESP0046-00	14-02-2020	New specification prepared
RMESP0046-01	14/02/2023	Periodic review.

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

	Safetab Life Science Puducherry			MASTER COPY
	STANDARD TESTING PROCEDURE			
Name of Product	PREGELATINIZED STARCH BP (STARCH 1500)			
STP No.	RMETP0046-01	**Revision No.**	01	**Item Code.:** RMEP0046
Supersedes	RMETP0046-00	**Effective Date**	14/02/2023	**Page No.:** 1 of 4

1. DESCRIPTION: < REFER GAM 001>

White or yellowish-white powder. It swells in cold water.

2. IDENTIFICATION: < REFER GAM 003>

A. Microscopic:

Examined under a microscope using a mixture of equal volumes of glycerol and water it presents irregular, translucent, white or yellowish-white flakes or pieces with an uneven surface. Under polarised light (between crossed nicol prisms), starch granules with a distinct black cross intersecting at the hilum may be seen.

B. By Chemical test:

Disperse 0.5 g in 2 mL of water without heating and add 0.05 mL of iodine solution. A reddish-violet or blue colour is produced.

3. pH: < REFER GAM 030>

Between 4.5 to 7.0

Weigh accurately about 3.0g of sample dissolved in 100ml carbon dioxide free water and stirring continuously. Determine the pH when a homogeneous solution is obtained.

Rinse the electrodes with distilled water and wipe dry with tissue paper. Set the instrument using buffer solution pH 6.87 by following instrument Operating Procedure. Clean the electrode. Immerse the electrode in the solution being examined and measure the pH.

4. OXIDISING SUBSTANCES:

Weigh accurately about 4.0g of sample to a glass-stoppered, 125ml conical flask and add 50.0ml water. Insert the stopper and swirl for 5 minutes. Transfer to a glass-stoppered 50ml centrifuge tube and centrifuge. Transfer 30.0ml of the clear supernatant liquid to a glass-stoppered 125-ml conical flask. Add 1ml of glacial acetic acid and 0.5g to 1.0g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25 to 30 minutes in the dark. Add 1ml of starch solution and titrate with 0.002M sodium thiosulphate until the starch-iodine colour disappears. Carry out a blank titration. Not more than 1.4ml of 0. 002M sodium thiosulphate is required (0.002 per cent, calculated as H_2O_2).

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

1 ml of 0.002M sodium thiosulphate is equivalent to 0.034mg of oxidising substances, calculated as H_2O_2.

Calculation:

$$= \frac{\text{Titer value x Molarity of 0.002M sodium thiosulphate x 0.034 x 100}}{\text{Sample weight in mg x 0.002}}$$

5. SULPHUR DIOXIDE:

Maximum 50 ppm.

Method II:

Introduce 150 mL of water into the flask (*A*) and pass carbon dioxide through the whole system for 15 min at a rate of 100 ± 5 mL/min. To 10 mL of dilute hydrogen peroxide solution add 0.15 mL of a 1 g/L solution of bromophenol blue in ethanol (20 per cent *V/V*). Add 0.1 M sodium hydroxide until a violet-blue colour is obtained, without exceeding the end-point. Place the solution in the test-tube (*D*). Without interrupting the stream of carbon dioxide, remove the funnel (*B*) and introduce through the opening into the flask (*A*) 25.0 g (*m*) of the substance to be examined with the aid of 100 mL of water. Replace the funnel. Close the tap of the funnel and add 80 mL of dilute hydrochloric acid to the funnel. Open the tap of the funnel to allow the hydrochloric acid solution to flow into the flask, making sure that no sulphur dioxide escapes into the funnel by closing the tap before the last few millilitres of hydrochloric acid solution drain out. Boil for 1 h. Open the tap of the funnel and stop the flow of carbon dioxide and also the heating and the cooling water. Transfer the contents of the test-tube with the aid of a little water to a 200 mL wide-necked, conical flask. Heat on a water-bath for 15 min and allow to cool. Add 0.1 mL of a 1 g/L solution of bromophenol blue in ethanol (20 per cent *V/V*) and titrate with 0.1 M sodium hydroxide until the colour changes from yellow to violet-blue (V_1 mL). Carry out a blank titration (V_2 mL).

