

Chapter 3

Experimental Procedure

3.1 Burner Systems Startup

3.1.1 Instrumentation power-up

The instrumentation of the burner including the PC need to be turned on, in order to provide safe ignition conditions. Without the instrumentation, there is no indication of what flow exists in the burner. The flow-meters are powered by a 5V DC-power supply. The air flow-meter output is connected to channel 0 and the methane gas flow-meter output is connected to channel 1 of the SC-1000 National Instruments data acquisition card. The LABVIEW routine 'flow.vi' is opened, and the maximum flow ratings for the flow-meters to be used are entered. Once the inputs have been verified to be correct for the current experimental configuration, the program is started. It is important to allow for flow-meter warm-up (30 minutes) prior to use in exact measurement. Periodically, at approximately weekly intervals, the flow-meter zeros are checked according to the Hastings flow-meter manual.(Hastings Instruments, 1994)

3.1.2 Lighting of burner

Before performing any experiments, the system must be completely free of leaks to prevent errors in flow-rate measurements and to maintain a safe working environment. Thorough leak checking including soap water application to every gas line joint is performed whenever the system is reassembled or modified.

Bunsen burner

The chimney is removed from the burner. The air co-flow is turned on first and set to 10 SCFH. The combustion air-flow is then turned on using the shut-off valve and set using the needle valve, monitoring the flow-rate reading on the computer monitor. Once the air-flow rates have been set, the fuel-needle valve is turned to a near (not completely) closed position and the methane gas line shut-off valve is opened. The methane gas flow readout on the computer screen should

be very low. The fuel flow is then slowly increased to an equivalence ratio in the stable burning range, between an equivalence ratio of 0.75 and 1.4. The equivalence ratio is also displayed on the computer screen. When a stable burning mixture has been reached, the flame is ignited using a match. Once the flame is stabilized the chimney is installed back on top of the burner. The air and methane gas flows are adjusted to near the desired operating condition. The system is allowed to warm up for 30 minutes before quantitative measurements are performed. Only after complete warm-up are air and methane gas flows adjusted to within a 0.5% tolerance of the desired operating condition.

Honeycomb burner

The air-flow is turned on first and set at the desired value by adjusting the air rotameter, observing the indicated flow-rate on the computer screen. The methane gas rotameter is adjusted to near its closed position, with the local shut-off valve closed. The methane gas line is then pressurized by opening all shut-off valves upstream of the local shut-off valve. Finally, the local shut-off valve is opened. The methane gas flow is adjusted to a point where the burner can burn in a stable fashion. The stable operating range for the honeycomb burner is between equivalence ratios of 0.6 and 1.4. Once in this region, the burner is lit using a match at the exit of the honeycomb burner chimney. The air and methane gas flows are adjusted to near the desired operating condition. The system is allowed to warm up for 30 minutes before quantitative measurements are performed. Only after complete warm-up are air and methane gas flows adjusted to within a 0.5% tolerance of the desired operating condition.

3.2 General procedure for Chemiluminescence measurements

The present section contains a description of how chemiluminescence measurements are set up, regardless of the particular experiment that is to be performed. The PMT and connected instrumentation amplifier are turned on and allowed to warm up for at least 30 minutes. The monochromator wavelength is set at the desired wavelength (308 nm for OH* and 431 nm for CH*). For spectral scans, the monochromator is set at a wavelength 1 nm smaller than the desired starting wavelength. The monochromator entrance and exit slit-widths are set to their desired values. For global chemiluminescence measurements the slit-width for both entrance and exit is set to $700\mu\text{m}$. For local chemiluminescence measurements both slit-widths are set to $1000\mu\text{m}$. Spectral scans are performed using either slit-width setting.

Once all preparations described above are done and ample warm-up time has been given, the DC-offset of the PMT voltage is zeroed. To perform this task, the LABVIEW program 'flow.vi' is started as described in Section 3.1.1. The output of the instrumentation amplifier is connected to channel 2 of the SC-1000 data acquisition board. The PMT voltage can now be read in the 'flow.vi' program window. The DC-offset adjustment on the instrumentation amplifier is used to zero the voltage displayed.

Finally, all unnecessary lights in the room are turned off and the monochromator slit is opened to its maximum height of 15 mm. The voltage now displayed in the 'flow.vi' program window is the PMT voltage due to the chemiluminescence from the flame.

3.3 Optimization of Optical System

The values given in Table 2.1 of Section 2.4.4 are the various optical system distances as calculated by an analytical model, described in Appendix C. The distances given are a good first guess at the final configuration of the system. A lot of time

must be invested to fine-tune the optical system to a point where the chemiluminescence signal is maximized. The following describes the steps that are taken to arrive at an optimal optic system. Where the procedures differ between local and global chemiluminescence, the procedures are described in separate paragraphs.

