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SOP No.	PIPL/ARD/001-00	12/02/25/					
Department	Analytical Research Development	12/02/25					
Superseded No.	Nil	Review Date	11/02/271				

### 1.0 OBJECTIVE

1.1 To lay down the procedure for Operation and Calibration of High-Performance Liquid Chromatography (Shimadzu P Series With PDA)

### 2.0 SCOPE

**2.1** This procedure is applicable for Operation and Calibration of High-Performance Liquid Chromatography (Shimadzu P Series With PDA) in ARD Department at Pritam International Pvt. Limited.

### 3.0 RESPONSIBILITY

- **3.1** Chemist/Executive/Designee-ARD/Person shall follow the cleaning, operation & calibration of HPLC.
- 3.2 ARD Section Person shall be responsible for calibration of HPLC.
- **3.3** Manager-Designee shall ensure efficacy of the procedure of cleaning, operation & calibration of HPLC.

### 4.0 ACCOUNTABILITY

- 4.1 ARD Designee
- 4.2 ARD & QA Manager

### 5.0 PROCEDURE

### 5.1 OPERATION PROCEDURE

- **5.1.1** Switch ON the Instrument (Pump, Injector, Detector, Column Oven and Computer loaded with Lab Solution Software for the Data computation)
- **5.1.2 Start the Lab Solution Software** by double-clicking the Lab Solution icon located on desktop.
- **5.1.3** Each time start an instrument application (by double clicking the instrument icon (HPLC written), an **Instrument Wizard** shall appear. This wizard is designed to direct to the basic functions of the instrument window.
- **5.1.4** Then go to the **File** option on the menu bar and then click on **Method**, then click on **new** or **open** to create or to follow the existing method respectively.
- 5.1.5 For each sample enter the run parameters required for the sample under test.
- **5.1.6** Data acquisition and instrument control parameters: –

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- (a) Instrument Setup: Select each tab and enter the correct parameters for running the sample.
- (b) Integration events: Additional integration events are not necessary for a test sample.
- (c) Calibration of Peaks/Groups: Calibration parameters are not necessary for a test sample.
- (d) Advanced method options: Advanced method options are not necessary for a test sample.
- (e) Reports: From the File menu, select Report Template and then click Open. From the list of templates, select Area%. rep to generate an Area %report when run the sample
- 5.1.7 Create a sequence: This button starts the Sequence Wizard that steps you through creation of an acquisition reprocessing sequence. Or for creating sequence enter file menu which display Sequence, from Sequence enter new method.
- **5.1.8** Single sample run: This button opens a dialog, from this select a previously saved method to run a single sample.
- 5.1.9 In the Single Run Acquisition dialog box, enter a number to be used as a Sample ID.
- 5.1.10 Click the method open button and then select the method file that was provided or the method you saved in the previous step.
- 5.1.11 Enter a **Data file** name you wish to use, or click the adjacent button to select a parameter that will be used to create the data file name for you.
- 5.1.12 Enter the Vial number and Injection Volume to be used.
- **5.1.13** Run sequence of samples: This button opens the Run Sequence dialog where you can start data acquisition using a stored sequence.
- **5.1.14** Save the method. When you have completed setting up the method parameters, from the File menu, followed by Method and click Save As. Type a name to be used for your method and then click OK.
- **5.1.15** Report any discrepancy observed during operation and calibration of instrument to Section In charge or his representative for corrective and preventive action.
- **5.1.16** Section In charge or his representative shall take the necessary action and report the same to Quality Manager. Affix 'Under Maintenance' label on HPLC

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### 5.2 CALIBRATION PROCEDURE

### 5.2.1 CALIBRATION FREQUENCY: HALF YEARLY $\pm$ 20 days.

### 5.2.2 PRECAUTIONS

- **5.2.2.1** For calibrating the Column oven, use only calibrated thermometer.
- **5.2.2.2** For calibration of pump flow rate use only calibrated stopwatch & Analytical Balance.

### 5.2.3 CALIBRATION PARAMETERS

The following components of system shall be calibrated.

A) Pump Calibration

i) Flow rate accuracy.

ii) Gradient composition accuracy.

B) Injector calibration:

i) Linearity & Carry over test.

ii) Injector Precision / Injector Reproducibility &

Repeatability

iii)Injector Volume Accuracy.

iv) Vial Positioning.

C) Column Oven Temperature

i) Temperature Calibration

D) Detector Calibration

i) Linearity of Detector Response

ii) Wavelength accuracy

iii) Noise and drift

E) Lamp Intensity

i) Lamp intensity to be verified and maintain

the record on monthly basis.

### A) PUMP CALIBRATION

### (i) FLOW RATE ACCURACY:

- a. Use Milli Q water (Degassed & filter) as a mobile phase for calibration of pump.
- b. Before starting the calibration purge the system two or three times to remove air bubbles.
- c. Before starting calibration set pump at constant flow and discard the volume for five minutes.
- d. Take a previously dried 10 ml Volumetric Flask and weigh it, note the weight of the Volumetric Flask (W1) and install a union in column compartment simultaneously start the required flow and stop watch for 5 minutes and collect it. After five minutes stop the flow and weight the collected volume in Volumetric Flask (W2) calculate the actual volume collected in Volumetric Flask (W2-

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W1=W3). Calculate the corresponding flow rate by using formula = (weight of collected volume /Time Taken x 0.997).

e. Select a flow rate of 0.5ml/min, 1.0 ml/min, 1.5ml/min, and 2.0 ml/min and perform the same exercise. Repeat the step same as above for calibration of pump Chanel B C D (if applicable).