6. IRON: < REFER GAM 007>

Maximum 20 ppm.

Dissolve the residue obtained in the test for sulfated ash in 20 mL of dilute hydrochloric acid. Filter. The filtrate complies with the test.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

7. FOREIGN MATTER:

Examined under a microscope using a mixture of equal volumes of glycerol and water, not more than traces of matter other than starch granules are present.

8. LOSS ON DRYING: < REFER GAM 026>

Not more than 15.0% w/w, Determined on 1.0g of sample by drying in an oven at 130°C for 90 minutes.

9. SULFATED ASH: < REFER GAM 032>

Maximum 0.6 per cent, determined on 1.0 g.

10. MICROBIAL CONTAMINATION:

Use 11.0g of sample for Total Microbial count and pathogen test.

Total Aerobic microbial count:

Procedure: Proceed as per the current general testing procedure GAM-035.

Total Yeast and mold count:

Procedure: Proceed as per the current general testing procedure GAM-036.

Escherichia Coli:

Procedure: Proceed as per the current general testing procedure GAM-037.

Salmonella species:

Procedure: Proceed as per the current general testing procedure GAM-038.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

REVISION HISTORY:

STP No.	Effective Date	Reason for Review
RMETP0046-00	14-02-2023	New STP prepared.
RMETP0046-01	14/02/2023	Periodic review.

****END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	09/02/2023	10/02/2023	11/02/2023

	Safetab Life Science Puducherry			
	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	SODIUM PROPYL HYDROXYBENZOATE BP			
Specification No.	RMESP0048-01	**Revision No.**	01	**Page No.:** 1 of 3
Supersedes	RMESP0048-00	**Effective Date**	30/08/2022	**Review Period:** 3 years

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	$C_{10}H_{11}NaO_3$
2	Molecular weight	202.2
3	Storage conditions	In an airtight container.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	8g
6	Quantity of reserve sample	16g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	Asst. Manager-QC	AGM-QC	AGM-QA
Signature			
Date	29/08/2022	30/08/2022	30/08/22

	Safetab Life Science Puducherry			
	RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	SODIUM PROPYL HYDROXYBENZOATE BP			
Specification No.	RMESP0048-01	**Revision No.**	01	**Page No.:** 2 of 3
Supersedes	RMESP0048-00	**Effective Date**	30/08/2022	**Review Period:** 3 years

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or almost white, hygroscopic, crystalline powder.
2.	*Solubility	Freely soluble in water, sparingly soluble in ethanol (96 per cent), practically insoluble in methylene chloride.
3.	*Identification **A.** By Melting point **B.** By IR **C.** By Thin-layer chromatography **D.** By Sodium Salts	 **A.** The precipitate melts at 96 °C to 99 °C. **B.** The Infrared absorption spectrum of sample should be concordant with that of reference spectrum propyl parahydroxybenzoate RS. **C.** The principal spot in the chromatogram obtained with test solution (b) is similar in position and size to the principal spot in the chromatogram obtained with reference solution (a). **D.** A dense white precipitate is formed.
4.	Appearance of solution	Solution S, examined immediately after preparation, is clear and not more intensely coloured than reference solution BY_6.
5.	*pH	Between 9.5 to 10.5
6.	*Related substances (i) Impurity A (ii) Unspecified impurities (iii) Total impurities (Excluding impurity A)	 Not more than 4.0% Not more than 0.5% Not more than 1.0%

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	Asst. Manager-QC	AGM-QC	AGM-QA
Signature			
Date	29/08/2022	30/08/2022	30/08/22

