STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Procedure for Operation and Calibration of FTIR	**Effective Date:**
Supersedes: Nil	**Review Date:**
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1.0 OBJECTIVE:

To lay down a procedure for operation and calibration of Fourier transform Infra-Red Spectrophotometer (FTIR).

2.0 SCOPE:

This procedure is applicable for operation and calibration of FTIR in quality control department.

3.0 RESPONSIBILITY:

Officer, Executive - Quality Control

Head – Quality Control

4.0 PROCEDURE:

4.1 Preliminary Check:

4.1.1 Check that the equipment and surrounding are clean, if not clean then clean with a soft cloth duster.

4.1.2 Ensure that equipment is connected to proper electric supply.

4.1.3 Ensure the calibration status of the equipment.

4.1.4 Ensure that the temperature in the area is not more than 25°C and RH is not more than 60 %.

4.2 Operation:

4.2.1 Switch ON the power by pressing the power button.

4.2.2 Switch ‘ON’ the personal computer attached to FTIR. The system is set up windows automatically.

4.2.3 Double click the Lab Solution IR on the desktop. Enter user ID and password into the login window and click ok.

4.2.4 The Lab Solution window is displayed. Select project and double click PC17 Instrument.

4.2.5 Click the desired program on the (shortcut) tab to start it.

4.2.6 Click the Spectrum than open the lab solution IR

4.2.7 Click the Instrument and Select the Initialization. A green rectangle is displayed in the status

monitor. Initialized is succeeded is displayed in the log

4.2.8 Click on main window of the menu and set parameters to select the **Data, Instrument, Advanced, More.**

4.2.9 **Select the data Tab and feed the following parameter:**

- Measurement Mode - % Transmittance
- Apodization - Happ-Genzel
- No of Scans - As required (1 to 4000)
- Resolution - 4.0 or as required offered the instrument
- Range (cm^{-1}) - Min.340 Max.4000

4.2.10 **Select the Instrument Tab choose the following parameter:**

- Beam - Internal
- Detector - Standard
- Mirror Speed - 2.8

4.2.11 **Select the More Tab and ensure the following parameter:**

- Gain - auto
- Aperture - auto
- Gain - "1"
- Mode - % Transmittance

4.2.11.1 **Select the Advanced Tab and ensure the following parameter:**

- IFG Noise - 1
- IFG Similarity - 1
- Aperture - 0
- IR Range - MID
- Light Source - Infrared

4.2.11.2 Select to the File Tab to save parameter set on the scan parameter tabs. To save the parameter. Write the file name as required to save parameter. To Load the save parameters click the button at the Left end .

4.2.11.3 To carry out the Scanning ,ensure that the inside of instrument .Sample compartment is accommodate as required for sample (Powder, liquid or Poly Film)

4.2.12 For fixation of HATR, Remove any sample holder present in the sample compartment. Place flat plate crystal through plate crystal on the accessory as per requirement. Fix down the accessory using fixing screw use flat plate crystal for spectra generation of LDPE, HDPE and blister foil. Use through plate crystal for spectra generation of powder material and liquids. Place the sample, close the door of the sample compartment and run the spectra. Spectrum data file as saved in specified file and give the extension of the name is" *. smf and comment on measure mode.

4.2.13 For the BKG put the previously dried KBr in to the sample cup and click to BKG button. Scanning will be start automatically and will show Spectrum is displayed.

4.2.14 Enter a file name and enter the sample information and click the mesurement.Put the sample into the sample cup (sample to be prepared as specified in the monograph) and click to sample scan.Scanning starts automatically and spectrum result appear in ready mode. Follow the same procedure for other sample.

4.2.15 The saved spectrum processing to select the manipulation window as required smoothing, Peak Pick & Data Calculation or given in the menu.

4.2.16 For the report print open spectrum file click on file select to print preview display will show generated report formats, select as required and click ok, Select the printer and print the reports.

4.2.17 Record the operation in instrument log book of FTIR.

4.3 Calibration:

4.3.1 Operate the instrument as per the operation procedure shown above procedure. And click Macro and select EP Validation than click to be open to be displayed

001 Program Stand. CLOSE, STOP, RUN PAUSE

002: Load Basic File.

003: Program End.

Click the Run Enter user ID and password into the login window and click ok

Give the massage and then ok

4.3.2 Replace the sample compartment and fix the polystyrene film holder in the sample compartment

4.3.3 EP Validation than window will be displayed

Setting, Load , Measurement & Cancel

4.3.4 Click on Setting mode and set following parameter.

4.3.5
- Instrument - IR Affinity-1
- User ID - A21965200901
- Temperature - 25°C
- Sample Name - POLYSTYRENE FILM
- Relative Humidity - 50 %
- Inspected By - Name of Analyst.

