

## Supporting Field-based Analysis for Respirable Crystalline Silica

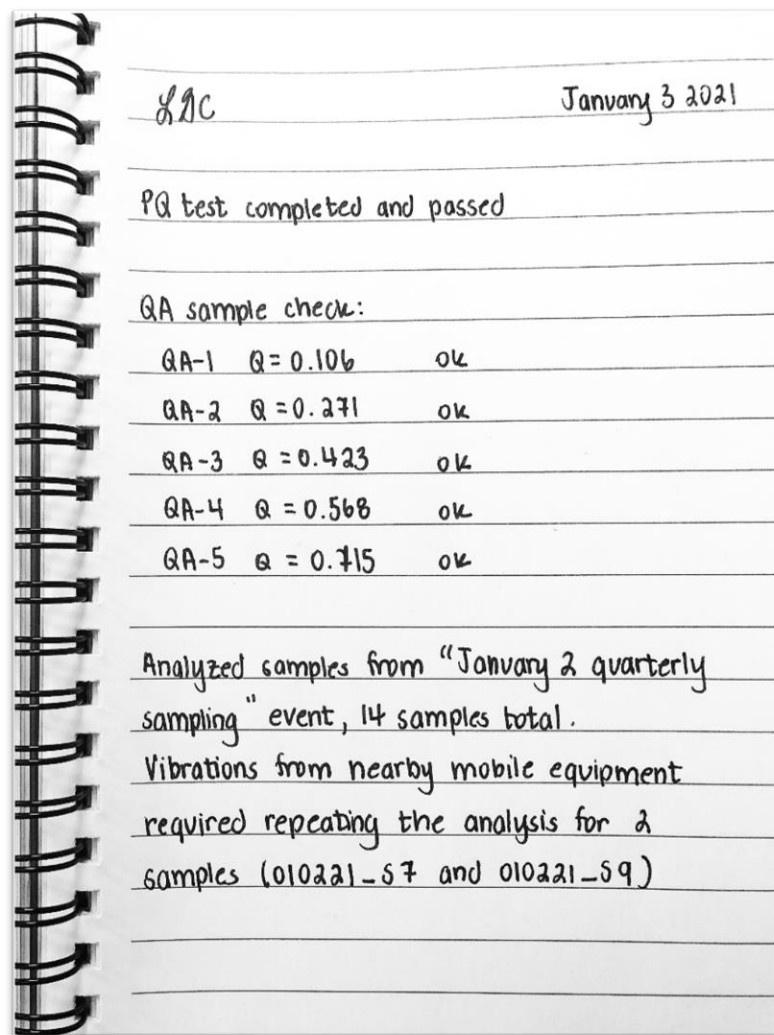
### Keeping a Logbook

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:

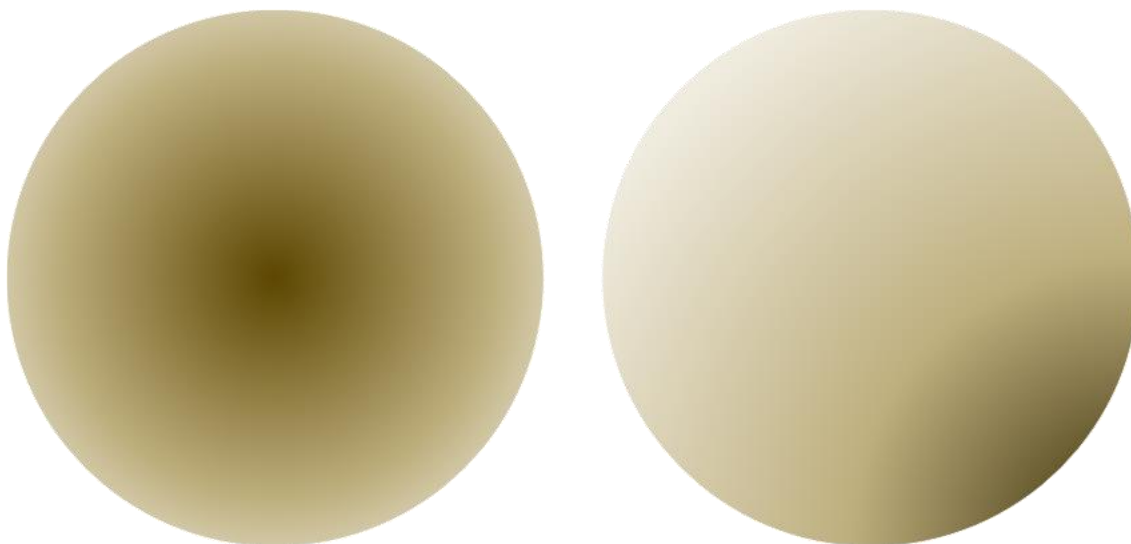
- The QA sample with the lowest quantity of RCS should contain approximately 20–40  $\mu\text{g}$  of RCS.
- The QA sample with the highest quantity of RCS should contain approximately 120–150  $\mu\text{g}$  RCS.
- The remaining QA samples should contain RCS quantities that are (approximately) evenly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