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	SODIUM PROPYL HYDROXYBENZOATE BP			
Specification No.	RMESP0048-01	**Revision No.**	01	**Page No.:** 3 of 3
Supersedes	RMESP0048-00	**Effective Date**	30/08/2022	**Review Period:** 3 years

S.NO	TEST (s)	SPECIFICATION (s)
7.	Chlorides	Not more than 350ppm
8.	Sulfates	Not more than 300ppm
9.	*Water content	Not more than 5.0%
10.	*Assay by HPLC (anhydrous basis)	Not less than 94.0% and Not more than 102.0% w/w.

Remarks: The above * Marked tests are to be performed while retesting the material.

REVISION HISTORY:

Specification No.	Effective Date	Reason for Review
RMESP0048-00	19-02-2020	New specification prepared
RMESP0048-01	30/08/2022	Name of the product in Specification has been changed to Sodium propyl hydroxybenzoate as per BP monograph. This changes captured as per Change Control number ST/CC/22/164.

**** END OF THE DOCUMENT ****

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	Asst. Manager-QC	AGM-QC	AGM-QA
Signature			
Date	29/08/2022	30/08/2022	30/8/22

	Safetab Life Science Puducherry			
	STANDARD TESTING PROCEDURE			MASTER COPY
Name of Product	SODIUM PROPYL HYDROXYBENZOATE BP			
STP No.	RMETP0048-01	**Revision No.**	01	**Page No.:** 1 of 10
Supersedes	RMETP0048-00	**Effective Date**	30/08/2022	**Review Period:** 3 years

1. DESCRIPTION: < REFER GAM 001>

White or almost white, hygroscopic, crystalline powder.

2. SOLUBILITY: < REFER GAM 002>

100mg of sample + 1mL of water	Freely soluble if the material dissolves.
100mg of sample + 10mL of ethanol (96%)	Sparingly soluble if the material dissolves.
10mg of sample + 100mL of methylene chloride	Practically insoluble if the material does not dissolves.

3. IDENTIFICATION:

First identification: B, D

Second identification: A, C, D

A) By Melting point: < REFER GAM 028>

Dissolve 0.5g of sample in 50mL of water. Immediately add 5mL of hydrochloric acid. Filter and wash the precipitate with water. Dry in vacuo at 80°C for 2 h. The precipitate melts at 96°C to 99°C.

B) By IR: < REFER GAM 003>

The Infrared absorption spectrum of sample should be concordant with that of reference spectrum propyl parahydroxybenzoate RS.

C) By Thin-layer chromatography:

Test solution (a):

Dissolve 0.10 g of the substance to be examined in 10 mL of water. Immediately add 2 mL of hydrochloric acid and shake with 50 mL of 1, 1-dimethylethyl methyl ether. Evaporate the upper layer to dryness and take up the residue with 10 mL of acetone.

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	**Asst. Manager-QC**	**AGM-QC**	**AGM-QA**
Signature			
Date	29/08/2022	30/08/2022	30/08/22

Test solution (b):

Dilute 1 mL of test solution (a) to 10 ml with acetone.

Reference solution (a):

Dissolve 10 mg of propyl parahydroxybenzoate RS in acetone and dilute to 10 mL with the same solvent.

Reference solution (b):

Dissolve 10 mg of ethyl parahydroxybenzoate RS in 1 mL of test solution (a) and dilute to 10 mL with acetone.

Plate: TLC octadecylsilyl silica gel F_{254} plate.

Mobile phase:

A mixture of 1 volumes of glacial acetic acid, 30 volumes of water and 70 volumes of methanol.

Application: 5 µL of test solution (b) and reference solutions (a) and (b).

Development: Over 2/3 of the plate.

Drying: In air.

Detection: Examine in ultraviolet light at 254 nm.

System suitability Reference solution (b):

The chromatogram shows 2 clearly separated principal spots.