**Acceptance Criteria**= The flow rate should be within  $\pm$  2% of the set value.

### (ii) GRADIENT COMPOSITION ACCURACY

Set the following chromatographic conditions

Column

: Restriction capillary

Solvent for Pump A

: Filtered and degassed purified water Solvent for Pump B,C& D : 10 ppm caffeine in purified water

Flow rate

: 2.0 ml/min

Wavelength Injection volume

: 272 nm : 0.00 µl

Run time

: 30 min

Gradient programming:

Step	Time in minute	Flow Rate	% Solvent A (Water)	%Solvent B (10 ppm	%Solvent C (10 ppm	%Solvent D (10 ppm
-		(ml/min)		Caffeine)	Caffeine)	Caffeine)
1	5.00	2.0	100.0	0.0	0.0	0.0
2	5.01	2.0	90.0	10.0	10.0	10.0
3	10.00	2.0	90.0	10.0	10.0	10.0
4	10.01	2.0	50.0	50.0	50.0	50.0
5	15.00	2.0	50.0	50.0	50.0	50.0
6	15.01	2.0	10.0	90.0	90.0	90.0
7	20.00	2.0	10.0	90.0	90.0	90.0
8	20.01	2.0	0.0	100.0	100.0	100.0
9	25.00	2.0	0.0	100.0	100.0	100.0
10	25.01	2.0	100.0	0.0	0.0	0.0
11	30.00	2.0	Stop			,

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### 5.2.4Procedure of Pump Calibration:

\* Purge all pump including rinse at required time.

Set the flow rate at 2.0 ml per minute. Keep the system in this flow rate for some time to equilibrate. Start the gradient programming. After completion of Gradient programming calculate actual concentration by using formula below

S. No.	Set Nominal Height (%)	Observed Height A X 100 B	Tolerance Limit 2%
1	0		0.00
2	10		9.8 - 10.2
3	50		49.0 – 51.0
4	90		88.2 – 91.8
5	100		98.0 – 102.0

### A= Actual Height

B = Maximum height at 100 %

Acceptance criteria: At each absorbance level  $\pm 2\%$  of set concentration

### 5.2.4.1PREPARATION OF CALIBRATION STANDARD SOLUTION:

### (A) Requirements:

- Standard caffeine
- Acetonitrile and water HPLC Grade.
- Diluent (70:30) Acetonitrile water.
- 100 ml, 50 ml and 20 ml Volumetric Flask.
- 1 ml, 2 ml, 5 ml, 10 ml Bulb pipette.

### (B) Preparation of caffeine stock solution

Weigh accurately about 50.0 mg of caffeine and transfer to a 100.0 ml volumetric flask, add 50 to 75 ml of mobile phase, sonicate for 5 minutes for dissolve and makeup the volume up to 100.0 ml with mobile phase. This solution contents about 0.5 mg/ml of caffeine.

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### (C) Working Solution of caffeine:

- (a) Dilute 1 ml of above stock solution to 100.0 ml with diluent (5 ppm)
- (b) Dilute 2 ml of above stock solution to 100.0 ml with diluent (10 ppm)
- (c) Dilute 2 ml of above stock solution to 50.0 ml with diluent (20 ppm)
- (d) Dilute 5 ml of above stock solution to 50.0 ml with diluent (50 ppm)
- (e) Dilute 10 ml of above stock solution to 50.0 ml with diluent (100 ppm)
- (f) Dilute 10 ml of above stock solution to 20.0 ml with diluent (250 ppm)

### (D) LINEARITY OF DETECTOR RESPONSE:

For Photo Diode Array Detector (PDA)

### Chromatographic parameter

Flow rate

: 1.0 ml/min.

Wave length

: 272 nm (200-400)nm

Column

: ODS, C18, 250mm X 4.6 mm,  $5\mu$  or any suitable column

Mobile Phase

: 70:30(Acetonitrile: Water)

### Procedure:

- (a) Set the detector at \( \lambda \) max.
- (b) Place the vials containing mobile phase i.e. blank and caffeine solutions having conc. of 5 ppm, 10 ppm, 20 ppm, 50 ppm & 100 ppm in the given sequence in the auto sampler. Program the system to successively inject 20 µl each of the mobile phase (MP) to serve as blank, therefore- mentioned five caffeine solutions and the MP again (second blank) at the end and record their chromatograms.
- (c) Read the areas of principal peaks in each chromatogram.
- (d) Enter the peak areas corresponding to each conc. And also, area of any caffeine peak in the chromatogram of the second blank.
- (e) A plot of the solution concentrations vs. the areas should be a straight line with its correlation coefficient greater than 0.999.

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### (E) CARRY OVER

Inject 20 µl solution as per Sequence Blank, Standard solution 250 ppm \_\_\_ Blank, calculate area if any peak at the same RT of standard. Operate the Instrument as per given parameters. Acceptance criteria is NMT 0.01%.

Calculate the carryover % & as shown below.

Peak area in Blank × 100

Carryover % = Peak area in Sample

### (F) INJECTOR PRECISION (Reproducibility & repeatability):

Inject 5 injection of Standard caffeine 20.0 ppm solution prepared above And calculate the RSD of area and RT

Acceptance Criteria: NMT 1.0% for area and NMT 0.5% for RT

### (G) WAVE LENGTH ACCURACY:

### For Photo Diode Array Detectors (PDA)

### Chromatographic parameter

Flow rate

: 1.0 ml/min.

Wave length

: (190-800)nm

Column

: ODS, C18, 250mm X 4.6 mm, 5µ or any suitable column

Mobile Phase

: 70:30(Acetonitrile: Water)

Set detector wavelength at 190 nm to 800 nm. Inject 20 µl of 0.01 mg/ml solution of caffeine. Record spectrum and report maxima and minima.