3.3.1 Initial lens system alignment

Once the lenses and fiber-optic cable have been arranged at the distances given in Table 2.1 and centered on the burner surface, a flash-light is used to illuminate the monochromator end of the fiber-optic cable. If the system is aligned properly, a spot of the desired size should be centered on the burner.

Global chemiluminescence measurements

In global chemiluminescence measurements, the spot should cover an area about 5% larger than the burner exit area. To change the size of the spot, the most effective tool is changing the distance from the second lens to the entrance of the fiber-optic cable. The distance between the two lenses is fixed in that their holders are bolted to each other. The distance between the burner and the first lens cannot easily be manipulated. Using the vertical adjustment on the fiber-optic cable holder, changing the distance between the last lens and the fiber-optic cable can be easily accomplished. If the light spot is not already centered on the burner surface, the horizontal adjustment on the fiber-optic cable holder is used to complete the initial alignment.

Local chemiluminescence measurements

Local chemiluminescence measurements benefited from better model predictions. It seems that since the lens to lens distance in the global chemiluminescence setup is so short, the predicted distances are more apt to be in error. Corrections in the distances for the local chemiluminescence lens system were very small in all cases. The difficult task for local chemiluminescence measurements is the proper centering of a light spot that is less than 1 mm in diameter. The light spot must be centered for the

traverse to perform a true radial pass through the flame. Centering is accomplished by adjusting the horizontal alignment of the fiber-optic cable holder.

3.3.2 Monochrometer end optical alignment

Once the lens-system has been aligned, the other end of the fiber-optic cable is attached to the collimating-beam lens in front of the monochrometer and the burner is lit according to the procedure described in Section 3.1. To optimize the amount of light transmitted to the diffraction grating, the collimating beam lens must be perfectly aligned with the light path in the monochrometer, i.e. parallel to the length of the monochrometer. The optical mount used for the collimating-beam lens allows for translation (control I) and rotation (control II) of the lens along and about an axis close to parallel to the slit width as well as rotation (control III) about the vertical axis.

Chemiluminescence measurements are started by following the procedure given in Section 3.2 with the wavelength set at 308 nm for OH* chemiluminescence. First, Control I is used to maximize the voltage reading. Next, the procedure is repeated for controls II and III. It may be necessary to repeat the adjustment of the controls several times.

3.3.3 Final alignment

Global chemiluminescence measurements

For global chemiluminescence measurements, some voltage signal increases may be obtained by further changing the distance between the second lens and the fiber-optic cable entrance. Other slight translational and rotational adjustments at the fiber-optic cable holder may also prove to provide slight increases in the maximum voltage collected. The change in voltage observed, is not due to the fact that more of the flame is being captured, rather the increase is due to the fact that more of the light is transmitted into the monochrometer and less is lost at the monochrometer entrance.

Local chemiluminescence measurements

For local chemiluminescence measurements, the alignment procedure is complete once the optimization process at the monochromator end of the fiber-optic cable is accomplished. The only further alignment check possible is to perform a traverse of the flame (as described in Section 3.5 and examine the data. If the distance over which chemiluminescence is detected is smaller than the inside diameter of the stainless steel tube, then the light spot was not properly centered.

3.4 Global Chemiluminescence Measurements

3.4.1 Preparation

Chemiluminescence measurements are performed only when the burner and all its instrumentation (including PMT and instrumentation amplifier) are properly warmed up as described in Section 3.1.2 and Section 3.2. An experimental strategy is also important to prevent transients from affecting the collected data. Large changes in the heat output of the burner are avoided because they lead to long thermal transients in the burner that must have reached steady state before data acquisition can resume.

3.4.2 Data acquisition

Once the burner is ready for measurements to begin, the LABVIEW routine 'flowavg.vi' is opened. The program behaves very similarly to the 'flow.vi' routine mentioned before. The difference is that 'flowavg.vi' has built in code that will allow for voltage offset measurements, data processing and finally data storage on the hard-drive. As with 'flow.vi' all program inputs are checked. The 'flowavg.vi' program is started and the desired experimental condition is set. The following paragraph describes how one global chemiluminescence data point is obtained.