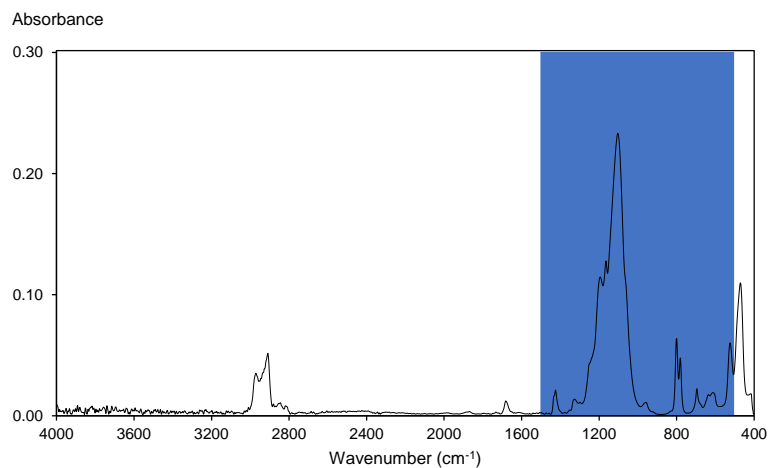
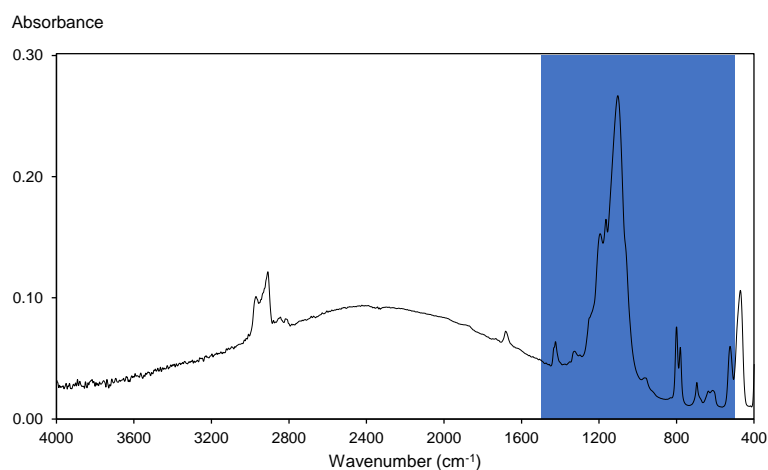
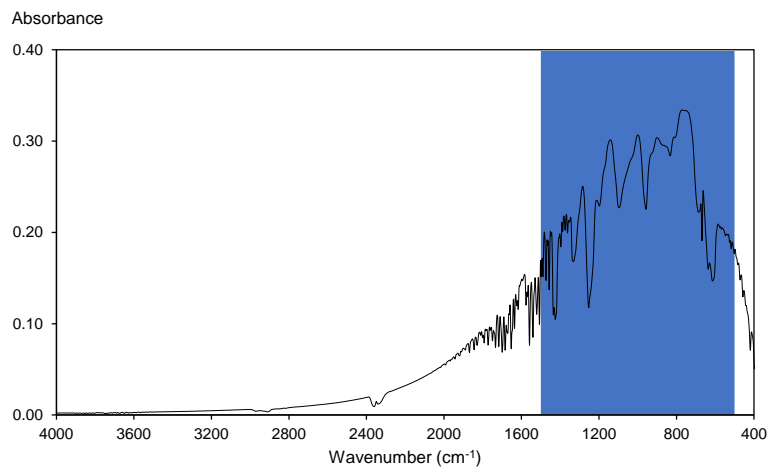
### Troubleshooting: What a Sample Spectrum Should (and Should Not) Look Like

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.

