

ABBOTT LABORATORIES

CORPORATE METROLOGY

INSTRUMENT
CALIBRATION PROCEDURE
Infrared Spectrophotometer
(General)

Abbott Laboratories Corporate Metrology

PROCEDURE: Infrared Spectrophotometer (General)

SECTION 1

INTRODUCTION AND DESCRIPTION

1.1 This procedure describes the calibration procedure of a infrared spectrophotometer used for laboratory purposes. The instrument being calibrated will be referred to herein as the Test Instrument (TI).

1.2 The instructions contained in this procedure are general instructions describing valid techniques to be employed in performing calibrations on infrared spectrophotometers. This procedure should be employed as a general guide that will vary with the model and manufacturer of the TI.

Table 1. Calibration Description

Test Instrument Characteristics	Performance Specifications	Test Method
Infrared Spectrophotometer	Range: (4000 to 200) cm^{-1} Resolution: $\leq 1 \text{ cm}^{-1}$	An Infrared Transmission Wavelength Standard (ITWS) will be placed in the sample compartment. A scan will be run covering all useful peaks of the calibration standard. Comparison against standard calibration certificate will determine the TI's accuracy.

NOTE

All measurement data shall be recorded on a Calibration/Test Record (C/TR). Tests performed to verify Test Instrument operating characteristics shall be noted on the data record by placing checks in the spaces provided.

SECTION 2

EQUIPMENT REQUIREMENTS

NOTE

Minimum use specifications are the principal parameters required for performance of the calibration, and are included to assist in the selection of alternate equipment. Satisfactory performance of alternate items shall be verified prior to use. All applicable equipment must bear evidence of current calibration.

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NOTE

The instructions in this procedure relate specifically to the calibration equipment listed below. If other equipment is substituted, the instructions must be interpreted accordingly. The Infrared Transmission Wavelength Standard is a NIST Standard Reference Material (SRM) 1921, and will be referred herein as the ITWS.

Table 2. Equipment Description

Item	Minimum Use Specifications	Calibration Equipment
2.1 ITWS	Range: (3 to 18) μm (540 to 3200) cm^{-1} Uncertainty: See Table 3	NIST SRM 1921

Table 3. Certified Peak Wavenumber (in Vacuum)

Peak Wavelength (cm^{-1})	Expanded Uncertainty, U^2
545.48	12.29
842.08	0.49
906.82	0.66
1028.34	0.27
1069.19	0.46
1154.63	0.54
1583.12	0.06
1601.34	0.07
2850.12	1.84
3001.39	0.12
3026.41	0.61
3060.02	0.14
3082.18	0.12

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NOTE

To ensure that a valid calibration is being performed, the accuracy tolerance ratio between the standard employed and the TI must be at least 4 to 1. If the ratio is less than 4 to 1, then this fact should be noted on the C/TR and the tolerance of the standard accounted for during the measurement process.

To determine the accuracy ratio, compute the tolerance value of the standard and the TI. Divide the standard tolerance value by the TI accuracy and verify that the result is 4.0 or greater. If the result is less than 4.0, either a different, more accurate, standard should be used, or the reduced accuracy ratio compensated for during the measurement process. Compensation can be accomplished by subtracting the standard tolerance value from the TI accuracy value and ensuring that all measured values are within resultant tolerance value.

SECTION 3

PRELIMINARY OPERATIONS

- 3.1 Review the entire procedure before starting the calibration process.
- 3.2 Inspect the TI for obvious defects or signs of damage that could impair its operation.
- 3.3 Turn power on TI. Allow sufficient time for sample chamber temperature to stabilize.
- 3.4 Setup TI according to the following table:

Table 4. Initial Setup.

SETUP	DESCRIPTION
Resolution	Set at 0.5 cm^{-1} or best resolution value achievable by the TI.
Scan Speed	Medium
Scan Range	$(4000 \text{ to } 600) \text{ cm}^{-1}$
Peak Threshold	Minimum value of full scale. Adjust accordingly as to obtain all peak wavenumbers of interest.

NOTE

Increasing TI resolution will decrease the scan speed.

- 3.5 Set up printer or plotter to follow the scan, and to print axes (Transmittance vs Wavelength), ordinate scale, and peak table.

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SECTION 4

CALIBRATION PROCESS

NOTE

Unless otherwise specified, verify the results of each test and take corrective action after all tests are completed. All "As Found Readings" must be recorded prior to any adjustment and/or repair.

NOTE

For TI's currently on the Calibration Program the calibration points should already be established on a pre-printed C/TR; however, if a C/TR is not available, as would be the case for an initial calibration, calibration points should be selected as per instructions on the Calibration Request Form.

4.1 Ensure that the TI reads 100% T (Transmittance) with no sample inside sample chamber. Otherwise, adjust TI to 100% T.

4.2 TI EQUIPPED WITH PEAK FUNCTION

NOTE

Instruments that do not provide a peak table will have to be calibrated manually, one peak at a time. Follow steps 4.3 of this procedure.

4.2.1 Make a reference scan with no sample inside the sample chamber. This will be considered as a baseline.

NOTE

Some TI's do not require a baseline or reference scan for accurate measurements.

4.2.2 Ensure ITWS is clean and lint free. Place ITWS on sample side of chamber.

4.2.3 Start the scan.

4.2.4 Use the TI's peak function to get a list of peak wavenumbers.

NOTE

If TI prints a peak table automatically, use it instead.

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4.2.5 Locate and compare each peak wavenumber listed on the C/TR parameters with the ones printed on the peak table.

NOTE

Due to the fact that the ITWS was certified at NIST under a vacuum environment, which has an index of refraction of 1.00000, the TI peak wavenumbers will be divided by 1.00026 to compensate for the air in the sample chamber and get the true readings.

4.3 TI NOT EQUIPPED WITH PEAK FUNCTION

4.3.1 Manually, use TI controls to slew the wavenumber towards the wavelength of interest. As you approach the first peak, slow down and observe the transmittance reading.

4.3.2 Continue slowly until the transmittance value is at its minimum. This will be your peak wavenumber. Verify the reading by slewing one step at a time, back and forth, around the peak wavenumber.

4.3.3 Repeat steps 4.3.1 and 4.3.2 for all peaks listed on the C/TR.

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