# DEVELOPMENT OF NEW CONTROL SOFTWARE FOR INTEGRATION OF AN ECHELLE GRATING SPECTROMETER FOR USE IN LASER INDUCED BREAKDOWN SPECTROSCOPIC MEASUREMENTS

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## ABSTRACT

A custom instrument control and data acquisition software package has been developed, in cooperation with Corning, Inc. under the DOE Office of Industrial Technologies GPLUS program, to integrate an echelle grating spectrometer into an existing Laser Induced Breakdown Spectroscopy (LIBS) instrument. The LIBS instrument has been and will be used in a variety of projects with relevance to the glass industry. The application of an echelle grating spectrometer to the LIBS instrument introduces the ability to simultaneously collect spectral information for the entire spectral range from the ultraviolet to the near-infrared (200 nm to 900 nm). This will afford the continuous, simultaneous, in situ monitoring of most atomic elements of interest to the glass industry for process characterization and control.

## INTRODUCTION

In this report we describe the development of control software for integration of an echelle grating spectrometer, the Mechelle 7500, into an existing Laser Induced Breakdown Spectroscopy (LIBS) instrument. The development and testing of this software has been carried out in cooperation with Corning, Inc. under the DOE Office of Industrial Technologies GPLUS program.

The key to process and emission characterization and control is the continuous and simultaneous monitoring of all relevant elements in the process or exhaust stream. The unique optical arrangement of an echelle spectrometer enables the simultaneous acquisition of a wide range of wavelengths. {Harrison, 1952 #70; Harrison, 1949 #69}

The motivation for development, in 1949, of the echelle optical configuration was the ever-increasing need for greater spectral resolution and range without the concomitant increase in photographic plate size. The echelle optical arrangement disperses the incident radiation twice. The first dispersion is in a linear fashion common to all diffraction gratings. In this case, the grating orders are not separated, but instead overlap. The second dispersion separates the orders refracting off of the diffraction grating orthogonally to the first dispersion. This produces a two-dimensional image of a large spectral range, typically several hundred nanometers in a relatively small space, e.g. a 2 inch by 20 inch plate. The same wavelength range only dispersed in a linear fashion with same resolution would require a much larger detector, a photographic plate on the order of tens of feet long. The advantages of this optical configuration translate well to the modern use of intensified charge-couple devices (ICCD) as optical detectors, since ICCDs can be compact devices only a few inches on each side. The echelle grating spectrometer combined with an ICCD is the next generation detector for laser-induced breakdown spectroscopy (LIBS). A simple schematic of the optical layout of an echelle grating spectrometer is depicted in Figure 1.

Laser-induced breakdown spectroscopy, also referred to as laser-induced plasma spectroscopy, dates to the 1960s. {Cremers, 1987 #72; Adrain, 1984 #71} The LIBS technique utilizes a small (typically <1 mm diameter) laser-induced plasma to dissociate all molecules and fine particles within the highly energetic plasma volume. Continuum emission (bremsstrahlung and electron-ion recombination) and atomic line emission are observed from the ensuing plasma emission. The atomic emission lines may be resolved both spectrally and temporally, enabling identification and quantification of the elements present. The LIBS technique has been applied to the analysis of solids, liquids, and gases, and a number of literature reviews are available that cover a wide range of LIBS-based analysis {Cremers, 1987 #72; Adrain, 1984 #71; Martin, 1999 #25}

The Mechelle 7500 spectrometer is shown in Figure 2. The main two components are the dispersing optics housed in the red section and the mated ICCD within the white section. Because the design of the spectrometer precludes the need for any moving parts, the spectrometer is especially well suited for surviving field tests. The atomic emission from

the laser-induced plasma is introduced into the spectrometer via a fiber optic cable (not shown).

The control software that originally shipped with the Mechelle 7500 did contain a number of useful features, but was not configurable to adapt to the varied reality of field tests in which Sandia's LIBS instrument has been and will be applied.

The main features missing from the commercial software were the ability to scale the spectrograph during data collection, in order to more closely observe key spectral features, and the ability to easily operate the data acquisition software in a continuous fashion while displaying real-time data. Additional desirable features include qualitative and quantitative analysis capabilities that were only offered in an off-line fashion.

