

Effect of Purging a Sealed and Desiccated FTIR Spectrometer Sample Compartment

Study demonstrating the inconsistency in background levels of water and carbon dioxide for an unpurged, sealed, and desiccated FTIR spectrometer as compared to a sealed and desiccated FTIR spectrometer with the sample compartment purged.

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Increase Accuracy of FTIR Analysis

Effect of Purging a Sealed and Desiccated FTIR Spectrometer Sample Compartment

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The purpose of providing a dry, CO₂-free purge to an FTIR spectrometer is twofold. The purge prevents deterioration of the beamsplitter by moisture and also eliminates undesired absorbance by water and carbon dioxide in the background. Consequently, the purge enhances the instrument's reliability by the reduction of the potential need for service and increases the accuracy of analysis by the elimination of inconsistencies in the background levels of water and carbon dioxide.

A study was undertaken to demonstrate the inconsistency in background levels of water and carbon dioxide for an unpurged, sealed, and desiccated FTIR spectrometer as compared to a sealed and desiccated FTIR spectrometer with the sample compartment purged.

All of the testing discussed in this application note was conducted by an independent consultant with extensive experience in the field of FTIR spectroscopy who is unbiased toward the instrument and the purge gas generator. The tests were performed on an FTIR spectrometer that was designed as a sealed and desiccated instrument to

be operated without purged gas. All data were collected in scan sets of four scans and each scan took approximately 15 sec. One scan was recorded for the background spectrum and a second was collected to produce the 100% transmittance line. For some spectra, scan sets were collected consecutively; for others, there was a time lag between the collection of the scans. The specific conditions are noted in the discussion of results. All spectra were apodized with the Norton-Beer strong function. The detector was a deuterated triglycine sulfate (DTGS) detector.

Experimental

Two sets of tests were conducted in order to demonstrate the effect of purging an FTIR spectrometer sample compartment. The first set of tests consists of various scans without the instrument sample compartment being purged. The second set of tests demonstrates the effect of purging the spectrometer sample compartment.

A model 75-45NA FTIR purge gas generator (Parker Hannifin Corporation, Haverhill, MA) was used as the source of purge gas in the experiments. The generator was connected directly to an air line and operated at 80 psig. The air was treated prior to entering the generator by a condensing unit in order to remove gross quantities of water, and by filtration to remove oil droplets. During experiments with the use of a purge gas, the gas entered the sample compartment at a flow rate of 0.4 scfm.

Results

Performance without sample compartment purge Figure 1 shows a single-beam spectrum of an unpurged, sealed, and desiccated FTIR spectrometer. Figure 2 shows the 100% transmittance line for the same, unpurged spectrometer. This line was produced from two consecutive scan sets. The spectrometer sample compartment had not been opened for at least 8 hr. The time between the two scan sets was less than 1 min. Despite the fact that there was a sealed atmosphere in the system, it can clearly be seen that there is a change in the carbon dioxide concentration.

The problem is that the interferometer compartment is sealed, but the sample compartment is not. The carbon dioxide and water levels in the atmosphere change because the operator is in the vicinity of the spectrometer. If the operator were to cease breathing, there would be no recorded change in the carbon dioxide or water levels. If the time between scans increases, the situation becomes worse. This is illustrated in Figure 3. The time between scans was 40 min. The spectrometer was not opened in this time, but the operator was in the laboratory during that period. The operator was in the laboratory during that period. The operator was across the room for most of the time, and was alone.

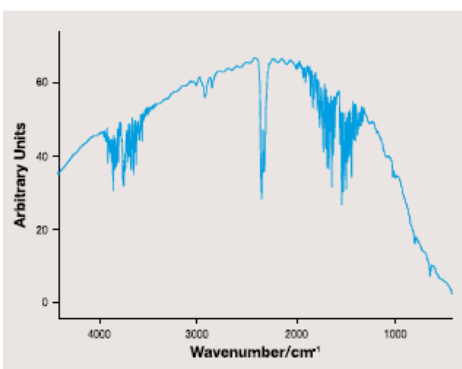


Figure 1:
Single-beam
spectrum from
unpurged,
sealed, and
desiccated
FTIR
spectrometer.

