APPLICATION NOTE

No. TN51508

Confident Data Collection in the QC Lab: Spectrometer Performance Assurance

Key words
Quality Assurance, quality control, raw material, SOP, System Performance Verification (SPV), system suitability, ValPro

Introduction

The central use of spectroscopy in quality control and quality assurance laboratories involves verification of a product's compliance with control specifications – product identity or purity. Strict standard operating procedures (SOPs) define each step. The workflow is defined, from sample collection to data analysis and report-out. Often the first step in this chain involves confirmation of instrument performance and the reliability of the instrument for the SOP task. Internal, regulatory and legal bodies require that an independent verification of the instrument be performed regularly, with signed, archived reports, for the final spectral evidence and report to have integrity.

Thermo Scientific Nicolet iS20: Performance and peace of mind

In the past, frequent performance testing was performed by personnel who started their careers during the age of "open cockpit" spectrometer operations. Knowledge of how the spectrometer worked – from properly loading chart paper to basic calibration methods – was required to obtain acceptable data. QC laboratories have evolved substantially, in terms of personnel, instrumentation and expectations.

This evolution has progressively placed more of the responsibility for performance verification on the instrument itself. Spectrometer performance criteria are increasingly stringent, as various oversight agencies require assurance that spectral data collected in the QC lab is trustworthy. Add-on software and hardware tools have made this process simpler, but the requirement for a set of routine tools fully integrated with the spectrometer (hardware and software) has become clear.

The latest generation mid-level FTIR spectrometer is the Thermo Scientific Nicolet™ iS20. Unlike previous FTIR spectrometers, this instrument and its software were designed and built with performance verification as a core metric. All Nicolet iS20 spectrometers ship with traceable standards mounted on an internal validation wheel, meaning any lab requiring system performance verification can now integrate suitability tests into laboratory procedures without additional purchases. Spectrometer Performance Verification (SPV) software tools provide a new level of operational and customizable functions for spectrometer validation. Finally, the new QCheck function within the OMNIC™ software, described in detail elsewhere, provides instant quality control activity through spectral comparisons or semi-quantitative analyses. The combination of these tools makes the Nicolet iS20 the best fit for any QC application.

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A QC laboratory scenario

The primary function of most QC laboratories involves determining if an incoming raw material or outgoing product meets specifications. If a problem arises downstream, resulting in a customer complaint, the data from the QC lab becomes a critical component in the investigation. Quality issues arising from suppliers can affect product, resulting in a need to take corrective or legal action upstream. Thus, much hinges on the reliability of the information. Customers or suppliers can shift the focus from addressing the problem to questioning the spectrometer, and time and money can be wasted.

This applies to forensics laboratories as well. An analysis may tie a suspect to the scene of a crime, but savvy defense lawyers can raise doubt regarding the data because there is nothing showing the spectrometer is reliable. The tiny doubt this can raise in a jury's malleable opinion may be sufficient to undermine the case.

The pharmaceutical industry has been living with scenarios like this for many years, as they are heavily regulated worldwide – all drug-related data must be backed with instrument performance verification and validation. The food and beverage industry is increasingly moving towards the same model, as the regulatory system becomes more involved. Sharpening instrument performance verification tools is more important than ever.

Ready for the lab: Inside and out

The Nicolet iS20 spectrometer comes standard with an internal verification wheel, shown in Figure 1. Containing NIST Traceable polystyrene and NPL Traceable NG11 Glass, the spectrometer is ready for the audited environment.

Figure 1: The Nicolet iS20 comes standard with a fully traceable verification wheel

Externally, the Nicolet iS20 is simplicity exemplified. All Nicolet iS20s ship with sealed and desiccated windows and two regenerable, dessicant packs. In purged environments, the gas is channeled around the sealed windows, so no modification is required to add or cease purging. The source is accessible from the sample compartment, ith no need to open the cover. In addition, the sample compartment is fully compatible with our line of Smart Accessories™ the Smart iTX™ attenuated reflectance accessory.

System Performance Verification (SPV)

The design process of making validation a core spectrometer metric required embedding system performance verification into the Nicolet iS20 hardware and software. The software tools are clustered beneath the SPV shield, shown in Figure 2. The shield indicates the status of both the results of SPV testing and whether the tests are up-to-date. A yellow indicator shows the SPV assurance is about to expire, and a red cross shows the SPV protection is expired, or the system is otherwise non-compliant.

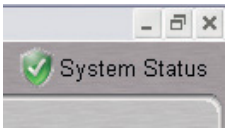


Figure 2: The System Performance Verification shield. The shield will be yellow if the SPV tests are nearing expiration, and red if there are any failures.