4.3.6 Ensure that above parameters are correct than click ok. Then click ok measurement and instrument Initialized is succeeded. Power spectrum (Base line Correction) will be start as per setup parameter.

4.3.7 Display will ask, insert the polystyrene film in the sample chamber.

4.3.8 Control of Resolution performance:

4.3.8.1 Resolution performance of apparatus should be carried out by spectrum obtained from polystyrene film.

4.3.8.2 The difference between the absorbances at the absorption minimum at 2870 cm^{-1} and the absorption maximum at 2849.5 cm^{-1} is greater than 0.33.

4.3.8.3 The difference between the absorbances at the absorption minimum at 1589 cm^{-1} and the absorption maximum at 1583 cm^{-1} is greater than 0.08. Record in Annexure-I .

4.3.8.4 In case of any major drift is observed, inform Head-QC for further action.

4.3.8.5 **Verification of the wave-number scale:**

3060.0 (± 1.0)

2849.5 (± 1.0)

1942.9 (± 1.0)

1601.2 (± 1.0)

1583.0 (± 1.0)

1154.5 (± 1.0)

1028.3 (± 1.0)

4.3.8.6 Take the print of polystyrene film spectrum note down in annexure-I.

4.3.9 HATR calibration:

4.3.9.1 Remove any sample holder present in the sample compartment. Scan back ground and place a flat plate on the accessory. Place the accesorry into the sample compartment. Fix down the accesory using the fixing screw on the baseplate.

4.3.9.2 Set the scan parameter as follows :

Mode : Transmittance

No of scan : 40

Resolution : 4 cm^{-1}

Apodization : Happ-genzel

Scan range : 4000 to 400

Detector : Standard

Mirror speed : 2.8 mm/sec

Aperture : Auto

4.3.9.3 With the accessory removed from the sample compartment, collect a background spectrum.place the accesorry in the sample compartment.collect a transmission spectrum using the same collection parameter as used to collect the back ground spectrum.

4.3.9.4 The tranmission value of this accessory should be at least 5 % at 1000 cm^{-1} for ZnSe prism plates.

4.3.9.5 Record the observations in the format as per Annexure - I.

4.3.10 If instrument is out of calibration, affix “OUT OF CALIBRATION” or “Do not use” label on the instrument and follow the Out of calibration SOP QAD/055.

4.3.11 Head-QC shall review the result and shall call the service personnel for rectification (If required).

4.3.12 **Frequency of calibration:**

Control of Resolution performance & Verification of the wave-number scale : Quarterly

HATR calibration: Once in Six months

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5.0 ANNEXURE (S) :

Annexure – I : Calibration Record of FTIR

6.0 REFERENCE (S):

SOP: Handling of Out of Calibration

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

7.0 ABBREVIATION (S)/DEFINITION (S):

FTIR : Fourier transform Infra-red

HATR : Horizontal Attenuated Total Reflectance

RH : Relative Humidity

LDPE : Low density polyethylene

HDPE : High density polyethylene

REVISION CARD

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

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ANNEXURE – I
CALIBRATION RECORD OF FTIR

Location		**Page No.**	7 of 2
Manufactured By		**Model No.**	
Date of Calibration		**Identification No.**	
Next Calibration Due On			

Calibration Frequency: Quarterly

1. Control of Resolution Performance:

The Absorbance at about 2849.5 cm^{-1} :_____

The Absorbance at about 2870 cm^{-1} :_____

Difference in absorbances :_____ (Limit : NLT 0.33)

The Absorbance at about 1583 cm^{-1} :_____

The Absorbance at about 1589 cm^{-1} :_____

Difference in absorbances :_____ (Limit : NLT 0.08)

2. Verification of the wave-number scale:

Wave Numbers (cm^{1})	Limits (cm^{-1})	Observed value (cm^{1})	Remark
3060.0	3059.0 to 3061.0		OK/Not OK
2849.5	2848.5 to 2850.5		OK/Not OK
1942.9	1941.9 to 1943.9		OK/Not OK
1601.2	1600.2 to 1602.2		OK/Not OK
1583.0	1582.0 to 1584.0		OK/Not OK
1154.5	1153.5 to 1155.5		OK/Not OK
1028.3	1027.3 to 1029.3		OK/Not OK

Opinion: The Instrument Calibration is OK / Not OK.

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Calibration Frequency: Half yearly

3. HATR Calibration:

Observation: The transmission value of HATR is _____ % at 1000 cm^{-1} for ZnSe prism plates.

Limits : The transmission value of HATR should be at least 5 % at 1000 cm^{-1} for ZnSe prism plates.

Opinion: The Instrument Calibration is OK / Not OK.