### **Acceptance Criteria**

Wavelength maxima found should be between 273±2 nm.

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Wavelength maxima found should be between 205±2 nm. Wavelength minima found should be between 245±2 nm.

### (H) INJECTOR CALIBRATION:

### a. Injector Volume Accuracy:

- Fill a HPLC vial with Degassed HPLC grade water.
- Seal the vial with septum and cap.
- Zero the analytical balance, and then carefully weigh the vial, Record the weight W1.
- Place the weighed vial in position 1 of the sample tray in the sample compartment.
- Fill the reservoir A with degassed 100% HPLC- grade water, then prime the solvent delivery system
- Prepare programmed in software as injection volume inject 15 injections of 20.0 μl.
- Remove the vial and reweigh the vial (W2). Repeat the process again as per above.
- To calculate the average volume of water injected per injection.
- Repeat it with 15 injections of 10 μl, 15 injections of 50 μl & 10 injections of 100 μl.

[{(W1-W2) X 1000} / {Number of injection X Density of Water}] = μL per injection Where density of water at 25°C is 0.997g/ml Acceptance criteria: ±2.0 %

### b. Injector Linearity:

• Inject separately 5, 10, 20, 50 and 100µl injection in duplicate of 20ppm solution.

### c. Test of Vial Positioning:

- Detector Linearity solution shall be use for the vial positioning test.
- Keep the Linearity solution vials at random position across the injector tray.
- All injector trays should be used with random position. Acceptance Criteria:
- All Vials to be picked/punctured as per program.

### (I) Column Oven Calibration (Temperature Calibration)

• Switch on the column oven and set Temperature at 40°C.

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- Remove the lift and right-side plastic opening center clip and insert the standard Calibrated thermometer to the middle of column compartment.
- Properly packs the side space of thermometer with cotton for proper insulation.
- Now wait up to ready position lamp (up to set temperature is achieved).
- Note down the Temperature in thermometer and repeat the exercise with temperature at 20°C & 60°C.

Acceptance Criteria: The observed reading (current) and standard thermometer reading do not differ by more than  $\pm$  2°C from the set temperature.

Note: Calibration of Column Oven Temperature from External Lab once in a year.

### (J) Noise and Drift

This procedure based on ASTM standard E 685-93 determines the noise & drift.

### **Conditions:**

Sequence : PQ PDA NOISE DRIFT

Program : PDA\_DET\_NOISE\_AND\_DRIFT

Flow : 1 mL/minute
Stop Time : 21.0 minutes
Solvent A : Milli Q water

Temperature : 40.0°C Wavelength : 254

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**Procedure:** The checks for noise, drift, and lamp intensity are included in the PDA Noise Drift sequence. For those checks, water is pumped through the cell at a flow rate of 1 ml/min. The PDA signal is recorded at 254 nm.

To calculate drift and noise, the measuring signal is split into 20 intervals of 1 minute each. For each interval, Chromeleon calculates a regression based on measured values, using the method of least squares. The slope of the curve indicates the drift of the measured signal; the absolute value of the slope indicates the absolute value of the drift. The noise is the distance between two parallel lines through the measured minimum and maximum values and the regression line. The calculated values are averaged for all 20 intervals to establish the final value.

Acceptance criteria: Noise: ±0.2mAU

Drift: ±2.0mAU/h (for VWD) ±5.0mAU/h (for DAD)

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### 6.0 REFERENCES

Indian Pharmacopoeia Commission, In-House

7.0 ABBREVIATION (S)

S. No	Abbreviation	Full Description
1	SOP	Standard Operating Procedure
2	PIPL	Pritam International Pvt. Ltd.
3	No.	Number
4	ARD	Analytical Research Development
5	RSD	Relative Standard Deviation
6	HPLC	High performance Liquid Chromatography
7	OQ	Operation Qualification
8	PDA	Photo Diode Array Detector

8.0 ANNEXURE (S)

Annexure No.	Details/Title of Annexure	Format No.
PIPL/ARD/001-A01	Format for Calibration Index for HPLC	PIPL/ARD/001/F01-00
PIPL/ARD/001-A02	Format for Calibration Data Sheet	PIPL/ARD/001/F02-00
PIPL/ARD/001-A03	Format for HPLC Calibration Summary	PIPL/ARD/001/F03-00
	Sheet	
PIPL/ARD/001-A04	Format for standard preparation sheet	PIPL/ARD/001s/F04-00

### 9.0 CHANGE HISTORY

S. No.	Supersedes Changes made		Effective Date	
01	Not Applicable	New Sop	12/02/25	

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10.0 DISTRIBUTION/ISSUANCE						
S. No.	Department	Issuance	S. No.	Department	Issuance	
1.	Quality Assurance	Yes \[ \sqrt{No} \]	10.	Planning & Purchase	Yes No V	
2.	Quality Control	Yes No	11.	Accounts	Yes No 1	
3.	Microbiology Lab	Yes No V	12.	F&D	Yes No V	
4.	Production (Mfg.)	Yes No 1	13.	NPD	Yes No 1	
5.	Production (Packing)	Yes No \(	14.	Human Resources	Yes No 1	
6.	RM Store	Yes No 1	15.	Maintenance	Yes No V	
7.	PM Store	Yes No V	16.	Information Technology	Yes \[ \sqrt{No} \]	
8.	FG Store	Yes No V	17.	House Keeping	Yes No 1	
9.	Warehouse	Yes No 1	18.	ARD	Yes \( \sqrt{No} \)	

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### ANNEXURE - PIPL/ARD/001-A01

