PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Bracketing of Standards during the analysis by HPLC, UV and GC	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**

1.0 OBJECTIVE:

To lay down a procedure for Bracketing of Standard during analysis by HPLC, UV & GC.

2.0 SCOPE:

This SOP is applicable during analysis of Assay, CU, Disso, RS and Residual Solvents by HPLC, GC and UV.

3.0 RESPONSIBILITY:

Officer, Sr.Officer ,Executive – Quality control

Head – Quality Control

4.0 PROCEDURE:

4.1 The bracketing standard is injected to ascertain the effectively instrument operation throughout the analysis.

4.2 System Precision shall be demonstrated with standard solution.

4.2.1 Before the Sample preparation analysis the System suitability shall be evaluated by replicate injections of standard preparation as mentioned in standard test procedure of respective product. Throughout the analysis the system precision shall demonstrated by injecting the standard preparation in-between and/or at the end of the analysis.

4.3 The bracketing standard is to be injected depending on the type of analysis.

4.4 For Analysis on HPLC:

4.4.1 For Assay:

4.4.1.1 During assay analysis by HPLC, the sample preparation to be made in single and two injections shall be performed from each sample preparation.

4.4.1.2 After completion of the system suitability as per respective STP, inject samples (Each duplicate) followed by one bracketing standard injection.

4.4.1.3 Bracketing standard is to be injected at the end of sequence or after set of 10 injections of sample,

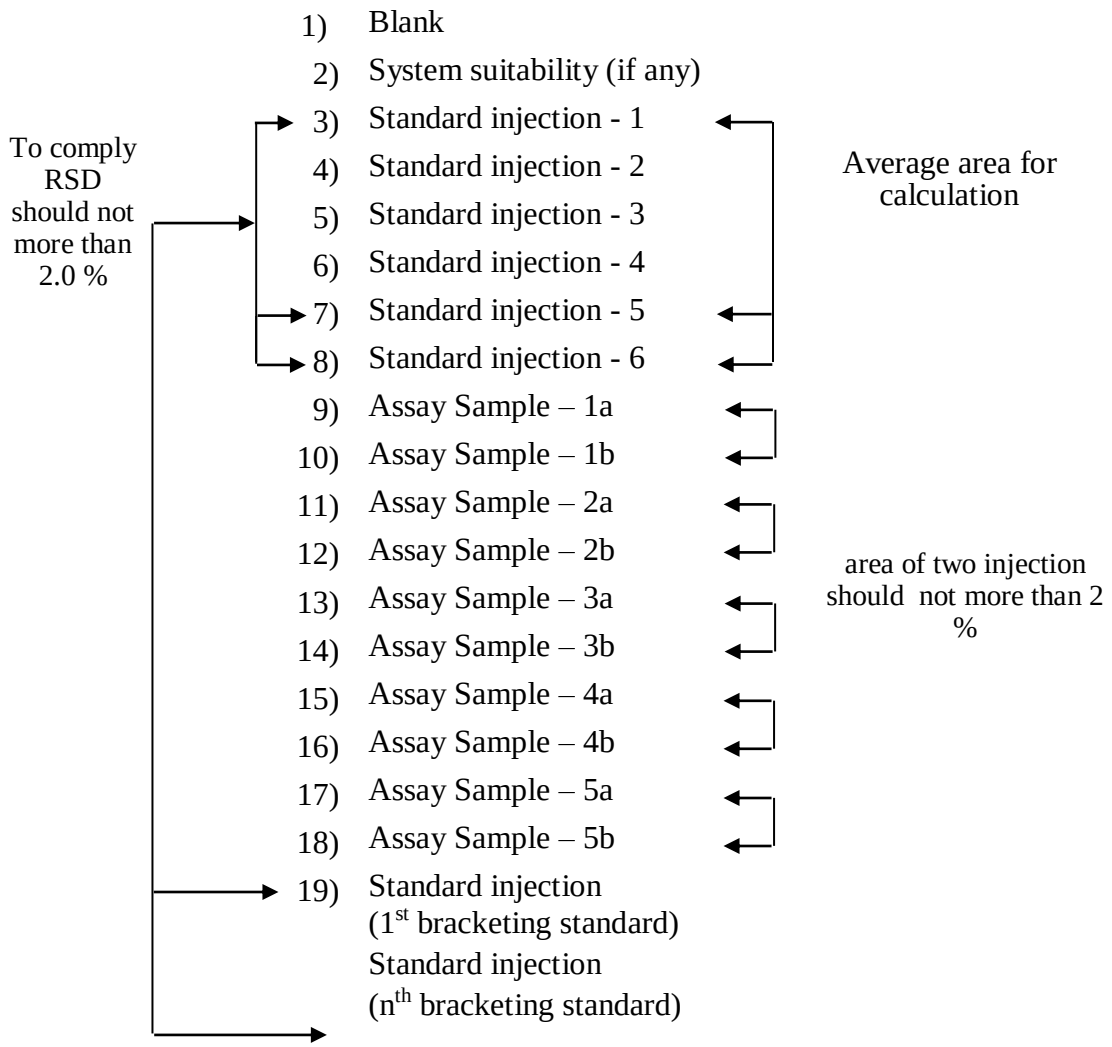
which ever earlier.