During our software development work the spectrometer manufacturer was in the process of generating developmental libraries for use in the LabVIEW programming environment. This made our effort possible, but also tied our progress to their pace and stage of development.

The front panel of the new control software is shown in Figure 3. The software developed under this program is a significant improvement beyond the original commercial software, but still is restricted by the state of driver development from the manufacturer.

Some of the features that we implemented in this version include: automated file naming and saving, front panel display and control of timing functions, and cursor bars that permit marking and monitoring.

Ongoing development will continue to refine the control software to fit the niche requirements of a field test oriented instrument.

## REFERENCES

Cremers, D. A., and Radziemski, L. J., "Laser Plasmas for Chemical Analysis," in *Laser Spectroscopy and Its Applications*, Radziemski, L. J., Solarz, R. W., and Paisner, J. A., Eds. (Marcel Dekker, New York, 1987), Chap. 5.

Adrain, R. S. and Watson, J., J. Phys. D: Appl. Phys. **17**, 1915 (1984).

Hahn, D.W., Carranza, J.E., Arsenault, G.R., Johnsen, H.A., Hencken, K.R. " Aerosol Generation System for Calibration of a Laser-Induced Breakdown Spectroscopy Instrument"

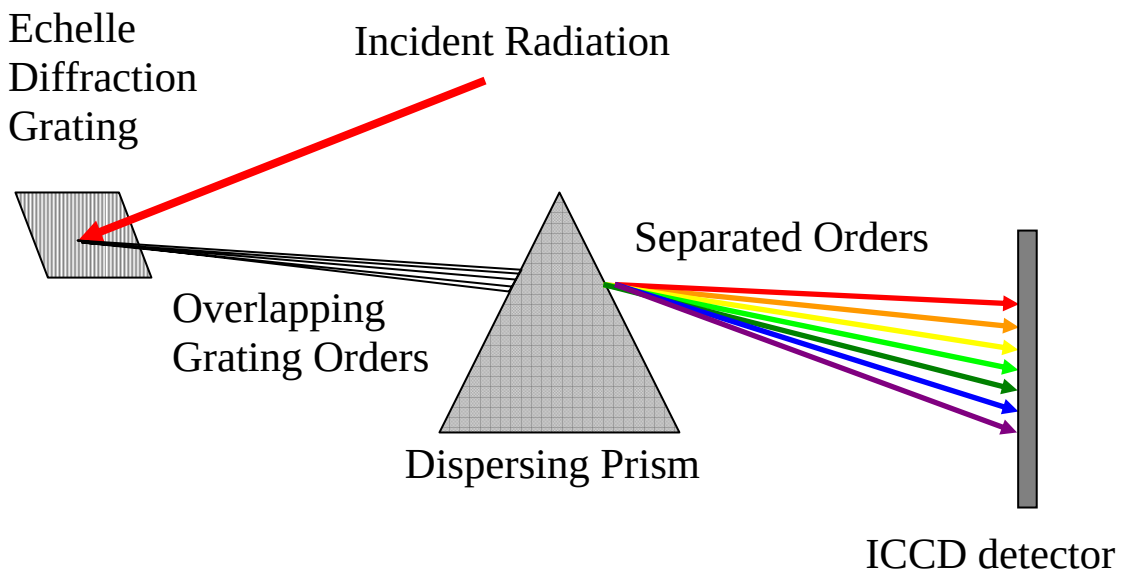


Figure 1. Schematic of echelle grating dispersive optics

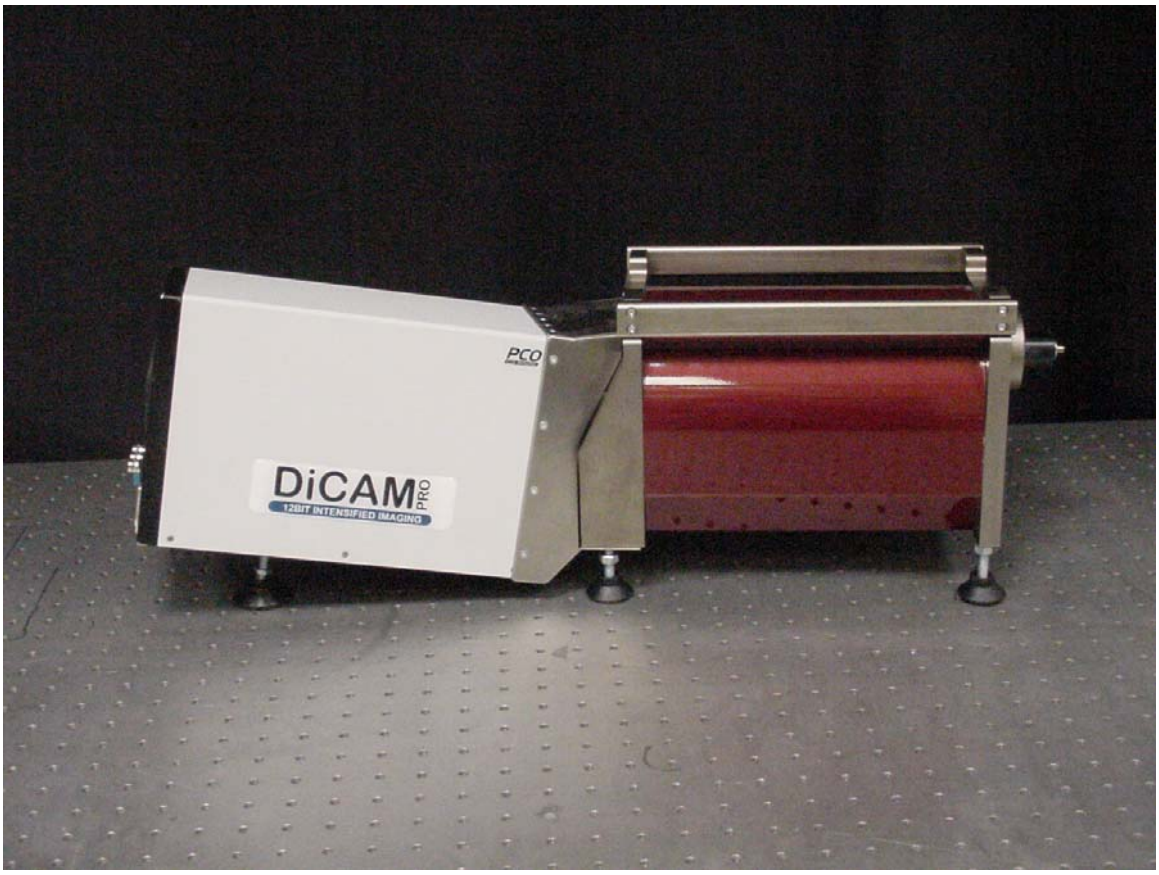


Figure 2. The Mechelle 7500 echelle grating spectrometer with DiCAM Pro ICCD.