Results:

The principal spot in the chromatogram obtained with test solution (b) is similar in position and size to the principal spot in the chromatogram obtained with reference solution (a).

D. By Sodium Salts:

To 1ml of solution S and 1ml water mix Add and 2mL of a 150 g/L solution of potassium carbonate and heat to boiling. No precipitate is formed. Add 4mL of potassium pyroantimonate solution and heat to boiling. Allow to cool in ice water and if necessary rub the inside of the test-tube with a glass rod. A dense white precipitate is formed.

Solution S:

Dissolve 5.0 g in carbon dioxide-free water prepared from distilled water, and dilute to 50 mL with the same solvent.

4. APPEARANCE OF SOLUTION:

Solution S examined immediately after preparation is clear and not more intensely coloured than reference solution BY_6.

5. pH: <REFER GAM 030>

Between 9.5 to 10.5, determined on solution S

Dilute 1 ml of solution S to 100 ml with water.

6. RELATED SUBSTANCES: (determined by liquid chromatography)

Chemicals/Reagents/Standards:

Propyl parahydroxybenzoate	:	Reference standard
4-Hydroxybenzoic acid (impurity A)	:	Reference standard
Ethyl parahydroxybenzoate (impurity C)	:	Reference standard
Potassium dihydrogen phosphate	:	AR grade
Methanol	:	HPLC grade
Purified Water	:	Milli Q water (or) Equivalent

Chromatographic Condition:

Column	:	Waters Xterra C18, (150 X 4.6mm), 5µm or equivalent
Flow rate	:	1.3 mL / minute
Detector wavelength	:	272 nm
Injection Volume	:	10µl
Run time	:	2.5 times the retention time of Propyl parahydroxybenzoate.
Retention time	:	Retention time of Propyl parahydroxybenzoate peak is at about 4.5 minutes

Mobile phase preparation:

A mixture of 35 volumes 6.8g/L solution of potassium dihydrogen phosphate and 65 volumes of methanol.

Test solution:

Weigh accurately about 50mg of the substance to be examined in 2.5ml of methanol and dilute to 50.0ml with the mobile phase. Dilute 10.0ml of this solution to 100.0ml with the mobile phase.

Reference solution (a):

Weigh accurately about 5 mg of ethyl parahydroxybenzoate RS (impurity C), 5 mg of 4-hydroxybenzoic acid (impurity A) RS and 5 mg of the substance to be examined 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.

Reference solution (b):

Weigh accurately about 50mg of propyl parahydroxybenzoate RS in 2.5ml of methanol and dilute to 50.0ml with the mobile phase. Dilute 10.0ml of this solution to 100.0ml with the mobile phase.

Reference solution (c):

Dilute 1.0ml of the test solution to 20.0ml with the mobile phase. Dilute 1.0ml of this solution to 10.0ml with the mobile phase.

Blank Preparation: Dilute 2.5ml Methanol in to 50ml with mobile phase

Relative retention:

With reference to Propyl parahydroxybenzoate (retention time = about 4.5 min): impurity A = about 0.3; impurity C = about 0.7.

System suitability Reference solution (a):

Resolution:

Minimum 5.0 between the peaks due to impurity C and Propyl parahydroxybenzoate.

Inject 10µl of the above solution as per following sequence.

Injection sequence:

S. No	Sample Name	No. of injections
1	Blank	1
2	Reference solution (a)	1
3	Reference solution (c)	1
4	Blank	1
5	Test solution	1

Impurity A: (NMT 4.0%)

$$= \frac{AT_A}{AS} \times \frac{WT}{50} \times \frac{10}{100} \times \frac{1}{20} \times \frac{1}{10} \times \frac{50}{WT} \times \frac{100}{10} \times 100 \times 1.4$$

Where,

AT_A = Area of impurity A peak in Test preparation.

AS =Area of the principal peak in the reference solution (c)

WT = Weight of the sample taken in mg.