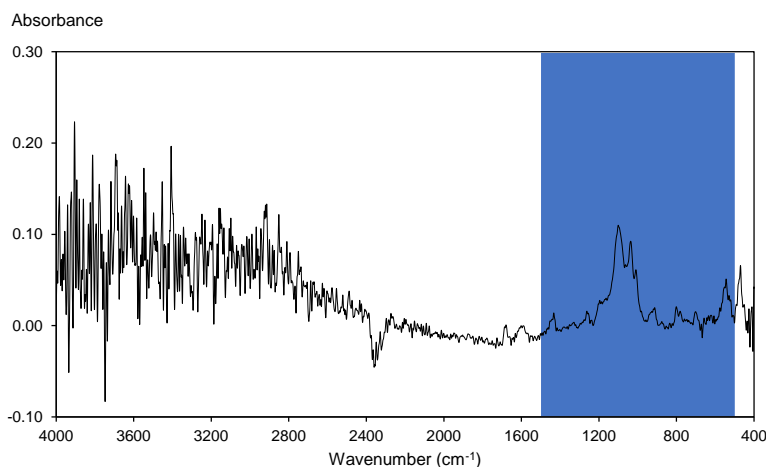
Figure 15 shows images of both good background and sample spectra, while Figure 16 shows examples of spectra with varying distortions and noise levels. Although sample spectra will all be slightly different (depending on how much material the sample contains and its composition), most will have similar 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.



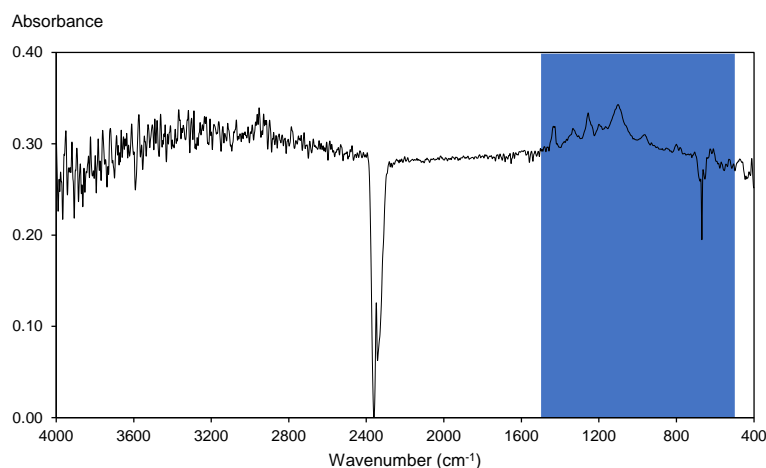
**Figure 15. Illustrations of three FTIR example spectra, background and sample. On the top is the spectrum of a clean filter, which has been collected as the background measurement. In most FTIR software programs, the background spectrum is not shown by default. In the middle is the spectrum of a sample 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  $\text{cm}^{-1}$ ) is shown in the shaded area.**



