

Method: CAL-1 Revision: 1 Final Revision Date: 06/02/03	Laboratory Instrument Calibration Method	INEOS Nitriles
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METHOD SUMMARY

This is a calibration procedure for the QC UV equipment for acrolein, aldehydes, peroxides, MEHQ and acetonitrile. It covers specific procedures for the Hitachi U-2000, Hitachi U-2001, Perkin Elmer Lambda EZ201, and Perkin Elmer Lambda 18 spectrophotometers. For other spectrophotometers, consult the instrument manual and adjust these procedures appropriately.

SAFETY

Acrylonitrile and acetonitrile are hazardous to the health and dangerous to handle. Use acrylonitrile and acetonitrile in a well ventilated hood. Review the MSDS for detailed information concerning toxicity, first aid procedures and safety precautions.

Refer to the appropriate safety section or site manual for the necessary protective equipment to use when handling any reagents or samples.

REFERENCE

CAL-070, "Calibration for UV Spectrophotometer," BP Green Lake Laboratory Procedure, Revision 7, 11/20/01.

APPARATUS AND REAGENTS

Spectrophotometers: Hitachi U-2000, Hitachi U-2001, Perkin Elmer Lambda EZ201, and/or Perkin Elmer Lambda 18.

Transmittance Standards, traceable to NIST.

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PROCEDURE

The Hitachi U-2000, Hitachi U-2001, and Perkin Elmer Lambda EZ201 will be calibrated externally by Hitachi. The Perkin Elmer Lambda 18 will be calibrated externally by Perkin Elmer. They will provide a certificate of calibration that shows traceability to a recognized standard. The Team Leader, or his representative, will keep this document in a calibration book. Checks on the instrument performance will be conducted by the Team Leader or his representative as described below.

For Hitachi U-2000, U-2001, and Perkin Elmer Lambda EZ201:

1. The calibration of wavelengths and transmittance/absorbance will be performed by the manufacturer's representative using NIST traceable standards. A tag will be placed on the instrument showing the date of calibration.
2. Either the Hitachi representative will check the transmittance standards while performing the instrument calibration or within 24 hours of the manufacturer's calibration, the Team Leader or his representative will check the calibration of a set of transmittance standards which the lab will maintain. The standards will measure transmittance between 8 and 11% and between 45 and 55% for specific wavelengths. The transmittance values of these standards will be recorded to the nearest 0.1 transmittance unit. They will be used as secondary standards to perform transmittance checks on the instrument as described below. New secondary standards may be purchased with an NIST traceable certificate. These new standards may be used for 1 year and then be recertified as described above.
3. The Team Leader or his representative will conduct the following performance check on the instrument:

Remove all cells from the spectrometer and auto-zero the instrument. Using the operator's manual and the parameters listed below, perform the indicated scan between 660 and 650 nm. A peak from the emission spectrum of the deuterium lamp should be detected between 655.7 and 656.nm.

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Data Mode	Single Beam
Start WL (nm)	660.0
Stop WL (nm)	650.0
Up Scale	10.000
Lo Scale	0.000
Scan Speed (nm/min)	100
Init Delay (sec)	0
Num Cycles	1
Cycle Time (sec)	0
Display Format	Sequential
Baseline	System
Response	Fast
Lamp Change WL (nm)	340.0
Vis Lamp	ON
UV Lamp	ON
Graph Print	OFF
Text Print	OFF
List Interval (nm)	10.0

- 4, Check the transmittance standards. They should be within 0.5 transmission unit of their know value. If either check is not within specifications, the Team Leader will complete a "Quality Incident Investigation" and the instrument will be recalibrated by Hitachi.

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For Perkin Elmer Lambda 18:

1. The calibration of wavelengths and transmittance/absorbance will be performed by the manufacturer's representative using NIST traceable standards. A tag will be placed on the instrument showing the date of calibration.
2. Either the Perkin Elmer representative will check the transmittance/absorbance standards and Abscissa standards while performing the instrument calibration or within 24 hours of the manufacturer's calibration, the Team Leader or his representative will check the calibration of a set of transmittance standards which the lab will maintain. The standards will measure absorbances between 9 and 50% for a specific wavelength. The absorbance values of these standards will be recorded to the nearest 0.1 Au unit. They will be used as secondary standards to perform transmittance/absorbance checks on the instrument as described below. New secondary standards may be purchased with an NIST traceable certificate. These new standards may be used for 1 year and then be recertified as described above.
3. The Team Leader or his representative will conduct the following performance check on the instrument:
Validation:
 1. Make sure that the Lambda 18 is on and there are no cuvettes in the cuvette holders.
 2. Select "Application" menu on the "UV Winlab" menu.
 3. Select "Validation" shown in the submenu.
 4. When the "Instrument Validation" screen pops up, select the "Perform Validation" button.
 5. The. Program will ask to insert a "Blank". Select "OK" to by-pass this step.
 6. After the Lambda runs a background check, a "Validation" submenu will display, requesting the insertion of the first of 4 standards (G1, G2, G3 and H) into the front cuvette holder. After placing the standard in the holder, press the "OK" button. After the validation of the first standard, repeat the validation for the remaining three standards.

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7. When all four standards have been run, the bottom left of the Validation screen will show "All tests passed; instrument is validated". The data will be automatically saved into a TXT file.
8. Print out a copy of the validation and put it into the ISO Calibration book. Exit the program.
9. Remove the last standard from the cuvette holder and store the standards properly.

If either check is not within specifications, the Team Leader will complete a "Quality Incident Investigation" and the instrument will be recalibrated by Perkin Elmer.