1.4 = Correction factor

Unspecified Impurity: (NMT 0.5%)

$$= \frac{AT_I}{AS} \times \frac{WT}{50} \times \frac{10}{100} \times \frac{1}{20} \times \frac{1}{10} \times \frac{50}{WT} \times \frac{100}{10} \times 100$$

Where,

AT_I = Area of Unspecified Impurity peak in Test preparation.

AS =Area of the principal peak in the reference solution (c)

WT = Weight of the sample taken in mg.

Total Impurities (Excluding Impurity A): (NMT 1.0%)

$$= \frac{AT_T}{AS} \times \frac{WT}{50} \times \frac{10}{100} \times \frac{1}{20} \times \frac{1}{10} \times \frac{50}{WT} \times \frac{100}{10} \times 100$$

Where,

AT_T = Area of Total Impurities peak in Test preparation.

AS =Area of the principal peak in the reference solution (c)

WT = Weight of the sample taken in mg.

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	**Asst. Manager-QC**	**AGM-QC**	**AGM-QA**
Signature			
Date	29/08/2022	30/08/2022	30/08/22

LIMITS:

Correction factor:

For the calculation of content, multiply the peak area of impurity A by 1.4

Impurity A:

Not more than 8 times the area of the principal peak in the chromatogram obtained with reference solution (c) (4.0 per cent)

Unspecified impurities:

For each impurity, not more than the area of the principal peak in the chromatogram obtained with reference solution (c) (0.5 per cent)

Total impurities (Excluding impurity A)

Not more than twice the area of the principal peak in the chromatogram obtained with reference solution (c) (1.0 per cent)

Disregard limit:

0.2 times the area of the principal peak in the chromatogram obtained with reference solution (c) (0.1 per cent).

7. CHLORIDE:<REFER GAM 008>

Maximum 350 ppm.

To 10 mL of solution S, add 1 mL of nitric acid and 30 mL of water and dilute to 50 mL with water. Shake and filter. Dilute 10 mL of the filtrate to 15 mL with water. Prepare the standard using 14 mL of chloride standard solution (5 ppm Cl) to which 1 mL of water has been added.

8. SULPHATE: <REFER GAM 009>

Maximum 300 ppm.

To 25 mL of solution S, add 5 mL of distilled water and 10 mL of hydrochloric acid and dilute to 50 mL with distilled water. Shake and filter. Dilute 10 mL of the filtrate to 15 mL with distilled water.

9. WATER: <REFER GAM 010>

Not more than 5.0% w/w, determined on 0.500g of sample.

10. ASSAY: (By HPLC)

Note: Chromatographic conditions, Mobile phase preparation, Reference solution (b) and Test solution described under related substances.

Inject reference solution (b) and the test solution.

Calculate the percentage content of $C_{10}H_{11}NaO_3$, from the declared content of propyl parahydroxybenzoate RS multiplied by a correction factor of 1.122.

Inject the reference solution (b). The test is not valid unless the column efficiency is not less than 2000 theoretical plates, the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 2.0 per cent

Inject 10µl of the above solution as per following sequence.

Injection sequence:

S. No	Sample Name	No. of injections
1	Blank	1
2	Reference solution (b)	5
3	Test solution (PPN-1)	2
4	Test solution (PPN-2)	1

Calculations:

Calculate the assay in % of Sodium propyl hydroxybenzoate as such basis of the sample as below.

$$= \frac{AT}{AS} \times \frac{WS}{50} \times \frac{10}{100} \times \frac{50}{WT} \times \frac{100}{10} \times \frac{P}{100} \times 1.122 \times 100$$

Where,

AT = Average area of the principal peak in Test solution.

AS = Average area of the principal peak in the Reference solution (b).

WS = Weight of the Propyl parahydroxybenzoate Reference standard in mg.

WT = Weight of the sample taken in mg.