### Format for Calibration of HPLC

Locatio	on		Instru	ment ID			
Make			Model	No.			
Calibra	ation Date		Calibr	ation due date			
Referen	ice SOP No		Frequ	ency	6 months ± 20 days		
Sr. No	Calibration paramete	•		Page No			
1	Index	A Company of the Comp					
2	Flow Rate Accuracy			Andrew Control of the			
3	Gradient Composition	n Accuracy					
4	Standard Preparation	Sheet					
5	Injection Precision/ In Repeatability	njector Reproducibil	ity &	×			
6	Injector Calibration						
7	Vial Positioning						
8	Carry Over Test						
9	Linearity of Detector	Response			1		
10	Wavelength Accuracy						
11	Noise and Drift						
Remarks: The Instrument Calibration & Performance is Complies / Not Complies							
Calibra	nted By	Checked By		Approve	ed By		
Date:		Date:	Date:				



# PRITAM INTERNATIONAL PVT. LIMITED PL ROOKK

ANNEXURE - PIPL/ARD/001-A02

	THANKA WYAVAL																				
	CALIBRATION DATA SHEET Calibration Parameter – 1: Flow Rate Accuracy																				
Inst	Instrument ID :																				
Cui	rent Loc	ation	ation : ARD Instrument Room-01																		
Calibration Frequency : HALF YEARLY ±20 Days																					
Dat	Date of Calibration : Next Due Date of Calibration :																				
Nar	Name of Equipment ID No. of Equipment Validity of Calibration																				
<b> </b>	lytical B																				
Stop	Watch								4												
Den	sity of W	ater at	25°(	C = 1	D (0.99	<b>)</b> 7)															
					7					1											
Cal	libration	of Pum	p -A																		
	Set Flow rate (ml/min)	Time	beaker beaker + water water (ml) Flow rate Limit																		
1.	0.5			1				-							0.49-0.51						
2.	1.0														0.98–1.02						
3.	1.5												,		1.47-1.53						
4.	2.0			***************************************					-						1.96-2.04						
Rei Tol	narks: C erance li	alibrati mits.	on o	f Pı	ımp fo	r Flow	rate	e accu	ıracy	Comp	lies/ do	es not	com	plie	s with the						
Cal	ibration	of Pum	p -B											2308							
1.	0.5					MANAGEM LANGE									0.49-0.51						
2.	1.0							1000000							0.98–1.02						
3.	1.5												3		1.47-1.53						
4.	2.0										(37)				1.96-2.04						
Ren	narks: C	alibrati	on o	f Pu	ımp fo	r Flow	rate	accu	racy	Comp	lies/ doe	es not	com	Remarks: Calibration of Pump for Flow rate accuracy Complies/ does not complies with the							



### PRITAM INTERNATIONAL PVT. LIMITED

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

### Tolerance limits.

	Set Flow rate (ml/m	Total Time (min) =T	Wt. of beaker (g) =W1	Wt. of beaker + water (g) =W2	Weight of water W2-W1= W	Volume of water (ml) W/D = V	Observed Flow rate (ml/min) =V/T	Tolerance Limit (ml/min)
1.	in) 0.5							0.49-0.51
2.	1.0							0.98-1.02
3.	1.5							1.47-1.53
4.	2.0	No de deservo						1.96-2.04

Remarks: Calibration of Pump for Flow rate accuracy Complies/ does not complies with the Tolerance limits.

Calibration of Pump -D	,
1. 0.5	0.49-0.51
2. 1.0	0.98-1.02
3. 1.5	1.47-1.53
4. 2.0	1.96-2.04

Remarks: Calibration of Pump for Flow rate accuracy Complies/ does not complies with the Tolerance limits.

The state of the s	Checked By: Sign. /Date:





# PRITAM INTERNATIONAL PVT. LIMITED PIPL ROORKE

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

### **CALIBRATION DATA SHEET** Calibration Parameter – 2: Gradient Composition Accuracy

Chro	matographic	Conditions:	-		
Para	meter	Details	P	arameter	Details
Run	Γime (Min.)		V	Vavelength (nm)	272 nm
Solve	ent for Pump		Mobile Phase	10 ppm Caffeine	
		Gradie	ent	Composition	
Sr.	Set	Observed H	eig	ht	Tolerance Limit (%)
No.	Nominal Height (%)	A X 100 B	II	(%)	
1.	0		=		0.00
2.	10		=		9.8 – 10.2
3.	50		=		49.0 – 51.0
4.	90		=		88.2 – 91.8
5.	100		=		98.0 – 102.0
	ctual Height Iaximum heig	ht at 100 %			·
Accer	otance criteri	a: ±2% of set concentration	on		
Rema	rks: Complie	es / does not comply			
g					
Calibr Sign. /	ration By: Date:			necked By: gn. /Date:	
	8				



# PRITAM INTERNATIONAL PVT. LIMITED DERES ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

Chromatographic Conditions:									
	meter	,	Details	P	arameter		Details		
Run '	Γime (Min).			V	Vavelength (nm)	272 nm			
Solve	ent for Pump			N	Mobile Phase	10 ppm Caffeine			
	Gradient Composition								
Sr.	Set		Observed 1	Heig	ht	T	olerance Limit (%)		
No.	Nominal		A X 100	T			(,,)		
	Height (%)		C	=	(%)				
1.	0						0.00		
				=			0.00		
	10						8		
2.	10						9.8 - 10.2		
				=					
3.	50						49.0 – 51.0		
				=			49.0 – 31.0		
4.	90		×				88.2 – 91.8		
				=					
5.	100						00.0 100.0		
٥.	100			=			98.0 – 102.0		
	ctual Height Iaximum heigl	nt at 100	) %						
Accep	tance criteria	n: ±2%	of set concentrat	ion					
Rema	rks: Complie	s / does	not comply			,			
Calibr	ation By:			Ch	ecked By:	-			
Sign. /					n. /Date:				
			2						