The entrance to the fiber-optic cable is blocked so that light cannot enter the fiber-optic cable. The 'SAVE' button in the program 'flowavg.vi' is pressed to cause the program to save the data being collected. However, since the light into the

fiber-optic cable is blocked, the voltage collected is due to dark-noise and stray light entering the monochrometer only. The program sorts the collected voltages into the 'dark-noise' category. When the block from the entrance to the fiber-optic cable is removed, the voltage immediately increases and the program sorts the collected voltages into the 'light' category. Ten dark-noise voltages are collected, then the light from the flame is allowed to reach the fiber-optic cable and 50 light-on data points are collected. Finally, the fiber-optic cable entrance is once again blocked and ten more dark-noise voltages are collected. When the 'SAVE' button is pressed again, the program averages the dark-noise data separately from the light-on data and finally subtracts the dark-noise average from the light-on average to obtain a voltage solely due to chemiluminescence from the flame. The chemiluminescence voltage is written to a separate array along with the average flow-rates and equivalence ratio during the light-on period. After processing the data, the program returns to displaying the measured voltages without saving the data points.

The procedure just described is repeated for every desired experimental condition. The PMT voltage display helps to verify the end of the heat-up or cool-down transient started with a change in operating condition.

3.4.3 Data post-processing

The only post-processing required for the data saved in the file is the correction discussed in Section 4.2 to account for the fact that the voltage measurement is not directly a measure of all of the OH* or CH* transition energy.

3.5 Local Chemiluminescence Measurements

3.5.1 Preparation

Chemiluminescence measurements are performed only when the burner and all its instrumentation (including PMT and instrumentation amplifier) are properly warmed up as described in Section 3.1.2 and Section 3.2. Local chemiluminescence measure-

ments require the use of an additional PC, running in MS-DOS mode, to control the traversing stepper motor via the parallel port. The monochromator is setup as for global chemiluminescence measurements (see Section 3.2) with the exception that a wider slit-width is used. The optical system is moved radially out from the center of the flame, to a point where the voltage displayed in the 'flow.vi' program is close to that observed when all light into the fiber-optic cable is blocked.

3.5.2 Data acquisition

To perform a traverse through the flame, another modified version of 'flow.vi' was written. The program 'flowavgtime.vi' contains features similar to 'flowavg.vi' in terms of data processing. The program 'flowavgtime.vi' has a timing feature that limits the number of data points to be averaged. In the program 'flowavg.vi', the number of data points to be averaged is user-controlled using the 'SAVE' button and not time-limited. The MS-DOS PC is programmed to move the stage at specified time intervals. The loop-timing in 'flowavgtime.vi' is such that data-collection occurs in the time between movements of the optical system. The coordination between the two PC's is entirely manual. Both programs are started as simultaneously as possible. The timing of the data acquisition program is such that over the course of 70 optical system movements, the slight difference in the starting times does not cause the data acquisition program to collect data during optical system movement.

Once the programs are started, the light to the fiber-optic cable is blocked during a small portion of data-collection time to provide the program with a dark-noise voltage to subtract from the light-on voltage. The program automatically recognizes the data-points representing dark-noise voltage only. The averaging and other processing of the data is as before for 'flowavg.vi'. Once the flame traverse is done, the data is written from memory to a file. The data in the file is corrected, similarly to the correction applied to the global chemiluminescence measurements.(see Section 4.2).

3.6 Spectral Scan Chemiluminescence Measurements

Spectral scan chemiluminescence measurements are used to identify the spectral content of a chemiluminescence signal. At one experimental condition, a whole range of wavelengths is studied. The data acquisition system used for these measurements differs from that used for other chemiluminescence measurements as described in Section 2.5.2. The data acquisition is externally triggered.

The monochromator wavelength is set to a wavelength 1 nm smaller than the beginning of the wavelength range of interest. The speed of the wavelength scan is set to the desired value. Most often, the wavelength scan speed is set to 12.5 nm/min. The program written to collect spectral scan data is called 'spectrum.vi'. The program has several important inputs that need to be correct for the program to work properly. The wavelength range and speed needs to be entered along with the desired wavelength resolution and number of voltages to be averaged for each data point. Typically, the wavelength range studied is 2000 Å, with 400 averages per data point, 1 Å wavelength resolution and a sample frequency of 2000 Hz. The trigger mechanism is set to 'external' for spectral scan chemiluminescence measurements. To perform these measurements, the burner and its instrumentation must be prepared as described in Section 3.1 and Section 3.2.

To begin the data acquisition, the program is started. The program is now waiting for the external trigger. The entrance-slit of the monochromator is opened if it has not already been opened and the wavelength scanning motor is turned on. The wavelength counter begins to turn, indicating the smooth ascent in wavelength. When the counter reaches the lower end of the wavelength of interest, the trigger-button attached to the data acquisition board is pressed, initiating the data collection.

As the data is collected, the data is processed and averaged. The program displays the processed data graphically in a separate window which opens immediately after data collection has begun. When the program has finished, the wavelength scanning motor on the monochromator is turned off. The data file written contains two data points for each resolved wavelength, the mean voltage and the standard

deviation of the measurement. No post-processing of the data contained in the file is necessary other than applying the correction for the optical train efficiency variation with wavelength.