P = Potency of the propyl parahydroxybenzoate Reference standard in % on as such basis

1.122 = Correction factor.

Calculate the assay in % of Sodium propyl hydroxybenzoate on anhydrous basis of the sample as below.

$$= \frac{\text{Assay as such basis}}{(100 - \%\ \text{Water})} \times 100$$

11. REVISION HISTORY:

STP No.	Effective Date	Reason for Review
RMETP0048-00	19-02-2020	New STP prepared
RMETP0048-01	30/08/2022	Name of the product in Specification has been changed to Sodium propyl hydroxybenzoate as per BP monograph. This changes captured as per Change Control number ST/CC/22/164.

**** END OF THE DOCUMENT****

	PREPARED BY	REVIEWED BY	APPROVED BY
Designation	**Asst. Manager-QC**	**AGM-QC**	**AGM-QA**
Signature			
Date	29/08/2022	30/08/2022	30/08/22

	Safetab Life Science Puducherry RAW MATERIAL SPECIFICATION			MASTER COPY
Name of Product	SODIUM STARCH GLYCOLATE (TYPE A) BP			
Specification No.	RMESS0029-01	**Revision No.**	01	**Item Code.:** RMAS0029
Supersedes	RMESS0029-00	**Effective Date**	28/09/2022	**Page No.:** 1 of 4

S.NO	RAW MATERIAL GENERAL SPECIFICATION (s)	
1	Molecular formula	NA
2	Molecular weight	NA
3	Storage conditions	In an airtight container, protected from light.
4	Precautions & Special instructions for sampling	Use hand gloves and nose mask while sampling. Reseal the containers immediately after sampling. Avoid inhaling.
5	Quantity of sample required for analysis	23 g
6	Quantity of reserve sample	46 g
7	Retest period	12 months from the date of release
8	Re-test Parameter	As mentioned in Specification
9	Reference	BP
10	Sampling instruction	Follow the standard operating procedure No.: ST/QC/040.
11	Destructions instructions	Follow the standard operating procedure No.: ST/QC/032.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	26/09/2022	27/09/2022	27/09/2022

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	SODIUM STARCH GLYCOLATE (TYPE A) BP			
Specification No.	RMESS0029-01	**Revision No.**	01	**Item Code.:** RMAS0029
Supersedes	RMESS0029-00	**Effective Date**	28/09/2022	**Page No.:** 2 of 4

S.NO	TEST (s)	SPECIFICATION (s)
1.	*Description	White or almost white, fine, free-flowing powder, very hygroscopic.
2.	*Solubility	Practically insoluble in methylene chloride. It gives a translucent suspension in water.
3.	*Identification **A.** By pH **B.** By Chemical test **C.** By Chemical test **D.** By Sodium salts	 **A.** Between 5.5 to 7.5 **B.** A suspension forms that settles after standing. **C.** The solution becomes blue or violet. **D.** A dense white precipitate is formed.
4.	Appearance of solution	Solution S1 is clear and colourless
5.	*pH	Between 5.5 to 7.5
6.	Sodium glycolate	Not more than 2.0%.
7.	Sodium chloride	Not more than 7.0%.
8.	Iron.	Not more than 20ppm.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Signature			
Date	26/09/2022	27/09/2022	27.09.2022

	Safetab Life Science Puducherry			MASTER COPY
	RAW MATERIAL SPECIFICATION			
Name of Product	SODIUM STARCH GLYCOLATE (TYPE A) BP			
Specification No.	RMESS0029-01	**Revision No.**	01	**Item Code.:** RMAS0029
Supersedes	RMESS0029-00	**Effective Date**	28/09/2022	**Page No.:** 3 of 4

S.NO	TEST (S)	SPECIFICATION (S)
9.	*Loss on drying	Not more than 10.0% w/w.
10.	*Assay (On dried basis)	Not less than 2.8% and not more than 4.2% w/w of Sodium. Calculated on the material washed with ethanol (80%) and dried as described under assay.
11.	*Microbial contamination Escherichia coli Salmonella Species	 Should be absent/1gm Should be absent/10gm

Remarks: The above * Marked tests are to be performed while retesting the material.