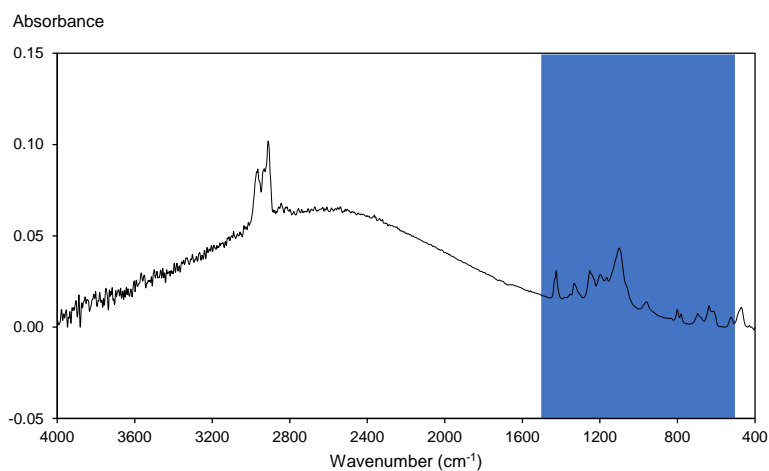
Figure 16 illustrates various types of noise and distortions that may arise in sample spectra. Noise is essentially random variation in a spectrum. When the intensity of the noise is high (i.e. there is a lot of random variation in the signal) relative to the intensity of the quartz signal (or the signal 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.



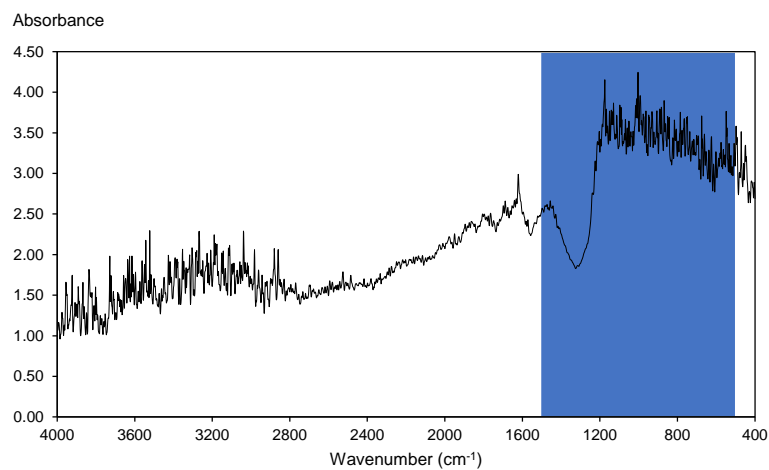
a. A spectrum with considerable noise outside of the fingerprint region.



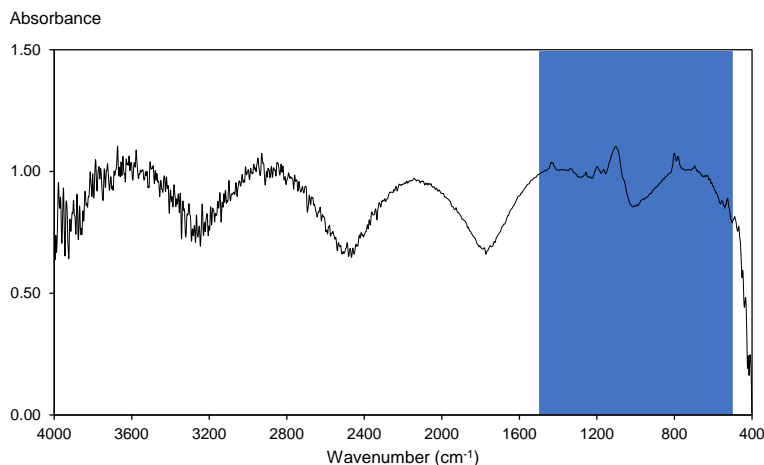
b. A spectrum with moderate noise outside of the fingerprint region. Note that the feature at approximately 2400 cm<sup>-1</sup> is not noise but rather is due to ambient carbon dioxide.