4.4.1.4 The RSD between the replicate standard injection and each respective bracketing standard injection should be not more than 2.0 %. Calculate the assay for each injection.

4.4.1.5 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.4.1.6 Schematically sequence is given with below diagram:

4.4.1.7 **Schematically it is represented as follows:**



4.4.2 For Content Uniformity:

4.4.2.1 During Content Uniformity analysis by HPLC, the sample to be made in single and single injection shall be performed from each sample.

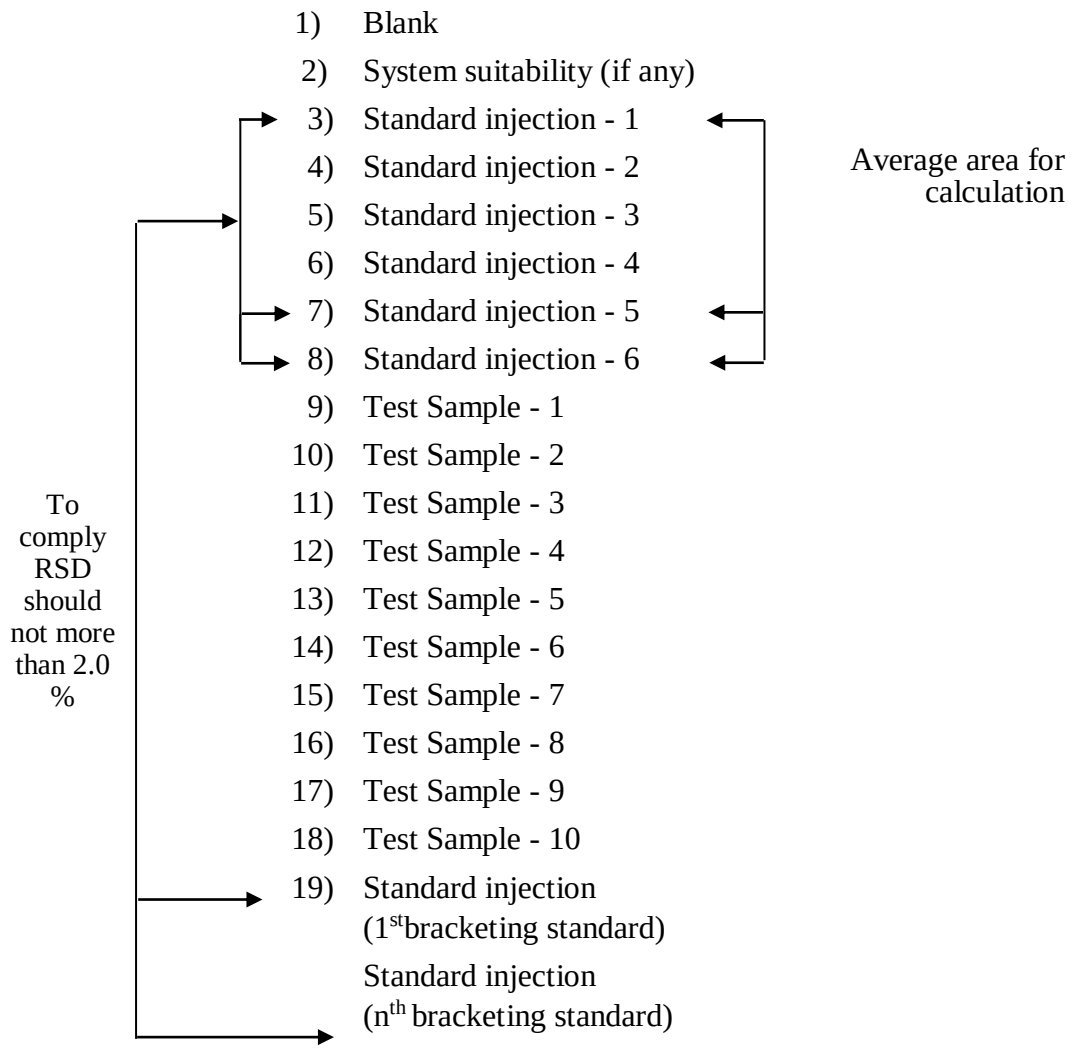
4.4.2.2 After completion of the system suitability as per respective STP, inject samples (Each single) followed by one bracketing standard injection.

