

# Direct-on-filter Analysis for Respirable Crystalline Silica Using a Portable FTIR Instrument



Centers for Disease Control  
and Prevention  
National Institute for Occupational  
Safety and Health

Information Circular 9533

## **Direct-on-filter Analysis for Respirable Crystalline Silica Using a Portable FTIR Instrument**

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January 2022

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NIOSH [2022]. Direct-on-filter analysis for respirable crystalline silica using a portable FTIR instrument. By Chubb LG, Cauda EG. Pittsburgh PA: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 2022–108, IC 9533. <https://doi.org/10.26616/NIOSH PUB2022108>

DOI: <https://doi.org/10.26616/NIOSH PUB2022108>

DHHS (NIOSH) Publication No. 2022-108

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January 2022

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## Acronyms and Abbreviations

CRADA	cooperative research and development agreement
FAST	Field Analysis of Silica Tool
FTIR	Fourier transform infrared spectroscopy
IR	infrared
LOD	limit of detection
LOQ	limit of quantification
MSHA	Mine Safety and Health Administration
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
PVC	polyvinyl chloride
QA	quality assurance
RCS	respirable crystalline silica
SIMPEDS	Safety in Mines Personal Dust Sampler

## Unit of Measure Abbreviations

°C	degrees Celsius
cm	centimeter
°F	degrees Fahrenheit
lpm	liters per minute
mg	milligram
mm	millimeter
µg	microgram
µm	micrometer
cm <sup>-1</sup>	reciprocal centimeter or inverse centimeter

## Definitions

Amorphous silica	a non-crystalline form of silica which, if present, interferes with the accurate quantification of crystalline forms, including quartz
Apodization	a mathematical process used in FTIR analysis to smooth the signal
Background (spectrum)	a measurement taken prior to sample measurement: if a clean filter is used the spectral features of the filter media are reduced, and environmental features such as water vapor and carbon dioxide are largely eliminated (or reduced) when the sample is analyzed
Baseline correction	a mathematical adjustment to decrease a curvature or slope in a sample spectrum; see Figure 6 for an illustration of the effect of baseline correction; various correction algorithms can be used
Commodity	a product or group of products that is mined at a particular operation; may also be considered a subsector within the mining industry
Correction factor	a mathematical adjustment made in the FAST software to account for mineral content of respirable dust and other factors that may otherwise decrease the accuracy of the respirable crystalline silica quantification; uses standard analysis results from an accredited laboratory as the reference method
Cristobalite	a form of crystalline silica, less commonly encountered than quartz
Desiccant	a material used to absorb excess moisture or humidity
Direct-on-filter	analysis that can be completed on the aerosol sampling filter on which the sample is collected, without altering the sample filter or the collected sample
Field-based monitoring	any exposure monitoring activity that can be completed where the sample is collected at the work site, without the need to send samples to an off-site laboratory for analysis
Focal point	the point at which the rays of the infrared beam converge and are most concentrated, and where the sample should be positioned

Fourier transform infrared spectroscopy	an analytical method capable of collecting a high resolution infrared (absorption) spectrum for a sample over a broad spectral range and converting the raw data in a spectrum using Fourier transform
Integration	the calculation of the area of a specific region of the sample spectrum; the integrated value is correlated to the amount of a substance that is present
Interference	a species that interferes with the analysis of quartz, possibly impacting the accuracy of the quantification of quartz
Limit of detection	the lowest amount of a substance that is detected with confidence by an analytical method; below this quantity, the signal may be due to random noise rather than to the presence of the substance
Limit of quantification	the lowest amount of a substance that is quantifiable with confidence by an analytical method; below this quantity, the amount cannot be quantified with confidence, though the substance can be <i>identified</i> with confidence if the limit of detection (which is always less than the limit of quantification) has been met
Macro	a series of software instructions that can be executed as one procedure; for field-based monitoring, the available macros (from NIOSH) first execute a baseline correction of the spectrum and then integrate the areas of interest; in some software packages, the NIOSH macro may also export sample results or generate a sample report
Non-destructive	any method of analysis where the sample is not altered or disturbed—that is, the sample is preserved in its original form and remains available for analysis using other analytical approaches
Quality Assurance (QA) sample	a sample taken for quality assurance purposes that is analyzed at the beginning of each session, with the results compared to previous data to confirm that the instrument hardware and software are generating consistent and reliable results
Quartz (alpha quartz)	the most common form of crystalline silica
Real-time monitoring	a specific type of field-based monitoring in which results are available after very short data collection periods (on the order of seconds), enabling changes in conditions (e.g., dust concentration) to be measured as they occur

Resolution	the minimum difference in frequency that can be distinguished within the spectrum; increasing the resolution increases the level of detail that is visible
Respirable crystalline silica	particles of crystalline silica small enough to reach the alveolar region of the lungs when inhaled
Respirable dust	particles of dust (of any composition) small enough to reach the alveolar region of the lungs when inhaled
Sample bracket	a NIOSH-designed component that is installed in the FTIR instrument (replacing the original bracket of the instrument) to accept the sample cradle or the sample holder, ensuring the correct position of the sample for analysis
Sample cradle or holder	a NIOSH-designed component that is inserted in the sample bracket to accept either the four-piece cassette (cradle) or the dust sampling capsule (holder), ensuring the correct position of the sample for analysis
Site-specific quantification	an adjusted quantification of RCS concentration specific to a particular location or operation, based on previous comparison of the (unadjusted) quantification generated by the FAST software and standard laboratory analysis of the same samples
Spectrum (spectra)	a plot of the sample data generated by the FTIR instrument and software (i.e., infrared absorbance at each wavenumber); spectrum can refer to the visual plot of the data or to the collection of individual data points
Standard analysis	the laboratory-based analysis method that would typically be used or accepted for exposure compliance purposes in a particular sector or subsector; for respirable crystalline silica analysis in the United States, see the following methods: MSHA P-2 [MSHA 2013a], MSHA P-7 [MSHA 2013b], NIOSH 7500 [NIOSH 2003], NIOSH 7603 [NIOSH 2017b], OSHA ID-142 [OSHA 2015]
Tridymite	a form of crystalline silica, less commonly encountered than quartz



# Direct-on-filter Analysis for Respirable Crystalline Silica Using a Portable FTIR Instrument

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## Introduction

Respirable crystalline silica (RCS) exposure occurs in many industries, and the potential health effects of RCS have been well-documented [NIOSH 2002]. Monitoring is an essential part of any RCS exposure control plan, but the timeliness of results from the lab and the cumulative cost of analysis may limit the extent and the effectiveness of the monitoring process.

Field-based monitoring procedures offer a supplement to sample analysis completed by an offsite laboratory. By eliminating the time spent transporting samples and waiting for laboratory results, results are available the same day or the next day to allow operators to make decisions regarding controls. This enhances the ability to collect data more effectively, enables better control over potential exposures, and protects worker health.

This document details how to implement field-based monitoring for RCS. It is primarily intended for industrial hygienists and other workers with health and safety responsibilities, specifically within the mining industry (although workers in other industries may also find it useful). The document has been written for a user with experience in respirable dust or RCS exposure assessment but who does not necessarily have specialized training in analytical techniques. The following topics are covered:

- General instructions for how to set up the equipment and the software required for field-based RCS monitoring.
- Technical details of the monitoring method with explanations of why they are important.
- Quality assurance procedures to ensure consistent data.
- Examples and case studies on how to use different types of samplers in conjunction with field-based monitoring.
- Links to additional resources for field-based monitoring (see Appendix A).
- Checklists to guide users through the process quickly (see Appendix B).

The NIOSH Manual of Analytical Methods contains sampling guidance for RCS in particular [NIOSH 2003a] and for aerosols in general [NIOSH 2016b].

This document contains guidance applicable to portable Fourier transform infrared spectroscopy (FTIR) instruments in general, though some instrument-specific guidance is also provided where necessary. While researchers from the National Institute for Occupational Safety and Health (NIOSH) have conducted extensive investigation on the use of four portable Fourier transform infrared spectroscopy (FTIR) instruments [Ashley et al. 2020], other models of FTIR instruments may also be available and compatible with field-based monitoring.

At the time this document was published, direct-on-filter analysis with portable FTIR was not a stand-alone NIOSH analytical method or a standard analytical method used by MSHA or OSHA

or other international bodies. The role of field-based monitoring should be to act in support of maintaining regulatory compliance by facilitating faster and more thorough assessment of RCS levels. When there is a need for a formal assessment of compliance status, the dust samples analyzed in the field should also be submitted to an accredited laboratory.

The field-based RCS monitoring approach presented here has been designed for respirable alpha quartz, which is the most common crystalline silica polymorph. The analysis is not currently optimized for other regulated crystalline silica polymorphs, cristobalite and tridymite, which can both impact the accuracy of the field-based analysis if they are present. In most mining environments, quartz is likely to be the only form of RCS present, but it is important to know if either cristobalite or tridymite has been reported in respirable samples (based on previous laboratory analyses). The term RCS will be used throughout this document except where more specificity is required.

## Technical Note

The approach to field-based monitoring described in this document and the quantification models used in the NIOSH-developed Field Analysis of Silica Tool (FAST) software were developed using 37-mm, 5- $\mu\text{m}$  pore polyvinyl chloride (PVC) filters housed in 4-piece conductive cassettes with stainless steel support (rather than a cellulose backing pad), or 37-mm, 5- $\mu\text{m}$  pore polyvinyl chloride (PVC) filters (within a capsule) housed in the dust sampling cassette with stainless steel support (see Figure 2). The models were calibrated using crystalline silica (quartz) particles (primarily Min-U-Sil 5, with NIST 1878a samples evaluated to confirm similarity) aerosolized in laboratory dust chambers. Calibration samples were analyzed using a Bruker Optics Alpha infrared spectrometer in transmission mode. Sixteen co-added sample scans were analyzed at a resolution of 4  $\text{cm}^{-1}$  using Blackman Harris 3-term apodization. Samples were subsequently processed using a concave rubberband baseline correction<sup>a</sup> (10 iterations) with 64 baseline points. Other options for instrument analysis parameters are listed in Table 2. The spectral region between 816 and 767  $\text{cm}^{-1}$  was integrated, and calibrated models were developed from the regression of the integrated area vs. gravimetric mass of crystalline silica (quartz).

When possible, field-based monitoring for crystalline silica is best completed using the same filters, cassettes, and operational settings listed above. Field-based monitoring may still be completed using different filter media, cassettes, and settings, but this may result in error or uncertainty in crystalline silica quantification.

For additional information regarding the development of field-based, direct-on-filter analysis, see Miller et al. [2012], Miller et al. [2013], Cauda et al. [2016], and Chubb and Cauda [2021]. This document is representative of all relevant findings to date, but research is ongoing in this area and additional findings will be published and made available to the public in the future.

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<sup>a</sup> Though a complete definition is beyond the scope of this document, a “concave rubberband correction” is a specific type of baseline correction that imagines a rubberband stretched between the two ends of the spectrum. The correction is applied iteratively, with the number of iterations determining the intensity of the correction.

## Overview of Field-based Monitoring

The process of field-based monitoring is depicted and described in Figure 1. Respirable dust samples are collected in the same way that they would be for conventional exposure monitoring practices. Following sample collection, the FTIR instrument is set up and its performance verified before respirable samples are analyzed. Sample data is processed using the FAST software, and the results can contribute to decision-making to decrease worker exposure to respirable crystalline silica. Additional samples can be collected and analyzed to confirm the efficacy of new or modified controls, beginning the process over again. The non-destructive analysis allows the same samples to be sent to an off-site laboratory following field-based analysis, if desired.

### What you need before you start:

- Obtain a portable FTIR instrument and its corresponding software
- Obtain the appropriate sample bracket for your FTIR instrument
- Obtain analysis ready cassettes and accessories:
  - four-piece cassette and sample cradle
  - coal dust sampling cassette and sample holder
- Obtain or create a set of quality assurance samples

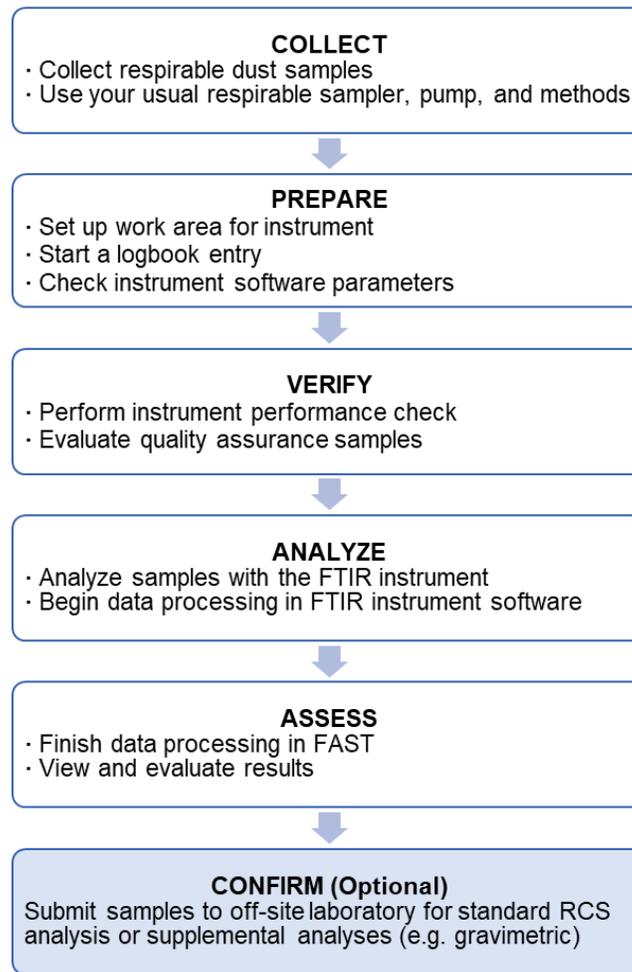


Figure 1. Overview of the field-based monitoring process.

Field-based monitoring does not require laboratory experience or training but does require careful execution to ensure reliable data; it should be part of a well-planned RCS exposure assessment strategy and used in conjunction with routine laboratory analysis of respirable samples. Previous experience in industrial hygiene or exposure assessment is recommended for users of field-based monitoring. Although FTIR instruments are generally designed to be user-friendly for non-expert users, NIOSH recommends contacting the manufacturer for assistance with initial instrument setup and with basic instrument training.

## **Getting Started: Hardware and Software**

Table 1 depicts and describes the hardware and software requirements. These requirements are in addition to the personal sampling pumps and respirable cyclone samplers that are typically used.

The analysis of samples collected by impactor-type respirable samplers is not currently supported by this field-based monitoring technique.

**Table 1. The hardware and software requirements and recommended materials for implementing field-based monitoring. These components are necessary to collect and analyze data from respirable dust samples. While it is possible to conduct field-based monitoring without the recommended materials, they facilitate a much faster, simpler, and more reliable process.<sup>b</sup>**

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### **Required Hardware**

- A portable FTIR spectrometer with transmission module (KBr windows and beam splitter). Available from Agilent Technologies, Bruker Corporation, PerkinElmer, and Thermo Scientific
- Laptop computer; see software specifications of the FTIR instrument software and Field Analysis of Silica Tool (FAST) for system requirements
- Sample bracket and sample cradle or sample holder (files of 3D models for 3D printing or traditional machining are freely available at <https://3dprint.nih.gov/discover/3dpx013209> or by searching (by author) for NIOSH at <https://3dprint.nih.gov/discover>)

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### **Required Software**

- FTIR spectrometer software (available from the manufacturer of the chosen instrument)
- Field Analysis of Silica Tool (FAST) <http://www.cdc.gov/niosh/mining/www.cdc.gov/niosh/mining/works/coversheet2056.htm>
- Spreadsheet software

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### **Recommended Materials**

- One of the cyclone samplers listed in FAST (see FAST documentation)
- “Analysis-ready” sample cassettes, in which the sample filter does not need to be removed from the cassette before analysis
  - A four-piece cassette
  - Dust sampling cassette (without check valve); in the mining industry this is often referred to as the coal dust sampling cassette, though it may also be known by different names in other industries
- “Quality assurance” samples
  - Commercially available filter samples or standards that contain quartz
  - A “do-it-yourself” option (as described in this Information Circular)

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<sup>b</sup> Links to instrument manufacturers and NIOSH products can be found at <https://www.cdc.gov/niosh/mining/content/HandwareandSoftwareforFAST.html>.

## Conducting Field-based Analysis for Respirable Crystalline Silica

After respirable dust samples have been collected, RCS is quantified by FTIR analysis. This section describes the analysis parameters that should be used to produce the most reliable results and outlines procedures for verifying that the instrument and its settings are appropriately configured before proceeding with sample analysis.

### Setting Instrument Analysis Parameters

Research conducted at NIOSH has indicated that the following parameters for analysis with a portable FTIR instrument provide the optimal balance necessary to obtain accurate and reproducible data in a reasonable period of time. For example, higher resolutions result in more data points per sample, and increased scan accumulations decrease noise (variation) in the data, but both come at a cost of longer analysis times. Table 2 summarizes the relevant parameters and settings used by NIOSH to develop the field-based method.

