

Purging of the Perkin-Elmer Spectrum 1000 FT-IR

Purging with inert, dry gas removes background water vapor and CO₂ from the optical pathway in the instrument. The presence of water vapor is indicated by the presence of a large number of narrow peaks between 4000 - 3200 and 2100 - 1400 cm⁻¹ and the presence of CO₂ is indicated by a major peak at 2200 cm⁻¹ and several smaller peaks below 1000 cm⁻¹ (Figure 1). These features often can not be successfully removed by background-subtraction. In our experience purging is required more often when using DRIFT rather than transmission measurements, although purging should be done whenever the features in Figure 1 are not removed by background subtraction.

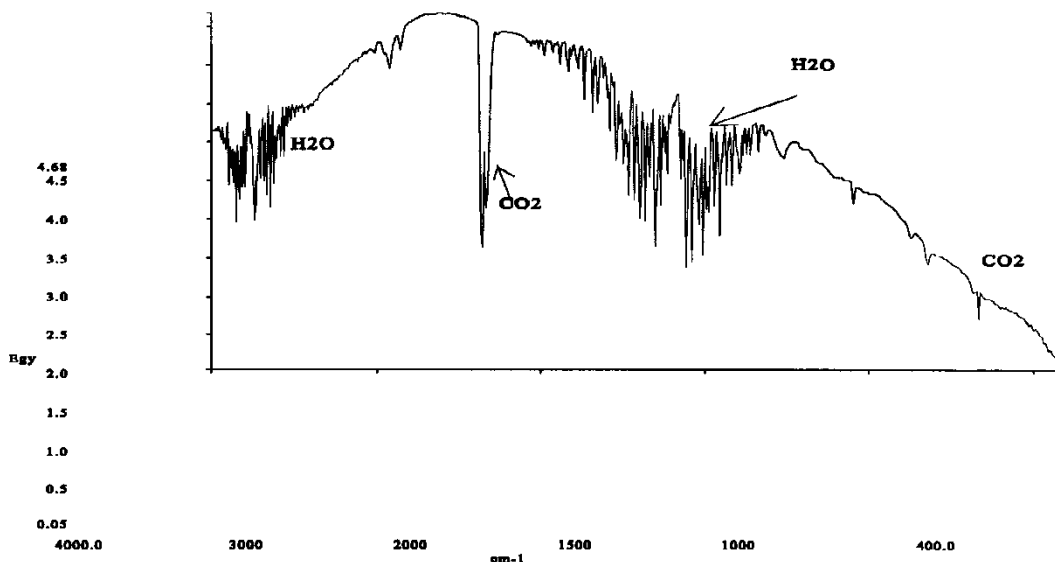


Figure 1. Background spectra without purging.

Flammable gases should not be used, and usually high-molecular-weight gases are preferred. Typically helium, nitrogen, and argon are used. Connect the purge tubing to a gas cylinder through a regulator because high pressure may damage the instrument by misaligning mirrors, etc.

Internal Purge

The optical system compartment should be purged after changing the dessicant or if the system has been open to air. In many cases, internal purging has already been performed by the

check the dessicant color meter on the internal purge cover. A pressure of approximately 1 lb/ in² (6.9 kPa) is the maximum suggested. Tygon[®] tubing (I.D. 3/8", O.D. 9/16", wall 3/32") connects the gas cylinder regulator to the instrument is connected by using Swagelok[®] fittings.

First, the cover is raised by using the larger of the two hexagonal wrenches in the kit (5/32") to turn the screw on the lower front cover in a counterclockwise direction. Usually the power is switched off before any work is done, but if not, a safety feature automatically switches off the power. Disconnecting the power cable also eliminates the hazards of electrical shock and laser radiation.

The cover is supported by inserting the free end of a prop arm into the notched receptacle. The plastic caps from the inlet and outlet ports are removed on the internal plastic cover, and tubing is connected to the inlet. Purge for approximately 10-20 min, although a purge may be run overnight at a lower flow rate. All caps should be replaced after purging in order to prevent moisture and CO₂ from re-entering the system. A background scan is usually performed and afterwards the results are printed and kept for future reference.

External Purge

The connection made in purging the sample compartment is different than the connection to the internal compartment. Tubing coming from the gas cylinder must be connected to the connection at the left rear corner of the instrument. An internal tube is already in place which allows the purge gas to flow into the sample compartment. Purge gas enters the sample compartment through an inlet on the left wall of the chamber. No special outlet is necessary because the gas dissipates at the edges of the compartment. The flow rate is set at approximately 1 ft³/min (28 L/min). The sample is placed in the compartment and purge gas is allowed to enter for approximately 2 min before the sample scan is initiated. On hot and humid days purge approximately 4 to 5 min before and continue during the sample scan. Because the sample compartment is opened each time a new sample is placed in the instrument, the purge gas must be left on to displace incoming air containing CO₂ and H₂O.