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Valid From: 01/04/23	Quantum Drugs & Chemicals Manamadurai	Department: Quality Control
Supersedes: FM/ QC/4002/ 05	**FINISHED PRODUCT SPECIFICATION**	SPC No: FM/QC/4002/06
	TITLE : TOLBUTAMIDE-BP/EP/IP/USP	Page 1 of 6

Tolbutamide-BP :

Sl. No	Tests	Standards
1	Appearance	White or almost white, Crystalline Powder
2	Solubility	Practically insoluble in water, soluble in acetone and in ethanol (96 per cent). It dissolves in dilute solutions of alkali hydroxides.
3 A.	IDENTIFICATION Melting point	 126 °C to 130 °C
B.	Ultraviolet and visible absorption spectrophotometry	Test solution (a) Dissolve 25.0 mg in methanol R and dilute to 100.0ml with the same solvent. Test solution (b) Dilute 10.0 ml of test solution (a) to 250.0 ml with methanol R. Spectral range: 245 - 300 nm for test solution (a); 220-235 nm for test solution (b). Absorption maxima; At 258 nm, 263 nm and 275 nm for test solution (a); at 228 nm for test solution (b). Shoulder: At 268 nm for test solution (a). Specific absorbance at the absorption maximum 480 to 520 for test solution (b).
C.	Infrared absorption spectrophotometry	Compare spectrum with the reference spectrum of Tolbutamide CRS
D.	Melting point after recrystallisation	135 °C to 140 °C
4	Appearance of Solutions	Dissolve 0.2 g in 5ml of dilute Sodium hydroxide solution R and add 5ml of water R. The solution is clear and colorless.
5	pH	4.5 to 5.5
6	**Related Substances(BY HPLC)** i) Unspecified impurities ii)PTSA(ImpurityA) iii)PTSU(Impurity B) iv) Total Impurities	 Not more than 0.10% Not more than 0.10% Not more than 0.10% Not more than 0.3%
7	Loss on drying (at 105°C)	Maximum 0.5%
8	Sulphated ash	Maximum 0.1%
9	Assay by Titration (ODB)	99.0% to 101.0%

	Prepared by:	Reviewed by:	Approved by:	Authorized by:
Name:	K.Dinakaraja	P.Muthuramalingam	K Soundararajan	M.B. Vijay Babu
Designation:	QC Executive	QC Manager	QA Manager	General Manager
Signature:				
Date:	24/03/23	25/03/23	27/03/23	29/03/23

Tolbutamide-EP :

Sl. No.	Tests	Standards
1	Appearance	White or almost white, Crystalline Powder
2	Solubility	Practically insoluble in water, soluble in acetone and in ethanol (96 per cent). It dissolves in dilute solutions of alkali hydroxides.
3 A.	IDENTIFICATION Melting point	 126 °C to 130 °C
B.	Ultraviolet and visible absorption spectrophotometry	Test solution (a) Dissolve 25.0 mg in methanol R and dilute to 100.0ml with the same solvent. Test solution (b) Dilute 10.0 ml of test solution (a) to 250.0 ml with methanol R. Spectral range: 245 - 300 nm for test solution (a); 220-235 nm for test solution (b). Absorption maxima; At 258 nm, 263 nm and 275 nm for test solution (a); at 228 nm for test solution (b). Shoulder: At 268 nm for test solution (a). Specific absorbance at the absorption maximum 480 to 520 for test solution (b).
C.	Infrared absorption spectrophotometry	Compare spectrum with the reference spectrum of Tolbutamide CRS
D.	Melting point after recrystallisation	135 °C to 140 °C
4	Appearance of Solutions	Dissolve 0.2 g in 5ml of dilute Sodium hydroxide solution R and add 5ml of water R. The solution is clear and colorless.
5	pH	4.5 to 5.5
6	**Related Substances(BY HPLC)** i) Unspecified impurities ii)PTSA(Impurity A) iii)PTSU(Impurity B) iv) Total Impurities	 Not more than 0.10% Not more than 0.10% Not more than 0.10% Not more than 0.3%
7	Loss on drying (at 105°C)	Maximum 0.5%
8	Sulphated ash	Maximum 0.1%
9	Assay by Titration (ODB)	99.0% to 101.0%

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Tolbutamide-IP:

S.No	Tests	Standards
1.	Description	A White, Crystalline Powder
2. A.	IDENTIFICATION Infrared absorption spectrophotometry	Compare spectrum with the reference spectrum of Tolbutamide CRS
B.	Ultraviolet and visible absorption spectrophotometry	Dissolve 25 mg in sufficient methanol to produce 100.0 ml. When examined in the range 245 nm to 360 nm, the resulting solution shows absorption maxima at about 258 nm, 263 nm and 275 nm and a shoulder at about 268 nm. Dilute the solution with methanol to produce a 0.001 per cent w/v solution. When examined in the range 220 nm to 235 nm, the resulting solution shows an absorption maximum at about 228 nm; absorbance at about 228 nm, about 0.50.
C.	Functional group	An orange – red colour is produced.
D.	Melting point after recrystallisation	135°C to 140°C
3.	Appearance of solution	Dissolve 0.2gm in 10ml of 1M Sodium hydroxide solution is clear and colorless.
4.	pH	4.5 to 5.5
5.	Non sulphonyl urea	Dissolve 0.5g in 1 ml of dilute ammonia solution and 9 ml of water, not more than a faint opalescence is produced.
6.	**Related Substances (BY HPLC)** i) Unspecified impurities ii)PTSA(Impurity A) iii)PTSU(Impurity B) iv) Total Impurities	 Not more than 0.10% Not more than 0.10% Not more than 0.10% Not more than 0.3%
7.	Heavy metals	Not more than 20 ppm
8.	Sulphated ash	Not more than 0.1%
9.	Loss on drying(dry at 105°C for 3hours.)	Not more than 0.5%
10.	Assay by Titration (ODB)	99.0% to 101.0%

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Tolbutamide-USP :

S. No	Tests	Standards
1.	**Identification**	
A.	Infrared Spectroscopy	Compare spectrum with the reference spectrum of USP Tolbutamide RS
B.	HPLC	The retention time of the major peak of the Sample solution corresponds to that of the Standard solution, as obtained in the Assay.
2.	Assay by HPLC (ODB)	97.0%- 103.0 %
3.	Selenium	Not more than 0.003 %
4.	Organic Impurities by HPLC	
	i) Tosylurea (PTSU)	Not more than 0.1%
	ii)Tosylamide(PTSA)	Not more than 0.1%
	iii) Tolazamide	Not more than 0.1%
	iv)Any other individual impurity	Not more than 0.1%
	v) Total Impurities	Not more than 0.3%
5.	Loss on drying (dry at 105°C for 3h.)	Not more than 0.5 %

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Specification - **Tolbutamide-BP/EP/USP :**

S.No	Test	Standard Limits
1	Appearance	White or almost white, Crystalline Powder
2	Solubility	Practically insoluble in water, soluble in Acetone and in ethanol(96%). It also dissolves in dilute alkali hydroxides.
3. A.	Identification Melting Point	 126°C to 130°C
B.	Ultraviolet and visible absorption spectrophotometry	Test solution (a) Dissolve 25.0 mg in methanol R and dilute to 100.0ml with the same solvent. Test solution (b) Dilute 10.0 ml of test solution (a) to 250.0 ml with methanol R. Spectral range: 245 - 300 nm for test solution (a); 220-235 nm for test solution (b). Absorption maxima; At 258 nm, 263 nm and 275 nm for test solution (a); at 228 nm for test solution (b). Shoulder: At 268 nm for test solution (a). Specific absorbance at the absorption maximum 480 to 520 for test solution (b).
C.	Infrared absorption spectrophotometry	Compare spectrum with the reference spectrum of Tolbutamide CRS.
D.	Melting point after recrystallisation	135°C to 140°C
E.	HPLC	The retention time of the major peak of the Sample solution corresponds to that of the Standard solution, as obtained in the Assay.
4.	Appearance Of Solution	Dissolve 0.2g in 5ml of dilute sodium hydroxide solution R and add 5ml of water R .The solution is clear and colorless.
5.	pH	4.5 to 5.5
6.	**Related Substances(BY HPLC)** i) Unspecified impurities ii)PTSA(Impurity A) iii)PTSU(Impurity B) iv)Total Impurities	 Not more than 0.10% Not more than 0.10% Not more than 0.10% Not more than 0.3%

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7.	Loss On Drying (dry at 105°C for 3h.)	Maximum 0.5 %
8.	Sulphated Ash (w/w)	Maximum 0.1 %
9.	a.Assay by Titration(ODB) b.Assay by HPLC (ODB)	99.0% to 101.0 % 97.0% to 103.0 %

PTSA : p-toluene sulphonamide
PTSU: p-toluene sulphonyl urea

HISTORY:

S.No	Date	Revision No.	Changes Made	Change Control Doc No.	Effective date
1	01/03/23	06	1. SPC Template was updated as per SOP /QC/501. 2. Administrative changes was done for better clarity 3. Specifications are separate from STP for better clarity and compliance 4. The specification and STP of USP are revised as per USP45 (2022) revision due to the update in Organic impurities and Assay by HPLC.	CCIF/2022-23/038	01/04/23

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