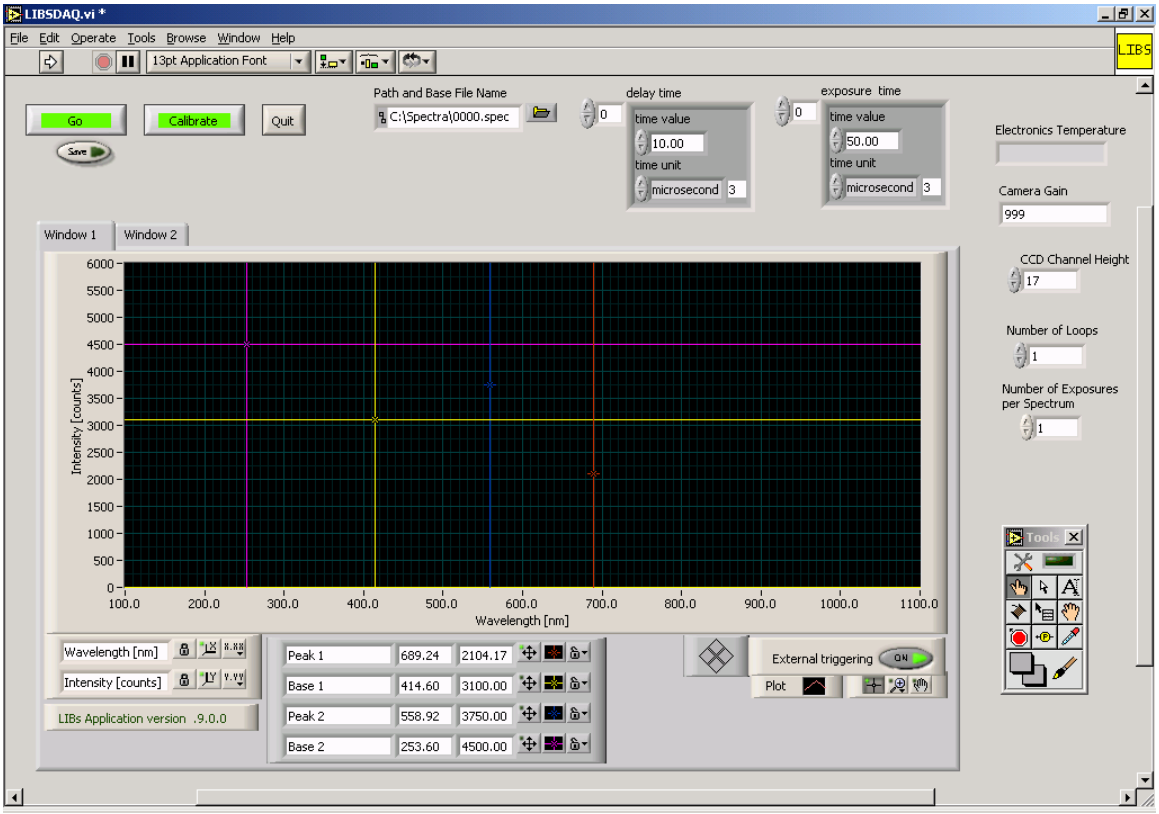


Figure 3. Image of front panel of LIBSDAQ.vi user interface.

# Appendix I:

# User Guide and Programming Instructions

LIBS Data Acquisition User Manual and Programming Guide
Version 1.0
Date: 9/19/2002
Author: Robert Steinhaus

**Program Overview:**

The following is a user's guide for the LIBS Data Acquisition software package. The purpose of this software package is to provide a method to allow a user to acquire by personal computer Laser Induced Breakdown Spectral Information from a Multichannel Instruments Mechelle 7500 Spectrometer. This package is designed to assist users making laboratory and field measurements using Laser Induced Breakdown Spectroscopy hereafter referred to as LIBS.

The software package consists of a number of LabVIEW modules that run on top of a LabVIEW advanced programming interface (API vO.3.3) provided by Multichannel Instruments of Skarpnack, Sweden. The API provides basic low-level camera/spectrometer routines known as sub-virtual instruments (or sub Vis) that are called by the LIBS data acquisition (LIBSDAQ.vi) program to obtain spectral information from the Mechelle 7500 spectrometer. The LIBS data acquisition program is modular in structure and is composed of one top-level virtual instrument (LIBSDAQ.vi) and 52 supporting subVis. Organizing the code into virtual instruments (subVis) tends to make the program modular and more easily modifiable.

**Required Files and Software:**

The LIBSDAQ program is supplied as a top level virtual instrument (LIBDAQ.vi) and a library of support subVis (LIBSDAQ.llb). The application and library virtual instruments are compiled under LabVIEW version 6.1 and LabVIEW 6.1 (or later) is required on your computer in order to run the application. Run time versions of the application can be generated which permit LIBSDAQ to be run on personal computers which do not have a LabVIEW license but such runtime versions of LIBSDAQ can not be altered or modified on the target non-licensed machine. In addition to the required LabVIEW system, application virtual instruments, and library subVis, an additional calibration file (typically generated by Mechelle software version 1.61) is required. The calibration file needed is caldic80.dat and it permits the display of wavelength-calibrated spectra on the LIBSDAQ.vi front panel spectrum graph. The file, caldic80.dat, needs to be placed in the correct folder specified later in this document under the topic of Calibration in order to be correctly referenced when wavelength correcting LIBSDAQ spectrum graphs.