4.4.2.3 Bracketing standard is to be injected at the end of sequence or after set of 10 injections of sample, which ever earlier.

4.4.2.4 The RSD between the Replicate Standard injections and each respective bracketing standard injection should be not more than 2.0 %.

4.4.2.5 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.4.2.6 Schematically it is represented as follows:



4.4.3 Dissolution and Dissolution profile By HPLC:

4.4.3.1 During dissolution analysis by HPLC make one injection of sample from each vessel. After the completion of system suitability, inject Blank and six sample injections from six vessels followed by standard injections.

4.4.2.2 After completion of the system suitability as per respective STP, inject samples (Each single) followed by one bracketing standard injection.

4.4.2.3 Bracketing standard is to be injected at the end of sequence or after set of 6 injections of sample, which ever earlier.

4.4.3.4 When the dissolution shall be perform for pooled sample than follow the sequence as per specified in the Assay test as per 4.4.1.7.

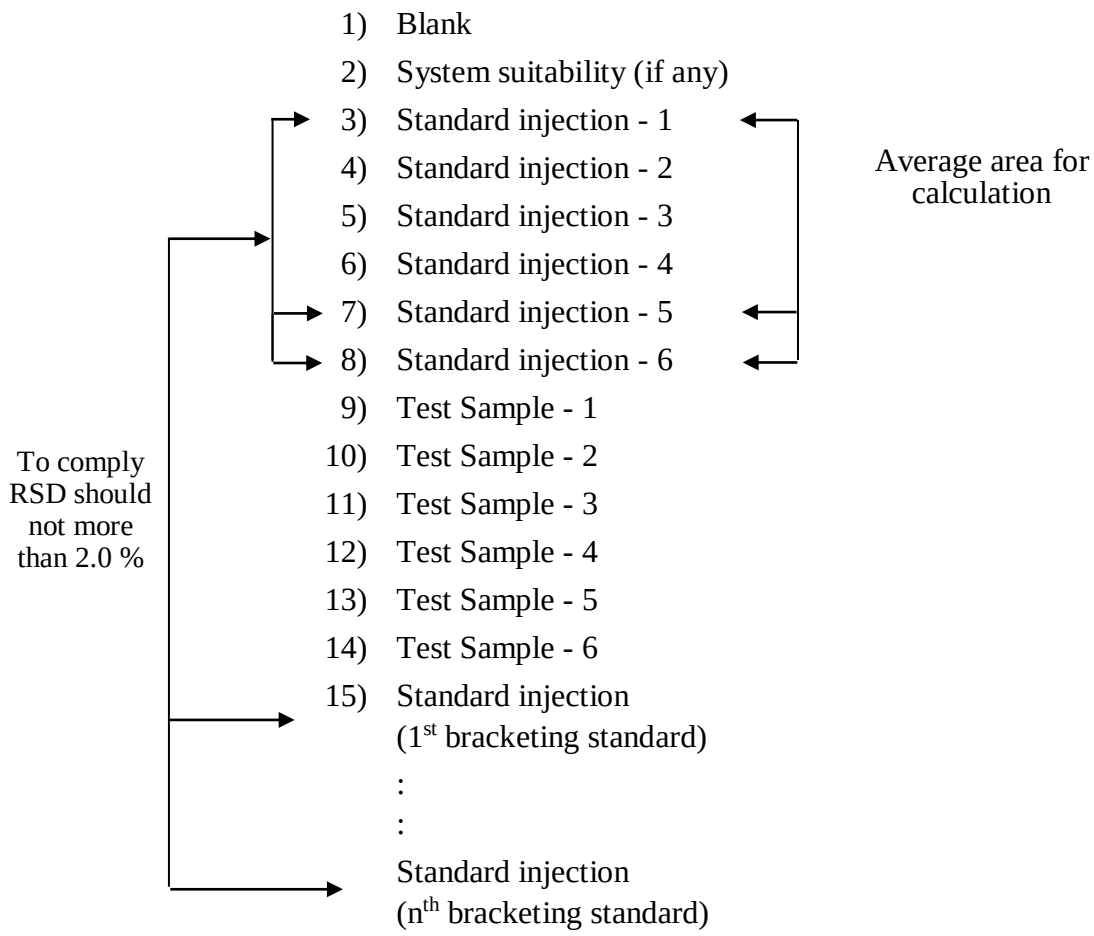
4.4.3.5 The RSD between the replicate standard injections and each respective bracketing standard injection should be not more than 2.0 %.

4.4.3.6 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.4.3.7 Schematically sequence is given with below diagram:

4.4.3.8 Schematically it is represented as follows:

STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Bracketing of Standards during the analysis by HPLC, UV and GC	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**



4.4.4 Related Substances (by HPLC):

4.4.4.1 The Related substances analysis by HPLC by using External standard method shall be performed in the following manner.

4.4.4.2 Perform the system suitability as per individual test method or STP.

4.4.4.3 For each test sample, solutions shall be prepared and each preparation shall be injected as mentioned in the standard test procedure.