These parameters are important to the integrity of the data, and therefore it is essential to ensure that the portable FTIR instrument is configured to these parameters before it is used for the application described in this document. The top (unshaded) portion of the table contains basic parameters that should be selected, regardless of the particular instrument or software used. Modifying these settings will impact analysis results and may invalidate the results. The bottom (shaded) portion of the table contains more advanced parameters, which may not be available for some instruments or software programs. While it is advisable to match these parameters, if available, they are less significant. Regardless, it is important that the same parameter options are used consistently from sample to sample and day to day.

**Table 2. Instrument analysis parameters**

<b>Parameter</b> (including possible alternate terminology)	<b>Value and unit</b>
Resolution (cm <sup>-1</sup> )	4
Sample scan time <i>Scans</i> <i>Accumulations (scans)</i> <i>No. of scans</i>	16 (scans)
Background scan time <i>Scans</i> <i>Accumulations (scans)</i> <i>No. of scans</i>	16 (scans)
Spectral range <sup>c</sup> <i>Min range limit and Max range limit Start (cm<sup>-1</sup>) and End (cm<sup>-1</sup>)</i> <i>Save data from __ cm<sup>-1</sup> to __ cm<sup>-1</sup></i>	4000 cm <sup>-1</sup> –400 cm <sup>-1</sup>
Result spectrum <i>Y-axis units</i> <i>Ordinate units</i> <i>Final format</i>	Absorbance or A
Phase correction <sup>d</sup> <i>Phase correct</i>	Mertz
Apodization <sup>e</sup>	Blackman Harris or Blackman Harris 3 or strong or NB Strong or Norton Beer Strong
Zero-fill factor <sup>d</sup> <i>Zero-filling factor</i>	2

<sup>c</sup> Some instruments may not be capable of acquiring data over the full range of 4000 cm<sup>-1</sup>–400 cm<sup>-1</sup>; however, if the range of approximately 1500–500 cm<sup>-1</sup> is acquired, the quantitative region for quartz will not be impacted by this limitation but the ability to identify and adjust for confounding minerals may be reduced. Data from as much of the range of 4000 cm<sup>-1</sup>–400 cm<sup>-1</sup> as possible should be acquired.

<sup>d</sup> A software parameter of FTIR analysis that impacts how the software program processes the raw data generated by the instrument, although this parameter may not be available or modifiable in some software packages.

<sup>e</sup> The quantification models used in FAST were calibrated with data processed using Blackman Harris 3-term apodization. More accurate results are likely to be obtained by using this same apodization, though using a different apodization (including using no apodization, if no option is available in the instrument software) is not likely to result in substantial differences in quantification of RCS, (for samples of respirable dust; see Appendix C). Regardless of the type of apodization selected, it should be used consistently for all samples analyzed.

## Verifying Instrument Integrity

When the instrument is not working properly, data quality will be impacted and the acquired data may be less accurate. In order for the FTIR instrument to produce generally reliable data, the internal optical components of the instrument must be in alignment and functioning according to specifications. It may not be possible to easily perform a visual check of these components; the software associated with each portable instrument has the capability to conduct a series of tests to evaluate the performance of the instrument. Depending on the instrument, these tests may be termed Systems Checks or Instrument Checks, Instrument Verification, Performance Qualification (PQ), or Operation Qualification (OQ). Regardless of terminology, the tests should generally include a check of the instrument signal-to-noise ratio and a check of the frequency and alignment.

While the instrument will function even if these tests are not performed, the acquired data may not be accurate. It is recommended to perform these tests on a regular basis, such as weekly, and in cases where the instrument may have been handled roughly (such as when shipped by a carrier service, or as checked baggage with an airline), or has changed environments drastically (such as from an arid climate to a humid climate or from a cold environment to a warm one). As the instrument and its internal components age, some components will need to be replaced, and regular performance testing can indicate when this is necessary.

When the performance tests are carried out routinely, they will indicate potential performance concerns before data quality is affected; if performance tests are not carried out regularly, low-quality data may go unnoticed. See the Quality Assurance (QA) Sample Spectra section for additional information on verifying that the instrument is producing accurate and reproducible data over time.

## Inserting the Sample in the Instrument

Once the portable FTIR is set up with the proper analysis parameters and the integrity of the instrument has been verified via performance tests, it is important to understand how to use the portable instrument for the analysis of respirable dust samples using the modality generally referred to as “direct-on-filter.” A few constraints and requirements need to be met to ensure that accurate results are obtained using direct-on-filter analysis.

When using a four-piece cassette, the top (inlet) piece and bottom (outlet) piece of the cassette should be removed from the two middle sections before being placed in the sample cradle. When using the dust sampling cassette, the foil capsule should be carefully removed from the plastic outer cassette before the capsule is placed in the sample holder.

The following pictures and directions (Figures 2–4) provide guidelines on how to position the samples inside the portable FTIR. In general, it is good practice to position the sample with the sample side facing the infrared source, which is typically on the left.



Photos by NIOSH

**Figure 2. Examples of suitable sample cassettes (which allow the filter media to remain secured in the cassette or capsule) for field-based analysis. The holder or cassette containing the filter media with the sample should be open on both sides, which can be accomplished by removing the top and bottom cassette pieces of the four-piece cassette (top) or removing the capsule from the dust sampling cassette (bottom). From one side the dust material on the filter (or a portion of the filter, in the case of dust sampling capsule) should be visible, and from the other side the underside of the filter media should be exposed and the support ring should be visible.**



Photos by NIOSH

**Figure 3. Inserting the sample into the cradle or holder. NIOSH has designed a cradle (top left) to accommodate the four-piece cassette (top right) and a filter holder (bottom left) to accommodate the dust sampling capsule (bottom right). The four-piece cassette should be inserted firmly into the cradle so that the sample is not tilted forward or backward in the cradle. The dust sampling capsule should be centered in the holder and secured so that the sample remains stationary when the holder is held vertically. Files of 3D models of the cradle, the filter holder, and instrument brackets (for each of the four portable instruments, for 3D printing or traditional machining) that fit either option are freely available at <https://3dprint.nih.gov/discover/3dpx-013209>.**



Photos by NIOSH

**Figure 4. Placing the cradle or holder in the transmission compartment of the FTIR instrument. The cradle (top) or the filter holder (bottom) should be inserted fully into the sample bracket in the instrument so that the bottom of the cradle or filter holder is tightly in contact with the sample bracket.**

## Importance of Positioning the Sample

The direct-on-filter analysis used for this application entails the use of *transmission* FTIR—i.e., the infrared beam needs to be free to pass through the center of the dust sample and the filter media and then reach the detector on the other side of the sample. Only a portion of the sample is evaluated directly (the center), and it is crucial that the same area of each sample is evaluated, since this is the area on which quantification models are based, enabling the quantity of RCS on the entire filter to be determined from the analysis of just a portion of the filter. As the infrared (IR) beam is invisible to naked eye, the correct setup of the sample (i.e. the cassette or capsule is correctly placed within the cradle or sample holder, which is in turn correctly placed in the bracket) ensures the proper conditions for the analysis.

When positioned correctly, the sample cassette (or capsule), the cradle (or filter holder), and the bracket work together to ensure that the sample is perpendicular:

1. to the transmission compartment (this relationship can be easily and quickly confirmed visually), and
2. to the IR beam (confirming this relationship visually is more complex and beyond the scope of this document).

These two criteria are essential to ensuring that the transmission beam is unimpeded and analysis of the sample is accurate. If the cassette or holder is tilted, the IR beam may be partially or fully blocked and may not reach the detector on the other side, or it may encounter a different portion of the filter instead of the center. Depending on the severity of the misalignment of the sample relative to the IR beam, the data may be incorrect or result in an invalid or failed sample. If the instrument “rejects” multiple sample scans (which indicates that not enough signal is reaching the detector), it is useful to check the alignment and positioning of the sample—for example by removing and reinserting the sample if there is any uncertainty.

The correct use of the cradle and bracket within the transmission compartment ensures two additional important requirements:

1. that the vertical plane of the filter will perpendicularly intersect the IR beam at its focal point, and
2. that the IR beam will be horizontally and laterally centered on the filter and dust deposit. The IR beam in each portable FTIR has a focal point in the transmission compartment, and the sample should be positioned at this focal point (see Figure 5) using the bracket. Modifications to this arrangement can have a negative impact on the quality of results. Note that in some instrument models, the bracket is easily moved, but in other models, the bracket is secured with a nut and bolt or a screw. Consult the instrument documentation to ensure the bracket is placed in the appropriate spot.



**Figure 5. Illustration of the relative position of the IR beam and sample cassette. When the sample cassette is correctly positioned in the instrument (within the removable cradle or filter holder), the IR beam will pass directly through the center of the sample at the focal point.**

Importantly, the dust sample needs to be analyzed in the center of the filter because the quantification performed by FAST of the RCS present on the entire filter is only possible by an accurate analysis of the center of the filter combined with a knowledge of the deposition of respirable dust and specifically RCS on the filter media.

## NIOSH-developed Accessories for FTIR Instrumentation

At the focal point, the infrared beam in the four portable FTIR instruments evaluated by NIOSH is fixed and ranges from 6 to 9 mm [Ashley et al. 2020], each of which is larger than the inlet openings commonly found in the traditional three-piece sampling cassette. The material of the cassette absorbs much of the IR beam, leaving insufficient signal for an accurate analysis. Additionally, the IR beam cannot penetrate the cellulose backing pad typically used to support the polyvinyl chloride (PVC) filter in the cassette, inhibiting the analysis of the sample. To address these issues, the PVC filter can be removed from the cassette with tweezers and inserted in a flat holder; however, this procedure requires additional handling which may induce sample losses. For these reasons, adjustments are needed to the cassette that is used to collect the respirable dust sample prior to the analysis, as described below.

NIOSH has designed specific cradles and brackets as accessories for a variety of holders, cassettes, and different FTIR units. Files of 3D models for 3D printing or traditional machining are freely available at <https://3dprint.nih.gov/discover/3dpx-013209>. These designs ensure the correct positioning of the sample inside a portable FTIR unit. These designs are not the only configurations possible, and other options can be created as long as the constraints described in the above section (that is, the correct vertical and horizontal positioning of the sample filter in the transmission compartment) are met.

Considerations for three types of samplers/cassettes are outlined below. Additional approaches may be explored in the future for available samplers and their related holders/cassettes.

### Four-piece Cassette

In order to minimize sample handling by keeping the filter in the cassette, NIOSH designed a four-piece cassette with top and bottom portions that can be removed to expose the filter to the IR beam while still containing the filter between two pieces of the cassette, and using a different support design to allow transmission FTIR analysis. The resulting cassette (see Figure 2) is compatible with a number of respirable samplers commonly used by industrial hygienists, many of which are named in the NIOSH 0600 [NIOSH 1998], NIOSH 7500 [NIOSH 2003b], NIOSH 7602 [NIOSH 2017a], NIOSH 7603 [NIOSH 2017b], and OSHA ID-142 [OSHA 2015] methods for respirable dust and RCS. The cassette was designed in collaboration with Zefon International via a Cooperative Research and Development Agreement (CRADA). The product that resulted from the collaboration is commercially available from Zefon International.

### Dust Sampling Cassette

The sampling cassette used in coal mines as part of the coal mine dust personal sampler unit uses a slightly different process than the four-piece cassette. The aluminum capsule that encloses the filter media and the aluminum ring that supports the filter media allow transmission of enough of the infrared beam to permit a reliable analysis of the sample. The capsule simply needs to be removed from the plastic cassette and inserted in a filter holder (illustrated in the photo on the bottom of Figure 3). Note that this sampling cassette is also available for compliance sampling and includes a check valve, but this version of the cassette is not compatible with direct-on-filter analysis.

While it is possible to use this sampling cassette for field-based monitoring, it is important to consider that the cassette was not designed for this use specifically, and other cassette options will likely provide more accurate results. This topic is discussed in Pampera et al. [2019]. Because of the potential for less accurate results, this type of cassette is most appropriate for relative determinations of RCS (e.g., higher-exposure potential areas or tasks vs. lower-exposure potential areas or tasks).

## Other Sampling Cassette Types

The four-piece cassette described above was designed specifically for use with field-based monitoring and is the preferred type of cassette for field-based monitoring. At the time this document was published, only calibration models for the four-piece cassette and dust sampling cassette (and respirable samplers that use these cassettes) were available in FAST. This section briefly describes other cassettes and samplers that are *physically* compatible with field-based analysis directly on the sample filter, but would require additional calibration models to be added to FAST to facilitate their use with field-based monitoring.

- A new respirable sampler has been designed and tested by NIOSH [Lee et al. 2017]. The sampler operates at 1.1 lpm and uses an innovative internal cassette with 25-mm PVC filter media. The cassette has been designed to allow direct-on-filter analysis of the dust sample with a portable FTIR. The performance of the sampler for respirable dust has been validated. The prototype sampler can be produced via 3D printing using the “Respirable size-selective sampler for end-of-shift quartz measurement” files available at <https://3dprint.nih.gov/discover/3dpx011762>.
- There is also an interest in direct-on-filter analysis of dust samples collected by real-time respirable dust monitors. The current analytical technologies used in real-time respirable dust monitors cannot distinguish RCS within respirable dust. Some units collect a dust sample on a filter after the dust sensor in order to protect the pump. The potential use of these collected samples is being explored by NIOSH. With the use of the proper filter media, the analysis of RCS is already possible (though RCS results are not available in real-time). NIOSH is continuing to explore ways to facilitate this additional implementation. Any change in the filter holder will need to be tested to confirm that the performance of the monitor (with respect to respirable dust) is not adversely impacted.
- Respirable dust samples can be collected with other cassettes that could be suitable to direct-on-filter analysis. The use of the Safety in Mines Personal Dust Sampler (SIMPEDS) [Harris and Maguire 1968; Stacey et al. 2014; Stacey et al. 2013] is common in countries outside the United States. Although this type of sampler is not currently recommended for field-based monitoring, the cassette may be compatible with direct-on-filter analysis, and the SIMPEDS sampler may be an option for field-based monitoring in the future.

## Generating Spectra

### Background Spectrum

Before any sample can be scanned or analyzed, every portable FTIR instrument requires analysis of a background spectrum. The FTIR analysis of a sample is a relative measurement—i.e., the interaction of the IR beam with a sample compared to the interaction of the IR beam with a reference. The background spectrum provides this reference. The general process used to generate a background spectrum is similar for each instrument, although each instrument and respective software has specific procedures. Consult the instrument software documentation (or Help file) for more information.

For the application described in this document, the sample consists of respirable dust on a filter, and the background spectrum is a clean filter (containing no respirable dust) of the same type. By using a clean filter for the background spectrum, the contribution of the filter media to the subsequent analysis of a sample (on similar filter media) will be minimized, resulting in a sample spectrum that is a function primarily of the respirable dust. This can be accomplished by one of two methods: analyzing a spectrum (with an empty transmission compartment used as the background spectrum) *for each filter* before any dust is collected and subtracting that from the sample spectrum later, or using a designated clean filter as the background spectrum for an entire set of filters. Both methods will ultimately produce results of approximately the same quality (see Appendix C). The designated clean filter method is preferred since it is much less cumbersome and much more efficient for the user, although normal differences in filter thickness mean that subtraction will reduce but not always eliminate the contribution of the filter.

If the clean filter method is used for the background spectrum, the following considerations apply:

- The clean filter should be the same type of media used for the collection of the respirable dust samples. In the United States, this is generally a 37-mm, 5- $\mu$ m pore size PVC filter, as indicated in the NIOSH methods 0600, 7500, 7602, 7603 [NIOSH 1998, 2003b, 2017a, 2017b].
- The clean filter used for the background spectrum can be selected from the blank filters in the set of cassettes used for sampling and should be from the same manufacturer lot or batch as the filters used for sampling. A good practice is to set aside a number of blanks equivalent to 10% of the sampling set. An additional sample can be set aside as a clean filter for the background spectrum. For example, if a health and safety professional is planning to collect 20 respirable dust samples one day, two cassettes should be set aside as blanks; an additional sample should be set aside as a clean filter to be used for the background spectrum. (20 samples+2 blanks+1 clean filter for the background spectrum).
- The subtraction procedure described below is *not* required when a clean filter is used for the background spectrum.

If the subtraction method is used, the following general procedure should be used:

1. Prior to sample collection, all filters to be used for sampling should be analyzed with the portable FTIR instrument, using an empty transmission compartment for the background spectrum. Data should be saved with filenames that correspond to the sample ID and

indicate that the spectrum represents a clean filter, before sample collection (e.g. sample1\_clean).

2. Following sample collection, all filters should be analyzed with the portable FTIR instrument, using the empty transmission compartment for the background spectrum. Data should be saved with filenames that correspond to the sample ID and indicate that the spectrum represents a sample filter that now contains respirable dust (e.g. sample1\_dust).
3. Using the instrument software, subtract the “clean filter” spectrum for the “sample filter” spectrum for each sample ID. Consult the software documentation for guidance on how to use the software to subtract spectra. Be sure to save the new spectrum files resulting from the subtraction procedure. It is recommended that the filename indicates that the spectrum is the result of subtraction (e.g. sample1\_subtracted).
4. All data processing to calculate RCS should use the subtracted spectrum files. The “sample filter” spectrum using an empty transmission compartment for the background measurement will *not* provide a reliable quantification of RCS.

