BRAZORIA COUNTY SHERIFF'S OFFICE
CRIME LABORATORY

INSTRUMENT QUALIFICATION and VALIDATION PLAN FOR THE LABORATORY'S FTIR SYSTEM

Section: Seized Drugs
Type of Analysis: Qualitative Identification by Fourier Transform Infrared Spectrometry
Date of Plan: December 2023

04 Jan 2024
Laboratory Director – Date

4 Jan. 2024
Quality Assurance Manager – Date

Kayla M. Baylor
Analyst – Date

1/2/24
Analyst – Date

Brazoria County Sheriff's Office Crime Laboratory
Seized Drugs Section
Analytical Instrument Qualification and Validation Plan for FTIR System

Abstract

To ensure that all instrumental data generated during the analysis of unknown samples submitted to the laboratory, all instruments and methods will be evaluated to determine their reliability and suitability. Analytical method validation is the collection of documented evidence that an analytical procedure is suitable for its intended use. Analytical instrument qualification is the collection of documented evidence that an instrument performs suitably for its intended purpose. The objective of this qualification plan is to establish the parameters by which the Laboratory's Fourier Transform Infrared Spectrometry (FTIR) will be evaluated to demonstrate its suitability for use in the identification of suspected controlled substances, dangerous drugs, and related materials. Additionally, due to the scope of the integration of the new equipment into the existing instrument platform, a performance qualification plan will be set forth for the Laboratory's FTIR system. This plan details the depth of the performance check when maintenance (preventative or corrective) is performed on the system and when those checks should occur.

Background

FTIR is an analytical technique used in the analysis of controlled substances due to its non-destructive nature, high level of discrimination, and its ability to identify and differentiate free base/acid and salt forms of various substances. As molecules are exposed to a specific energy of infrared radiation, the molecule will absorb the radiation by vibrating. At room temperature, molecules sit in their ground electronic and ground vibrational states. If the infrared radiation the molecule is exposed to has the appropriate energy, the molecule will absorb the radiation exciting it to a higher vibrational state. The FTIR directs infrared radiation to an unknown sample (compound of interest) and measures the radiation absorbed by the sample. The chemical bonds in a compound will vibrate at characteristic frequencies as the compound absorbs infrared radiation. The functional groups of the molecule absorb the infrared radiation in the same frequency range regardless of the structure of the rest of the molecule. Therefore, there is a direct correlation between the frequencies of infrared radiation a molecule absorbs and its structure. The measured absorption signals from the FTIR are transmitted into a graphical spectrum where the absorbance is plotted as a function of energy, typically noted as wavenumbers. The spectrum plot depicts the characteristic vibrational and rotational absorption bands present in a sample vs. the intensity of those bands over a range of infrared radiation.

Attenuated total reflectance (ATR) is a common sampling technique used in FTIR spectrometry. This sampling technique can be used on solid or liquid samples with minimal to no sample preparation required. The ATR attachment is placed into the path of the infrared beam in the spectrometer. The infrared beam is directed into a diamond crystal by the mirrors in the ATR attachment. The sample is placed on the diamond crystal and a portion of the infrared beam exits the crystal coming into contact with the sample where it will be absorbed (attenuated) at the appropriate wavenumbers. As the attenuated infrared beam leaves the crystal it is directed out of the ATR attachment and on to the detector and data processor for conversion into an absorption spectrum.

An infrared spectrum can provide very useful information on the structure of a compound by identifying the presence of functional groups. However, the identification of an unknown is best performed by comparison of the absorption bands and their intensities over a full spectral range (4000- 600 cm^{-1}) with

comparison of a known sample performed under the same conditions. Infrared spectra contain so many peaks that the possibility that two compounds have the same infrared spectra is extremely small, the only exception to this is optical isomers which will produce the same spectra. Therefore, the infrared spectrum provides analysts with valuable information when analyzing and identifying unknown substances.

Instrument Set-up and Installation Qualification:

The Seized Drugs purchased an FTIR from Thermo Scientific to expand the sections instrumental techniques to aid in the identification of unknown substances. Due to the FTIR's ability to differentiate between the salt and base form of drugs and its ability to analyze sample mixtures with little to no sample preparation the instrumentation will provide a valuable tool in the section.

The new instrument designated FTIR-1 was installed by Thermo Scientific technician Jared Stevens on August 1, 2022. A qualification software program referred to as ValPro System Qualification (ValPro) was included with the instrument system. The ValPro program incorporates tests for energy ratio, noise level, optical resolution, wavenumber accuracy, repeatability, and detector linearity. FTIR transmission spectra are collected in ValPro using two reference standards: a 1.5 mil polystyrene standard and a NG11 glass standard which are mounted on a serialized wheel inside of the instrument. A qualification report is generated with pass/fail results of these tests based on the manufacturer's specifications once the program has finished running. As part of the installation, the service technician ran the ValPro program and the instrument passed all qualification tests. After the installation was complete and the qualification tests passed the instrument was released to the section.

All relevant manufactured Declaration of Conformities and installation documentation for the instrument can be found in System Qualification Package Binder including the results of the onsite ValPro qualification. Refer to Table 1 for instrumental specifications.