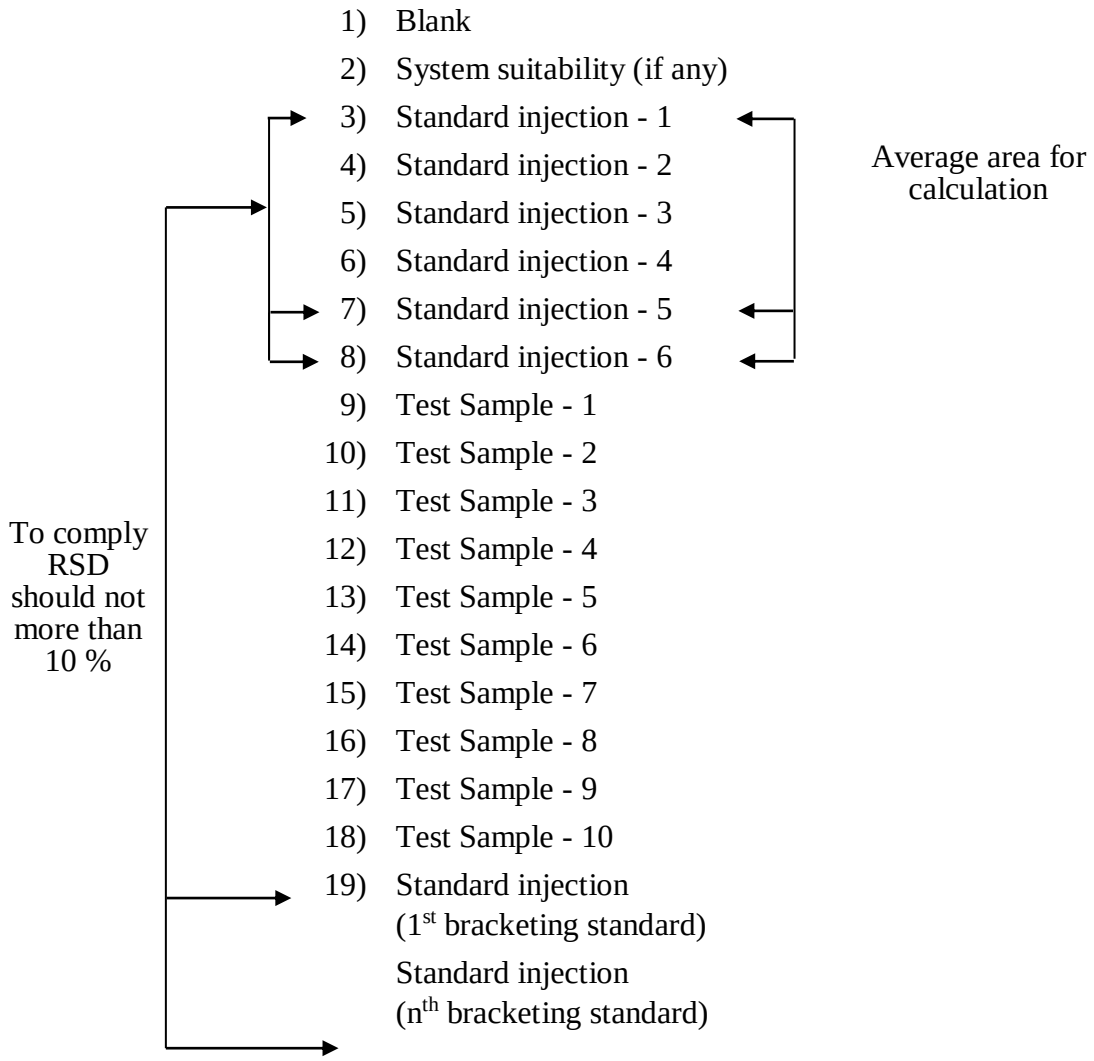
4.4.4.4 Bracketing standard is to be injected at the end of sequence or after set of 10 injections of sample, which ever earlier.

4.4.4.5 The RSD between the replicate standard injections and each respective bracketing standard injection should be not more than 10%

4.4.4.6 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.4.4.7 Schematically sequence is given with below diagram:

4.4.4.8 Schematically it is represented as follows:



4.5 For analysis on GC:

4.5.1 Assay, Related substances:

4.5.1.1 During analysis for assay and related substances by GC followed the method given by HPLC except acceptance criteria

4.5.1.2 The % RSD between the replicate standard injections and each bracketing standard injection should be not more than 15.0.

4.5.2 Residual solvent by GC

4.5.2.1 During residual solvents analysis by GC, the sample preparation to be made in duplicate and single injection shall be performed from each samples.