**Required Hardware:**

LabVIEW, the language in which the LLBSDAQ application is written, is available on a variety of computers including computers running Windows, Apple Macintosh OS, Linux, HP-UX, and SUN OS. High-level programs written in LabVIEW can be ported from one LabVIEW platform and executed on another LabVIEW platform with excellent transportability. Unfortunately, the Multichannel Instruments LabVIEW API subVis are

largely coded in a Intel X86 “C/C++” programming language and these special “Code Vis” will not successfully port to Macintosh or UNIX hosts. You will need to acquire an Intel/Windows based personal computer capable of supporting the current version of LabVIEW in order to run the LLBSDAQ data acquisition application. Recommended hardware requirements to run LabVIEW have evolved over the years. The current suggested minimum hardware configuration that could be used to run LabVIEW is

Windows NT/2000/XP/9X LabVIEW Minimum Hardware Requirements

- 32 MB RAM minimum, 64 MB recommended
- 65 MB disk space for minimal LabVIEW installation, 200 MB for full installation
- Pentium processor or equivalent recommended

An up to date suggestion for current minimum system configuration to support Labview and LabVIEW Applications can be found through the National Instruments support webpage

http://www.ni.com

For any LIBS system, more memory, a faster processor, and larger amounts of local fast storage will certainly provide benefits to the LIBS software. In general, LIBS requires a large amount of data to be accessed, processed, and saved. For these reasons, it is recommended that the LIBS control PC be Windows 98 or above with at least 128MB of RAM, a minimum of a Pentium PII class processor, and a hard drive of at least 10GB. A method for off-line data storage and data archiving (tape, CD-R drive, or DVD-R drive) should be strongly considered, as a typical LIBS collection routine will store well over 30MB of data per day.

The hardware interface to the Mechelle Spectrometer is currently a PCI bus card manufactured by PCO GmBH in Germany. Your computer will need to support PCI cards and include at least one PCI slot to plug in and use the camera interface card. It is essential that the PCI slot chosen for installation of the camera interface PCI card be a Master PCI slot. The PCO camera interface card operates as a PCI Bus Master. This simply means that the PCO camera interface card can take control of the PCI bus, in order to transfer image data to and from the computer’s RAM and the Mechelle Spectrometer. Multiple bus masters can coexist in a single system, as long as they leave enough time for one another to operate; this is why it is sometimes necessary to scale back video or SCSI acceleration on some systems when installing a PCI bus mastering interface card like the PCO camera interface.

The PCO camera interface will only work in a bus master slot; fortunately, most recent

computers running both Mac OS and Windows support bus master cards in all slots.

On some early Windows-compatible motherboards, not all PCI slots supported PCI bus mastering. On these motherboards, it is necessary to make sure that the PCO camera interface PCI card is in a bus master slot; if the slot does not support bus mastering, the PCO camera interface board will not work at all.

Modem Windows-compatible motherboards generally support bus masters in all PCI slots, so you should be able to place the PCO PCI camera interface board in any PCI slot.

**Installation:**

The LIBSDAQ program is built on the Mechelle LabVIEW API. It is recommended that you first obtain a current copy of the Multichannel Instruments LabVIEW API for the Model 7500 Spectrometer before you install the LIBSDAQ software. The Multichannel Instruments LabVIEW API can be found on the Multichannel Instruments Web Site

http://www.multichannel.se

under a subheading of “Mechelle/Software”. The Multichannel Instruments LabVIEW API installer will create a folder

C:\…\Multichannel Instruments\LabVIEW MIAB API\labview

And it is recommended that you install the LIBSDAQ.vi virtual instrument and the LIBSDAQ.llb library into this folder. The calibration file caldic80.dat created while doing calibrations using the Mechelle vi .61 spectrometer support software should be placed into the following location so as to be read by the LIBSDAQ software and used to correct the wavelength calibration of LIBSDAQ front panel spectrum graphs.

C:\…\Multichannel Instruments\Lab VIEW MIAB API\instrument\SensiCam

**Calibration:**

LIBSDAQ does not yet have its own wavelength calibration facilities. The Mechelle 7500 tends to slowly drift in its wavelength calibration as a function of time. To take accurate wavelength corrected spectra with LLBSDAQ is possible but currently requires help from the Mechelle vi.6 camera software. Mechelle vl.6 camera software contains a calibration facility and as part of its function writes an instrument calibration file named caldic80.dat out to the hard disk. LIBSDAQ can use the calibration file (caldic80.dat) created by the Mechelle vi .6 software to wavelength correct all of the spectra it acquires. LLBSDAQ needs to find a copy of the caldic80.dat file in the

C :\…\Multichannel Instruments\Lab VIEW MIAB API\instrument\SensiCam

Directory created when the MIAB LabVIEW API installer installs the LabVIEW API. If it is necessary to update the calibration it is necessary to completely exit and close the

LLBSDAQ program and start the Mechelle vl.61 camera software (LIBSDAQ and Mechelle camera software can not be in memory at the same time as they each try to acquire the PCO camera interface card exclusively as a resource and will not share the camera interface card). Using Mechelle camera software v1.61 you can run a calibration and generate an updated caldic80.dat calibration file. You must close the Mechelle camera software v1.61 and copy the calibration file to the

C:\…\Multichannel Instruments\LabVIEW MIAB API\instrument\SensiCam

directory in order for the newly acquired LLBSDAQ spectra to be wavelength corrected.