Regardless of which approach to the background spectrum is taken, it is crucial that the filter used as a background is clean (i.e., does not contain any dust and has not been touched) and is as similar to the sample filters as possible. For instance, a filter that is perceptibly thicker or thinner than other filters should not be used as the background because this could impact the quality of sample results, especially for small quantities of RCS.

## Quality Assurance (QA) Sample Spectra

It is important to ensure that the instrument performance remains consistent over time. One element of regular performance checks should be the analysis of a set of four to seven quality assurance, or QA, samples, which remain with the instrument and are each analyzed every time the instrument is used. The calculated value of RCS should remain consistent over time for the set of QA samples, indicating that the instrument performance is accurate; however, note that these samples are not used to calibrate the instrument, as this is generally not necessary. See *Creating a Set of Samples to be Used as Quality Assurance Samples* for guidance on establishing a set of QA samples.

To track the QA sample results over time, the QA Sample Tracking spreadsheet (see Appendix A for a complete list of resources for field-based monitoring) should be used together with this document. The spreadsheet can accommodate up to ten QA samples. If multiple sets of QA samples will be used (for example, for multiple instruments), it is recommended that a unique spreadsheet is used for each set of QA samples.

To use the spreadsheet, the column headers in the “Input QA Sample Data” tab should be named according to the sample IDs of the QA samples. Each time the QA samples are analyzed, the results should be entered. The data should be typed directly into each cell rather than copied and pasted, as this can interrupt some of the formatting that makes the spreadsheet function. The spreadsheet calculates basic summary statistics for the data over time; this allows the spreadsheet to flag any data point that is a statistical outlier. Approximately 10–20 data points are needed before this function becomes a reliable indicator of true outliers; when there are only a few data points, the spreadsheet may flag a data point that is only slightly different from the others. This

process can be accelerated by analyzing QA samples several times per day (e.g., in the morning and again in the afternoon) for the first several days.

Each time the instrument is used (or daily, when the instrument is used multiple times throughout the day), the QA samples should be analyzed and the data entered into the spreadsheet. Any of the QA samples that are outliers will be highlighted automatically in the spreadsheet (unless there are still too few data points for a reliable indication). The incidence of one outlier (while all other QA samples are within normal variation) indicates that the problem is likely unique to that particular sample and is therefore not likely to be indicative of a problem with the instrument itself. Often this is a matter of a simple error during the analysis—e.g., the QA sample was not inserted properly in the cradle (or filter holder) or the cradle was not properly inserted in the bracket, the wrong QA sample was analyzed (or the spectrum was labeled incorrectly), or the result was incorrectly entered in the spreadsheet (e.g. transposition of digits). It is also possible that the QA sample was physically damaged prior to analysis, changing the result. Note that over time, some QA samples may begin to lose material due to repeated use; QA samples should be discarded when there is evidence of lost material. When one QA sample is an outlier, the recommended action is to re-analyze the QA sample. If the result is within the normal range for that sample (as indicated by previously saved results in the QA Sampling Tracking Spreadsheet), then sample analysis should proceed as usual. If the result is still outside of the normal range, the QA sample was likely damaged and should be discarded. The spreadsheet should be updated accordingly and then sample analysis can proceed as usual.

The incidence of multiple outliers indicates a potential problem with the instrument. This could be due to either hardware or software changes. It is important not to ignore irregular QA sample data, because the problem will likely also impact the accuracy of subsequent sample data. Irregularities should be noted in a logbook. A number of issues may occur that result in irregular sample data, and a number of steps may help to identify the cause of the issues, including:

- Check that all data points are entered correctly, that samples have been labeled correctly, and that no values have been transposed.
- Make sure that the background was properly scanned; this will typically require scanning the clean filter again to produce a new background, paying particular attention to the positioning of the clean filter in the transmission compartment. QA samples should be reanalyzed after scanning the new background.
- Check the software analysis parameters to ensure that all parameters are correct (see the Setting Instrument Analysis Parameters section).
- Complete the applicable performance verification/diagnostic tests to identify components that may be beyond their service life or otherwise need to be replaced.
- Examine the instrument to observe any physical abnormalities that may be present (i.e., cracked, cloudy, or smudged windows and mirrors); if faulty components are identified, replace them if possible. If faulty components are identified but cannot be immediately replaced, contact the manufacturer for service recommendations.
- Replace the internal desiccant if there is reason to believe that humidity could be a factor (note that the desiccant is not a user-serviceable part on all instrument types). While excess humidity can affect the sample spectrum, it is mainly a concern due to the

potential for condensation on internal instrument components, which the desiccant mitigates.

- Feel the outer casing of the instrument and note if it is warmer than usual. If the instrument seems particularly warm, turn the instrument off and allow it to come to room temperature before turning it on again.

If the source of the problem cannot be identified and resolved using the steps above, it may be necessary to communicate directly with the manufacturer of the instrument for more advanced diagnostic tools or to return the instrument to the manufacturer for service.

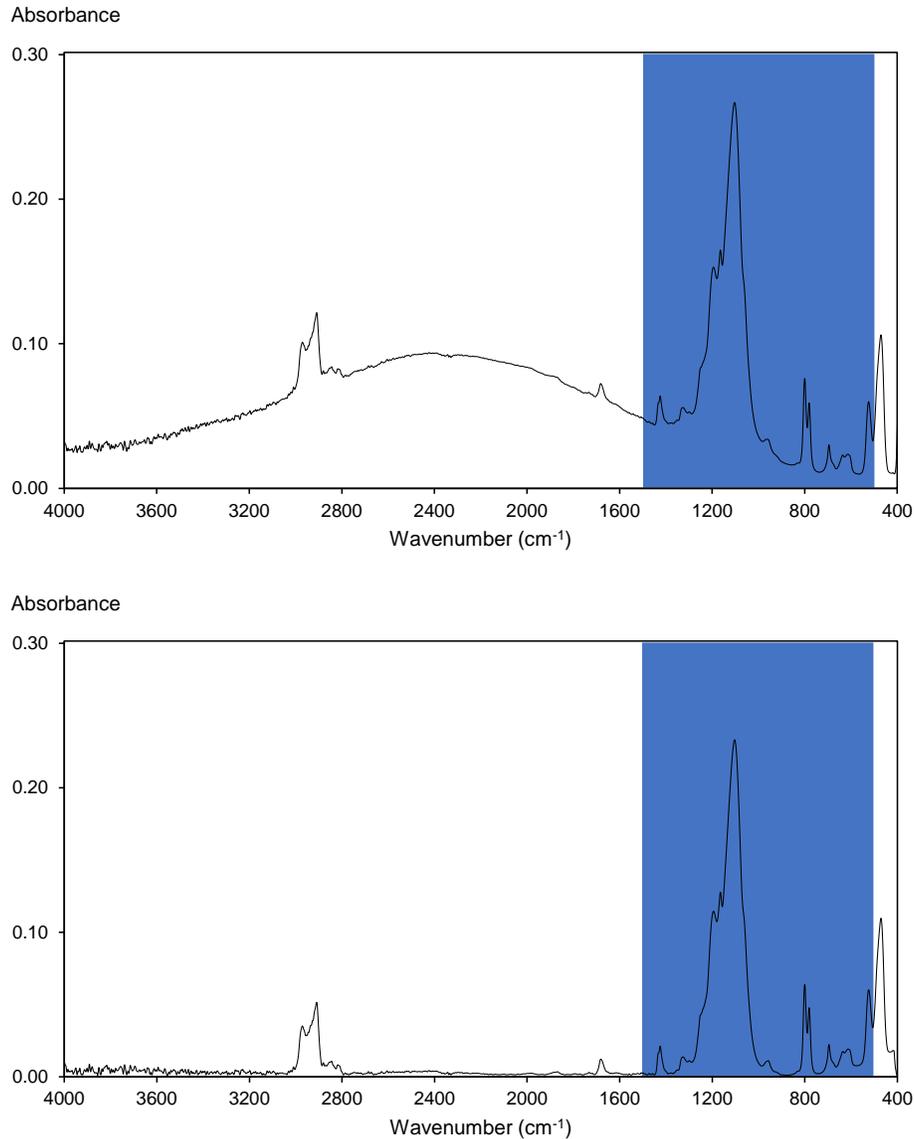
## Sample Spectra

After the background spectrum has been generated and the instrument performance has been verified by the QA sample analysis, sample spectra may be analyzed. Sample spectra should be given unique identifiers that can be easily linked back to the identifiers on the physical samples. The following section describes how sample data are processed and then interpreted to provide meaningful results.

## Processing Sample Data

After the spectrum of a sample is generated, the spectrum must also be processed by two procedures: (1) a baseline correction, and (2) integration of areas of interest for crystalline silica (quartz) and other minerals that might have a confounding effect in the quantification of RCS in the sample. NIOSH has designed macros for the four FTIR instruments evaluated to facilitate the processing of the analyzed samples. Each macro serves as a combination procedure that performs the baseline correction, integrates the portions of the spectrum used for identification and quantification, and reports values for these ranges of the spectrum. These macros are available for users to download (<https://github.com/niosh-mining/fast/>) and import into their version of the software, and they will be updated as areas are added and/or modified based on ongoing research. To properly load macro and template files, see the FTIR software documentation.

The baseline correction adjusts the spectrum to remove distortions such as slope or curvature in the spectrum, as shown in Figure 6. While it is important to perform a baseline correction in general, different software programs perform the correction differently. In some programs, only a generic “baseline correction” option may be available. In other cases, there may be multiple baseline correction options, often including an “auto baseline correction”. The most important practice is to use the same option consistently for both QA samples and other samples; see Appendix C for a comparison of several common options. In general, baseline correction procedures can typically be accessed through the *manipulate* or *transform* menu; search or use the Help file for the FTIR software if needed. Baseline correction should be performed over the full range of the spectrum (e.g. 4000–400 cm<sup>-1</sup>). The NIOSH macros for the Bruker, Perkin Elmer, and Thermo instruments include a baseline correction; spectra collected using the Agilent software should be baseline corrected manually.



**Figure 6. Illustration of a spectrum baseline correction. The two spectra show the same sample before a baseline correction is performed (on the top) and after a baseline correction is performed (on the bottom).**

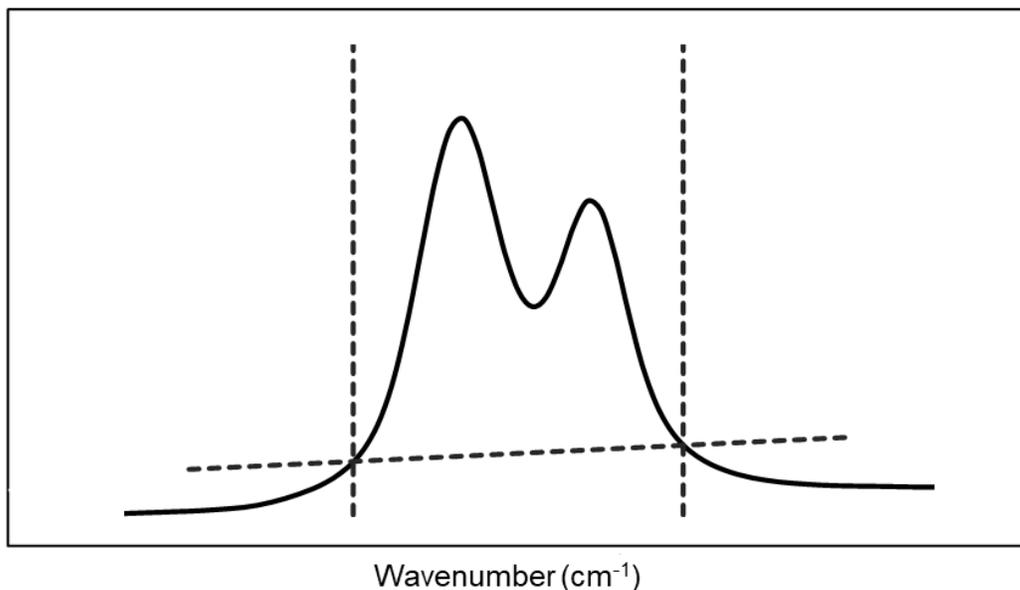
The FAST software analyzes multiple wavelength ranges of a sample spectrum when calculating RCS mass and concentration. The ranges each correspond to a specific mineral or group of minerals, and the ranges that are used depend on information about the sample that the user enters in FAST (such as commodity).

The range corresponding to quartz (the Q value in FAST), which is sometimes referred to as the doublet area, is always used. Even if no other information about the sample is known or provided in FAST, an estimated mass and concentration for RCS can be determined from the result of integrating this area, as described below. While the other ranges used by FAST are not shown here, they are defined in a similar way, between a left and a right endpoint and a straight line at the base of the peak. Over time, as more is learned about the minerals that may be found in

respirable dust and how they might impact the accuracy of field-based RCS quantification, more ranges for analysis may be added to FAST, or the existing ranges may be modified to improve overall accuracy.

The lower boundary of each area is determined by drawing a straight line from the intersection of the spectrum with the left response boundary to the intersection of the spectrum with the right response boundary (see Figure 7).

Absorbance



**Figure 7. Illustration of the quartz doublet. The area between the spectrum and the lower boundary (defined by the left and right boundaries) is used to quantify RCS (quartz).**

## Interpreting Sample Data Using FAST

After sample spectra have been analyzed and processed in the FTIR software, the results must be exported to a spreadsheet, which can then be imported into FAST, which is available on the NIOSH Mining Program webpage at:

<https://www.cdc.gov/niosh/mining/works/cover sheet2056.html/>. General instructions are included below for how to export sample results from the software programs that accompany four FTIR instruments, as well as a generic format that will work regardless of the instrument used. This information is also included in the FAST software Help file.

For macro files, template files, and their descriptions, see <https://github.com/niosh-mining/fast/>. To properly load macro and template files, see the FTIR software documentation.

## Exporting Results from Agilent

### *In RESOLUTIONS PRO*

1. Open a new Multi-Spectral Document by selecting **File >> New >> Multi-Spectral Document >> OK**
2. Open all spectra files to be included in the report by selecting **Open >> (select files) >> Open**
3. Add each sample spectrum into the Multi-Spectral Document by dragging or copying the file:
  - a. To drag:
    - i. Click the + next to the sample name to expand.
    - ii. Click on the spectrum entry and drag it to the Multi-Spectral Document.
    - iii. Check the Multi-Spectral Document to confirm that the sample file was successfully transferred or copied.
  - b. To copy:
    - i. Click the + next to the sample name to expand.
    - ii. In the data grid in the lower portion of the screen, highlight Row 1. iii. Right-click and select **Copy** (or use **Ctrl+C**).
    - iv. Click on the + next to the Multi-Spectral Document to expand.
    - iii. In the data grid in the lower portion of the screen, place the cursor in the first empty row.
    - iv. Right-click and select **Paste** (or use **Ctrl+V**).
4. When all samples have been added to the Multi-Spectral Document, use the Ctrl key to highlight each row of the grid.
5. Select **Analysis >> Peak Options >> New Peak Template (all spectra)**.
6. In the **Creation Choices** field (top), select **Create a set from file**.
7. Select **Browse** and choose the **Peak-area.def** file. Select **Open >> OK**.
8. Use the Ctrl key to select the columns for Name, K, Q, C, D, and M.
9. Right-click and select **Copy** (or use **Ctrl+C**).

### *In EXCEL*

1. Place the cursor in cell A1; right-click and select **Paste** (or use **Ctrl+V**).
2. Save the file; the resulting file (to be imported into FAST) should have the following format, as shown in Figure 8.

	A	B	C	D	E	F
1	Name	K	Q	C	D	M
2	sample1_0000(1)	0.034	0.704	-0.043	-0.036	-0.03
3	sample2_0000(1)	0.019	0.145	-0.006	-0.006	-0.02
4	sample3_0000(1)	0.015	1.722	-0.015	-0.012	-0.017

**Figure 8. Results format from the Resolutions Pro software (Agilent). The results report exported from Resolutions Pro displays data for each sample in a row format. The Sample ID should appear in Column A, followed by the result areas beginning in Column B.**

## Exporting Results from Bruker

### *In OPUS*

1. Select **Print >> Generate Report**.
2. On the **Select Files** tab of the menu that appears, select **Multi sample report**, and load the template file **silica\_report.art**.
3. Select **Load** and navigate to the **Processed\_Spectra** folder (or the location where processed files have been saved), then select the sample files to be included in the report.
4. Select the **Output** tab; on the menu that appears, check that the **csv file** option is selected, then specify a name for the report file.
5. Specify a file path for the saved report, then select **Generate**.

### *In EXCEL*

1. Open the saved report spreadsheet and highlight Column A.
2. Select the **Data >> Text to Columns**.
3. Select **Delimited** and click **Next**; select **Semicolon** and click **Finish**.
4. Save the file; the resulting report (to be imported in FAST) should have the following format, as shown in Figure 9.