Table 1 – FTIR-1 Instrumental Specifications

Thermo Scientific Nicolet iS20 Fixed Gold KBr Beamsplitter Spectrometer System Serial # BEP2210424	
Component	**Serial #**
IR Source	BCA2210729
Laser	BDH2210473
Modulator	BBU2200859
Source Mirror	ASG2210958
Aperture	BEA2210467
DTGS KBr Detector	BEN2210385
Validation Wheel	AJZ15486
Smart iTX ATR base and diamond crystal plate	Y92890
Computer	GWWV1N3
Software	OMNIC version 9.13.1224 ValPro Qualification version 2.9.99

Operational Qualification and Validation

Operational qualification is the documented collection of activities necessary to demonstrate that an instrument is operating as intended by the manufacturer and as required by the user. The FTIR instrument includes ValPro System Qualification software that performs test to demonstrate the instrument is operating according to the manufacturer's specifications. ValPro tests will be run at various times throughout the validation process following installation.

The use of validated methods in a qualified instrument provides confidence in the reliability of identification for analyzed samples. Commonly encountered controlled and non-controlled substances, and dangerous drugs will be analyzed to see if the method is able to produce accurate identifications and meets criteria for selectivity, repeatability, and matrix effects.

The Smart iTX Diamond.exp method parameters that will be validated in this study are

Number of Sample Scans:	16
Resolution:	4 cm^{-1}
Final Format:	Absorbance
Correction:	None
Background:	Before every sample
Scan Range:	4000-600 cm^{-1}
Gain:	1
Optical Velocity:	0.4747
Aperture:	230

Procedure

Standards will be analyzed using the Smart iTX Diamond Method by placing a small amount of sample directly on the ATR sampling area. These standards were chosen to test the instrument's selectivity, its ability to discriminate salt from base form, and its ability to distinguish diastereomers. To evaluate the instrument's repeatability, ten samples of verified cocaine HCl and ten samples of cocaine base will be analyzed over two days. Wavenumbers must be less than or equal to 2 cm^{-1} to meet acceptance for repeatability. Lastly, nine mixtures of controlled substances and commonly encountered adulterants and diluents will be analyzed to assess the impact of matrix effects on ability to identify unknown substances. The mixture samples will consist of four samples of cocaine HCl with adulterants or diluents, four samples of cocaine base with adulterants and diluents, and one methamphetamine sample with an adulterant or diluent. The spectra data files for each standard run will be made available electronically. Analysis of the standards and samples will be compared to an outside library for identification. The data collected during validation will be used to make an in-house BCCL library on FTIR-1.

The analysis of mixtures to assess matrix effects demonstrates the limitation of FTIR spectrometry. FTIR does not include a separation technique therefore, the spectra produced is the result of absorptions from all substances present within the sample. Due to this the mixture samples may not result in identifiable spectra if they are not relatively pure. Additionally, adulterants and diluents in high concentration may often mask the presence of the controlled substance. This limitation may be overcome through the use of sample purification techniques prior to performing FTIR analysis. It is also

possible to use library search routines to give an indication as to what substances other than those of interest may be present in a mixture. For example, lactose in a tablet, procaine in a cocaine sample, or dimethylsulfone with methamphetamine. The ability to know what other substances are present may allow the analyst to use software subtraction functions to remove the other substances contribution to the spectrum to generate a spectrum that can be used to identify the presence of any controlled substances or dangerous drugs.

Performance Plan

Instrument performance must be assessed prior to and during instrument use and after maintenance to the system in order to utilize the FTIR technique on a routine basis. A performance qualification is the documented collection of activities performed in order to demonstrate that an instrument consistently performs according to specifications set forth by the laboratory and is performing as expected for its intended use. This will be accomplished by implementing a maintenance/performance plan.

The seized drugs section will perform monthly performance checks on the FTIR system. The performance check will consist of running the ValPro System Qualification check in addition to checking the instruments alignment and running a known standard. Acceptable performance will be indicated if the instrument passes all of the qualification tests based on the manufacturer's specifications. The known standard that is run must be documented and have an acceptable library match. The results obtained from the ValPro checks will be compared to prior results to verify that the system is working consistently over time.

FTIR-1 will be evaluated each day of use by performing a system status check and alignment. The system check is performed automatically when the OMNIC operating software is opened and electronic connection to the instrument is made. A successful status check is indicated on the instrument by the instrument with a green check mark and a note that all systems passed. Performing a system alignment optimizes the internal instrument components and maximizes the infrared signal that is received by the detector. Monitoring trends in the numerical value of the signal can indicate when maintenance of the system is needed.

Preventative maintenance will be performed annually on FTIR-1 and will include a ValPro performance check to demonstrate that the instrument is operating as expected. Extensive maintenance includes the replacement of internal components such as the source, laser, interferometer, mirror, detector, ATR accessory or major electronics such as boards, or integrated circuits. When extensive maintenance on the instrument set-up is performed a ValPro performance check will be performed after all maintenance has been completed.

If an instrument parameter is not deemed acceptable (e.g. system check fails or one of the ValPro tests fail), the instrument will be taken out of service until maintenance can be performed and the instrument is brought back into service. When the instrument is not being utilized due to servicing or malfunction it will be clearly marked to avoid unintended use.

Reducing the risk of contamination is an important consideration when performing analysis on an FTIR. To ensure this does not occur the diamond and stage will be cleaned between each sample using a mild

acid (e.g. 0.1N HCl) and acetone. The mild acid will clean the area and the addition of acetone will remove any water present from the cleaning.

Conclusion

The objective of this validation plan is to qualify the FTIR-1 instrumental set-up, validate a method on the instrument for the identification of unknown compounds commonly submitted to the Seized Drugs section, and establish an in-house library for identification purposes.

References

1. Principles of Instrumental Analysis-6th Edition, Douglas Skoog, Chapter 16 – An Introduction to Infrared Spectrometry.