



Fig. 15 Schematic diagram of the experimental setup used to calibrate the diode laser frequency scan rate ($\text{cm}^{-1}/\text{point}$)

The mass flow rate, dm/dt , (g/s) is calculated using Eq. 11 by the product of the measured number density (N , molecules cm^{-3}), the gas flow velocity (u , cm/s), and the cross-sectional area of the flow duct (A , cm^2 ; and the appropriate conversion factors).

$$dm/dt = N \cdot u \cdot A \cdot (\text{g/s}) \quad (11)$$

The standard measurement angles θ_1 and θ_2 are 45° and 135° . These angles combined with typical laser frequency tuning rates of approximately 1 MHz/data point result in a velocity measurement sensitivity of 1 m/s/data point shift. The application of the BRD-based detection technique and the resulting high signal-to-noise ratios enables peak shift measurements of less than one data point and the measurement of gas flow velocity and mass flow rates throughout both the primary and secondary drying phases of freeze-drying.

All three measured parameters, water vapor temperature, water vapor number density, and gas flow velocity use the same instrument calibration factor, the diode laser frequency scan rate (MHz/point or $\text{cm}^{-1}/\text{point}$). The diode laser frequency scan rate ($\text{cm}^{-1}/\text{point}$) is determined by launching the fiber-coupled diode laser output into a Fabry-Perot interferometer (FPI) optical cavity. The measurement principle and an experimental schematic of the calibration technique are illustrated in Fig. 15.

The laser light is transmitted through the mirrored cavity only when cavity conditions and the laser wavelength or frequency results in constructive interference of the oscillating light waves. The transmission maxima occur separated in frequency by $\Delta\bar{\nu}$ (free spectral range, FSR) as described by Eq. 12:

$$\frac{1}{2nL} = \Delta\bar{\nu}(\text{cm}^{-1}) \quad (12)$$

where n is the index of refraction of the medium between the two cavity mirrors (typically air, $n=1$) and L is the separation between the mirrors M_1 and M_2 .

Figure 16 shows a water vapor absorption lineshape and an FPI fringe spectrum simultaneously recorded using a TDLAS monitor. The peak locations of the FPI interference spectrum in combination with the interferometer FSR (2000 MHz) are used to calculate the laser frequency scan rate calibration. Typical laser tuning rates