Clicking on the shield brings up the screen shown in Figure 3. Five oversight features are interrogated: Instrument Status, Scheduled Maintenance, System Suitability, Performance Verification and Spectral Quality. The Instrument Status and Spectral Quality indicators rely on tools long embedded in OMNIC, such as checking spectrometer communication and water contamination. Scheduled Maintenance keeps a record of when the spectrometer was last serviced, and a notification interval prior to expiration ensures this is duly noted by turning the shield yellow.

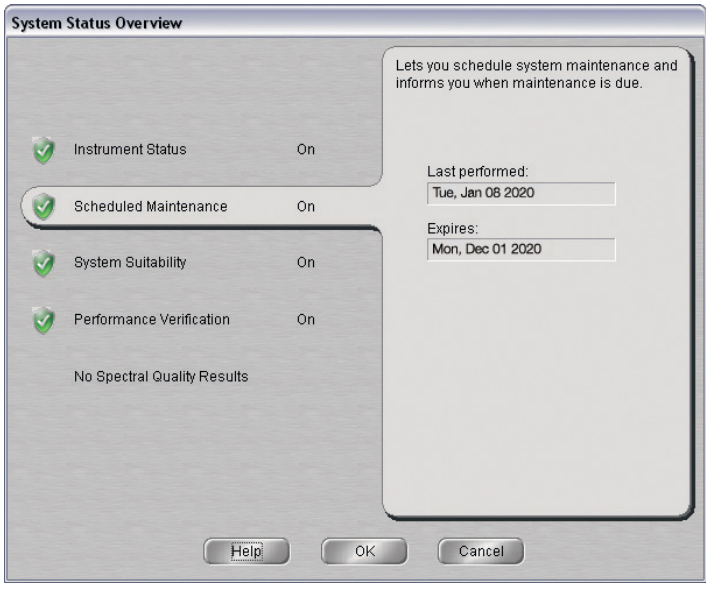


Figure 3: The SPV control panel, showing the ability to track dates of the last check of each feature.

System suitability

The System Suitability test can be configured by the QC lab to meet its own needs, including the specific accessory used in the analysis. As an example, Figure 4 shows the suitability tests set up for a packaging lab, where the "gold standard" sample is low density polyethylene (LDPE) run on a diamond ATR. Some user interaction is required since a real sample is involved.

System Suitability checks for contamination and throughput versus a stored reference spectrum. The test also examines the signal to noise in critical regions, ensuring confidence in detecting small spectral features. The most demanding test is based on a sample representative of the type of materials seen by the QC lab. Items 4 and 5 within Figure 4 report on peaks from the LDPE sample. The spectrum is checked for both peak location and intensity. The latter test, unique to OMNIC, provides confidence in the responsiveness of the spectrometer to the customer's specific samples. This greatly enhances the confidence in spectral comparison, especially when combined with the high sensitivity feature of QCheck.