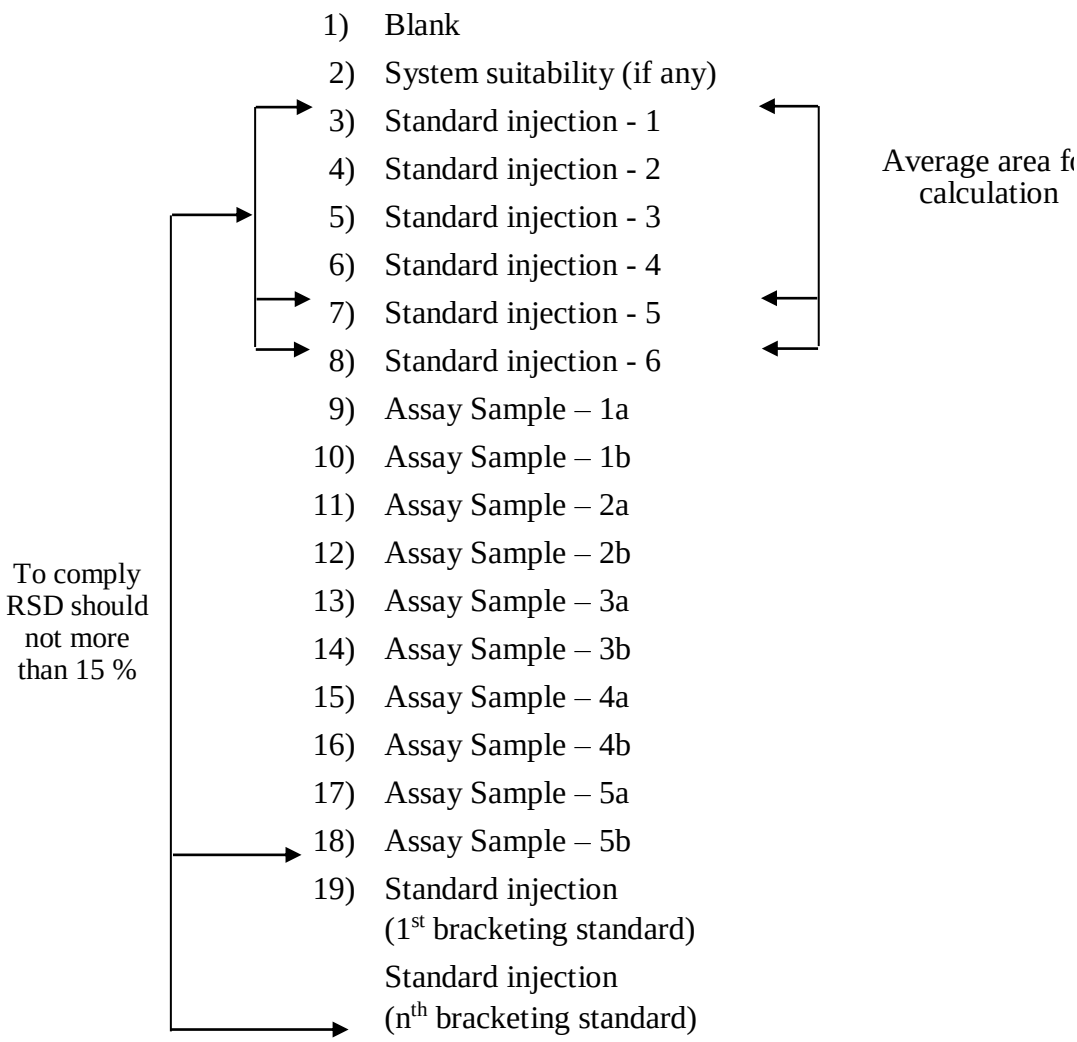
4.5.2.2 Bracketing standard is to be injected at the end of sequence or after set of 10 injections of sample, which ever earlier.

4.5.2.3 The % RSD between the replicate standard injections and each respective bracketing standard injection should be not more than 15.

4.5.2.4 Schematically sequence is given with below diagram:

4.5.2.5 Schematically it is represented as follows:

STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Bracketing of Standards during the analysis by HPLC, UV and GC	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**



