

OUALITY CONTROL DEPARTMENT

| STANDARD OPERATING PROCEDURE | | | |
|--|------------------------|--|--|
| Department: Quality Control | SOP No.: | | |
| Title: Operation, cleaning and calibration of FTIR | Effective Date: | | |
| Supersedes: Nil | Review Date: | | |
| Issue Date: | Page No.: | | |

1.0 OBJECTIVE:

To lay down procedure for operation, cleaning and calibration of FTIR.

2.0 SCOPE:

This SOP is applicable for operation, cleaning and calibration of FTIR (Make: Metrohms, Model: Nicolet 380).

- **3.0 RESPONSIBILITY** Execution- Executive QC Checking -Assistant Manager QC
- **4.0 ACCOUNTABILITY** Manager Quality Control
- **5.0 PROCEDURE:**
- 5.1 Switch "**ON**" the power supply for FTIR.
- 5.2 Turn "ON" the Computer & wait till the Instrument gets initialized.
- 5.3 Wait for 30 minutes for Instrument stabilization.
- 5.4 After stabilization double click the Omnic software icon.
- 5.5 Omnic software window will open and right sign ✓ will appear at the top right hand side of the window.
- 5.6 If the cross "X" appears check the interface cable between Computer and Instrument & restart the software.
- 5.7 From the tool bar click the collect background icon. Instrument will take the background & prompts whether to add the window or not. Click "Yes" to proceed further
- 5.8 Click the "COLLECT SAMPLE" after making the pallet using Hydraulic Press.
- 5.9 Enter the spectrum title when prompted; insert the pallet holder along with pallet to the sample compartment transmission accessory. Press "**OK**". Now instrument will collect the spectra. Click "**OK**" to add the spectrum in window. From file menu save the spectra after entering the file name.
- 5.10 Make the file by material / product name & store the data by giving date of analysis & B.No.
- 5.11 For analysis using ATR open the sample compartment, remove the direct transmission base plate by pulling it up wards. Now insert the ATR accessory from top and press it desired position. Once the accessory is positioned, SW will show the message for accessory and test the accessory. As these accessory are small it will be automatically recognized by the instrument and parameter will change accordingly.
- 5.12 Collect the background with bare crystal (Diamond / ZnSe) then put the sample on crystal & adjust the pressure using pressure device. For liquid sample you need not to put the pressure



OUALITY CONTROL DEPARTMENT

| STANDARD OPERATING PROCEDURE | | | |
|--|------------------------|--|--|
| Department: Quality Control SOP No.: | | | |
| Title: Operation, cleaning and calibration of FTIR | Effective Date: | | |
| Supersedes: Nil | Review Date: | | |
| Issue Date: | Page No.: | | |

device down. As the contact between crystal and liquid is always better.

5.13 For peak positioning after collection the sample click on find Peek tool from the tool bar and replace on that on the top right of the spectral window. All the peak positions will be numbered according to their wave numbers.

5.2 CALLIBRATION PROCEDURE:

Frequency: Once in a month.

- 5.2.1 Clean the instrument with dry and clean cotton cloth.
- 5.2.2 Switch 'ON' the computer.
- 5.2.3 Place sample holder in the chamber.
- 5.2.4 Choose spectra.
- 5.2.5 Click on 'measure'.
- 5.2.6 Message "Remove sample from holder" shall be appeared.
- 5.2.7 Click "OK".
- 5.2.8 "Background graph" shall be appeared.
- 5.2.9 Message "Set polystyrene film" shall be appeared.
- 5.2.10 Place poly styrene film in the holder.
- 5.2.11 Click 'OK'.
- 5.2.12 IR appears on the screen.
- 5.2.13 Results appear on the screen.
- 5.2.14 Check following parameters from the print out:
 - 1. Resolution
 - 2. Wave number accuracy
 - 3. Repeatability of wave number
 - 4. Repeatability of Transmittance
- 5.2.15 Enter the results of the above parameters as per Annexure -I.
- 5.2.16 If the instrument is not giving satisfactory results, recalibrate the instrument.

5.3 CLEANING PROCEDURE:



OUALITY CONTROL DEPARTMENT

| STANDARD OPERATING PROCEDURE | | | |
|--|-----------------|--|--|
| Department: Quality Control SOP No.: | | | |
| Title: Operation, cleaning and calibration of FTIR | Effective Date: | | |
| Supersedes: Nil | Review Date: | | |
| Issue Date: | Page No.: | | |

Frequency: Daily or after each use.

- 5.3.1 Open the front part of the instrument.
- 5.3.2 Wipe out any material in the sample holder assembly by means of tissue paper.
- 5.3.3 Clean all the sampling accessories with tissue paper after analysis and keep them in proper place.
- 5.3.4 Clean the outer surface of the instrument with dry cotton cloth.
- 5.3.5 Place the 'self indicating' coarse silica gel bag in the sample holding assembly after analysis.
- 5.3.6 If the colour changes of the silica bag shown by the instrument (changes from blue to pink), regenerate the silica by keeping it in oven at 70° C for some time till it gets blue colour again.
- 5.3.7 Record the details of cleaning in log card.