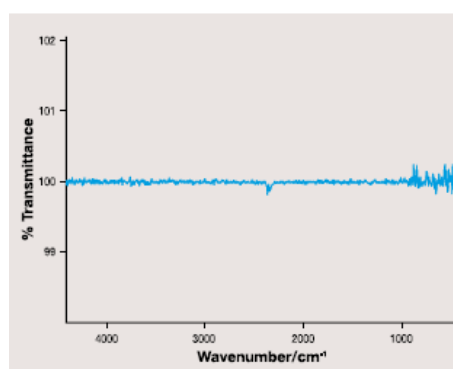


Figure 2:
100%
Transmittance
from
unpurged,
sealed, and
desiccated
FTIR
spectrometer

Figure 3 indicates that the carbon dioxide and water content of the air change as a result of one person being in a room of approximately 7200 ft³. In this case, the water level actually decreased, but there was a marked increase in the amount of CO₂ in the air. In an actual experiment, scan sets should never be collected 40 min apart. This Figure simply illustrates how severe the lack of purge can be even for a sealed system.

A new background scan set was collected, and the sample cell was opened for 15 sec; then another scan set was collected immediately after the sample compartment was closed. The resulting 100% transmittance line is shown in Figure 4. As can be seen from this spectrum, the level of atmospheric water has decreased and the level of carbon dioxide has increased. Without purge, it is virtually impossible to maintain the atmospheric absorption bands as constant while a sample is changed. The levels of these vapors and gases are constantly

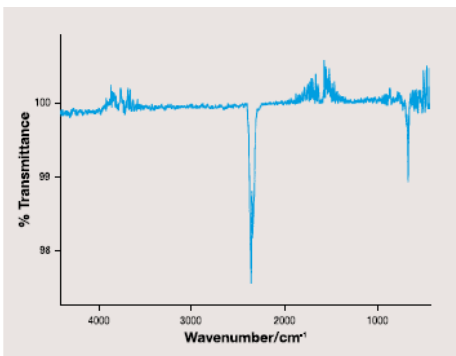


Figure 3: 100% Transmittance from unpurged, sealed, and desiccated FTIR spectrometer: 40 min between scans.

changing, and the spectrometer is a very good detector for the change. Even if the operator were to hold his or her breath while opening the sample compartment, the ambient level of water and CO₂ in the atmosphere has enough fluctuation that it is impossible to maintain a constant level of water and CO₂.

Performance with the sample compartment purged

The sealed and desiccated spectrometer used in this study has a purge port with a line that leads directly to the sample compartment. This is provided so that the atmosphere within the sample compartment can be purged and maintained in a water-free and carbon dioxide-free state. Figure 5 is a single-beam spectrum of the spectrometer once the sample compartment had been totally purged by the purge gas generator. When this is compared with Figure 1, it is evident that the water and the CO₂ bands have reduced in intensity, but there is still plenty of water

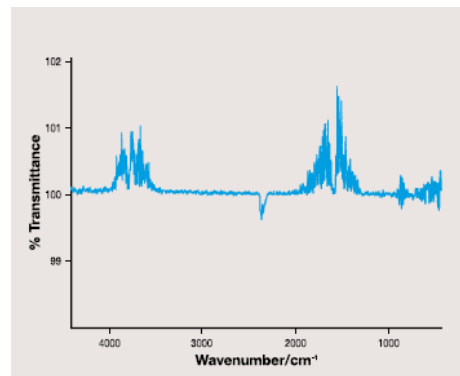


Figure 4: 100% Transmittance from unpurged, sealed, and desiccated FTIR spectrometer: 15 sec between scans.

in the desiccated compartment. It should be mentioned, however, that the desiccant was at the end of its cycle; consequently, there is more water than there would be if the desiccant were fresh. Herein lies another problem with sealed and desiccated systems: The dryness of the sealed interferometer compartment changes as the desiccant becomes saturated. The corresponding 100% line from the purged sample compartment is shown in Figure 6. The sample compartment door had not been opened between the two consecutive scan sets, so the 100% line is again very good.