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Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	26/09/2022	27/09/2022	27/09/2022

REVISION HISTORY:

Specification No.	Effective Date	Reason for Review
RMESS0029-00	17-02-2020	New specification prepared
RMESS0029-01	28/09/2022	Title name has been changed from Sodium starch glycolate to Sodium starch glycolate (Type A) as per BP monograph. This changes captured as per Change Control number ST/CC/22/189.

**** END OF THE DOCUMENT ****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	26/09/2022	27/09/2022	27/09/2022

	Safetab Life Science Puducherry			
	STANDARD TESTING PROCEDURE			MASTER COPY
Name of Product	SODIUM STARCH GLYCOLATE (TYPE A) BP			
STP No.	RMETS0029-01	**Revision No.**	01	**Item Code.:** RMAS0029
Supersedes	RMETS0029-00	**Effective Date**	28/09/2022	**Page No.:** 1 of 5

1. DESCRIPTION: < REFER GAM 001>

White or almost white, fine, free-flowing powder, very hygroscopic.

2. SOLUBILITY: < REFER GAM 002>

10mg of sample + 100mL of Methylene chloride	Practically insoluble if the material does not dissolves.

It gives a translucent suspension in water.

3. IDENTIFICATION:

A. By pH: < REFER GAM 030>

Between 5.5 to 7.5

B. By Chemical test:

Prepare with shaking and without heating a mixture of 4.0 g of the substance to be examined and 20 mL of carbon dioxide-free water. The mixture has the appearance of a gel. Add 100 mL of carbon dioxide-free water and shake. A suspension forms that settles after standing.

C. By Chemical test:

To an acidified solution, add iodinated potassium iodide solution. The solution becomes blue or violet.

D. Sodium salts:

a) Pipette 2ml of solution S2 and Add 2 mL of a 150 g/L solution of potassium carbonate and heat to boiling. No precipitate is formed. Add 4 mL of potassium pyroantimonate solution and heat to boiling. Allow to cool in iced water and if necessary rub the inside of the test-tube with a glass rod. A dense white precipitate is formed.

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4. APPEARANCE OF SOLUTION:

Solution S1:

Centrifuge the suspension obtained in identification test B at 2500RPM for 10 min. Collect carefully the supernatant.

Solution S2:

Place 2.5g in a silica or platinum crucible and add 2 mL of a 500 g/L solution of sulfuric acid. Heat on a water-bath, then cautiously over a naked flame, raising the temperature progressively, then incinerate in a muffle furnace at 600 ± 25 °C. Continue heating until all black particles have disappeared. Allow to cool, add a few drops of dilute sulfuric acid, heat and incinerate as above. Allow to cool, add a few drops of ammonium carbonate solution, evaporate to dryness and incinerate cautiously. Allow to cool and dissolve the residue in 50 mL of water.

5. pH: < REFER GAM 030>

Between 5.5 to 7.5, Disperse 1.0 g in 30 mL of carbon dioxide-free water.

6. SODIUM GLYCOLATE:

Maximum 2.0 per cent. Carry out the test protected from light. Test solution Place 0.20 g in a beaker. Add 5 mL of acetic acid and 5 mL of water. Stir until dissolution is complete (about 10 min). Add 50 mL of acetone and 1 g of sodium chloride. Filter through a fast filter paper impregnated with acetone, rinse the beaker and filter with acetone. Combine the filtrate and washings and dilute to 100.0 mL with acetone. Allow to stand for 24 h without shaking. Use the clear supernatant.