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### ANNEXURE - PIPL/ARD/001-A02

Chromatographic Conditions:									
Parai			Details	P	arameter		Details		
Run T	Time (Min).			V	Vavelength (nm)		272 nm		
Solve	nt for Pump			N	Iobile Phase		10 ppm Caffeine		
	Gradient Composition								
Sr.	Set		Observed I	Ieigl	nt	T	olerance Limit (%)		
No.	Nominal		A X 100		hand sales				
	Height (%)		D	=	(%)				
1.	0		(1) (1) (1) (1) (1) (1) (1) (1) (1) (1)				0.00		
				=					
2.	10			-			9.8 – 10.2		
2.				=			7.0 10.2		
	1	-14							
3.	50			=			49.0 – 51.0		
4.	90		Y,			***************************************	88.2 – 91.8		
				=					
5.	100	2.					98.0 – 102.0		
J.	100			=			96.0 - 102.0		
	ctual Height								
B = V	laximum heig	ht at 10	0 %						
Accep	otance criteri	a: ±2%	% of set concentrat	ion					
D		/ 1			9 <b>4</b>				
Rema	rks: Complic	es / aoe	s not comply						
Calibration By: Checked By:									
Sign. /					gn. /Date:				
							s		



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Sign./Date: ....

### PRITAM INTERNATIONAL PVT. LIMITED ROOK

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

### **CALIBRATION DATA SHEET**

Calibration Parameter - 3: Injection Precision (Reproducibility & repeatability)

2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By: Checked By:		Standard Caffeine Solution		
No.  1  2  3  4  5  Mean  SD  RSD (%)  Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 %  2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.	Solvent/Dil	ent : Y	Water: Acetonitrile (30:70)	
1 2 3 4 5 Mean SD RSD (%)  Acceptance Criteria 1. Retention Time = RSD should not be more than 0.5 % 2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.		Retention Time	Area	
3 4 5 Mean SD RSD (%)  Acceptance Criteria 1. Retention Time = RSD should not be more than 0.5 % 2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.				
4 5 Mean SD RSD (%)  Acceptance Criteria 1. Retention Time = RSD should not be more than 0.5 % 2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.	2			
Mean  SD  RSD (%)  Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 % 2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.	3			
Mean  SD  RSD (%)  Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 %  2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By: Checked By:	4			
SD  RSD (%)  Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 %  2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By:  Checked By:	5			-
Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 %  2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By: Checked By:	Mean			
Acceptance Criteria  1. Retention Time = RSD should not be more than 0.5 %  2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By: Checked By:	SD			
1. Retention Time = RSD should not be more than 0.5 % 2. Area = RSD should not be more than 1.0 %  Remarks: Complies / does not comply.  Calibration By: Checked By:	RSD (%)			
NA PRO	2. Area =	RSD should not be more than 1	more than 0.5 % 1.0 %	
		•	,	
		ky:	A CONTRACT OF THE CONTRACT OF	
		By:	A CONTRACT OF THE CONTRACT OF	
		;y:	A CONTRACT OF THE CONTRACT OF	
		By:	A CONTRACT OF THE CONTRACT OF	





# PRITAM INTERNATIONAL PVT. LIMITED PIPL ROORKEE

ANNEXURE - PIPL/ARD/001-A02

			TION DATA SHEET
Calibra	atio	on Paramo	eter – 4: Injector Calibration
Chromatographic conditions:		XX7-4 (1	1000()
Mobile phase/Diluent	:	Water (1	100%)
Injection volume (µl)	:	10 μ1	
Run Time (Min.)	:		
A. Calibration of Injection V	olu	ıme Accui	
Injection Volume			10μl from Vial.
Number of injections			15
Weight of vial with water (g) [V	W1]	]	
Weight of vial after injection (g	) [V	W2]	
W1-W2=W (in g)			
Density of water at 25°C			0.997
Observed Volume [W x1000/0.	997	7x15]	·
Acceptance criteria (μl) (±2% o	f Ir	njection	9.8 to 10.2
volume)		u	
	-		
Calibration By: Sign. /Date:			Checked By: Sign. /Date:
			,



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### ANNEXURE - PIPL/ARD/001-A02

Chromatographic conditions:					
Mobile phase/Diluent	: Wate	r (10	0%)		
Injection volume (µl)	: 20 μl				
Run Time (Min.)					
B. Calibration of Injection Vo	olume Ac	cura	cy: -		·
Injection Volume				20μl from Vial.	
Number of injections				15	
Weight of vial with water (g) [V	V1]				
Weight of vial after injection (g)	) [W2]		XXVXXXXXXXX		
W1-W2=W (in g.)					
Density of water at 25°C				0.997	
Observed Volume [W x1000/0.9			я	(4)	
Acceptance criteria (µl) (±2% o	f Injectior	ı		19.6 to 20.4	
volume)					-
Calibration By:		·	Checked By:		
Sign. /Date:			Sign. /Date:	3	
			7 .	ž.	