6.0 SAFETY & PRECAUTIONS:

- 6.1 Vibrations can affect the performance of FTIR. Minimize the noise & vibration wherever possible.
- 6.2 Intense magnetic field, such as procedure by the super conducting magnets, can affect spectrometer performance. The FTIR should away from such fields.
- 6.3 FTIR parts are hygroscopic & become irreversible opaque when exposed to moisture. Thus excess humidity & rapid changes in room temperature that may cause condensation must be avoided.

7.0 REVISION HISTORY:

| Revision No. | Reason for Revision | Superseded from & date |
|-----------------|---------------------|------------------------|
| | | |

8.0 DISTRIBUTION:

| Сору | Issuance Record | | | | Withdrawal Record | | Destruction Record | |
|------|-----------------|-----------------|---------------------------------|-------------------------------|----------------------|---------------|-----------------------|---------------|
| No. | Date | Dept. issued | Name / Signature of receiver | Issued By Name / Signature | Ву | Sign/ Date | Ву | Sign/ Date |
| | | | | | | | | |



QUALITY CONTROL DEPARTMENT

| STANDARD OPERATING PROCEDURE | | | |
|--|------------------------|--|--|
| Department: Quality Control SOP No.: | | | |
| Title: Operation, cleaning and calibration of FTIR | Effective Date: | | |
| Supersedes: Nil | Review Date: | | |
| Issue Date: | Page No.: | | |

9.0 REFERENCES:

Not Applicable

10.0 ABBREVIATIONS & ANNEXURES:

SOP : Standard Operating Procedure

No. : Number

QC : Quality Control

° C : Degree Celsius

I.R : Infra red

FTIR : Fourier Transform Infra red

ATR : Attenuated transmitted reflectance

Annexure I: Calibration Record of FTIR



Remarks: Satisfactory / Not Satisfactory

PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

| | STANDARD OPERATING I | PROCEDURE |
|---|------------------------------------|----------------------------------|
| artment: Quality Control | | SOP No.: |
| e: Operation, cleaning and calibration of FTIR ersedes: Nil | | Effective Date: |
| | | Review Date: |
| Date: | | Page No.: |
| | ANNEXURE-I CALIBRATION RECORD O | F FTIR |
| | REF. No. ISSUE DATE: ISSUED BY : | Page 1 of 3 |
| PERFORMANCE DATE | DATE OF LAST PERFORMAN DONE | NCE NEXT DUE FOR PERFORMANCE |
| INSTRUMENT NAME | INSTRUMENT DETAIL INSTRUMENT MAKE | S INSTRUMENT IDENTIFICATION NO. |
| | OBSERVATION | |
| 1.Resolution: | | |
| Wave number | Measured (T%) | Standard peak depth (%T) |
| 2870, 2851 | | Not less than 18 |
| 1589.0 , 1588.3 1583.0 , 1582.5 Not le | | Not less than 10 |
| Remarks : Satisfactory/Not Satisfactory | y | |
| | REF. No. ISSUE DATE: ISSUED BY: | Page 2 of 3 |
| | | |
| 2.Wave number accuracy: | , | |
| 2.Wave number accuracy: Wave number | Measured wave number | Error Tolerance |
| | | Error Tolerance ±0.5 |
| Wave number | | |
| Wave number 3082.18 | | ±0.5 |
| Wave number 3082.18 3060.23 | | ±0.5 ±0.5 |



QUALITY CONTROL DEPARTMENT

| | S | TANDARD OPERA | TING PRO | CEDURE | |
|--|----------------------------|---|---------------------|--------------------------|--|
| Department: Qua | lity Control | | | SOP No.: | |
| Fitle: Operation, cleaning and calibration of FTIR | | | Effective Date: | | |
| Supersedes: Nil | | | | Review Date: | |
| Issue Date: | | | | Page No.: | |
| . Repeatability of wave n | umber : | | | | |
| Wave number | Measured- I wave number | Measured -II wave number | Error | Tolerance | |
| 2850.7 | | | | ± 0.5 | |
| 1601.4 | | | | ±0.5 | |
| 1181.4 | | | | ± 0.5 | |
| Remarks : Satisfactory/No | - | | | | |
| Wave number | Observation -I %T | Observation-II %T | Error | Tolerance | |
| 2850.7 | | | | ± 0.5 | |
| 1601.4 | | | | ± 0.5 | |
| 1181.4 | | | | ± 0.5 | |
| Remarks : Sati | sfactory/Not Satisfactory | | | | |
| Calibration done by : | | Date: | | | |
| Checked by : | | Date : | | | |
| | | REF. No ISSUE DATE: _ ISSUED BY : | | Page 3 of 3 | |
| | | CONCLU | CLON | • | |
| | | CONCLU | SIUN | | |
| INSTRUMI | ENT WORKING | SATISFACTORY | INSTRUM SATISFAC | ENT NOT WORKING CTORY | |



QUALITY CONTROL DEPARTMENT

| STANDARD OPERATING PROCEDURE | | |
|--|------------------------|--|
| Department: Quality Control | SOP No.: | |
| Title: Operation, cleaning and calibration of FTIR | Effective Date: | |
| Supersedes: Nil | Review Date: | |
| Issue Date: | Page No.: | |

| PERFORMED BY | | | CHECKED BY | Y | |
|--------------|------|------|------------|------|------|
| Name | Sign | Date | Name | Sign | Date |
| | | | | | |