	A	B	C	D	E
1	No	Spectrum	Result	Freq.1	Freq.2
2	1	sample1	0.704	816	767
3			0.034	930	900
4			-0.03	740	720
5			-0.036	743	718
6			-0.043	890	865
7	2	sample2	0.145	816	767
8			0.019	930	900
9			-0.02	740	720
10			-0.006	743	718
11			-0.006	890	865
12	3	sample3	1.722	816	767
13			0.015	930	900
14			-0.017	740	720
15			-0.012	743	718
16			-0.015	890	865

**Figure 9. Results format from the Opus software (Bruker). The results report exported from Opus displays data for each sample in a column format rather than a row format. Column B must contain the sample ID, and column C must contain the result areas for samples.**

## Exporting Results from Perkin Elmer

### *In SPECTRUM 10*

1. Open all samples files to be included in the report.
2. Select **Process >> Macro >>Silica est.**
3. With the **Results Table** open, select **File >> Send To >> Excel.**

### *In EXCEL*

1. To simplify the spreadsheet, keep the **Sample Name, Q, K, M, D,** and **C** columns (Columns A, E, F, G, H, I) for the processed files; all other cells can be deleted.

2. Save the file; the resulting report (to be imported in FAST) should have the following format, as shown in Figure 10.

	A	B	C	D	E	F
1	<b>Sample Name</b>	<b>q</b>	<b>k</b>	<b>m</b>	<b>d</b>	<b>c</b>
2	sample1.SPA_2	0.704	0.034	-0.03	-0.036	-0.043
3	sample2.SPA_2	0.145	0.019	-0.02	-0.006	-0.006
4	sample3.SPA_2	1.722	0.015	-0.017	-0.012	-0.015

**Figure 10. Results format from the Spectrum software (PerkinElmer). The results report exported from Spectrum displays data for each sample in a row format. The Sample ID should appear in Column A, followed by the result areas beginning in Column B.**

## Exporting Results from Thermo Fisher

### *In TQ ANALYST EZ EDITION*

1. Open the **TQ Analyst EZ Edition** program.
2. From the **File** menu, select **Open Method...**, then select the **Silica\_int.qnt** method and click **Open**.
3. From the **Diagnostics** menu, select **Multiple Summary – Select Files...** Select the sample files to be included in the report. TQ Analyst EZ Edition will generate and open a spreadsheet containing the results.

### *In EXCEL*

1. Depending on the version/settings of TQ Analyst EZ Edition, the format of the results spreadsheet may differ slightly; delete columns as needed so that the format of the spreadsheet is as shown below.
2. Save the file; the resulting report (to be imported in FAST) should have the following format, as shown in Figure 11.

	A	B	C	D	E	F	G
1	Tue Mar 07 12:10:21 2017 (GMT-05:00)						
2	Method filename: C:\mydocuments\omnic\Quant\Silica_int.qnt						
3	Method Revision 4 Last Saved Date: Tue Feb 28 12:33:02 2017 (GMT -05:00)						
4	Method title: Silica - Integration						
5							
6	Spectrum	Spectrum	q	k	m	d	c
7							
8	Sample de	C:\...sample1	0.704	0.034	-0.03	-0.036	-0.043
9	Sample de	C:\...sample2	0.145	0.019	-0.02	-0.006	-0.006
10	Sample de	C:\...sample3	1.722	0.015	-0.017	-0.012	-0.015

**Figure 11. Results format from the Omnic software (Thermo Scientific). The results report exported from Omnic/TQ Analyst displays data for each sample in a row format, but also includes a header. Sample data should begin in Row 8; Sample ID should appear in Column B, followed by result areas beginning in Column C.**

### Exporting Results in a Generic Format

The generic format for exporting sample results should resemble Figure 8 or Figure 10.

Notable items about the generic import format are as follows:

- Each row (after the header row) describes one sample.
- Each sample is defined by 6 values:
  - the sample name;
  - the Q, K, M, D, and C values.

### Importing Results to FAST

1. Open FAST and select **Data >> Import Sample Data...**
2. On the **Select Method** screen, select **Import from file**.
3. Beside the **Select File** field, use the **Browse** button to select the report file (exported from the instrument software) for the sample dataset.
4. In the **Select Importer** field, choose the appropriate instrument option; if using an instrument that is not listed, select the **Generic** option. When the **Generic** option is selected, columns must be mapped so that FAST can identify each component of the dataset. Note that for any instrument, an option exists to indicate a .csv file or an .xlsx file. The option selected should match the format in which the report file was saved.

5. Select **Import**. FAST will indicate the number of samples found in the report. Confirm that this number is correct, then select **Next** (at the bottom).
6. On the **Create Sample Event** screen, enter the following information, which will apply to each sample in the report file:
  - a. In the **Event Name** field (*optional*), give a short name to the sample set.
  - b. In the **Sample Date** field (*required*), enter the date that the sample set was collected.
  - c. In the **Sample Analysis Date** field (*required*), enter the date that the sample was analyzed by the portable method.
  - d. In the **Location** field (*optional*), enter a general description of where the sample set was collected geographically (e.g., Tucson, or New Mexico, or the U.S. Southwest). To add a new location to the dropdown list, select **Manage Locations**, click **Add**, and type the name of the location to be added.
  - e. Beside the **Use Correction Factor** field (*optional*), check the box if you would like to use the current correction factor. Note that correction factor is tied to location, and the correction factor must be calculated using the correction factor tool within FAST. If a correction factor has not been previously calculated for a particular location, the **Use Correction Factor** option will not appear. A correction factor should only be applied to samples of the same commodity type.
  - f. In the **Commodity** field (*required*), select either **Coal** or **Other** according to the type of mine where samples were collected. This information is (for **Coal**) or will be (for **Other**) used to make mathematical adjustments to the data based on anticipated mineralogy.
  - g. In the **Instrument** field (*optional*), select the type of FTIR instrument being used. To add a new instrument to the dropdown list, select **Manage Instruments**, click **Add**, and type the name of the instrument to be added.
  - h. In the **Tags** field (*optional*), enter any descriptive words or phrases that you want to associate with this entire data set; use the Tab key to separate multiple tags. Examples of tags could be: July, longwall move, new dust suppression, etc. These tags will apply to the entire sample event.
7. Select **Next**. On the **Enter Sample Data** screen, do *not* edit the data in the M, D, C, Q, or K fields; this is the FTIR data that has been exported for the sample, and changing it will change the estimated mass and concentration of RCS for the sample. Enter the following information, which will apply only to the sample indicated in the **Sample ID** field:
  - a. In the **Location** field (*optional*), enter the specific location where the sample was collected (e.g., primary crusher, 5 belt, mill, etc.).
  - b. In the **Worker** field (*optional*), enter the worker with whom the sample is associated. This does not necessarily need to be the worker's full name—first name only, initials, job title, or a numeric identifier are all accepted formats. This field can be left blank if the sample is not associated with a specific worker or if you do not wish to identify the worker.

- c. In the **Sampling Duration** field (*required*), enter the sampling time in minutes (e.g., 100).

Alternatively, select the box beside **Use Sampling Times** to indicate the start and stop times for the sample; FAST will calculate the sampling duration from the times entered.

- d. In the **Sampler Type** field (*required*), select the type of sampler used. This information is used in calculations. Selecting the incorrect sampler type may result in less accurate determination of RCS.
- e. In the **Filter Size** (mm) field (*required*), select the size of the filter used to collect the sample. This is typically 37 mm.
- f. In the **Respirable Dust Mass** (mg) field (*optional*), enter the gravimetric mass of the sample, if known. You can add this information at a later time if you do not have the data yet but will have it in the future.
- g. In the **Laboratory Silica** ( $\mu\text{g}$ ) field (*optional*), enter the RCS (quartz) mass reported by an accredited laboratory using the standard method, if known. You can add this information at a later time if you do not have the data yet but will have it in the future.
- h. In the **Average Flow Rate** (lpm) field (*required*), enter the pump flow rate in liters per minute. The entered flow rate should match the nominal flow rate for the selected sampler type ( $\pm 5\%$ )  
  
Alternatively, select the box beside **Use Pre & Post Flow Rate** to indicate the pre-sampling (calibrated) flow rate and the post-sampling flow rate; FAST will calculate the average flow rate from these two values.
- i. In the **Spectrum File** field (*optional*), choose the raw spectrum file associated with this file.
- j. In the **Tags** field (*optional*), enter any descriptive words or phrases that you want to associate with this specific sample; use the Tab key to separate multiple tags.

Examples of tags could be: wearing respirator; control ON; follow-up sample, etc. These tags will apply to a specific sample.

8. Use the arrows to navigate to the first/previous/next/last sample. Some fields (such as sampler type and filter size) are “sticky” and will not change from one sample to the next unless you opt to change them. FAST assumes that these fields remain the same within a specific sampling event, but you can change any of these fields for a particular sample if appropriate.

Alternatively, switch to **Grid View** (from the default **Sample View**) to quickly enter data for multiple samples. *Tip:* before switching to Grid View, indicate the sampler type and filter size for the first sample.

When all required fields have been completed, select **Finish**. Newly entered sample data can then be reviewed in either the **Events** or **Samples** tab.

## Supporting Field-based Analysis for Respirable Crystalline Silica

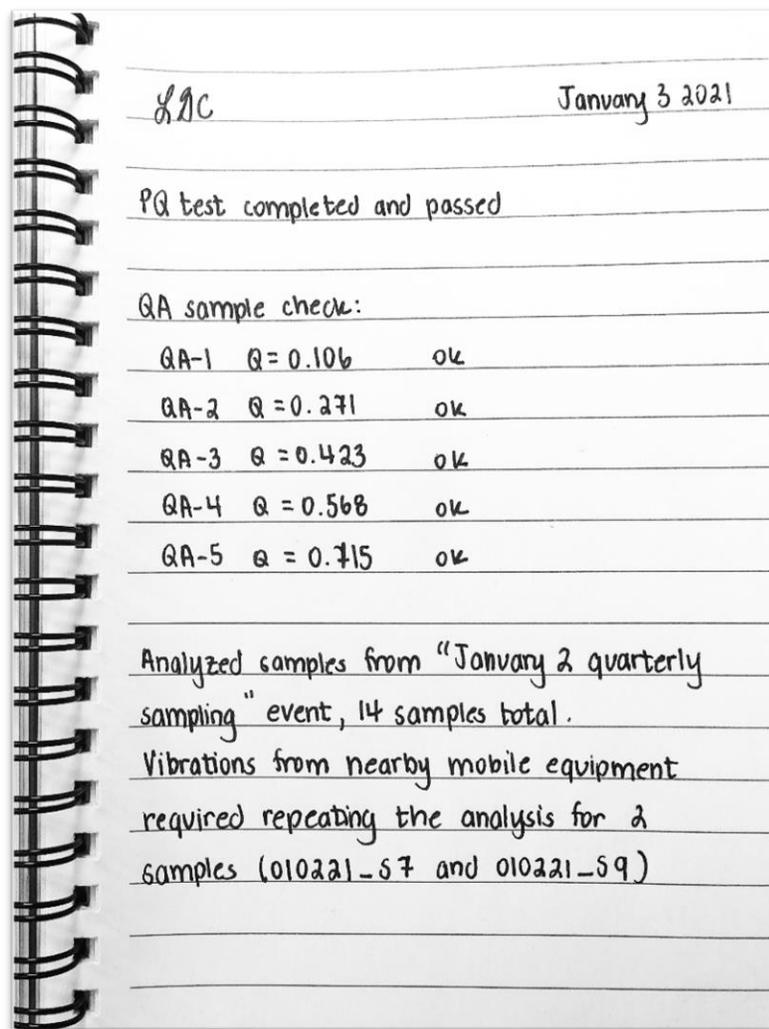
### Keeping a Logbook

It is important to keep detailed and accurate records of the analyses that have been completed as well as notes indicating quality assurance measures. This is particularly helpful in situations where multiple people are using the instrument, or the instrument is used infrequently by the same person.

To maintain an accurate record of what has been done, a logbook entry should be recorded *every time that the instrument is used*. Suggested information to include is as follows:

- Date
- Name or initials of the analyst
- Whether instrument checks were performed
- Result of instrument checks (if performed)
- Which QA samples were analyzed
- Results of QA sample checks
- Any anomalies related to the QA sample check or the QA samples themselves
- Other anomalies during analysis
- Details identifying the sample set
- Location of saved sample report

A typical logbook entry might look like the example shown in Figure 12.



**Figure 12. An example of a suggested logbook entry. This entry shows the details related to analysis of QA samples, the samples analyzed, the location of the sample report, and notes on environmental conditions. The entry also includes the date and the initials of the instrument operator.**

### Organizing Spectrum Files to Support Field-based Monitoring

When generating sample spectra and naming spectrum files, it is important to give each file a unique name, since that name will be used to generate a report of the results from the FTIR software and will then be imported into FAST. For instance, the unique sample ID associated with the sample filter/cassette could be used, or the sample date plus the sample number (e.g., 2019-05-05\_22).

In some cases, such as for the analysis of QA samples, it may make more sense to save data within distinct folders, organized by date. This allows samples with the same name (e.g., "QA Sample 12") to be saved unambiguously.

Note that the default method of naming sample files may vary for the different FTIR software packages. Therefore, checking to ensure that each spectrum file is saved with a unique filename is also recommended.

## Creating a Set of Samples to be Used as Quality Assurance Samples

In the Quality Assurance (QA) Sample Spectra subsection of the Generating Spectra section, the purpose of analyzing and tracking a set of QA samples over time is discussed. This set of QA samples is unique to a particular instrument and can be created by the user in the field.

Commercially available samples of quartz on filter media (such as standard reference materials or similar products) may also be used as QA samples. Since the quartz mass for this kind of sample is known with a high level of confidence (which is not a requirement for QA samples), they may be expensive.

To create a set of QA samples, four to seven respirable dust samples should be collected in the workplace. Each QA sample should contain a different quantity of respirable dust and a different quantity of RCS. It may be necessary to collect a larger set of samples (ten to twenty); after analyzing these samples with the FTIR instrument, the FAST software should be used to determine the relative amount of RCS in each sample, and a subset of four to seven samples should be selected so that the set as a whole satisfies the following criteria:

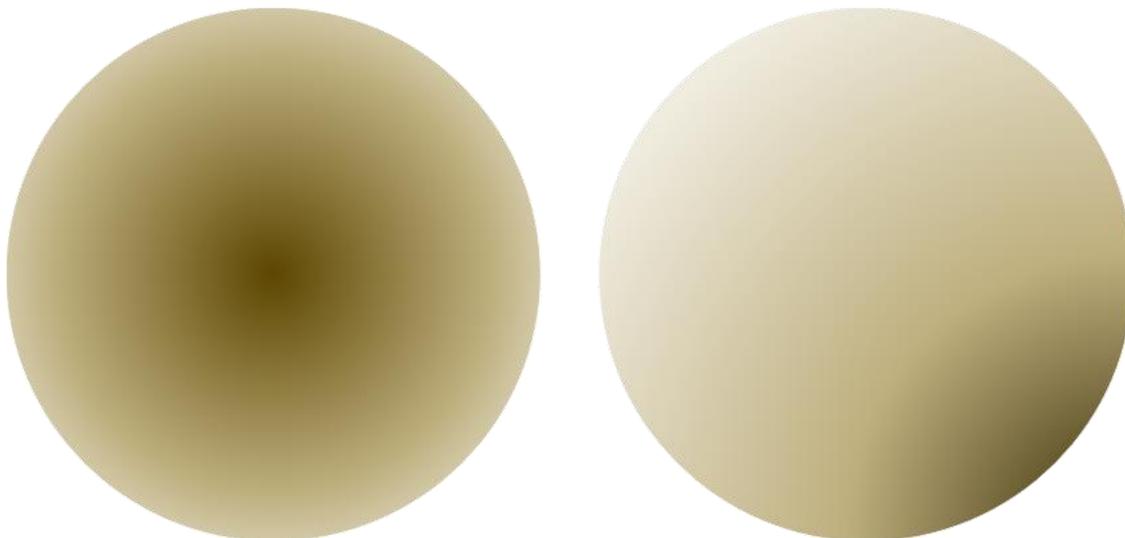
- The QA sample with the lowest quantity of RCS should contain approximately 20–40  $\mu\text{g}$  of RCS.
- The QA sample with the highest quantity of RCS should contain approximately 120–150  $\mu\text{g}$  RCS.
- The remaining QA samples should contain RCS quantities that are (approximately) evenly spread between 20 and 150  $\mu\text{g}$ .

Other considerations for selecting samples to be used as QA samples are the following:

- If the respirable dust is visible on the sample filter, the deposition should appear radially symmetric. It is not uncommon to see more material at the center of the filter (which may cause the center to appear darker than the rest of the filter). This is sampler-dependent and does not present an issue for the reliability of the QA sample analysis, as long as the deposition is symmetric (see Figure 13).

If the filter is heavily loaded with respirable dust, there is a higher probability that some material may come loose from the filter over time. When selecting QA samples, it is not recommended to select samples with more than approximately 5 mg of respirable material or samples where the deposit of respirable dust looks very thick or “cakey.” If there is visible dust loss from the QA sample filter (e.g. after repeated use), that sample should no longer be used.

If the respirable dust in the specific environment is known to have a fairly low RCS content (<5%) that requires more heavily loaded samples to reach 120–150  $\mu\text{g}$  of RCS, it is recommended to create a slightly larger set of QA samples (at least 6–8 samples). Over time, as the QA samples are used repeatedly, some may begin to lose material; a larger sample set will allow damaged samples to be discarded without the set of QA samples becoming too small.



