



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

1.0 OBJECTIVE:

To lay down a procedure for operation and calibration of FTIR Spectrophotometer.

2.0 SCOPE:

This procedure is applicable for operation and calibration of FTIR Apparatus in Quality Control Department.

3.0 RESPONSIBILITY:

Officer, Executive - Quality Control Department
Head - Quality Control Department

4.0 DEFINITION(S):

NA

5.0 PROCEDURE:

Make: Shimadzu, Model: 8400S

5.1 Operation:

- 5.1.1 Ensure that the instrument is clean & free from dust.
- 5.1.2 Ensure that the temperature in the area is not more than 25°C and RH is not more than 60 %.
- 5.1.3 Switch 'ON' the power of the FTIR.
- 5.1.4 Switch 'ON' the personal computer attached to FTIR. The system is set up windows automatically. The windows Program Manager will appear on the screen.
- 5.1.5 Click the (IR Solution) icon. After initialization, the IR Solution Window will appear.
- 5.1.6 Click on Environment and go to Instrument preference, Click Instrument and select FTIR 8000 series.
- 5.1.7 Ensure that displayed window is on measure mode.



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

5.1.8 Click on measurement, select initialize and wait for Instrument Initialization.

5.1.9 See the status of instrument for ready with green signal in the right side of the appeared window.

- **Interface**

- **Mirror**

5.1.10 Four Tabs appear on the right side of window.

Data, Instrument, More, File

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.400 Max. 4000

Select the Instrument Tab choose the following parameter:

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

Select the More Tab and ensure the following parameter:

- Gain - auto
- Aperture - auto
- Gain - "1"
- Mode - Power



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

Select the Files Tab and click

Save As

to save the Parameters.

Write the file name as required to save the parameter. To Lock the saved parameter put the check mark to [Locked] so that the setting and changes of all parameter are prohibited.

- 5.1.11 To carry out the Scanning, ensure that the inside of instrument. Sample compartment is accommodated as required for sample (Powder, Liquid or Poly Film)
- 5.1.12 Open the data file as saved in specified file and give the extension of the name is” * . smf and comment on measure mode.
- 5.1.13 For the BKG put the previously dried KBr in to the sample cup and click to BKG button. Scanning will be start automatically and display will show ready.
- 5.1.14 For the sample scanning give the comment and extension name .Put the sample into the sample cup (sample to be prepared as specified in the monograph) and click to “SAMPLE” mode. Scanning starts automatically and spectrum results appear in ready mode. Follow the same procedure for other sample.
- 5.1.15 To Process saved spectrum select the manipulation1 as required smoothing peak tables or given in the menu.
- 5.1.16 For the report print open spectrum file, click on file and select to print preview display will show specified generated reports formats , select as required and click OK, Select the printer and print the reports.
- 5.1.17 Record the operation in instrument log book of FTIR.

5.2 Calibration :

- 5.2.1 Operate the instrument as per the operating procedure shown above procedure.
- 5.2.2 Replace the sample compartment and fix the polystyrene film holder in the sample compartment.



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

5.2.3 Click on measurement menu and select diagnostic mode (Appeared FTIR base diagnostic window, click on “Done” to ensure the parameter as per requirement of instrument.

5.2.4 Again click on the measurement menu and ensure nothing is inside of the sample compartment and select to “EP 5.0 Validation” mode then window will display.” Select the Method”

5.2.5 Click on mode and set following parameter.

- Instrument - FTIR8400S
- User ID - A21014000313
- Temperature - 25°C
- Sample Name - Polystyrene film
- Relative Humidity - 50 %
- Inspected By - Name of Analyser

Ensure that above parameter are correct then click ok.

Again click on measure mode and click to OK. Power spectrum (Base line correction) will start as per setup parameter.

5.2.6 Display will ask insert the polystyrene film in the sample chamber.

5.2.7 Click on view mode then go for manipulation 1 for Spectrum processing as required.

5.2.8 Verification of the followig wave numebr scale (cm⁻¹)

- 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)



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

5.2.9 Take the print of polystyrene film spectrum and note down in Annexure I.

5.2.10 Resolution performance :

Resolution performance of apparatus should be carried out by spectrum obtained from polystyrene film. The difference between the maximum absorption at 2849.5cm^{-1} to the minimum at 2870cm^{-1} should be greater than 0.33 absorbance and that from the maximum at 1583cm^{-1} to the minimum at 1589cm^{-1} should be greater than 0.08 absorbance.

5.2.11 Frequency - Quarterly

5.2.12 Fill the calibration status on metallic calibration label of the instrument, record the calibration results in annexure-I

5.2.13 If instrument is out of calibration, affix "UNDER MAINTENANCE" and call for service engineer.

5.2.14 Note down the Calibration activity in Instrument logbook.

5.3 Cleaning :

5.3.1 The outside body of the FTIR should be wiped with a clean dry lint free cloth and the inner sample compartment should be cleaned using non shredding brush ensuring that the optics are not disturbed.

6.0 ABBREVIATION(S) :

FTIR – Fourier transform Infra red

QCD – Quality Control Department

SOP – Standard Operating Procedure

NA – Not Applicable

IP- Indian Pharmacopoeia.

7.0 REFERENCE(S):

IP-1996



STANDARD OPERATING PROCEDURE

Department: Quality Control

SOP No.:

Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)

Effective Date:

Supersedes: Nil

Review Date:

Issue Date:

Page No.:

ANNEXURE I

Calibration Record: Fourier Transform Infra Red Spectrophotometer (FTIR)

PARAMETER:

(1) WAVE NUMBERS ACCURACY:

Wave Numbers (cm ⁻¹)	Limits (cm ⁻¹)	Observation (cm ⁻¹)	Remark
3060.0	3059.0 to 3061.0		
2849.5	2848.5 to 2850.5		
1942.9	1941.9 to 1943.9		
1601.2	1600.2 to 1602.2		
1583.0	1582.0 to 1584.0		
1154.5	1153.5 to 1155.5		
1028.3	1027.3 to 1029.3		



STANDARD OPERATING PROCEDURE

Department: Quality Control	SOP No.:
Title: Operation and calibration of Fourier Transform Infra Red Spectrophotometer (FTIR)	Effective Date:
Supersedes: Nil	Review Date:
Issue Date:	Page No.:

2. RESOLUTION PERFORMANCE:

The absorbance at the 2849.5 cm^{-1} maximum and that at 2870.0 cm^{-1} minimum should be greater than 0.33.

The absorbance at the 1583.0 cm^{-1} maximum and that at 1589.0 cm^{-1} minimum. Should be greater than 0.08.

Absorbance at 2849.5 cm^{-1} = _____ (A)

Absorbance at 2870.0 cm^{-1} = _____ (B)

Resolution = _____ =-----

Absorbance at 1583.0 cm^{-1} = _____ (C)

Absorbance at 1589.0 cm^{-1} = _____ (D)

Resolution = _____ =-----

Remarks: The Instrument Calibration **Complies / Does Not Comply.**

Calibrated By :

Date

Checked By :

Date