If the sample compartment is open for 15 sec, then closed, and a new 100% line is measured with the background spectra from before the door had been opened, the resulting 100% line is shown in Figure 7. Clearly, there is a great deal of water vapor in the spectra. If the system were allowed to purge for 3 min, the water vapor and CO₂ that were in the sample compartment are completely removed. This can be seen in Figure 8 where a 100% line is obtained from the background spectra before the door was opened, and a new scan was collected 3 min after the door was closed. Figure 6, 7, and 8 illustrate that simple purging the sample compartment will provide good results. This, of course, does not remove all of the water and CO₂, and the water vapor and the CO₂ gas are still present in the desiccated chamber. Nonetheless, if a system is purged only in the sample compartment, it is far better than not purging at all.

Conclusion

Changing levels of CO₂ and water vapor in a laboratory atmosphere can result in interference from these contaminants for unpurged FTIR spectrometers. The changing levels of CO₂ and water are primarily a result of operator breathing, and the extent

of interference to the FTIR spectra is dependent on the amount of time elapsed between the background spectra and the sample spectra. Purging of the sample compartment increases the reliability and consistency of FTIR spectra. The experiments described above clearly identified

the need for purge, even for the purge-less sealed and desiccated FTIR spectrometer sample compartment. The purge gas generator proved to be a suitable source of purge gas for such FTIR spectrometers.

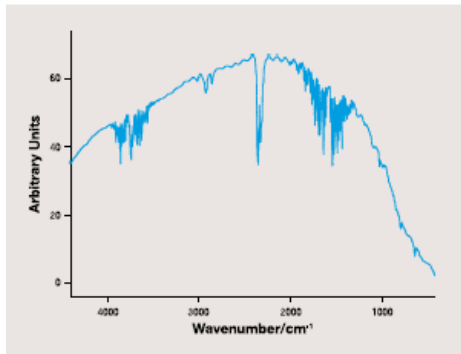


Figure 5: Single-beam spectrum from sealed and desiccated FTIR spectrometer with purged sample compartment.

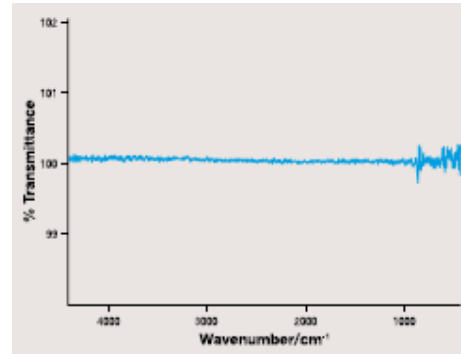


Figure 6: 100% Transmittance from sealed and desiccated FTIR spectrometer with purged sample compartment.

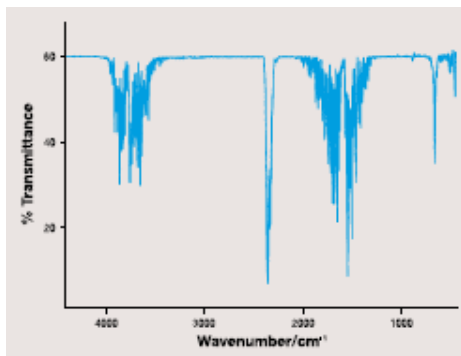


Figure 7: 100% Transmittance from sealed and desiccated FTIR spectrometer with purged sample compartment. Sample compartment opened for 15 sec between scans.

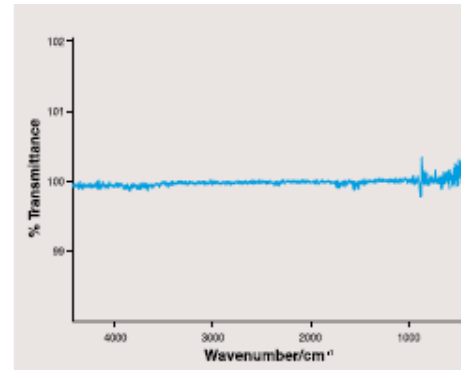


Figure 8: 100% Transmittance from sealed and desiccated FTIR spectrometer with purged sample chamber. Second scan collected 3 min after sample compartment was opened.