**Figure 13. Illustration of the deposition of material in respirable dust samples. Many samplers result in a visible concentration of material at the center of the filter compared to the edge of the filter. This is not a problem, provided that the deposit is centered on the filter (left) rather than skewed towards the edge of the filter (right).**

### Generating a Site-specific Correction Factor

The quality of the quantification of RCS in respirable dust samples using the portable direct-on-filter FTIR approach can be affected by the presence of other minerals in the samples [Foster and Walker 1984; Ojima 2003; Pickard et al. 1985; Taylor et al. 1970]. Accredited laboratories have the experience and knowledge to address this issue during the FTIR analysis and the processing of the data as indicated by NIOSH method 7603 [NIOSH 2017b], which adopts the FTIR technique for RCS analysis. In addition, the NIOSH 7603 method provides guidance for sample preparation procedures that can reduce the presence of certain minerals.

Most users of the field-based method will likely not have the same level of experience as a laboratory infrared spectroscopy expert and, for this reason, quantification models have been developed to standardize the RCS quantification in the presence of confounding minerals. Specific quantification models have been designed for samples collected in coal mines.

For samples collected in any environment designated in the FAST software as “other” (which includes non-mining environments, FAST does not include tailored quantification models. Additional tailored quantification models may be added in future updates to FAST. Until that time, a different strategy—the “site-specific quantification model”—can be used to address the presence of mineral interferences. As the name suggests, the site-specific quantification model uses a correction factor that is unique to a particular site. A site can be one or several workplace environments (within the same operation) where it is reasonable to assume that the characteristics of the respirable dust (including percent quartz content) are similar. Qualitative information (such as the color of the respirable dust on the sample filters), quantitative information (such as the percent silica reported by the external laboratory for previous samples collected in an area), and operational information (such as knowledge of processes that use additives or chemical that could also be present in the dust sample) can be used to determine if two areas are similar enough to be considered the same site.

Ultimately, a site is determined at the user's discretion. For example, a small quarry operation, including the pit, the plant, and the lab, might be considered a single site if the respirable dust has a similar appearance and quartz content throughout. A large multi-pit metal mine might encompass several different sites (i.e. each pit, each processing area, and the lab) if respirable dust has different characteristics depending on the area of the operation. In certain situations, such as in industries including construction or manufacturing, correction factors that are unique to specific tasks could be used instead (e.g., a correction factor specific to cutting brick and a correction factor specific to breaking concrete). Publications describe the application of this method [Cauda et al. 2018; Hart et al. 2018].

Regardless of how big or small a site is, the correction factor for that site should be periodically evaluated via laboratory analysis. This evaluation should include samples from all areas of the facility to which the correction factor is applied; the ratio of the result of the field-based analysis to the results of the lab-based analysis should be approximately the same for all samples. For any samples where this ratio is different, a unique correction factor should be generated and used.

To generate a site-specific quantification model, the procedure outlined below should be followed.

1. Collect a set of at least fifteen to twenty samples. The sample set should contain a range of respirable dust loadings. The final sample set should contain at least ten samples, ranging from 20–30  $\mu\text{g}$  RCS per sample to 275–325  $\mu\text{g}$  RCS per sample, which may be determined using either the direct-on-filter approach or the standard analysis through an off-site laboratory. Ideally, sample loadings will be (approximately) evenly spread between 20 and 325  $\mu\text{g}$  of RCS. While a total respirable dust loading can be estimated from expected concentration and sampling time, it can be difficult to estimate the RCS loading accurately in this way, so additional samples are recommended. Gravimetric analysis (such as NIOSH 0600 [NIOSH 1998]), if available, may be helpful. These guidelines are summarized within the **Calculate Correction Factor** function of the FAST software.
2. Analyze the samples using the FTIR instrument and import the data into FAST. At this point, choose which samples will remain in the sample set and which will be excluded (i.e., if they are outside of the loading range; if they are duplicates or extras). When importing the data into FAST, be sure to indicate the location for which you plan to use the correction factor.
3. Submit the sample set to an off-site laboratory for the standard RCS analysis.
4. When the sample results are received from the laboratory, add each laboratory result to the appropriate sample in FAST by right clicking on a sample, selecting “edit,” and adding the result to the Laboratory Silica field.
5. To calculate the correction factor, select **Calculate Correction Factor** from the Data menu in FAST. Select the appropriate event location. FAST will display samples that may be used to calculate the correction factor; it will not display samples for which laboratory results are not available, or samples outside of the range of 20–325  $\mu\text{g}$  RCS. Click on samples to select them; at least 10 samples must be selected. Use the “Data Quality Evaluation Results” at the bottom of the screen (see Figure 14) to guide you as you select the samples to include; the correction factor generated with the currently

selected samples is shown below this section. When you have selected all samples you wish to select, click **Save Correction Factor**.

6. The saved correction factor can be selected in FAST during the **Create Sample Event** step; the correction factor is applicable to any future sample events occurring in the same location.
7. Once a correction factor is established, additional samples should periodically be sent to the off-site laboratory to verify the validity of the correction factor. This should also be done any time that changes in the occupational environment are perceived such that the existing correction factor may need to be adjusted. This may also be necessary in large operations, where an operator may decide to establish individual correction factors for specific areas of the operation. The frequency depends on how stable conditions are. For conditions that remain consistent over time, verification of the correction factor might occur once or twice a year. For conditions that are less stable (or where the stability of conditions is unknown), more frequent verification might be needed.

**Data Quality Evaluation Results**

	Target	Selected Data	
Samples (n)	>10	8	Dataset has too few samples - add data for additional samples
Min value	20-30	25	
Max value	275-325	225	Maximum value is too low - add data for additional sample(s) at higher sample loading(s)
Quartile 1	94	95.2	
Quartile 3	231	187.5	Q3 value is too low - add data for additional sample(s) at higher sample loading(s)

**Correction Factor = 0.79**

**Figure 14. Screenshot from the “Calculate Correction Factor” function of FAST. This tool enables RCS results calculated in FAST and RCS results from an off-site laboratory to be used to calculate a correction factor that can be applied to future sample events. The Data Quality Evaluation Results feature provides guidance for determining an appropriate correction factor.**

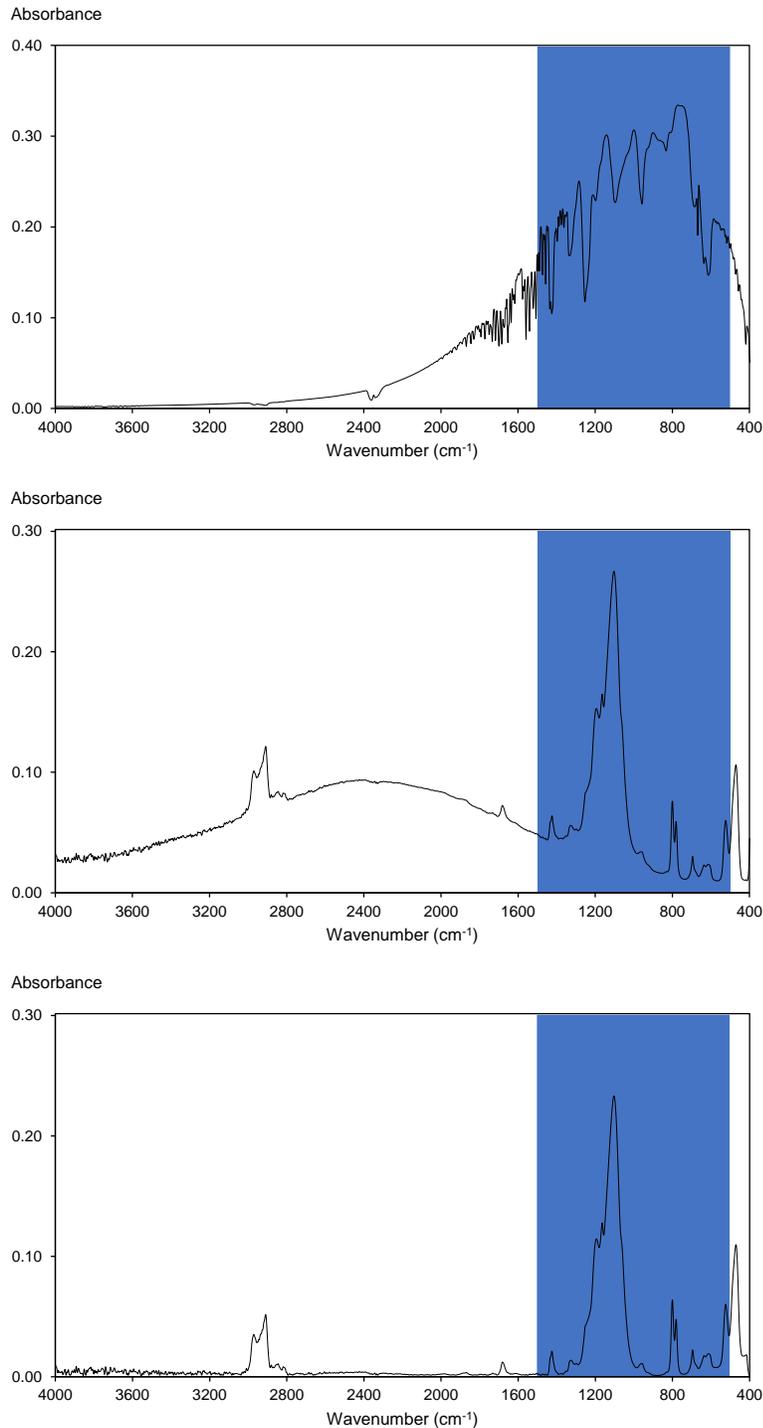
### Troubleshooting: What a Sample Spectrum Should (and Should Not) Look Like

The sample spectrum—the source of data used by FAST to calculate the amount of RCS in a sample—is actually the product of two spectra: the background spectrum (a clean filter) and the sample filter spectrum. The background spectrum is used to adjust the sample filter spectrum to reduce the impact of the filter material; therefore, what is ultimately used for RCS quantification is the primary representation of the respirable dust sample’s mineral composition. In order to obtain a reliable spectrum, it is important that both the background spectrum and sample spectrum are correct. A visual inspection can provide information on the quality of the spectrum: some software programs provide the option to display the background as it is scanned, or they provide the option to view the background component of a sample spectrum. Note that while some programs do enable this, they may not provide it as the default option; they may refer to it as the reference rather than the background; and they may not provide the background spectrum in the same units (generally absorbance or transmittance) as the sample spectrum.

The exact appearance of the spectrum will depend on the composition of the sample, but in general, the background and sample spectra should both appear as a relatively smooth line (as

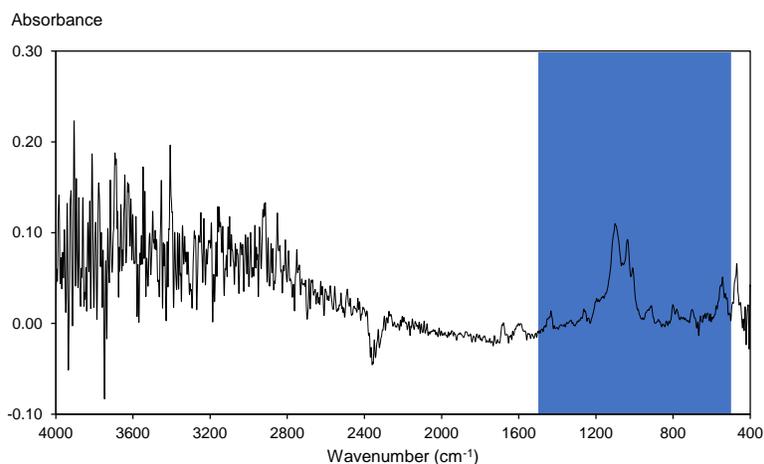
opposed to a “spiky” line with many abrupt changes), especially in the right-hand side (also known as the fingerprint region) of the spectrum (closer to  $600\text{ cm}^{-1}$ ). Depending on the instrument or the software, there may be a region of “noisy” data on the left-hand side (closer to  $4000\text{ cm}^{-1}$ ); as long as the noise is relatively consistent from day to day and does not occur to the right of approximately  $3000\text{ cm}^{-1}$ , this is acceptable. If noise suddenly increases or appears in an area that previously was not noisy, this indicates that a problem needs to be addressed before continuing to analyze samples.

Figure 15 shows images of both good background and sample spectra, while Figure 16 shows examples of spectra with varying distortions and noise levels. Although sample spectra will all be slightly different (depending on how much material the sample contains and its composition), most will have similar qualities, and over time, many users will be able to readily distinguish a high quality sample spectrum from one of lower quality. These examples are intended to facilitate that but should not be considered comprehensive of all the spectra or issues that may arise. Note that spectra have not been baseline-corrected in Figures 15 and 16, except where noted.

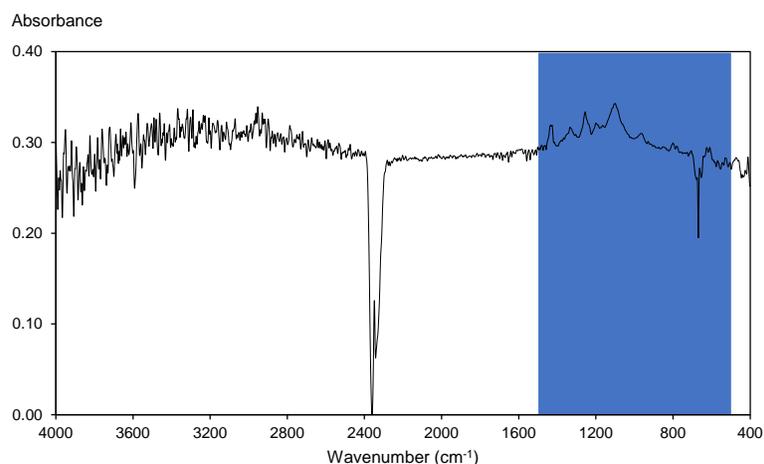


**Figure 15. Illustrations of three FTIR example spectra, background and sample. On the top is the spectrum of a clean filter, which has been collected as the background measurement. In most FTIR software programs, the background spectrum is not shown by default. In the middle is the spectrum of a sample filter, after the background spectrum (clean filter) has been removed by the FTIR software. On the bottom is the same spectrum after baseline correction. In each spectrum, the fingerprint region (1500–500 cm<sup>-1</sup>) is shown in the shaded area.**

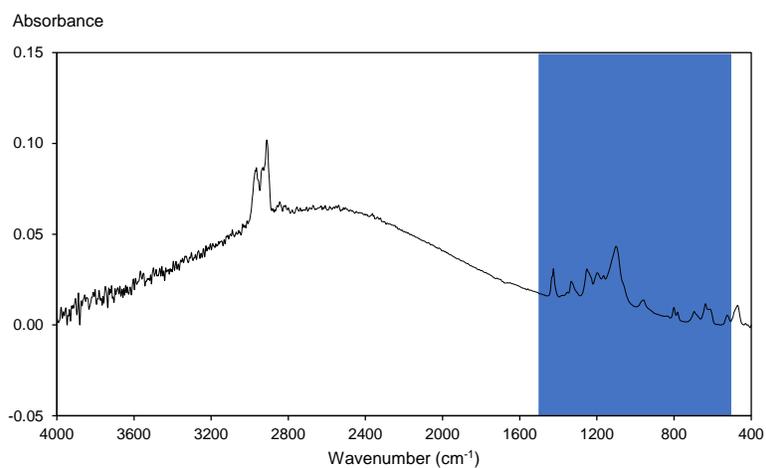
Figure 16 illustrates various types of noise and distortions that may arise in sample spectra. Noise is essentially random variation in a spectrum. When the intensity of the noise is high (i.e. there is a lot of random variation in the signal) relative to the intensity of the quartz signal (or the signal of any other species of interest), the ability to accurately measure quartz (or other species of interest) is compromised. Noise can be present in any portion of the spectrum and may occur over the most or all of the spectrum (such as in 16d) or be isolated to specific regions (such as 16a, b, and e). It is of particular concern when it is present in the fingerprint region of the spectrum (such as in 16d), since the quantity of quartz present in a sample is calculated from this region. When noise is visible in a spectrum, consider both the location of the noise (whether it impacts the fingerprint region) and the relative magnitude. In lightly loaded samples, noise may seem more pronounced relative to the low overall absorbance intensity of the sample, but might not indicate a problem with instrument performance. Users may need to exercise judgement on whether the sample is reliable; examining the fingerprint region more closely may be helpful. When in doubt, it is advisable to perform instrument performance verification checks and/or analyze the QA samples, then repeat the sample analysis.



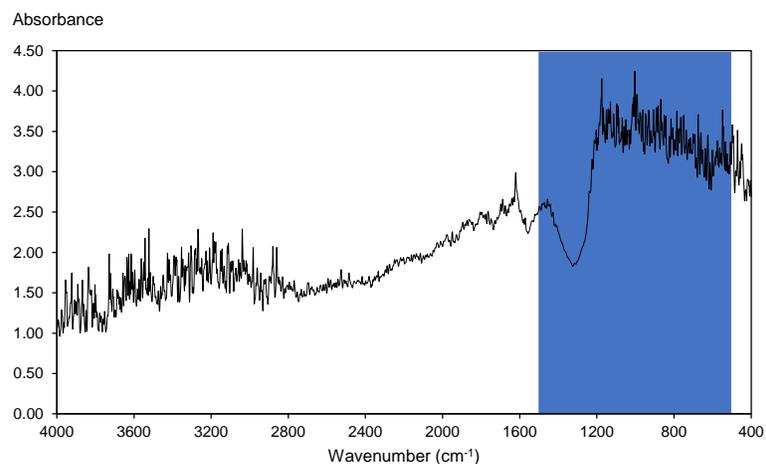
a. A spectrum with considerable noise outside of the fingerprint region.