The MIAB LabVIEW API vO.3.3 does have a variety of subVi primitives for performing calibration in the LabVIEW environment. At some point LIBSDAQ may be improved to do its own calibrations of the Mechelle 7500 camera but at this point it must rely on the calibration facilities built into Mechelle camera software v 1.61.

**Operating the LIBSDAQ Application/Front Panel Controls:**

Operating the LIBSDAQ application generally involves first entering desired spectrometer measurement parameters into front panel controls and then starting the LJBSDAQ application by pressing the “Run Arrow” button located in the extreme upper left corner of the LabVIEW Application (typically right below the EDIT pull down menu item on the LabVIEW user interface). An annotated description of the front panel controls into which parameters must be entered to effect correct operation of the LIBSDAQ program follows.

**Controls:**

LIBSDAQ front panel controls orchestrate the entire flow of LIES data collection. (Controls are described on the LIBSDAQ front panel roughly in a top to bottom and left to right order):

[Go/Measuring Button] The Go/Measuring Button normally displays “Go” on the button legend when the application is waiting to be commanded to take data. Pressing the Go/Measure button causes camera data acquisition to start and the button legend to change to “Measuring...“ while the measurement takes place. When the measurement completes the button returns to the “Go” position.

[Save Button] The save button works in conjunction with the Go/Measure button and causes data acquisition spectrum measurement files to be saved to disk when pressed and illuminated with the button’s green LED color.

[Quit Button] The quit button causes spectrometer data acquisition to halt. If the quit button is depressed while a looped data acquisition measurement is taking place the loop completes the current loop iteration taking however many exposures were entered in the”

Number of Exposures per Spectrum” control and then terminates.

[Path and Base File Name Control] The Path and Base File Name string entry control allows a user to specify the path and base file name to be used when recording a spectrum file to disk. In the case of a looped measurement the base file name is only the name of the first spectrum file written to disk. If the loop controls specify more than one data collection loop to be performed subsequent spectrum file names will iterate the final digit of the base file name entered in the Path and Base File Name Control. Initially, the path and base file name control initializes to the following specification

C:\Spectra\0000.spec

You should make sure to actually create on disk a folder with the specified value filled in to the path and file name control. LIBSDAQ will generate errors if it cannot find on disk the path and directory entered in the Path and Base File Name Control.

[Delay Time Control] The Delay Time Control allows a user to enter timing information to be used when making a LIBSDAQ measurement. The user should select a time value and a time unit value to apply to the current data acquisition measurement. The delay time control specifies the time the application will wait after the edge of a camera trigger signal to the TRIG BNC input of the PCO DiCam Pro camera integrated into the Mechelle 7500 spectrometer before starting to take an exposure. Timing units can be from milliseconds to nanoseconds depending on the needs of the experimenter.

[Exposure Time Control] The Exposure Time Control sets the amount of time the shutter on the DiCam Pro camera is open. The time for the shutter to be open to make the exposure should be placed in the time value control. Units should be entered in the time unit control. The exposure time can be varied from milliseconds to nanoseconds depending on the needs of the experimenter.

[Electronics Temperature Indicator] The Electronics Temperature Indicator displays the temperature of the camera electronics integrated into the model 7500 spectrometer. The temperature of the CCD incorporated into the Mechelle 7500 spectrometer is not available to a user through the LIBSDAQ application because the MIAB LabVIEW API does not support this temperature measurement.

[Camera Gain] Sets the analog electronic camera gain (typically from 50 to 999) to match the analog gain of the camera to the light intensity of the measurement.

[CCD Channel Height] specifies the “binning” used for integrating signal on the CCD array, the possible values range from 1 to 31. The intensity of the pixels in several rows for one channel are summed to form the intensity of one spectral channel.