Reference solution:

Dissolve 0.310 g of glycollic acid previously dried in vacuo over diphosphorus pentoxide at room temperature overnight, in water and dilute to 500.0 mL with the same solvent. To 5.0 mL of this solution add 5 mL of acetic acid and allow to stand for about 30 min. Add 50 mL of acetone Rand 1 g of sodium chloride . Filter through a fast filter paper impregnated with acetone, rinse the beaker and filter with acetone.

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Combine the filtrate and washings and dilute to 100.0 mL with acetone. Allow to stand for 24 h without shaking. Use the clear supernatant. Heat 2.0 mL of the test solution on a water-bath for 20 min. Cool to room temperature and add 20.0 mL of 2, 7-dihydroxynaphthalene solution. Shake and heat in a water-bath for 20 min. Cool under running water, transfer to a volumetric flask and dilute to 25.0 mL with sulfuric acid, maintaining the flask under running water.

Within 10 min, measure the absorbance at 540 nm using water as the compensation liquid. The absorbance of the solution prepared with the test solution is not greater than that of a solution prepared at the same time and in the same manner with 2.0 mL of the reference solution.

7. SODIUM CHLORIDE:

Maximum 7.0 per cent. Place 0.500 g in a beaker and suspend in 100 mL of water. Add 1 mL of nitric acid. Titrate with 0.1 M silver nitrate, determining the end-point potentiometrically using a silver-based indicator electrode and a double-junction reference electrode containing a 100 g/L solution of potassium nitrate in the outer jacket and a standard filling solution in the inner jacket.1 mL of 0.1 M silver nitrate is equivalent to 5.844 mg of NaCl.

Calculation:

$$= \frac{\text{Titer value} \times \text{Molarity of 0.1 M silver nitrate} \times 0.005844\ \text{g} \times 100}{\text{Sample weight in g} \times 0.1}$$

8. IRON: < REFER GAM 007>

Maximum 20 ppm, determined on 10 mL of solution S2.

9. LOSS ON DRYING: < REFER GAM 026>

Not more than 10.0 per cent, determined on 1.000 g by drying in an oven at 130 °C for 1.5 h.

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Date	26/09/2022	27/09/2022	27/09/2022

10. ASSAY :

Shake about 1 g with 20 mL of ethanol (80 per cent V/V), stir for 10 min and filter. Repeat the operation until chloride has been completely extracted and verify the absence of chloride using silver nitrate solution. Dry the residue at 105 °C to constant mass. To 0.700 g of the dried residue, add 80 mL of glacial acetic acid and heat under a reflux condenser for 2 h. Cool the solution to room temperature. Titrate with 0.1 M perchloric acid, determining the end-point potentiometric ally. Carry out a blank titration.

1 mL of 0.1 M perchloric acid is equivalent to 2.299 mg of Na.

Calculation:

$$= \frac{\text{Titer value} - \text{Blank value} \times \text{Molarity of 0.1M perchloric acid} \times 0.002299 \times 100}{0.1 \times \text{Sample weight in g}}$$

11. MICROBIAL CONTAMINATION:

a. Esherichia Coli:

Procedure: Proceed as per the current General Analytical Method GAM-037.

b. Salmonella Species:

Procedure: Proceed as per the current General Analytical Method GAM-038.

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
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Date	26/09/2022	27/09/2022	27/09/2022

12. REVISION HISTORY:

STP No.	Effective Date	Reason for Review
RMETS0029-00	17-02-2020	New STP prepared
RMETS0029-01	28/09/2022	Title name has been changed from Sodium starch glycolate to Sodium starch glycolate (Type A) as per BP monograph. This changes captured as per Change Control number ST/CC/22/189.

****END OF THE DOCUMENT****

Particulars	PREPARED BY	REVIEWED BY	APPROVED BY
Name	K.SARAVANAN	M.VIJAYAKUMAR	A.G.KANNAN
Designation	Asst. Manager-QC	AGM-QC	GM-QA
Signature			
Date	26/09/2022	27/09/2022	27/09/2022