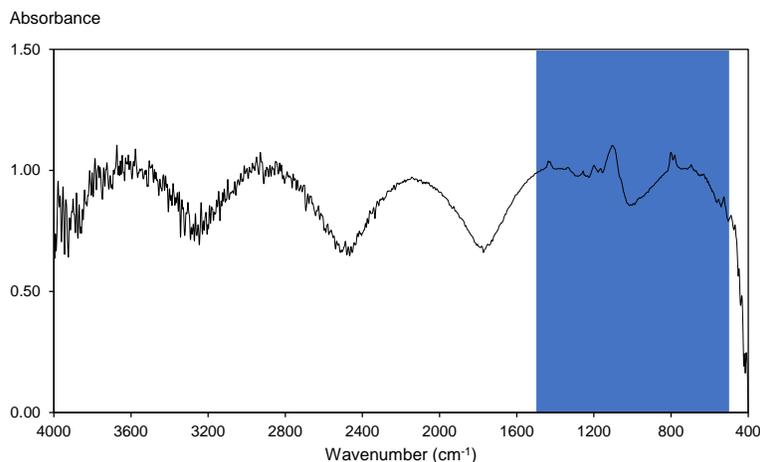
b. A spectrum with moderate noise outside of the fingerprint region. Note that the feature at approximately 2400 cm<sup>-1</sup> is not noise but rather is due to ambient carbon dioxide.



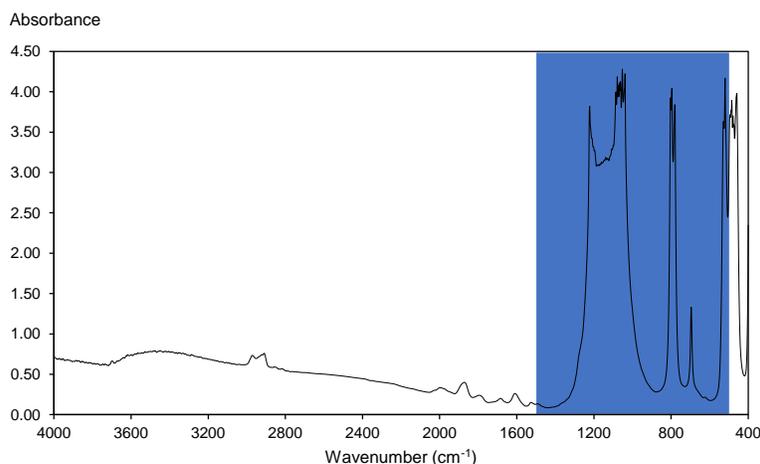
c. A spectrum with minor noise outside of the fingerprint region. Note that the feature at approximately 3000 cm<sup>-1</sup> is not noise but rather is due to ambient humidity (water vapor).



d. A sample spectrum with considerable distortion, including substantial noise within the fingerprint region.



e. A spectrum with considerable distortion that has resulted in an oscillating appearance throughout the spectrum, including the fingerprint region.



f. A spectrum with a considerable distortion in the fingerprint region.

**Figure 16. Illustrations of six FTIR example spectra with noise and/or distortions. Beneath each example spectrum is a brief description of key features, and in each spectrum, the fingerprint region (1500–500 cm<sup>-1</sup>) is shown in the shaded area. A more detailed discussion is provided in the text.**

A distortion—a larger change in the overall shape of a spectrum—may appear with or without excess noise and could be due to a variety of causes. Spectra with distortions such as those in 16d and 16e, where virtually the entire spectrum, including the fingerprint region, is abnormal (see the middle and bottom panels of Figure 15 for comparison), should not be used for calculating quartz concentrations, as such distortions are almost certain to impact the reliability of the spectrum data. These types of distortions might arise as a result of an overloaded sample filter, the instrument or sample being disturbed while the analysis is in progress (including when the instrument indicates that scans have been rejected), or a malfunction of the instrument. Reanalyzing the sample and/or analyzing the QA sample may help to identify the problem. The spectrum of an overloaded or heavily loaded sample may resemble 16f, where portions of the

spectrum appear normal but the tops of some of the peaks in the fingerprint region are distorted. This happens when there is so much sample that the instrument detector is “overwhelmed” by the sample signal.

Note that peaks at approximately  $2400\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$  (see Figure 16b and 16c, respectively) are not noise but are normal features due to ambient carbon dioxide and water vapor, respectively. These peaks may be more prominent, less prominent, or even absent depending on sample loading and how similar the ambient conditions are when the background is scanned and when the sample is scanned.

#### *Possible Causes of Spectrum Problems*

A number of causes might be responsible for spectrum problems, as described below, followed by their solutions.

**Problem:** Incorrect sample analysis settings.

**Solution:** Check that all the sample analysis parameters match those in Table 1; correct any settings that do not match.

**Problem:** Background spectrum is faulty.

**Solution:** Re-scan; choose a different clean filter if necessary.

**Problem:** Unsuitable analysis conditions (heavy vibrations, sample bracket is loose, sample is moving during analysis, etc.)

**Solution:** Make sure that the bracket and sample holder are secure so that the sample is stationary when analysis begins. Light vibration is typically not an issue if it is steady but may be problematic if it is intermittent during analysis. If vibrations are present, place the instrument on a sturdy surface (such as a heavy desk) or wait until vibrations decrease.

**Problem:** Instrument windows are smudged, dirty, cloudy, or cracked.

**Solution:** Clean or replace windows. Note that windows are very difficult to clean without smudging or scratching; therefore it is recommended to replace dirty windows (and store the instrument so that dust and humidity are minimized).

**Problem:** Other hardware issue.

**Solution:** Run instrument diagnostics to determine the problem; consult software help documentation or the instrument manufacturer to repair/replace hardware.

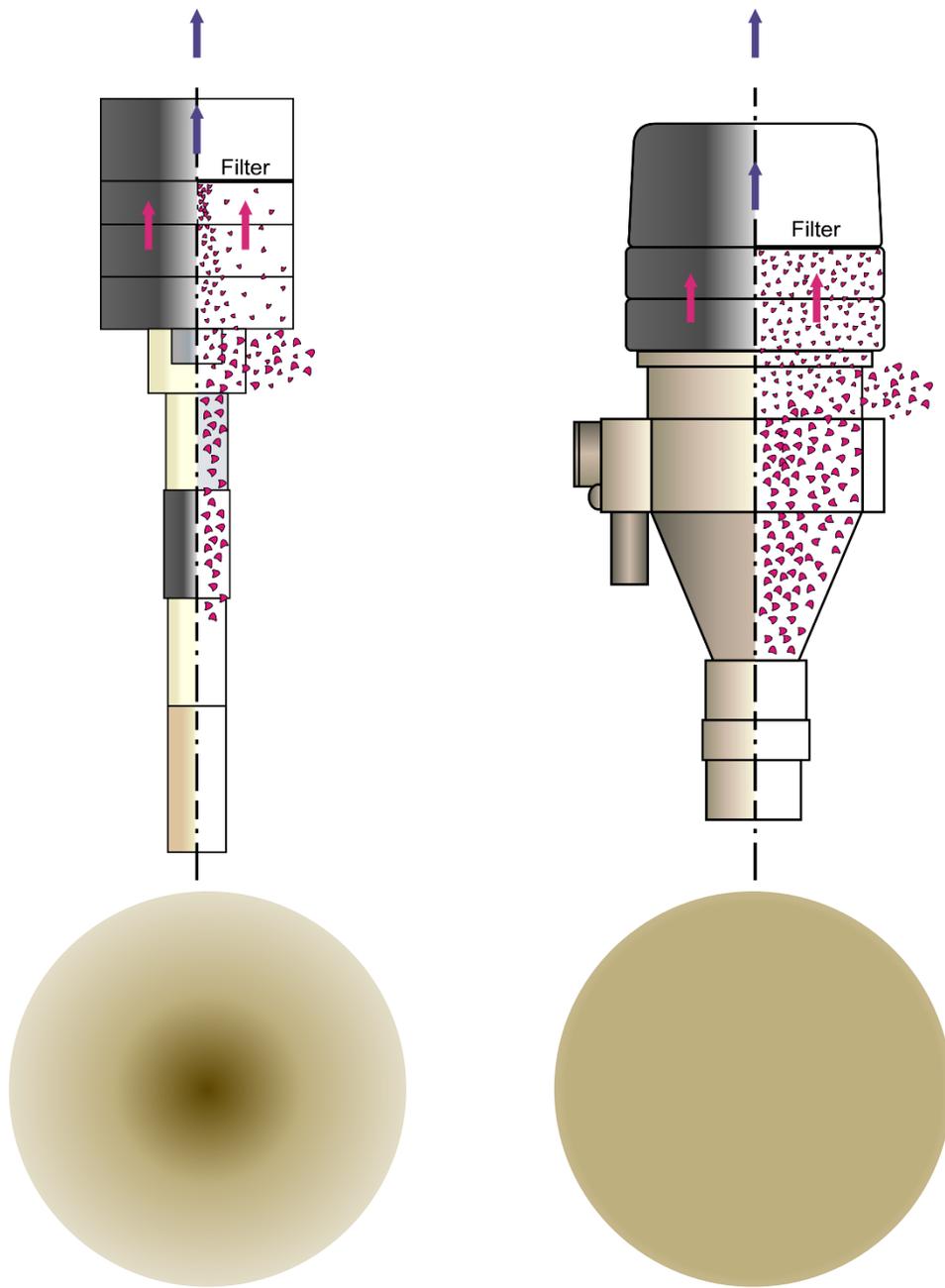
#### *Selecting the Appropriate Sampler Type in FAST*

The sampler used to collect the dust sample for direct-on-filter analysis has an important role in the analytical approach. The choice of sampler must ensure that the collected sample is representative of respirable dust in the environment monitored. The sampler also affects the quantification model for RCS and the appropriate sampler must be selected in the FAST software in order for the RCS result to be accurate.

As described in the previous sections, this specific direct-on-filter approach entails the analysis of a small portion of the dust sample deposited on the filter. This portion is a circular area approximately 6–9 mm in diameter in the center of the filter. The quantification of RCS is then calculated by using established quantification models to correlate the analysis in the center of the filter with the quantity of RCS present on the entire filter. The quantification model is a function of how the dust sample is distributed across the filter, or the *deposition pattern* of respirable dust on the filter.

Respirable dust particles enter a sampling cassette as an aerosol in a stream of air (see Figure 17, top). Once inside the cassette, the aerosol is distributed throughout the volume of the cassette while moving towards the filter media. Eventually, the dust particles are collected on the filter and the air stream exits the cassette. Several factors affect the aerodynamic behavior of the aerosol inside the cassette and, ultimately, the deposition pattern of the dust on the filter: the air velocity at the inlet of the cassette, residence time of the air inside the cassette, the shape of the cassette, and the sampler outlet. The combination of these factors induces different deposition patterns of the dust on filter. The size of the filter also has an influence on the deposition pattern: most samplers collect respirable dust on 37-mm-diameter filters, although some samplers use 25mm or 47-mm diameter filters.

Examples of deposition patterns induced by two samplers/cassettes configurations (see Figure 17, bottom) are presented here. Both samplers use 37-mm diameter filters, which are the most common type used in the United States. Note that the figure refers to the nylon Dorr-Oliver sampler with a luer fitting; the conductive style of this sampler and the nylon version with coupler will result in samples with similar but not identical patterns of dust deposition.



**Figure 17. Illustrations of dust particles entering a respirable sampler and typical patterns of dust deposition from two respirable samplers. Respirable samplers separate particles according to size (top left and right), though samplers of different types display distinct patterns of respirable dust deposition on the sample filter. The nylon Dorr-Oliver sampler results in a high density of respirable dust deposited at the center of the filter relative to the perimeter of the filter (bottom left), whereas the GK2.69 cyclone sampler results in a more uniform density of respirable dust across the diameter of the filter (bottom right).**

The two samplers are the GK2.69 cyclone (BGI by Mesa Labs, Butler, NJ) [Stacey et al. 2014; Stacey et al. 2013] and the 10-mm nylon Dorr-Oliver cyclone (Zefon International, Ocala, FL) [Lidén and Kenny 1993; Maynard 1993; Bartley et al. 2010]. The GK2.69 sampler is operated at a flow rate of 4.2 lpm for respirable dust collection; the Dorr-Oliver sampler is operated at a flow rate of 1.7 lpm except when it is used in U.S. coal mines, where it is used at 2.0 lpm as specified in 30 CFR 70.205 [2019]. The GK2.69 sampler is coupled with a 37-mm diameter cassette in a configuration called an “open-face cassette,” in which the aerosol exits the sampler directly into the volume of the cassette. In contrast, the Dorr-Oliver sampler uses an enclosed cassette and the aerosol moves from the sampler to the cassette through a narrow coupler. Because of these differences and the factors presented above, the deposition pattern for respirable dust samples collected by the two samplers is different. The respirable dust particles collected with the GK2.69 sampler are spread evenly across the 37-mm diameter filter. The respirable dust particles collected with the DO sampler tend to be more concentrated in the center of the filter than at the edges. Both deposition patterns have been found to be consistent for a wide range of respirable dust (and RCS) loadings.

Note that electrostatic effects can adversely affect the sample (changes to sample deposition, increased wall losses, etc.). Samples collected using samplers and cassettes that are composed of conductive or static-dissipative materials are less susceptible to these effects than samples collected using more insulating materials [NIOSH 2016a]. In general, the minimization of wall losses is preferred for direct-on-filter analysis, since only the material deposited on the filter can be analyzed by the instrument.

#### *How Deposition Pattern Affects the Quantification for Respirable Crystalline Silica Using the Direct-on-filter Analysis*

The deposition pattern influences the relationship between the area in the center of the filter that is analyzed directly by the FTIR and the remaining portion of the sample that is not analyzed by the FTIR. Different deposition patterns translate into different models for quantification of RCS on the entire filter. In other words, two samples may contain the same mass of RCS, but the raw FTIR results would be different if they were collected with different samplers. For this reason, the quantification model for the sampler used to collect the sample needs to be used for the quantification of RCS in the sample. In FAST, selecting the correct sampler type will ensure that the quantification model developed for that sampler is used.

NIOSH has developed quantification models for samples collected with a number of commonly used samplers which have been incorporated into FAST. NIOSH is working on developing additional quantification models for samples collected with other samplers. Once the models for additional types of samplers have been developed, they will be added to the FAST software.

#### **“Housekeeping” Suggestions for FTIR Instruments**

Because the use of the FTIR instrument at a site might be intermittent, several housekeeping suggestions are recommended for optimum performance, as follows.

Once an instrument is set up and operating, it may be left powered on for several days (or longer) between analysis sessions; leaving the instrument on is preferable to frequently turning it on and off. When the instrument will not be used for an extended period of time (several weeks or

more), it is strongly recommended to store the instrument in a protective case and to store the case in a suitable environment. If the conditions of the storage environment (especially temperature and humidity) are different from the environment where the instrument will be used, the instrument should be allowed to equilibrate with the new environment for at least 24 hours before it is powered on. This helps to ensure that any condensation has time to dissipate, as this could harm the internal electrical components of the instrument.

Note that the performance of an FTIR instrument relies on internal optical components. In some instruments, some of the optical components are partially exposed and can be affected or damaged by dirt and humidity if care is not taken. Best practices include keeping the lid or door to the transmission compartment closed when the instrument is not actively being used and exercising care not to touch or disturb internal components when the transmission compartment is opened to insert or remove a sample.

## Environment for Storage and Use of FTIR Instruments

The environment where the FTIR instrument is stored and where it is operated should be as described below. Note that while FTIR instruments may be used to analyze samples collected in an underground environment, the instruments themselves are not intended for underground use and should only be used in above-ground environments.

### *Low in Humidity*

**Storage:** If storing the instrument in a low-humidity environment is not possible, the FTIR instrument should be stored in an airtight container (such as a large, sealed bag) with a desiccant (such as a silica gel pack) to absorb excess moisture.

**Operation:** Most instruments can operate in up to 80% non-condensing humidity; however, high humidity will degrade some components over time, negatively impacting data quality and eventually requiring replacement.

### *Moderate in Temperature*

Temperature should be between approximately 18°C and 35°C (65°F and 95°F).

### *Relatively Clean and Free of Dust*

**Storage:** If storing the instrument in a clean environment is not possible, the FTIR instrument should be stored in an airtight container (such as a large, sealed bag).

**Operation:** While small amounts of dust are generally not harmful, larger quantities can contaminate samples and may begin to accumulate in the FTIR instrument sample chamber and potentially in internal chambers, which could negatively impact data quality or cause equipment damage over time. A site office or a similar location is generally suitable; cleaning the work surface with a damp cloth before setting up the instrument can also help to control dust.

### *Not Prone to Physical Disturbances*

**Storage:** Prolonged periods of physical disturbance (such as vibrations from nearby heavy equipment) are a concern. While short-duration vibrations and small bumps should not affect the instrument, prolonged movement may loosen or misalign internal components.