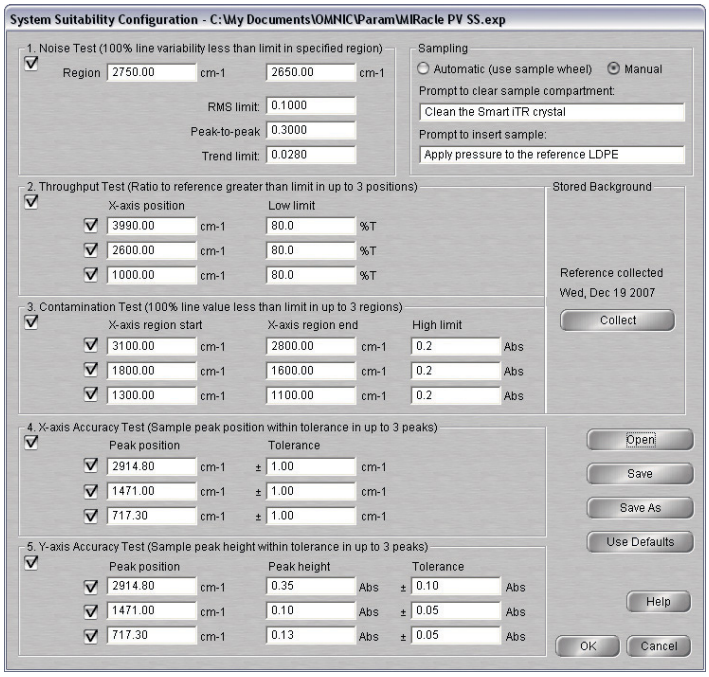
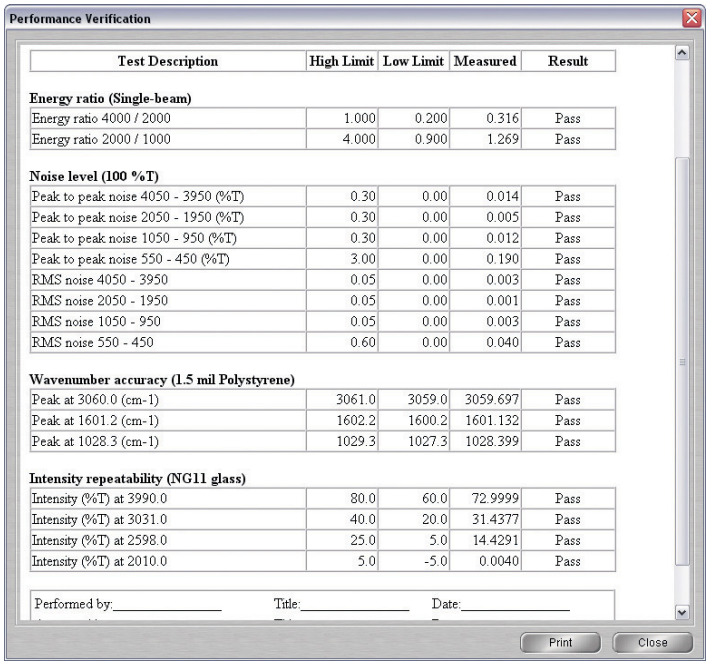


Figure 4: The System Suitability set-up window. Each parameter can be edited or turned off. The test configured here uses a low density polyethylene film as a reference material.

Performance verification

The Performance Verification routine ensures spectrometer performance using published ASTM methods. These preset tests rely on an internal wheel equipped with NIST traceable polystyrene and NPL traceable NG11 glass. PV operates fully automatically, with no accessory present, so the spectrometer can be PV-qualified continuously.

PV first executes a noise test, and then rotates the internal wheel to permit transmission through a polystyrene film and NG11 glass. A report of the test, shown in Figure 5, is then prepared These tests give confidence versus the traceable standards that both the wavenumber and intensity axes are reporting reliable values.


Performance Verification

Test Description	High Limit	Low Limit	Measured	Result
Energy ratio (Single-beam)				
Energy ratio 4000 / 2000	1.000	0.200	0.316	Pass
Energy ratio 2000 / 1000	4.000	0.900	1.269	Pass
Noise level (100 %T)				
Peak to peak noise 4050 - 3950 (%T)	0.30	0.00	0.014	Pass
Peak to peak noise 2050 - 1950 (%T)	0.30	0.00	0.005	Pass
Peak to peak noise 1050 - 950 (%T)	0.30	0.00	0.012	Pass
Peak to peak noise 550 - 450 (%T)	3.00	0.00	0.190	Pass
RMS noise 4050 - 3950	0.05	0.00	0.003	Pass
RMS noise 2050 - 1950	0.05	0.00	0.001	Pass
RMS noise 1050 - 950	0.05	0.00	0.003	Pass
RMS noise 550 - 450	0.60	0.00	0.040	Pass
Wavenumber accuracy (1.5 mil Polystyrene)				
Peak at 3060.0 (cm-1)	3061.0	3059.0	3059.697	Pass
Peak at 1601.2 (cm-1)	1602.2	1600.2	1601.132	Pass
Peak at 1028.3 (cm-1)	1029.3	1027.3	1028.399	Pass
Intensity repeatability (NG11 glass)				
Intensity (%T) at 3990.0	80.0	60.0	72.9999	Pass
Intensity (%T) at 3031.0	40.0	20.0	31.4377	Pass
Intensity (%T) at 2598.0	25.0	5.0	14.4291	Pass
Intensity (%T) at 2010.0	5.0	-5.0	0.0040	Pass

Performed by:_____ Title:_____ Date:_____

Print Close

Figure 5: The report window from a successful Performance Verification run.

Intensely regulated laboratories

The Thermo Scientific ValPro™ validation package is a proven industry leader designed for pharmaceutical laboratories and other industries needing even stricter validation protocols. ValPro provides DQ, IQ, OQ and PQ compliance, testing using ASTM, Chinese Pharmacopeia, European Pharmacopeia, Japanese Pharmacopeia and US Pharmacopeia guidelines, and trend-charting for long term evaluation. ValPro is an add-on package for any laboratory requiring additional regulatory compliance. When active, ValPro supersedes PV, as it contains all of the PV tests plus trend charting and more.