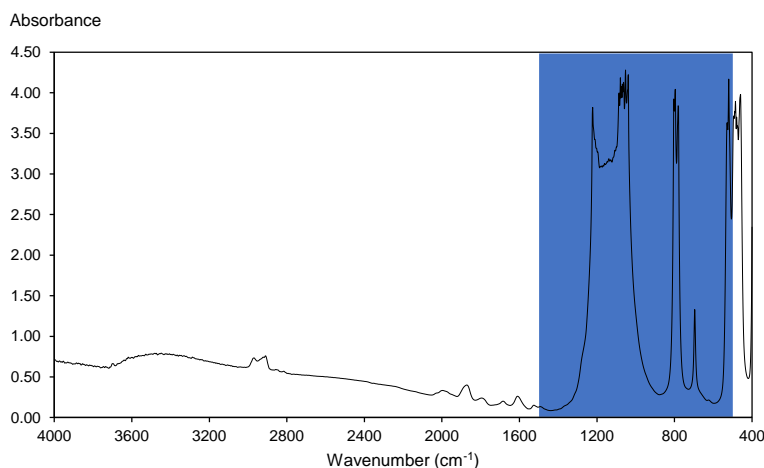
c. A spectrum with minor noise outside of the fingerprint region. Note that the feature at approximately 3000 cm<sup>-1</sup> is not noise but rather is due to ambient humidity (water vapor).



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.

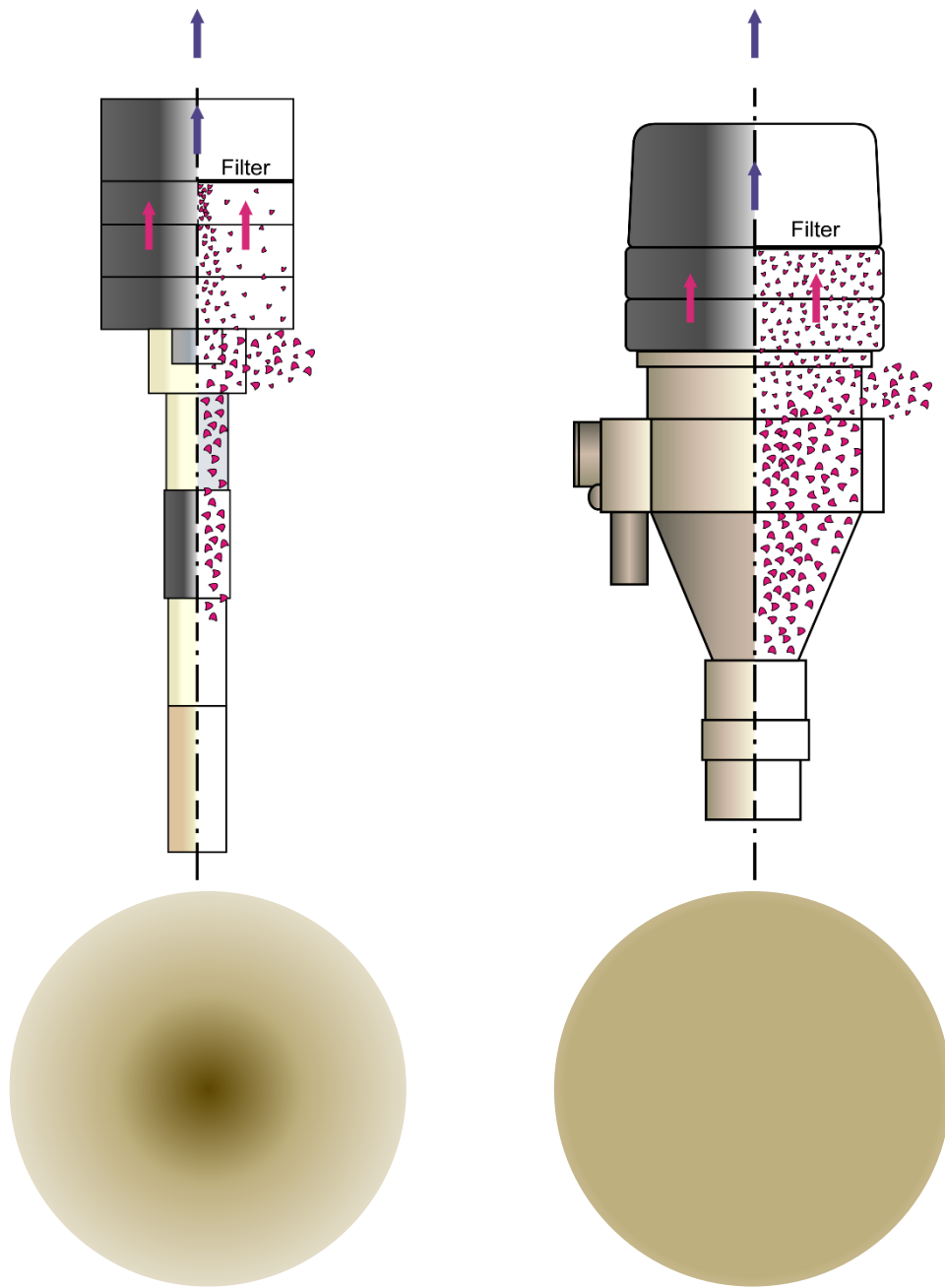
#### *Selecting the Appropriate Sampler Type in FAST*