**Operation:** Sudden shocks or vibrations are a concern. While occasional moderate vibrations are not likely to harm the instrument, they can affect the data quality.

## Guidelines for Performing Standard Laboratory Analysis on Samples Analyzed in the Field

This section provides basic guidance on when it may be advantageous to conduct laboratory analysis on samples that have already been analyzed in the field with the portable FTIR, using methods such as MSHA P-2 [MSHA 2013a], MSHA P-7 [MSHA 2013b], NIOSH 7500 [NIOSH 2003], NIOSH 7603 [NIOSH 2017b], and OSHA ID-142 [OSHA 2015]. Because the laboratory analysis is an additional cost (per sample) that the operator must account for, it is important to use this option strategically.

Because of its nature as direct-on-filter, non-destructive analysis, field-based RCS monitoring does not degrade the dust samples, which allows users to analyze the same samples with the standard analysis as well if desired. This can be done by submitting the samples to an off-site accredited laboratory. The possibility of conducting laboratory analysis on the same samples analyzed in the field is an important feature of the monitoring approach and is an option that users should consider to maximize the impact of this tool. When field-based RCS monitoring is implemented initially, new users may find it helpful to have the same-sample comparison of field-based results and laboratory results for a majority of samples. This approach can be used to confirm that users are acquiring reliable results, as well as to determine if a single site-specific correction factor is appropriate for an operation, or if different correction factors would be appropriate for different work areas. As more experience is gained with field-based monitoring, laboratory analysis does not necessarily need to be conducted on all samples. Instead, laboratory analysis can be conducted periodically on specific sample sets to 1) confirm overall reliability of field-based analysis (including when new personnel become involved in field-based monitoring, when a new instrument is acquired or when an old instrument is serviced, or when an instrument has not been used for an extended period of time), 2) check site-specific correction factors, and 3) compose reports where laboratory accredited data is required.

The FAST software has been designed to allow users to add laboratory results to existing samples in the FAST database. The user can edit the “laboratory mass ( $\mu\text{g}$ )” field to add laboratory RCS results for each sample at any time. In addition, the user can also edit the “respirable dust mass (mg)” for the same sample. These data are available from the laboratory results. In this way, the user can have a comprehensive set of results from the field and laboratory on the same sample, with the data all stored together in FAST.

At the time this document was published, direct-on-filter analysis with portable FTIR was not a stand-alone NIOSH analytical method or a standard analytical method used by MSHA or OSHA or other international bodies. For this reason, when there is a need for a formal assessment of compliance status, the dust samples analyzed in the field should then be sent to an accredited laboratory. In this case, the role of field-based monitoring should be to act in support of maintaining regulatory compliance. The goal is that the faster, more comprehensive assessment of the RCS exposure levels that is facilitated by the use of field-based monitoring should increase the probability of compliance status for the operator and protection of workers. This is accomplished by decreasing the RCS concentration and exposure, which is the primary goal of the monitoring approach.

The standard laboratory analysis should also be used for samples collected to generate a site-specific quantification model, described in more detail in the Generating a Site-specific Correction Factor section. The laboratory analysis on selected dust samples is essential to create the site-specific correction factor for an individual operation. Once a correction factor is established (using the “FAST—Site-specific Correction Factor Tool” spreadsheet), additional samples should periodically be sent to the off-site laboratory to verify the validity of the correction factor. This should also be done anytime the user perceives changes in the occupational environment such that the existing correction factor may need to be adjusted. Finally, the standard laboratory analysis should be used for confirmation when field-based monitoring provides results close to an “action value” for the operator. This could be the permissible exposure limit or another value that would trigger an action such as modification of control technologies or work practices in order to decrease exposure. If the field-based method provides a result that is very close to an action value, the standard laboratory analysis can be used to confirm the result. Conversely, standard laboratory analysis is not necessary if the field-based method provides a result that is substantially above or below the action value, since there is little ambiguity in such a result.

### Using Field-based Monitoring to Evaluate Efficacy of Controls

One of the most valuable uses of field-based monitoring is to quickly evaluate the efficacy of newly implemented or modified controls with respect to decreasing airborne RCS concentrations. When a new or modified control is introduced, adjustments to the control may need to be made for optimal performance. With traditional laboratory-based evaluation of RCS, this process may take weeks, but it can proceed much faster with the aid of field-based analysis. The procedure below should be followed and is depicted in Figure 18.

1. Collect a set of samples that will serve as a baseline, before any changes have been made. Be sure to record the sampling times and pump flow rates so that the RCS concentrations can be calculated.

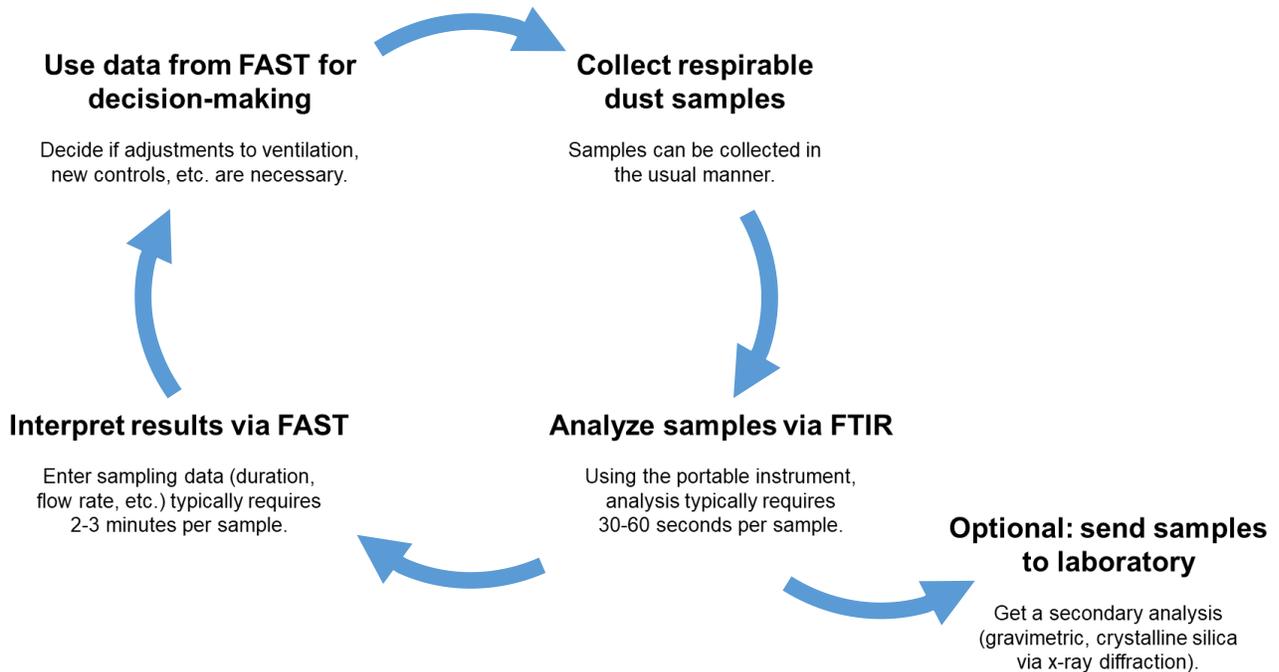
*Note:* If the results of the baseline samples are very close to the limit of detection/limit of quantification (LOD/LOQ, as indicated by FAST), it may be necessary to collect samples for a longer duration, or collect samples with a high-volume sampler, so that more material can be collected; otherwise, it may be difficult to determine if the control has truly been effective.

2. Implement the new or modified control. This could be an engineering control or a change to work practice.
3. Allow enough time for the effects of the control to be fully realized, and then collect another set of samples (the “evaluation sample set”). Ideally, the sampling times and pump flow rates should be similar to the baseline sample. The same type of sampler should be used for both sample sets if possible.

*Note:* In FAST, the same commodity option should be chosen for the baseline sample set and the evaluation sample set. If a correction factor is used, it should be identical for both sample sets. A correction factor is always advisable to lower bias due to minerals and other factors. Nevertheless, provided that the samples are all collected in the same location, under the same conditions, and in a relatively short span of time—within

approximately a day of each other—a correction factor may not be necessary, because the samples will most likely be very similar in composition to one another, and thus can be directly compared to determine if RCS concentrations after the control have decreased relative to the baseline.

4. Continue to modify the control and collect evaluation samples until the desired reduction has consistently been achieved.



**Figure 18. Illustration of the cyclic process of field-based monitoring. As shown, samples are collected, analyzed, and the data are interpreted in a short period of time.**

## Conclusion

When used appropriately, field-based monitoring for RCS enables the timely evaluation of workplace exposure to crystalline silica and can be a valuable component of successful RCS control strategies. The accuracy of results obtained via field-based monitoring are reliant upon field conditions as well as upon the conscientious sampling and analysis by the user. This document provides users with guidance to achieve reliable and consistent results for monitoring purposes, while FAST can provide additional information regarding limitations of sampling data (such as flagging data that fall below the LOD or LOQ). Traditional laboratory analysis should be used for periodic evaluation of results and for circumstances where formal reporting of results is required.

This document can also be used as a reference to help users procure the necessary equipment and software (see Getting Started: Hardware and Software), set up a portable FTIR instrument for field-based analysis (see Setting Instrument Analysis Parameters), and follow appropriate quality

assurance procedures (see Verifying Instrument Integrity; Creating a Set of Samples to be Used as Quality Assurance Samples; and Keeping a Logbook). Appendix A contains links to additional resources that can be used in the field-based monitoring process, Appendix B contains checklists to guide users through the most vital field-based monitoring procedures, and Appendix C contains comparisons of several FTIR analysis methods and parameters discussed in this document.

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## Appendix A: Additional Resources for Field-based Monitoring

Listed below are tools that can support and facilitate field-based monitoring. Some tools are used in conjunction with the instrument software; several versions are available to accommodate different software packages. Other tools are independent of the instrument software and the same version can be utilized by any user.

All of the following resources are freely available online.

### Software-specific Resources (<https://github.com/niosh-mining/fast/>)

- Experiment setup files (see Table 2)
- Macros for spectral processing
- Report templates

### General resources (<https://github.com/niosh-mining/fast/>)

- QA Sample Tracking spreadsheet

### 3D Model Files (<https://3dprint.nih.gov/>)

- Sample bracket (instrument-specific)
- Sample cradle (for use with the four-piece cassette)
- Sample holder (for use with dust sampling cassette)
- Prototype respirable sampler (see Lee et al. 2017)

### Additional guidance for using the provided 3D Model Files:

- The 3D Models available from the link above were designed for a fused deposition modeling (FDM) layer thickness of 0.005 inches when using acrylonitrile butadiene styrene (ABS) material. Different materials and printer formats can affect the accuracy of the printed model, and these specifications should be used as a benchmark. For instance:
  - An FDM layer thickness of 0.015 inches or greater will have a cross section with edges that appear amplified in their rounded coarseness. Printer manufacturers try to compensate for an amount of layer flow, but much is dependent on local conditions.
  - A printer utilizing a cured liquid deposition may exhibit a much finer edge section resulting in greater accuracy.
- Note that the models are provided in units of inches; users who typically print in metric units should be mindful of the conversion.
- Consider how the item is oriented relative to the printer head or nozzle as it is being printed. Generally, the accuracy of the sample cradle, either as a full or half circle, will be greater when oriented perpendicular to the printing head, and greater accuracy will be achieved when the part is printed lying flat on the print platen.

## Appendix B: Operational Checklists for Field-based Monitoring

### Field-based monitoring: checklist for daily operation

- Set up the work area.
- Area is free of excessive dust.
- Work surface is level and stable.
- Humidity is 80% RH or less.
- Temperature is between 65°F and 95°F (18°C and 35°C).
- Initiate instrument/software and check software parameters.
- Background scans: 16
- Sample scans: 16
- Spectral range: 4000–400  $\text{cm}^{-1}$
- Resolution: 4  $\text{cm}^{-1}$
- Phase correction: Mertz
- Zero-fill factor: 2
- Apodization: Blackman Harris (preferred) or medium (second choice); if neither option is available, choose an option consistent with previous analyses
- Results spectrum: absorbance
- Begin a logbook entry for the analysis session.
- Complete an instrument performance check.
- Evaluate the instrument using the QA samples.
- Generate a background spectrum for the QA samples.
- Analyze the QA samples.
- Process QA sample data and generate a report of the results using the macro(s).
- Enter QA sample results in the tracking spreadsheet to verify instrument performance.
- Analyze samples.
- Generate a background spectrum from one of the sample set blanks.
- Analyze the samples.
- Process sample data and generate a report of the results using the macro(s).
- Import results to FAST and enter sampling information to determine RCS concentration.

### **Field-based monitoring: checklist for creating a quality assurance (QA) sample set**

- Collect 10–20 respirable dust samples. The final set should contain 4–7 samples; extra samples may be required if RCS content is relatively low (<5%), requiring more heavily loaded samples to achieve 120–150 RCS.
- Analyze samples via field-based monitoring/FAST. They should meet the following criteria.
- The QA sample with the lowest quantity of RCS contains 20–40  $\mu\text{g}$  of RCS.
- The QA sample with the highest quantity of RCS contains 120–150  $\mu\text{g}$  of RCS.
- The remaining QA samples contain RCS quantities that are (approximately) evenly spread between 20 and 150  $\mu\text{g}$ .
- The dust deposition is radially symmetric (see Figures 13 and 17).
- Enter the results (from either the exported report file or from FAST) in the QA Sample Tracking spreadsheet.
- Analyze QA samples every time the instrument is used and enter results in the spreadsheet to track the performance of the instrument over time.

### Field-based monitoring: checklist for determining a site-specific correction factor

- Collect 15–20 respirable dust samples. The final set should contain at least 10 samples.
- Analyze samples via field-based monitoring/FAST. They should meet the following criteria.
- The sample with the lowest quantity of RCS contains 20–30  $\mu\text{g}$  (0.020–0.030 mg) of RCS.
- The sample with the highest quantity of RCS contains 275–325  $\mu\text{g}$  (0.275–0.325 mg) of RCS.
- The remaining samples (at least 8, for a total of at least 10 in the sample set) contain RCS quantities that are (approximately) evenly spread between 20 and 325  $\mu\text{g}$  (0.020–0.325 mg) of RCS.
- Remove any samples outside the range of 20–325  $\mu\text{g}$  (0.020–0.325 mg) RCS or any extraneous samples within the range.
- Collect and analyze additional samples to meet the above criteria, if necessary.
- Send samples to off-site laboratory for the standard analysis for RCS.
- Use the **Calculate Correction Factor** function in FAST to determine the appropriate correction factor (see Figure 14).
- To use the correction factor, select it in FAST during the **Create Sample Event** step.
- Periodically repeat the process above with new samples to ensure that the correction factor has remained consistent or to modify the correction factor as respirable dust composition evolves.

## Appendix C: Comparison of FTIR data obtained using different methods or parameters

As discussed in the Generating Spectra section, there are two methods for generating a background spectrum, and as summarized in Table 2, there are several available options for instrument software settings such as apodization and baseline correction. The tables in this appendix illustrate the differences that could be expected in sample results for several common options for these settings.

Note that differences seen in actual samples for these types of settings may be greater or less than the differences shown here for laboratory prepared samples. Tables C1, C2, and C4 show data for samples containing *only* quartz to illustrate a “best case scenario”, while Tables C3 and C5 show data for samples that are approximately 25% quartz, in addition to other mineral phases (such as albite/anorthite, microcline, phlogopite, and clinocllore) in order to illustrate a more complex sample. Since different mineral phases can have different impacts on sample spectra and actual samples may be even more complex, these results should not be considered representative of every potential composition of respirable dust that might be encountered.

Regardless of the composition of samples or the settings chosen, it is important to use the same settings consistently, rather than switching between different settings for different samples.

### How the samples for Tables C1, C2, and C4 were collected and analyzed:

The following procedures were used to collect this data with a portable FTIR instrument with the parameters shown in Table 2:

1. Clean PVC filters were first analyzed by FTIR, using the empty sample compartment (ambient air) as the background scan.
2. Samples of a high purity quartz material (Minusil 5) were collected on the filters using the Dorr-Oliver nylon cyclone, operated at a flow rate of 1.7 lpm. The mass of quartz collected ranged from approximately 15  $\mu\text{g}$  to 300  $\mu\text{g}$  of quartz crystalline silica.
3. Sample filters were then analyzed again by FTIR, again using the empty sample compartment (ambient air) as the background scan.
  - a. From each sample spectrum, the initial spectrum of the clean filter (from Step 1) was subtracted.
  - b. The macro procedure was used to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the right column of Table C1.
4. Sample filters were analyzed once more by FTIR, using a clean filter (from the same lot as the rest of the sample filters) as the background scan.
  - a. The macro procedure was used to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software (with “other” for the selected commodity and using no correction factor) was then used to estimate the silica mass. These data are reported in the left column of Table C1, Table C2, and Table C4.

