Raman Spectrometer Performance Monitoring and Maintenance

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1 INTRODUCTION

This document addresses the performance monitoring and maintenance of Raman Spectrometers (sample compartment and/or microscope). A Raman spectrometer can be used to analyze samples in larger quantities in the sample compartment, if available, or in smaller quantities on the microscope, utilizing one or more objectives (e.g., 10X, 50X, 100X). In general, the signal for opaque samples can be maximized with a high numerical objective utilizing a microscope, while the signal for transparent samples can be maximized using a macro lens (i.e., sample compartment) or small magnification objective. The Raman spectrometer may utilize one or more excitation lasers (e.g., 785 nm, 780 nm, 532 nm). The signal will be more intense with a shorter wavelength excitation source; however, there is a trade off as samples may fluoresce and/or overheat with higher energy. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/subdisciplines: Explosives Chemistry, General Chemistry, Seized Drugs, and Paints and Polymers.

3 EQUIPMENT

- Instrumentation
    - Thermo Almega XR Dispersive Raman Spectrometer System with sample compartment and microscope, Omnic Software (or equivalent)
    - Thermo DXR3 Dispersive Raman Spectrometer System with Omnic Software (or equivalent)
    - Thermo DXR3 Dispersive Raman Spectrometer System with microscope accessory with Omnic Software (or equivalent)
- Materials
    - Polystyrene slide, rod or film (Thermo or equivalent)
    - Alignment tool containing calibration slide with pinhole and white light (Thermo or equivalent)
    - General laboratory supplies

4 STANDARDS AND CONTROLS

- Performance Verification Standard

  Polystyrene is used to assess daily operating performance and continued integrity of the Thermo Raman systems. Polystyrene requires no preparation and does not expire.

- Alignment Tool

  The alignment tool is used as needed to verify that the sample compartment and microscope are aligned and functioning properly. There is no sample preparation involved. The tool does not expire.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter the information in the appropriate log to indicate completion.

5.1.1 Microscope

A. Start Omnic and turn on the desired laser (if applicable) under 'Experiment Setup.' Allow time for the laser(s) to warm up and for the detector/CCD to cool. If the detector/CCD is not cool, a message will be displayed when data collection is attempted. The current temperature can also be viewed under the Advanced Tab in Experiment Setup. When the detector/CCD temperature setpoint has been reached, data collection can begin.
B. Turn the microscope illuminator on.
C. Set the operating parameters as listed in the 'Instrumental Conditions' section.
D. Place the polystyrene slide on the stage and focus. Ensure that the illuminator is off, then collect the sample spectrum. Perform a peak analysis by using the 'Find Peaks' option under the 'Analyze' menu. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the labeled spectrum.
E. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.1.2 Sample Compartment

A. Start Omnic and turn on the desired laser (if applicable) under 'Experiment Setup'. Allow time for the laser(s) to warm up and for the detector/CCD to cool. If the detector/CCD is not cool, a message will be displayed when data collection is attempted. The current temperature can also be viewed under the Advanced Tab in Experiment Setup. When the detector/CCD temperature setpoint has been reached, data collection can begin.
B. Set Operating Parameters as listed in the 'Instrumental Conditions' section.
C. Place the polystyrene rod or film in the sample compartment. Collect the sample spectrum. Perform a peak analysis by using the 'Find Peaks' option under the 'Analyze' menu. Evaluate the results using the ‘Acceptance Criteria' section. If the results are acceptable, print the labeled spectrum.

D. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 As Needed Checks

The following procedure will be performed as needed based on performance. Enter the information in the appropriate log to indicate completion.

A. Alignment of the Spectrometer
    1. Perform the appropriate alignment for the instrument in use. See appropriate instrument personnel for assistance if needed.
B. System tuning
    1. Focus the alignment tool on the white light. Verify calibration by choosing 'Calibrate Instrument' under the 'Collect' pulldown and, if applicable, check the boxes for:
        i. Laser frequency calibration
        ii. Wavelength calibration
C. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

6 INSTRUMENTAL CONDITIONS

6.1 Microscope

Collect Tab	
Exposure Time:	20 seconds
Number of Exposures:	2
Final Format:	Shifted Spectrum (cm^{-1})
Cosmic Ray Rejection:	Yes (or Medium)
White Light Correction:	Yes (if applicable)
Background Exposures:	2
Bench Tab	
Beam Path/Accessory:	Microscope
Laser:	Select to coincide with analysis setup
Focus:	Adjust to achieve maximum signal
Aperture:	Select to coincide with analysis setup
Resolution:	Same conditions as sample analysis
Grating Positions:	Multiple
Max Range Limit:	3300 cm^{-1}
Min Range Limit:	200 cm^{-1}

6.2 Sample Compartment

Collect Tab	
Exposure Time:	20 seconds
Number of Exposures:	2
Final Format:	Shifted Spectrum (cm^{-1})
Cosmic Ray Rejection:	Yes (or Medium)
White Light Correction:	Yes (if applicable)
Background Exposures:	2
Bench Tab	
Beam Path/Accessory:	180-degree or applicable compartment accessory
Laser:	Select to coincide with analysis setup
Focus:	Adjust to achieve maximum signal
Aperture:	Select to coincide with analysis setup
Resolution:	Same conditions as sample analysis
Grating Positions:	Multiple
Max Range Limit:	3300 cm^{-1}
Min Range Limit:	200 cm^{-1}

7 Acceptance Criteria

7.1 Polystyrene

The Polystyrene spectrum is acceptable if all peaks are within ±5 cm^{-1} of the expected values, listed below (in cm^{-1}):

621 1001 1602 3054

8 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

9 Safety

Lasers used in Raman Spectrometers can be hazardous and must be used following best practices for eye safety and other potential hazards. Instruments are clearly marked with laser warnings and the laser safety class of the enclosure.

10 Revision History

Revision	Issued	Changes
02	07/01/2022	Revised to match new format requirements. Removed references to Horiba Raman and NXR Raman accessory. No other substantive changes to content.
03	11/15/2022	Section 2- Updated Scope to remove location. No other substantive changes to content.