4.6 Analysis by UV-VIS spectrophotometer

4.6.1 Dissolution analysis by UV

4.6.1.1 Photometric mode:

4.6.1.1.1 Measure the standard absorbance in photometric mode at specified λ_{max} followed by six samples.

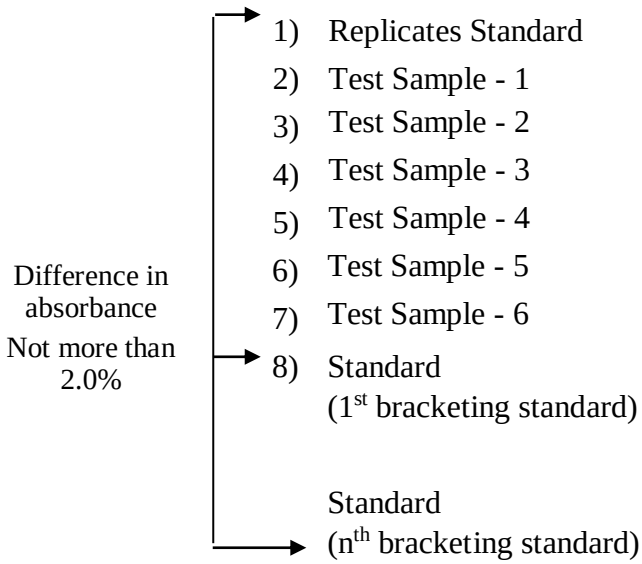
4.6.1.1.2 Bracketing standard is to be taken after every 6 samples.

4.6.1.1.3 The difference in absorbance between the replicate standards and bracketing standard should not be more than 2.0 %.

4.6.1.1.4 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.6.1.1.5 Schematically sequence is given with below diagram:

4.6.1.1.6 Schematically it is represented as follows:



4.6.2 Content Uniformity Test by UV-VIS spectrophotometer

4.6.2.1 Photometric mode:

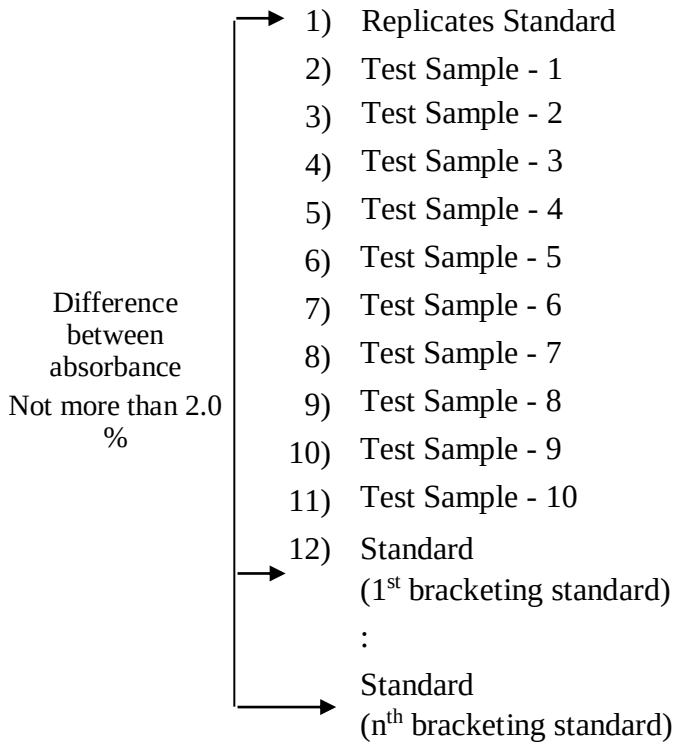
4.6.2.1.1 Measure the standard absorbance in photometric mode at specified λ_{max} followed by samples

4.6.2.1.2 Bracketing standard is to be taken after every 10 samples.

4.6.2.1.3 The difference in absorbance between the replicate standard and bracketing standard should not be more than 2.0 %..

4.6.2.1. Schematically it is represented as follows

STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Bracketing of Standards during the analysis by HPLC, UV and GC	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**



4.6.3 Assay by UV-VIS spectrophotometer

4.6.3.1 Scan mode:

4.6.3.1.1 Prepare sample preparation as per respective standard test procedure for one sample and analyze.

4.6.3.1.2 Perform the standard scan.

4.6.3.1.3 For each sample two scans to be performed.

4.6.3.1.4 Bracketing standard is to be taken after every 5 samples (Each duplicates).

4.6.3.1.5 The difference in absorbance between the first standard scan and the bracketing standard should not be more than 2%.

4.6.3.1.6 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.6.3.2 Photometric mode:

4.6.3.2.1 Measure the standard absorbance in photometric mode at specified λ_{max} followed

by samples.