5. The original sample spectra from Step 4 (before the macro procedure was applied) were reprocessed to assess differences in results for three apodization options:
  - a. Original spectra were converted from Blackman Harris 3 term apodization to medium apodization. The macro procedure was used on the new spectra to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the center column of Table C2.
  - b. Original spectra were converted from Blackman Harris 3 term apodization to boxcar apodization. The macro procedure was used on the new spectra to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the right column of Table C2.
6. The original sample spectra from Step 4 (before the macro procedure was applied) were reprocessed to assess differences in results for four baseline correction options. Note that while the macro procedure itself was not used here (since the macro procedure includes a concave rubberband baseline correction), the equivalent steps were performed each time (a baseline correction followed by calculating the peak area for quartz).
  - a. Original sample spectra were baseline corrected using a scattering baseline correction, the peak area for quartz was calculated, and the FAST software was then used to estimate the silica mass. These data are reported in the second from the left column of Table C4.
  - b. Original sample spectra were baseline corrected using a rubberband baseline correction, the peak area for quartz was calculated, and the FAST software was then used to estimate the silica mass. These data are reported in the second from the right column of Table C4.
  - c. The peak area for quartz was calculated from the original sample spectra with no baseline correction, and the FAST software was then used to estimate the silica mass. These data are reported in the right column of Table C4.

**How the samples for Tables C3 and C5 were collected and analyzed:**

1. Samples of a material from a granite quarry (approximately 25% quartz) were collected on PVC filters using the Dorr-Oliver nylon cyclone, operated at a flow rate of 1.7 lpm. The total mass of respirable dust collected ranged from approximately 100 µg to 1700 µg (approximately 30 to 400 µg quartz).
2. Sample filters were analyzed by FTIR, using a clean filter as the background scan.
  - a. The macro procedure was used to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the left column of Tables C3 and C5.

3. The original sample spectra from Step 2 (before the macro procedure was applied) were reprocessed to assess differences in results for three apodization options:
  - a. Original spectra were converted from Blackman Harris 3-term apodization to medium apodization. The macro procedure was used on the new spectra to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the center column of Table C3.
  - b. Original spectra were converted from Blackman Harris 3-term apodization to boxcar apodization. The macro procedure was used on the new spectra to perform a baseline correction (concave rubberband correction) and calculate the peak area for quartz, and the FAST software was then used to estimate the silica mass. These data are reported in the right column of Table C3.
4. The original sample spectra from Step 2 (before the macro procedure was applied) were reprocessed to assess differences in results for four baseline correction options. Note that while the macro procedure was not used here (since the macro procedure includes a concave rubberband baseline correction), the equivalent steps were performed each time (a baseline correction followed by calculating the peak area for quartz).
  - a. Original sample spectra were baseline corrected using a scattering baseline correction, the peak area for quartz was calculated, and the FAST software was then used to estimate the silica mass. These data are reported in the second from the left column of Table C5.
  - b. Original sample spectra were baseline corrected using a rubberband baseline correction, the peak area for quartz was calculated, and the FAST software was then used to estimate the silica mass. These data are reported in the second from the right column of Table C5.
  - c. The peak area for quartz was calculated from the original sample spectra with no baseline correction, and the FAST software was then used to estimate the silica mass. These data are reported in the right column of Table C5.

### **Comparing the two methods for background scans**

Table C1 shows a comparison of estimated quartz mass for the same sample filter containing only quartz, using two different methods for the background (as described above and in the Generating Spectra section). For these samples, differences in results between the two methods range from 0–10  $\mu\text{g}$  RCS, with a mean difference of 3  $\mu\text{g}$ .

**Table C1. Comparison of the two methods for background scan**

<b>Designated clean filter method result (µg RCS)</b>	<b>Subtraction method result (µg RCS)</b>
10	11
16	12
16	14
11	15
11	15
11	15
15	19
14	19
18	19
14	19
16	19
18	20
15	20
17	21
19	21
14	22
16	22
18	22
22	23
20	23
16	23
27	23
18	24
25	26
21	26
28	27
25	28
24	28
26	29
27	30
31	30
30	30
29	30
31	30
28	31
27	31

<b>Designated clean filter method result (µg RCS)</b>	<b>Subtraction method result (µg RCS)</b>
29	31
29	32
34	33
32	33
37	33
31	33
33	34
34	34
33	35
39	36
33	37
31	37
37	37
38	38
30	39
39	41
38	42
42	42
38	42
43	44
43	45
41	45
46	46
44	47
48	48
51	52
53	52
60	60
67	67
85	78
83	84
84	85
85	88
93	95
111	108
134	127
141	138

<b>Designated clean filter method result (<math>\mu\text{g}</math> RCS)</b>	<b>Subtraction method result (<math>\mu\text{g}</math> RCS)</b>
143	143
148	148
157	158
195	190
193	193
205	195
212	212
222	221
222	228
234	232
241	235
250	251
250	253
289	290

### Comparing three apodization options

Table C2 shows a comparison of estimated quartz mass for the same sample filter containing only quartz, using three different methods for apodization (as described above and in Table 2). Depending on the portable instrument and its corresponding software, other apodization options may also be available. As most differences for these samples are less than 1  $\mu\text{g}$ , one decimal place is shown to better demonstrate the small differences, though normally only integer values would be reported (i.e. 10.2  $\mu\text{g}$  vs. 10  $\mu\text{g}$ ). For these samples, differences range from 0–15  $\mu\text{g}$  between Blackman Harris 3-term and medium apodization, with a mean difference of less than 1  $\mu\text{g}$ , and from 0–14  $\mu\text{g}$  between Blackman Harris 3-term and boxcar, with a mean difference of less than 1  $\mu\text{g}$ .

**Table C2. Comparison of three types of apodization for quartz-only samples**

<b>Blackman Harris 3-term result (<math>\mu\text{g}</math> RCS)</b>	<b>Medium result (<math>\mu\text{g}</math> RCS)</b>	<b>Boxcar result (<math>\mu\text{g}</math> RCS)</b>
10.0	10.0	10.0
16.0	15.8	16.0
15.8	15.8	15.8
10.8	10.6	10.8
10.6	10.2	10.4
10.8	10.6	10.8
14.5	14.2	14.3
14.0	13.6	13.8
17.6	17.4	17.7
13.8	13.6	13.8
15.7	15.5	15.7
15.3	15.1	15.3
17.5	17.4	17.5
16.8	16.6	16.8
19.4	19.2	19.4
13.8	13.4	13.6
16.4	16.2	16.4
18.4	18.1	18.3
22.3	22.3	22.3
19.5	19.2	19.4
16.2	16.0	16.0
27.4	27.4	27.4
18.3	17.9	18.1
24.6	24.5	24.7
21.4	21.1	21.3
27.5	0.0	0.0

<b>Blackman Harris 3-term result (<math>\mu\text{g RCS}</math>)</b>	<b>Medium result (<math>\mu\text{g RCS}</math>)</b>	<b>Boxcar result (<math>\mu\text{g RCS}</math>)</b>
25.4	25.3	25.5
23.9	23.6	23.8
25.8	25.5	25.8
27.2	27.0	27.2
29.6	29.4	29.6
30.6	30.4	30.6
28.6	28.5	28.7
30.8	30.6	30.8
26.9	26.6	26.8
28.2	28.1	28.3
28.9	28.7	28.9
28.7	28.5	28.7
34.1	34.0	34.2
31.7	31.5	31.7
36.8	36.8	37.0
31.3	31.1	31.3
32.6	32.3	32.6
34.1	31.7	31.9
33.2	33.0	33.2
38.7	38.7	38.9
32.9	32.5	32.8
31.2	30.9	31.1
36.8	36.6	36.8
38.3	38.1	38.3
30.1	29.6	30.0
39.4	39.2	39.4
38.4	38.1	38.3
41.8	41.5	41.9
38.2	37.9	38.1
43.3	41.5	41.7
43.4	43.4	43.4
41.3	41.1	41.1
46.2	45.8	46.2
44.1	43.8	44.0
47.5	47.2	47.5
50.7	50.4	50.8
53.3	53.0	53.4

<b>Blackman Harris 3-term result (<math>\mu\text{g RCS}</math>)</b>	<b>Medium result (<math>\mu\text{g RCS}</math>)</b>	<b>Boxcar result (<math>\mu\text{g RCS}</math>)</b>
59.9	59.4	60.0
67.4	67.0	67.4
85.0	84.7	85.1
83.4	82.8	83.4
84.1	78.5	79.1
84.8	84.2	84.7
92.5	91.9	92.5
111.1	110.4	111.1
134.1	133.4	134.3
141.4	140.6	141.5
143.4	142.5	143.4
147.8	146.8	147.7
156.7	155.5	156.6
195.1	194.2	195.3
193.2	178.1	179.4
205.4	204.3	205.7
211.7	210.2	211.7
221.6	220.2	221.7
222.2	220.6	222.3
233.9	232.6	234.2
241.0	239.4	241.1
249.8	248.3	249.8
250.1	248.5	250.0
288.6	286.4	288.5

Table C3 shows a comparison of estimated quartz mass for the same sample filter containing quartz and other minerals, using three different methods for apodization, in samples of mixed mineral composition. As in Table C2, differences between the three methods are often less than 1  $\mu\text{g}$  for these samples and one decimal place is shown to better demonstrate the small differences, though normally only integer values would be reported (i.e. 10.2  $\mu\text{g}$  vs. 10  $\mu\text{g}$ ). For these samples, differences range from approximately 0.2–2.6  $\mu\text{g}$  between Blackman Harris 3-term and medium apodization, with a mean difference of about 1.1  $\mu\text{g}$ , and from approximately 0.0–0.8  $\mu\text{g}$  between Blackman Harris 3-term and boxcar (i.e. no apodization), with a mean difference of about 0.3  $\mu\text{g}$ .

**Table C3. Comparison of three types of apodization for mixed-mineral samples**

<b>Blackman Harris 3-term result (<math>\mu\text{g RCS}</math>)</b>	<b>Medium result (<math>\mu\text{g RCS}</math>)</b>	<b>Boxcar result (<math>\mu\text{g RCS}</math>)</b>
27.4	27.2	27.5
28.3	28.1	28.5
48.9	48.3	48.9
51.5	51.1	51.7
65.7	65.3	65.7
108.5	107.7	108.7
136.8	135.8	137.0
137.4	136.4	137.5
147.0	146.0	147.4
148.5	147.5	148.7
154.0	153.2	154.3
177.9	176.6	178.1
212.8	211.5	213.2
234.5	233.0	235.1
236.6	235.3	237.2
247.9	246.4	248.5
302.5	300.6	303.0
385.1	382.6	385.8
403.2	400.6	404.0

### Comparing four baseline correction options

Table C4 shows a comparison of estimated quartz mass for the same sample filter containing only quartz, using four different methods for baseline correction (as described above and in the Processing Sample Data Section). As all differences for these samples are less than 1  $\mu\text{g}$ , one decimal place is shown to better demonstrate the small differences, though normally only integer values would be reported (i.e. 10.2  $\mu\text{g}$  vs. 10  $\mu\text{g}$ ). For these samples, differences range from approximately 0.0–0.9  $\mu\text{g}$  between the concave rubberband correction and the scattering correction, with a mean difference of about 0.2  $\mu\text{g}$ ; from approximately 0.0–0.6  $\mu\text{g}$  between the concave rubberband correction and the rubberband correction, with a mean difference of about 0.2  $\mu\text{g}$ ; and from approximately 0.0–0.6  $\mu\text{g}$  between the concave rubberband correction and no baseline correction, with a mean difference of 0.2  $\mu\text{g}$ .

**Table C4. Comparison of four types of baseline correction for quartz-only samples**

Concave rubberband correction result ( $\mu\text{g}$ RCS)	Scattering correction result ( $\mu\text{g}$ RCS)	Rubberband correction result ( $\mu\text{g}$ RCS)	No baseline correction result ( $\mu\text{g}$ RCS)
10.0	10.0	10.0	10.0
16.0	16.2	16.2	16.2
15.8	15.8	16.0	16.0
10.8	10.6	10.6	11.1
10.6	10.6	10.6	10.6
10.8	11.5	10.9	10.9
14.5	14.9	14.7	14.7
14.0	14.9	14.2	14.2
17.6	17.7	17.7	17.7
13.8	14.5	14.0	14.0
15.7	16.2	15.8	15.8
15.3	15.1	15.5	15.5
17.5	17.7	17.7	17.7
16.8	17.0	17.0	17.0
19.4	19.4	19.4	19.4
13.8	14.2	14.2	14.2
16.4	17.4	16.8	16.8
18.4	18.5	18.5	18.5
22.3	22.3	22.5	22.5
19.5	19.8	19.8	19.8
16.2	17.2	16.4	16.4
27.4	27.5	27.7	27.5
18.3	18.9	18.5	18.5
24.6	24.7	24.7	24.7
21.4	21.5	21.5	21.5
27.5	27.5	27.5	27.5

<b>Concave rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Scattering correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>No baseline correction result (<math>\mu\text{g}</math> RCS)</b>
25.4	25.7	25.7	25.7
23.9	24.2	24.2	24.2
25.8	25.8	26.0	26.0
27.2	27.4	27.4	27.4
29.6	29.8	29.8	29.8
30.6	30.6	30.8	30.8
28.6	28.7	28.7	28.7
30.8	30.9	30.9	30.9
26.9	27.2	27.2	27.2
28.2	28.3	28.3	28.3
28.9	29.1	29.1	29.1
28.7	28.9	28.9	28.9
34.1	34.2	34.2	34.2
31.7	31.9	31.9	31.9
36.8	36.8	37.2	37.0
31.3	31.5	31.5	31.5
32.6	32.8	32.8	32.8
34.1	34.2	34.2	34.2
33.2	33.2	33.2	33.2
38.7	38.7	39.1	38.9
32.9	33.0	33.0	33.0
31.2	31.3	31.3	31.5
36.8	36.8	36.8	36.8
38.3	38.3	38.5	38.5
30.1	30.4	30.4	30.4
39.4	39.4	39.4	39.4
38.4	38.5	38.7	38.7
41.8	41.9	41.9	41.9
38.2	38.3	38.3	38.3
43.3	43.4	43.6	43.6
43.4	43.6	43.6	43.6
41.3	41.5	41.5	41.5
46.2	46.2	46.2	46.2
44.1	44.3	44.3	44.3
47.5	47.5	47.5	47.5
50.7	50.9	50.9	50.9
53.3	53.4	53.4	53.4
59.9	60.0	60.0	60.0

<b>Concave rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Scattering correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>No baseline correction result (<math>\mu\text{g}</math> RCS)</b>
67.4	67.5	67.5	67.5
85.0	85.1	85.5	85.5
83.4	83.6	83.6	83.6
84.1	84.2	84.2	84.2
84.8	84.9	84.9	84.9
92.5	92.6	92.6	92.6
111.1	111.1	111.3	111.3
134.1	134.2	134.5	134.5
141.4	141.5	141.7	141.7
143.4	143.6	143.6	143.6
147.8	147.9	147.9	147.9
156.7	157.0	157.0	157.0
195.1	195.3	195.7	195.7
193.2	193.4	193.6	193.4
205.4	205.5	205.8	205.8
211.7	212.1	212.1	212.1
221.6	221.9	221.9	221.9
222.2	222.6	222.6	222.6
233.9	234.2	234.5	234.3
241.0	241.3	241.3	241.3
249.8	250.2	250.2	250.2
250.1	250.6	250.6	250.6
288.6	289.1	289.2	289.2

Table C5 shows a comparison of estimated quartz mass for the same sample filter containing quartz and other minerals, using four different methods for baseline correction (as described above and in the Processing Sample Data Section). As in Table C3, differences between the four methods are often less than 1  $\mu\text{g}$  for these samples and one decimal place is shown to better demonstrate the small differences, though normally only integer values would be reported (i.e. 10.2  $\mu\text{g}$  vs. 10  $\mu\text{g}$ ). For these samples, differences range from approximately 0.0–0.9  $\mu\text{g}$  between the concave rubberband correction and the scattering correction, with a mean difference of about 0.3  $\mu\text{g}$ ; from approximately 0.2–1.1  $\mu\text{g}$  between the concave rubberband correction and the rubberband correction, with a mean difference of about 0.5  $\mu\text{g}$ ; and from approximately 0.2–0.9  $\mu\text{g}$  between the concave rubberband correction and no baseline correction, with a mean difference of 0.5  $\mu\text{g}$ .

**Table C5. Comparison of four types of baseline correction for mixed-mineral samples**

<b>Concave rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Scattering correction result (<math>\mu\text{g}</math> RCS)</b>	<b>Rubberband correction result (<math>\mu\text{g}</math> RCS)</b>	<b>No baseline correction result (<math>\mu\text{g}</math> RCS)</b>
27.4	27.5	27.7	27.7
28.3	28.3	28.5	28.5
48.9	48.9	49.1	49.1
51.5	51.5	51.7	51.7
65.7	65.7	65.8	65.8
108.5	108.5	108.7	108.7
136.8	137.0	137.2	137.2
137.4	137.5	137.7	137.7
147.0	147.4	147.4	147.4
148.5	148.7	148.9	148.9
154.0	154.2	154.5	154.5
177.9	178.5	178.5	178.5
212.8	213.4	213.4	213.4
234.5	235.1	235.3	235.3
236.6	237.0	237.4	237.4
247.9	248.5	248.7	248.7
302.5	303.2	303.2	303.2
385.1	386.0	386.0	386.0
403.2	404.0	404.3	404.2



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**DHHS (NIOSH) Publication No. 2022-108**

**DOI: <https://doi.org/10.26616/NIOSH PUB2022108>**