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.

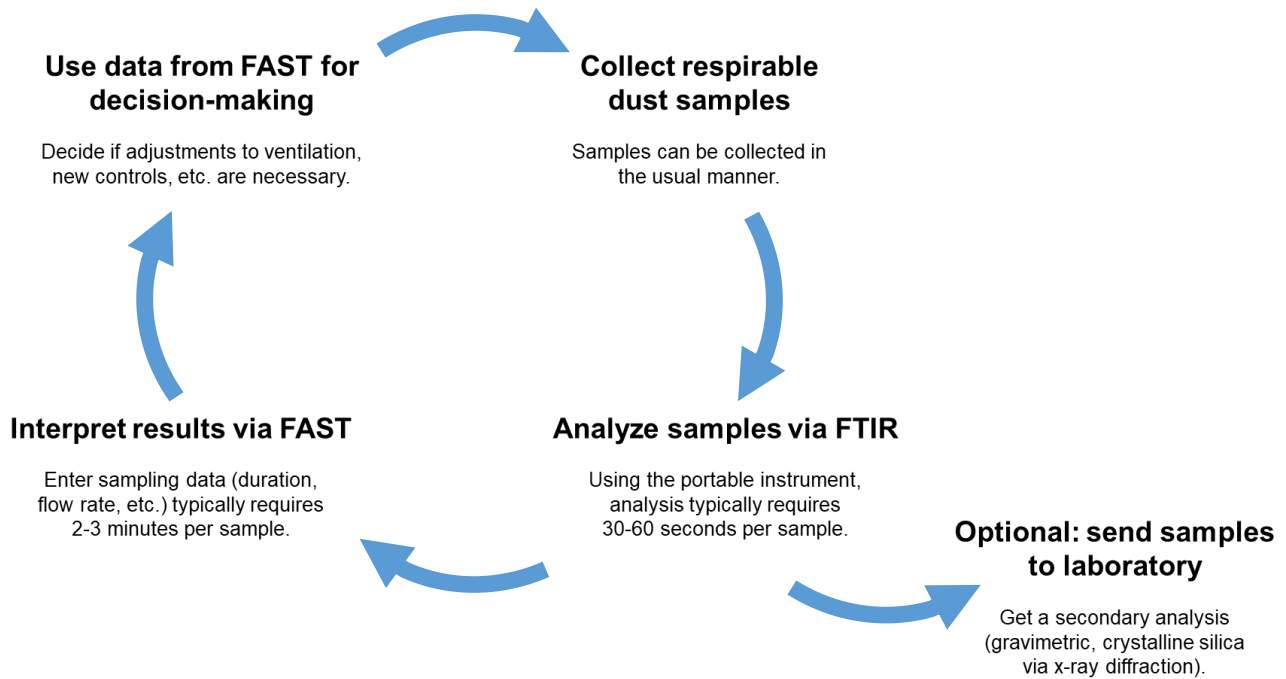
*Note:* If the results of the baseline samples are very close to the limit of detection/limit of quantification (LOD/LOQ, as indicated by FAST), it may be necessary to collect samples for a longer duration, or collect samples with a high-volume sampler, so that more material can be collected; otherwise, 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 µg of RCS.
- The QA sample with the highest quantity of RCS contains 120–150 µg of RCS.
- The remaining QA samples contain RCS quantities that are (approximately) evenly spread between 20 and 150 µg.
- 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). The verification code for this document is 222554
  - 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