4.6.3.2.2 For each sample two measurements to be performed.

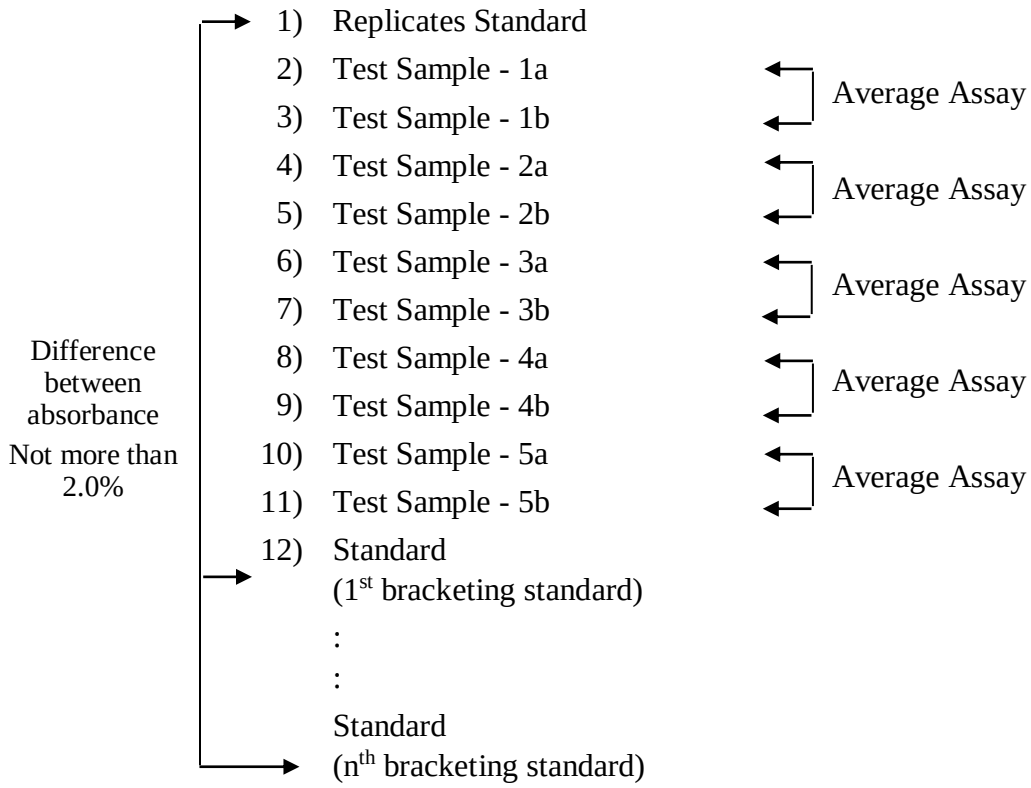
4.6.3.2.3 Bracketing standard is to be taken after every 5 samples (Each duplicates).

4.6.3.2.4 The difference in absorbance between the replicate standard and the bracketing standard should not be more than 2 %.

4.6.3.2.5 Ensure to comply the standard solution stability given in a respective standard test procedure.

4.6.3.2.6 Schematically sequence is given with below diagram:

4.6.3.2.7 Schematically it is represented as follows:



4.7 If the system is idle for 1 hour or more due to some minor problems that does not affect the system, then confirm the system is still suitable by injecting a bracketing standard before proceeding further.

4.8 When the % RSD of the area not mentioned in system suitability criteria in STP than use for bracketing standard initial system suitability solution (resolution solution etc.), and follows the procedure of respective test.

5.0 ANNEXURE (S):

Nil

6.0 REFERENCE (S):

SOP: Preparation, approval, distribution, control, revision and destruction of Standard Operating Procedure (SOP).

7.0 ABBREVIATION (S)/DEFINITION (S):

RSD : Relative Standard Deviation

HPLC : High Performance Liquid Chromatography

GC : Gas Chromatography

UV-VIS : Ultra Violet Visible

STP : Standard Test Procedure

NMT : Not more than

REVISION CARD

S.No.	REVISION No.	REVISION DATE	DETAILS OF REVISION	REASON (S) FOR REVISION	REFERENCE CHANGE CONTROL No.
1	00	---	---	New SOP	---