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ANNEXURE - PIPL/ARD/001-A02

Chromatographic conditions:				
Mobile phase/Diluent	:	Water (10	00%)	
Injection volume (µl)	  -	50 μl	0070)	
Run Time (Min).	:			
C. Calibration of Injection		ıme Accura	acv: -	
Injection Volume			50μl from Vial.	
Number of injections			15	
				117 - 1
Weight of vial with water (g)	[W1	]		
Weight of vial after injection	(g) [	W2]		
W1-W2=W (in g)				1300
Density of water at 25°C			0.997	
Observed Volume [W x1000/	0.99	7x15]		
Acceptance criteria (µl) (±2%	of I	njection	49.0 to 51.0	×
volume)				
Calibration By: Sign. /Date:  Checked By: Sign. /Date:				
	***************************************			
,		40	•	
	2			



# PRITAM INTERNATIONAL PVT. LIMITED L ROORKEE

ANNEXURE - PIPL/ARD/001-A02

Chromatographic condition	~ .		
Chromatographic condition  Mobile phase/Diluent	:	Water (1	00%)
Injection volume (µl)	:	100 µl	
Run Time (Min.)	·  :	Τουμι	
D. Calibration of Injection		ime Accur	*QAV* _
Injection Volume	VOIC	me Accui	100μl from Vial.
Number of injections			10
Weight of vial with water (g)	[W1	]	
Weight of vial after injection (	(g) [	W2]	
W1-W2=W (in g)			
Density of water at 25°C			0.997
Observed Volume [W x1000/0	).99′	7x15]	
Acceptance criteria (μl) (±2%	of I	njection	98.0 to 102.0
volume)			
Calibration By: Sign. /Date:			Checked By: Sign. /Date:



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# PRITAM INTERNATIONAL PVT. LIMITED ROORKEEN OF THE PRITAM INTERNATIONAL PVT.

ANNEXURE - PIPL/ARD/001-A02

E Injector Lincovity				
E. Injector Linearity:  Chromatographic conditions:				
Mobile phase/Diluent	Wator Ao	otonitrilo (20.70)		
	water: Ac	etonitrile (30:70)		
Injection volume (μL)	20 μl			
Column ID				
Flow rate (ml/min)	1.0 ml/min			
Detector Wavelength (nm)	272 nm	,		
Column Oven temperature (°C)	30°C			
Inject separately 5 μl, 10 μl, 20 μ Standard Solution	սl, 50 µl & 1	00 μl injection in duplicate of 20 ppm Caffeine		
Injection Volume (μl)		Mean Area		
5 µl				
10 μ1				
20 μ1		,		
50 μl				
100 μl				
Acceptance Criteria = Area vs. Injection volume graph should be linear and Correction coefficient should be greater than 0.999				
Result:				
1. Area vs. Injection volume gr	aph is linear	/not linear.		
2. Correction coefficient is great	2. Correction coefficient is greater/not greater than 0.999. (Correction coefficient			
).				
Calibration By:		Checked By:		
Sign. /Date:		Sign. /Date:		



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### PRITAM INTERNATIONAL PVT. LIMITED

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

### F. Vial Positioning:

Procedure: Inject Blank and single injection of standard solution of each dilution.

Note: Use the all concentration solution of Detector Linearity test dilution.

Sr. No.	Vial Program	Tray Name	Concentration	Vial Analysed	Remark
1			5 ppm		
2			10 ppm		
3			20 ppm		
4			50 ppm		
5			100 ppm		
		Acceptance Cri	iteria: All Vials to be pic	ked/punctured as j	per program.

Calibration By:	Checked By:
Sign. /Date:	Sign. /Date:



# PRITAM INTERNATIONAL PVT. LIMITED ROORKEE

### **HPLC Calibration**

Calibration Parameter – 5: Carry Over		
<b>Chromatographic conditions:</b>		
Mobile phase/Diluent	:	Water: Acetonitrile (30:70)
Injection volume (μL)	:	20μ1
Column ID	:	
Detector Wavelength (nm)	:	272 nm
Use 250 ppm Caffeine standard so	luti	on:
Calculation for Carry over (%)		
Area observed of any peak due to Ca	ıffei	ne in post blank (A) :
Area of Principal peak in the chroma	itog	ram of the Caffeine :
solution (B)		
Carry over (%) = (Ax100/B)		÷
Acceptance Criteria: NMT 0.01%		
Remarks	:	Complies / does not comply.
		-
C. III		
Calibration By: Sign. /Date:		Checked By: Sign. /Date:
Sign. /Date.		Sign. /Datc.
,		
		·
-		
		-

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Sign./Date: .....

### PRITAM INTERNATIONAL PVT. LIMITED ROOR

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

### CALIBRATION DATA SHEET

Calibration Parameter – 6: Linearity of Detector Response		
Chromatographic conditions:		
Mobile phase/Diluent	Water: Acetonitrile (30:70)	
Injection volume (μL)	20 μL	
Column ID		
Flow rate (ml/min)	1.0 ml/min	
Detector Wavelength (nm)	272 nm	
Column Oven temperature (°C) 30°C		
Preparation of Caffeine Standard Solution		
Use 5 ppm, 10 ppm, 20 ppm, 50 ppm & 100 ppm Caffeine Standard Solution		

Inject triplicate injection (each) of the above 5 ppm, 10 ppm, 20 ppm, 50 ppm, 100 ppm solution separately. Find out area of the resulting chromatogram. Plot the Graph of Area vs. Concentration.

Concentration of caffeine solution	Mean Area	
(in ppm)		
5 ppm		
10 ppm		
20 ppm		
50 ppm		
100 ppm		
Acceptance Criteria = Area ver. conc	centration graph should be linear and Correction	
	be greater than 0.999.	
<ol> <li>Area vs. concentration graph is linear /not linear.</li> <li>Correction coefficient is greater/not greater than 0.999. (Correction coefficient).</li> </ol>		
Calibration By:	Checked By:	
Sign. /Date:	Sign. /Date:	



### PRITAM INTERNATIONAL PVT. LIMITED ROORKEE V2

### ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

# CALIBRATION DATA SHEET Calibration Parameter – 7: Wavelength Accuracy

Chromatographic conditions:		
Mobile phase/Diluent	Water: Acetonitrile (30:70)	
Injection volume (μL)	20 μL	
Column ID		
Flow rate (ml/min)	1.0 ml/min	
Detector Wavelength (nm)	268 nm to 276 nm	
Column Oven temperature (°C)	30 °C	
Use Caffeine standard	20 ppm	

Wavelength(nm)	Area of Caffeine solution of 20 ppm
269	
270	
271	
272	:
273	
274	
275	
276	

**Observation:** λ max obtained at: .....nm.

ACCEPTANCE CRITERIA = Area should be maximum at wavelength  $272 \pm 2$  nm.