[Number of Loops] This numerical control is used to set the number of loops requested for a measurement (data acquisition). Normally, Values that are legitimate entries for this control are positive values ranging from 1 to $2^{32}$ or 4,294,967,296.

[Number of Exposure per Spectrum] This numerical control is used to set the number of exposures to sum in the camera before displaying and storing (if SAVE button is selected) files to disk. Because of artificial limits in MIAB LabVIEW API v0.3.3 the legitimate entries for this control range from 1 to 250. Future releases of the MIAB LabVIEW API will probably remove this artificial restriction and will permit much larger numbers of exposures per spectrum to be accumulated in the camera.

[External Triggering] The external triggering button is located just to the right and below the Spectrum graph. This button controls the triggering mode of the mechelle spectrometer. The default triggering mode is external trigger. This triggering mode requires a rising TTL signal pulse be applied to the TRIG input BNC connector on the back of the Mechelle spectrometer.

[Wavelength Cursor Controls] Four wavelength cursor numerical controls are provided below the Spectrum Graph for allowing an operator to specify the location of up to four cursors on the Spectrum Graph display to help identify features displayed on the Spectrum Graphs. Entries in the wavelength cursor controls are organized in three fields. These fields are (from left to right) Name Label, x-position (wavelength), and y-position (intensity).

**Programming Information:** Revising and Extending the LIBSDAQ Program

LIBSDAQ at the time of writing of this manual is incomplete. Several important capabilities have been left out of the current program. These capabilities include calibration, longer term integrated exposures (>250 exposures/spectrum), overlay of elemental spectra, and data analysis. The LIBSDAQ program has been written in such a way as to permit adding functions and capability (the program is written in a modular fashion employing multiple levels of sub Vis). New releases of the MIAB LabVIEW API should be relatively easy to incorporate into future releases of LIBDAQ (the current MIAB LabVIEW API is v0.3.3 is an experimental version). The following notes may be of help as a suggestion for how to integrate new releases of the MIAB LabVIEW API into LLBSDAQ.

Integration of New Releases of MIAB LabVIEW API

LabVIEW is currently a compiled language and the MIAB LabVIEW API and LIBSDAQ code typically have to be recompiled when the code changes or is moved to a new location or platform. Since LIBSDAQ sits on top of the MIAB LabVIEW API any changes to the API can have impacts on the viability and operation of LIBSDAQ. Incremental releases of the MIAB LabVIEW API have tended to retain the same essential structure and sub Vi names. When receiving a new version of the MIAB LabVIEW API you should make an effort to determine what has changed in the new release. Changes in the “C++” code in the internals of the API subVis causes no problem but changes to the connector pane or the organization and naming of the subVis will give rise to problems when compiling LIBSDAQ. If there are changes to the connector pane of subVis you will

typically see “broken wires” in the LIBSDAQ code built on top of the API subVis. Parts of LIBSDAQ may have to be recoded if the changes to the Sub Vis and their connector panes are too extensive. If new API subVis have the same name and connector pattern you may substitute the new API sub Vis for the old API subVis and sub Vi libraries and the system will automatically detect the presence of the updated subVis and recompile and relink the LIBSDAQ application.

**Updating the LIBSDAQ front panel/user interface**

LIBSDAQ includes a tab selectable paging user interface. It is intended that at some point LIBSDAQ be upgraded to include tab selected pages with customized spectrum graphs optimized for each of the various elements of interest. At present two tab selectable pages are defined but additional pages can be easily added (use the “add element” to the front panel tab control). Additional code will need to be added to the diagram of LIBSDAQ to support the additional tab selected spectrum graphs and pages.

**Current LIBSDAQ Documentation**

A copy of the LabVIEW diagrams of the current implementation of LIBSDAQ is provided in an appendix to this manual. All of LIBSDAQ may be examined and modified. Some of the MIAB LabVIEW API is diagram protected and can not be altered by end users.

Password protected diagram LabVIEW Code can be recompiled when new versions of LabVIEW come out. The LIBSDAQ application with password-protected subVis will be automatically updated and run under the new LabVIEW systems. Machine-readable source code and a copy of the current MIAB LabVIEW API is available on a CD included at the back of the LIBS Data Acquisition User Manual.