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As the Nicolet iS20 comes with the appropriate traceable validation wheel installed, there are no additional hardware requirements for ValPro. The software allows validation of certain accessories as well as the spectrometer itself, ensuring compliance at all levels.

Confidence before, during and after

Each function of SPV is watched by the OMNIC software. Dates for all instrument operations – Performance Verification, System Suitability, normal maintenance and ValPro – are recorded, and expiration dates set. The shield shown in Figure 2 will turn yellow to warn of impending expiration of any of the operations, and red when they are expired. A green shield indicates the system has passed all assigned tests, within the prescribed interval, and data collection can proceed with confidence.

Further, the status of the SPV tools at the time of data collection is recorded in the experiment information header. This provides an audit trail for each spectrum, ensuring the instrument operation was verified within the proper time interval before a critical data set was collected.

The flow chart shown in Figure 6 summarizes this process. Critical decision points are monitored, averting risk at every step, so even the most intensive audit will find everything in order. In a logical sequence, the Spectrometer Performance Verification process takes you down the left column, so a sample measurement leading to an “Out of spec” or “Pass” conclusion can be reached with confidence. Less demanding processes can lead to risk and a questionable conclusion at the end. With the integrated SPV tools and Nicolet iS20, you are directed to corrective actions when a “No” arises, which leads directly back to the path of confident data collection.

Clearly, the Nicolet iS20 covers all aspects needed in demanding laboratory settings: verification before, status indication during and an audit trail afterwards. Never has it been this easy to cover all your verification needs.

Conclusion

When verifying raw materials or finished goods with the Nicolet iS20, quality control laboratories will now have more confidence than ever before in their answers, every day. System Performance Verification ensures that the infrared spectrometer, the accessory, and the method are meeting the “fit-for-purpose” laboratory requirements, eliminating any risk or doubt inherent in using analytical equipment. Further improvements in both confidence and productivity can be achieved using OMNIC QCheck software, the SOP builder and the simple operation achievable through the Nicolet iS20 touch panel, by eliminating manual procedures and doubtful comparisons. All these QC-focused tools come standard with any Nicolet iS20 FTIR spectrometer, delivering performance, peace of mind and compliance from day one.

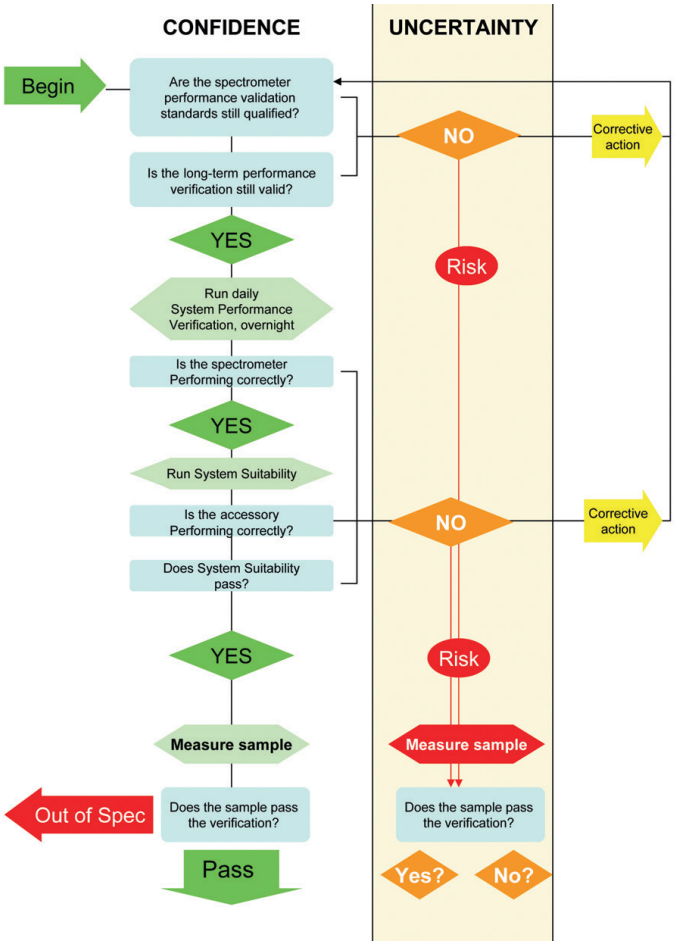

Figure 6

Find out more at **thermofisher.com/FTIR**

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