### PRITAM INTERNATIONAL PVT. LIMITED

### ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

Chromatographic conditions:	
Mobile phase/Diluent	Water: Acetonitrile (30:70)
Injection volume (μL)	20 μL
Column ID	
Flow rate (ml/min)	1.0 ml/min
Detector Wavelength (nm)	241 nm to 248 nm
Column Oven temperature (°C)	30 °C
Use Caffeine standard	20 ppm

Wavelength(nm)	Area of Caffeine solution of 20 ppm
241	
242	
243	
244	
245	
246	
247	
248	

**Observation:** λ min obtained at: .....nm.

ACCEPTANCE CRITERIA = Area should be minimum at wavelength  $244 \pm 2$  nm.

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Sign./Date: ..... Sign./Date: ....

### PRITAM INTERNATIONAL PVT. LIMITED 8

### ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

Chromatographic conditions:		
Mobile phase/Diluent	Water: Acetonitrile (30:70)	
Injection volume (μL)	20 μL	
Column ID		,
Flow rate (ml/min)	1.0 ml/min	
Detector Wavelength (nm)	202 nm to 209 nm	
Column Oven temperature (°C)	30 °C	
Use Caffeine standard	20 ppm	

Wavelength(nm)	Area of Caffeine solution of 20 ppm
202	
203	
204	
205	
206	
207	
208	
209	

ACCEPTANCE CRITERIA = Area should be maximum at wavelength 205 ± 2 nm

Remarks: Complies/does not complies



### PRITAM INTERNATIONAL PVT. LIMITED

ANNEXURE - PIPL/ARD/001-A02

### **HPLC Calibration**

## **CALIBRATION DATA SHEET Calibration Parameter – 8: Column Oven**

	Date of Calibration
Digital	
Thermometer ID	Valid up to

1. Cleanliness of the Instrument:

Ok/Not Ok

**Column ID:** 

2. Calibration of Column Oven: 2.1 Set Temperature: at 20°C

Sr. No.	Observed Temperature	Limit
01		±2°C
Mean		(18-20) °C

### 2.2 Set Temperature: at 40°C

Sr. No.	Observed Temperature	Limit
01		±2°C
Mean		(38-40) °C

### 2.3 Set Temperature: at 60°C

Sr. No.	Observed Temperature	Limit
01		±2°C
Mean		(58-60) °C

Calibration By:	Checked By:
Sign. /Date:	Sign. /Date:



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Sign./Date:

### PRITAM INTERNATIONAL PVT. LIMITED ROORKEE

### ANNEXURE - PIPL/ARD/001-A02

Calibration Param Observation:	neter – 9: Noise and Drift
Remarks: Complies/does not complies	
Note: Calibration of Column Oven Temper	ature from External Lab once in a year.
	*
Calibration By: Sign. /Date:	Checked By: Sign. /Date:





# Format for HPLC Calibration Summary Sheet

Calibration Bus Evariation         Calibration of the pump of the pum	INSTRUMENT ID :					Calibration Date	ate		
Test Name         Observation         Acceptance Criteria           bration of the pump flow rate flo	a					Calibration D	ue Date		
Flow Rate   Flow Rate   Flow Accuracy   (±2 % of set flow rate)	Test Name			Obse	ervation			Acceptance Criteria	Status
flow rate         (ml/min)         A         B         C         D           0.5         0.5         0.5         0.49-0.51           1.0         1.0         0.98-1.02           1.5         0.98-1.02         1.47-1.53           1.5         1.0%         50%         90%         100%         At each absorbance level ±2%           1 min Oven         Limit — • 0.00         9.8 to 10.2         49.0 to 51.0         88.2 to 91.8         98.0 to 102.0         ± 2.0°C of set value           1 corducibility & rate bility by atability by g20 ppm Caffeine}         RSD of Area         RSD of Area         RSD of Area         RSD of Area         RSD of area NMT 1.0 %	Calibration of the pump	Flow	, Rate		Flow Ac	curacy		(±2 % of set flow rate)	
1.0   1.0	flow rate	/lm)	min)	A	В	C	Q		
1.0   0.98-1.02   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.96-2.04   1.06 %   1.06 %   1.06 %   1.00		0	5.					0.49-0.51	
1.5   1.6   1.0   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.47-1.53   1.96-2.04     Indicact Composition   0 %   10 %   50 %   90 %   100 %   At each absorbance level ±2%   1.96-2.04     Indicact Composition   0 %   10 %   50 %   100 %   At each absorbance level ±2%   1.96-2.04     Indicact Composition   0 %   1			0.					0.98-1.02	Complies / Does
dient Composition  2.0  dient Composition  0 % 10 % 50 % 90 % 100 %  Limit —			5.					1.47-1.53	not Complies
dient Composition         0 %         10 %         50 %         90 %         100 %         At each absorbance level ±2% of set concentration           Limit —— Limit —— Dration / External corration / External oration / External oration / External oration / External oroducibility & RSD of Area         25°C         40°C         60°C         ± 2.0°C of set value           ctor Precision or adability)         RSD of Area		2	0.					1.96-2.04	
Limit         → 0.00         9.8 to 10.2         49.0 to 51.0         88.2 to 91.8         98.0 to 102.0         of set concentration           umn Oven oration / External oration / External oration / External oroducibility & recision         £ 25°C         40°C         60°C         ± 2.0°C of set value           ctor Precision or oration / External or adability or adability)         RSD of RT NMT 0.5%         RSD of Area         RSD of Area	Gradient Composition	%0	10 %	% 05	% 06	100 %	40004	100   100	
Limit         →         0.00         9.8 to 10.2         49.0 to 51.0         88.2 to 91.8         98.0 to 102.0           mn Oven oration / External ctor Precision / External oroducibility & Resention Time atability)         A 40°C         40°C         60°C         ± 2.0°C of set value           ctor Precision atability         RSD of Area         RSD of Area         RSD of Area         RSD of Area	Accuracy						of set c	absorbance level ±270 oncentration	Complies / Does
tmn Oven oration / External oration / External ctor Precision atability)25°C 40°C External RSD of Retention Time atability)40°C ± 2.0°C of set value RSD of RT NMT 0.5% RSD of Areactor Precision atability ng 20 ppm Caffeine}RSD of AreaRSD of Area	Limit —	0.00	9.8 to 10.2	49.0 to 51.0	88.2 to 91.8	98.0 to 102.0			not Complies
ctor PrecisionRSD ofRSD of RT NMT 0.5%producibility & atability)RSD of AreaRSD of Areang 20 ppm Caffeine}RSD of AreaRSD of Area	Column Oven calibration / External	7	2°C	40°C		J <sub>0</sub> 09	# 2	.0°C of set value	Complies / Does
isionRSD ofRSD of RT NMT 0.5%lity &Retention TimeRSD of AreaCaffeine}RSD of AreaRSD of Area	Lab								
Caffeine RSD of Area	Injector Precision (Reproducibility &	RSD of Retenti	f on Time	·		,	R	SD of RT NMT 0.5%	Complies / Does
	repeatability) {Using 20 ppm Caffeine}	RSD of	f Area				R.	SD of area NMT 1.0 %	not Complies





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ANNEXURE - PIPL/ARD/001-A03

Injector Linearity	Correlation coefficient `r' =		NLT 0.999	Complies / Does not Complies
Injector volume Accuracy	15 injection of 10 μL		9.8-10.2	Complies/ Does not complies
Injector volume Accuracy	15 injection of 20 μL		19.6-20.4	Complies/ Does not complies
Injector volume Accuracy	15 injection of 50 μL		49.0-51.0	Complies/ Does not complies
Injector volume Accuracy	10 injection of 100 μL		98.0-102.0	Complies/ Does not complies
Vial Position	Refer to Detector Linearity Vials		All Vials to be picked/punctured	Complies/ Does not complies
Wavelength accuracy	Wavelength =(Using Caffeine 20 ppm)	- nm	272.0 nm ± 2.0 nm (Maxima)	Complies / Does not Complies
Wavelength accuracy	Wavelength =(Using Caffeine 20 ppm)	mu -	244.0 nm ± 2.0 nm (Minima)	Complies / Does not Complies
Wavelength accuracy	Wavelength =(Using Caffeine 20 ppm)	- nm	205.0 nm ± 2.0 nm (Maxima)	Complies / Does not Complies

# PRITAM INTERNATIONAL PVT. LIMITED

Format for HPLC Calibration Summary Sheet ANNEXURE - PIPL/ARD/001-A03

Linearity of Detector Response	Correlation coefficient `r' =	NLT 0.999	Complies / Does not Complies
Carryover Test	Carryover =%	Carryover of Caffeine NMT 0.05% of area of 20 μl injection of 250 ppm Standard Solution.	Complies / Does not Complies
Noise & Drift (VWD)		Noise: ±0.2 mAU Drift: ±2.0 mAU	Complies / Does not Complies

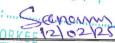
Remarks: All the parameters for the calibration as per predefined acceptance criteria are under limit hence the instrument is suitable for routine analysis

Checked By	Sign./Date
	•
Calibrated By:	Sign./Date:



# PRITAM INTERNATIONAL PVT. LIMITED te: .... Concern

ANNEXURE - PIPL/ARD/001-A04



### **Standard Preparation Sheet**

Mobile Phase: 70 Volume of ACN & 30 Volume	ne of water
Column Name: Inertsil ODS, C18, 250 mm x	4.6 mm, 5 μ Column ID No:
Injection Volume: As Per Required Parameters	
Use Std.: Caffeine	
<b>Detector:</b> As Per Required Parameters	
Flow Rate: 1.0 ml/min	
Name of Standard:	
Batch No.:	
Valid up to:	
Stock Solution:	
Weight aboutgm of Caffeine in phase,Sonicate 5 min for dissolve, make volume	100 ml Volumetric Flask add 50-75 ml of mobile up to mark with mobile phase.
5 ppm: Take 1 ml of this solution into 100 ml of	mobile phase.
10 ppm: Take 2 ml of this solution into 100 ml of	of mobile phase
20 ppm: Take 2 ml of this solution into 50 ml of	mobile phase
<b>50 ppm:</b> Take 5 ml of this solution into 50 ml of	mobile phase
<b>100 ppm:</b> Take 10 ml of this solution into 50 ml	of mobile phase
<b>250 ppm:</b> Take 10 ml of this solution into 20 ml	of mobile phase
Blank: Mobile phase	
Remarks: The Prepared Solution is OK/Not OK.	
Prepared By:	Checked By:
Date:	Date: