Industrial Monitor and Control Corporation

Imacc Cross-Stack
FTIR Monitoring System

The Imacc Cross-Stack Monitor

The Imacc cross stack system was developed to monitor industrial stacks and ducts in-situ. Cross-stack monitoring has the advantage that the sample is never touched, as it is in an extractive monitor, so there is no question of the analysis system modifying mix. Cross-stack monitoring is ideal for monitoring gases like SO_3, H_2SO_4, HF, etc. which have serious loss or equilibrium problems in extraction lines. A photograph of the Imacc monitor on an industrial incinerator is shown in Figure 1. The unit is coupled to the duct in the rear as shown in the schematic of Figure 2 which shows the beam path within the instrument. The infrared light source is in the FTIR base unit at the top of the figure. Light from this source is modulated by the FTIR and sent to the accessory as a 2.0 inch diameter beam. The accessory has a beam splitter that allows half-of the light to pass through and get collimated by the optics shown at the bottom of the accessory. This collimated light is sent across the duct/stack where it hits a retro-reflecting mirror on the far side. The light returning from the retro mirror reenters the accessory, bounces off the beam splitter, and is focused onto the detector. In this way, the light double passes the duct providing a path twice the duct/stack dimension. A variation on this configuration, is the fiber-optic linked probe shown in Figure 3. This probe has a small beam path so it is appropriate for detection of high concentration gases. It was actually developed for detection of lower explosive limits (LELs) in a manufacturing process.

In what follows the basic Imacc FTIR system is described as is the software which allows for fully automated operation. The software also allows for control of external devices such as valves or pumps, so the FTIR system alone can provide automated sample selection, daily background generations, and even automated calibration checks.

The FTIR modulator itself is built for Imacc by Nicolet Instruments and we custom integrate it into our monitor. The modulator is typically used at a true 0.5 cm^{-1} resolution *although it is capable of providing resolutions as high as 0.125 cm^{-1} for those applications where higher resolution is required to isolate compounds in complex mixtures*. The instrument is controlled and data reduced by a Pentium computer, optionally supplied with the instrument. All software is MS Windows® based for ease of

Figure 1 Imacc Cross-stack FTIR on an industrial incinerator in Europe.

use although we can interface to other programs like Wonderware or LabView. The control and display software packages were custom developed by Imacc to make operation easy even for non-technical people.

Of particular importance are the analytical methods incorporated into our system. These are standard equipment on all Imacc systems. Our FTIR uses Classical Least Squares (CLS) for analysis of spectra, as do many other FTIR systems. However, Imacc has made substantial enhancements to the basic CLS routines allowing for dynamic selection of references to keep up with process or ambient air changes, automatic correction for non-linearity, dynamic line shift correction (correcting for sample turbulance), and automatic correction of interferences caused by non-analyte gases. *These algorithms consequently provide the best stability and highest accuracy of any system we are aware of on the market today.* It is because of the instrument stability and the rigor of the analysis methods that we do not require the acquisition or subtraction of background spectra to properly quantitate data, as required by many other FTIR systems. The instrument is also stable enough that the I_o spectrum (background instrument response spectrum) needs to be generated only if the instrument has sustained rough treatment in shipping or if the system optics are changed in some way. If it is required by regulation, generation of I_o can be automated and synthetic I_o spectra (noiseless mathematical I_o) can be generated directly by the software from any single beam spectrum at prescribed intervals.

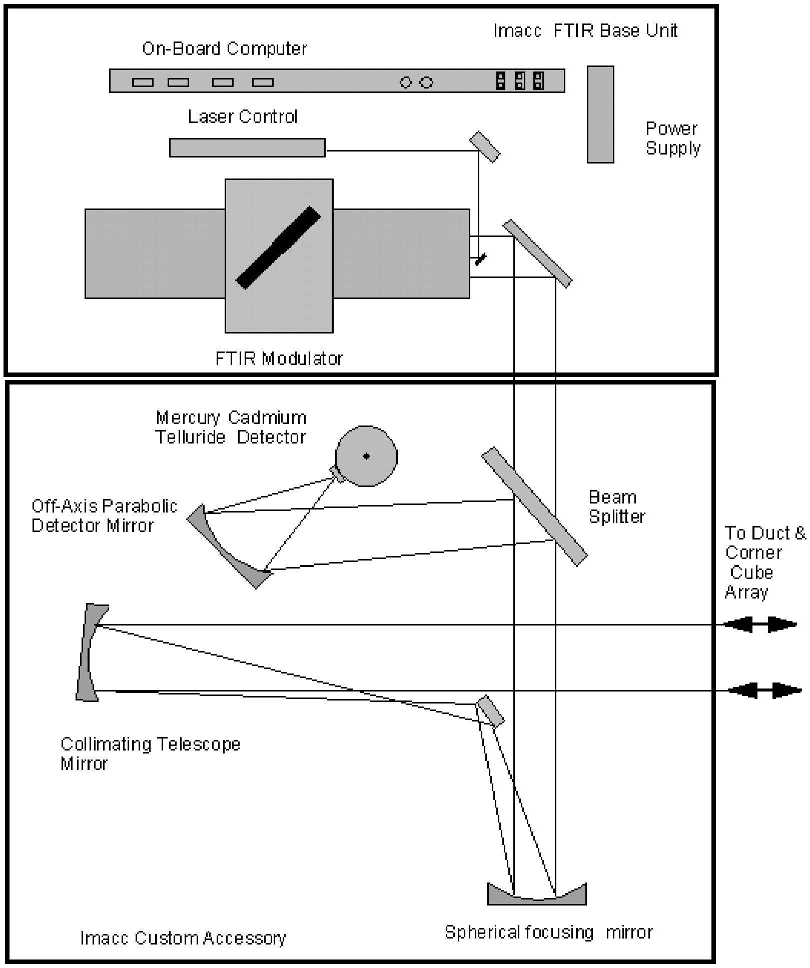


Figure 2 Schematic of beam path in Imacc Cross-stack monitoring system.

Figure 3 Imacc fiber-optic linked probe for monitoring in industrial ducts/stacks.

The unique combination of rugged hardware and sophisticated software allows our systems to be used "unattended" for months at a time while also providing trouble-free operation in manned tests.

Critical Analytical Issues for FTIR

The software incorporated into the Imacc system is unique in the industry. Imacc developed specialized software algorithms that eliminate essentially all of the problematic issues with field FTIR units. The main issues, mentioned above, are: non-linearity, spectral line shift (especially in open-path systems), and interferences by other compounds. Each of these are discussed below.

Non-linearity arises from two sources the infrared detector and the finite resolution of the instrument. The detector non-linearity (only a problem with cooled quantum detectors and not DTGS detectors) has been resolved through linearization circuits built into the detector preamplifier. The non-linearity caused by finite resolution must be corrected in software. This has been done in the Imacc system with algorithms that automatically analyze a training set of spectra, spanning the range of concentrations of concern, and produce linearity correction factors which are incorporated into the compiled analysis routines. An example of linearization of Acetaldehyde, over a broad range of concentrations encountered in an industrial process stream, is shown in Figures 4 and 5. Note that the algorithms corrected for both offset (dc bias) and linearity. Linearity corrections, if ignored, can cause factor of two to five errors in many compound concentrations, they are significant.

Line shift is a hardware issue. If the FTIR system is not highly stable *every* spectrum gathered will have small wavelength shifts. In addition, atmospheric turbulance causes the IR beam to be displaced and this in itself causes some line shift. As shown in Figure 6, line shift can cause large errors in the analysis routines. These arise because the collected spectra no longer match the reference standards. As demonstrated in Figure 6, a shift of $1/10^{th}$ of a resolution element can cause an error of 8% in the gas concentration results. In the Imacc systems, this is all but eliminated by the dynamic alignment systems in the FTIR. However, to assure that shift can never be a problem, the Imacc software corrects for line shift to better than $1/100^{th}$ of the instrument resolution after each spectrum is collected and before it is analyzed. These correction algorithms totally eliminate quantification problems due to line shift. It should be noted however, that this shift is not just a moving of the spectrum in wavenumbers but a true shift of the spectrum relative to the fixed digital grid specified by the laser controlling the system. What this means is that the lines must all change shape depending on how they fall in the digital grid. This is shown in Figure 7, where the spectrum on the bottom has been shifted by about 0.1 cm^{-1} resulting in the trapezoidal lines becoming triangular. If this type of line shape is not done, the measured spectra will never match the spectra in the reference library.

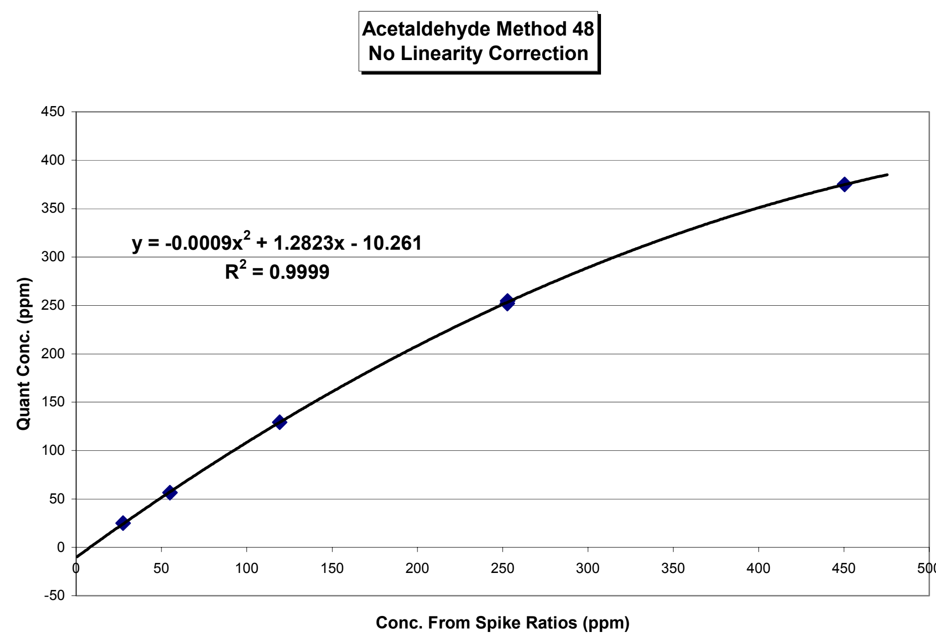


Figure 4 FTIR response for Acetaldehyde before linearization. The low level concentrations near normal process values were linear but the very high values deviated from linearity. The intercept is also non-zero.

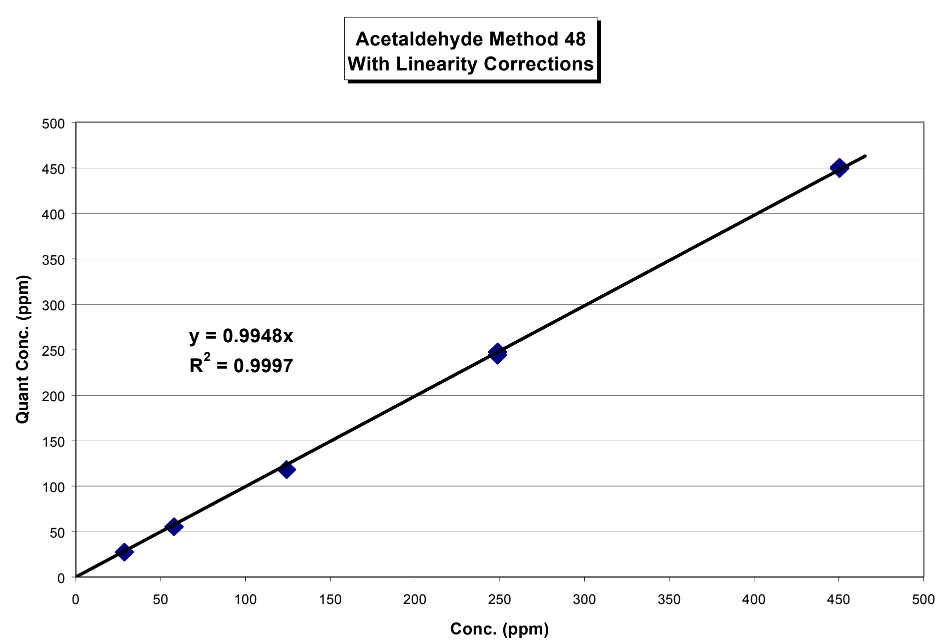


Figure 5 FTIR response after linearization with the Imacc software. The system response is linear throughout and has zero intercept.

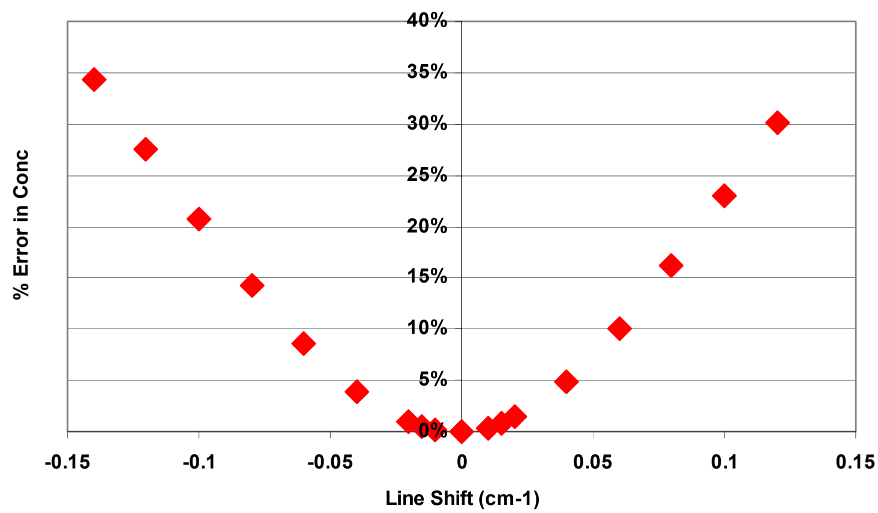


Figure 6 Percent error in gas concentration with small line shifts in the spectra. (Resolution 0.5 cm^{-1})

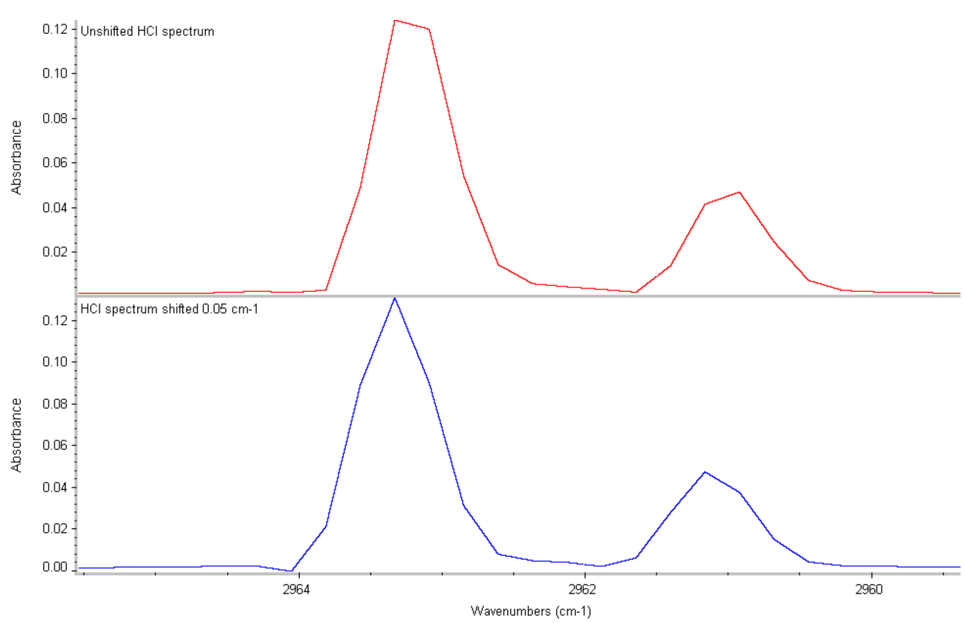


Figure 7 Example of line shift showing the alteration of line shape as required by the fixed digital grid of the laser.

The last issue, interference, arises when other compounds absorb infrared light at the same wavelength as an analyte. This can cause bias on the concentration results for the analyte particularly when the interfering gas is at high concentration. The software in the Imacc system corrects for these interferences by again using a training set (typically blank samples containing only the interfering compounds and none of the analyte). The software analyzes the training set and automatically produces bias correction factors for the full range of interfering concentrations. These corrections are then incorporated into the analysis routines guaranteeing bias free concentration results for all data. All correction procedures are a fundamental property of the analysis method and not of the FTIR itself. Consequently, as long as the FTIR is not modified by changing optics, beam splitter, or the detector these functions do not need regeneration or revalidation once they have been set up initially.

Imacc Software Suite

There are three software packages, included with the instrument, that comprise the user interface to the instrument and its data. These are Imacc Script Editor (the control software), Imacc Monitor (the display software), and Imacc Quantify (the analytical method generator). There are also other tools provided such as a synthetic Io generator, a Batch Reprocessor, and a Spectral Averager.

The script editor is a simple "drag and drop" package that allows the user to set up sequences of events for the FTIR. Objects are simply dropped into the window in the order in which they are to be executed. Typical objects are FTIR data acquisition, valve control, or background collection. One can set up loops that can be continuous or started and stopped based upon time or external events. A simple loop is shown in Figure 8. This loop will collect and process data continuously until 23:30:00, at which time processing stops, valve 1 is closed, valve 2 is opened, and a background spectrum is collected. After this is complete, valve 2 is closed, valve 3 re-opened, and collections continues (looping back to the top). Various scripts can be created and saved so they can be opened and executed at will.

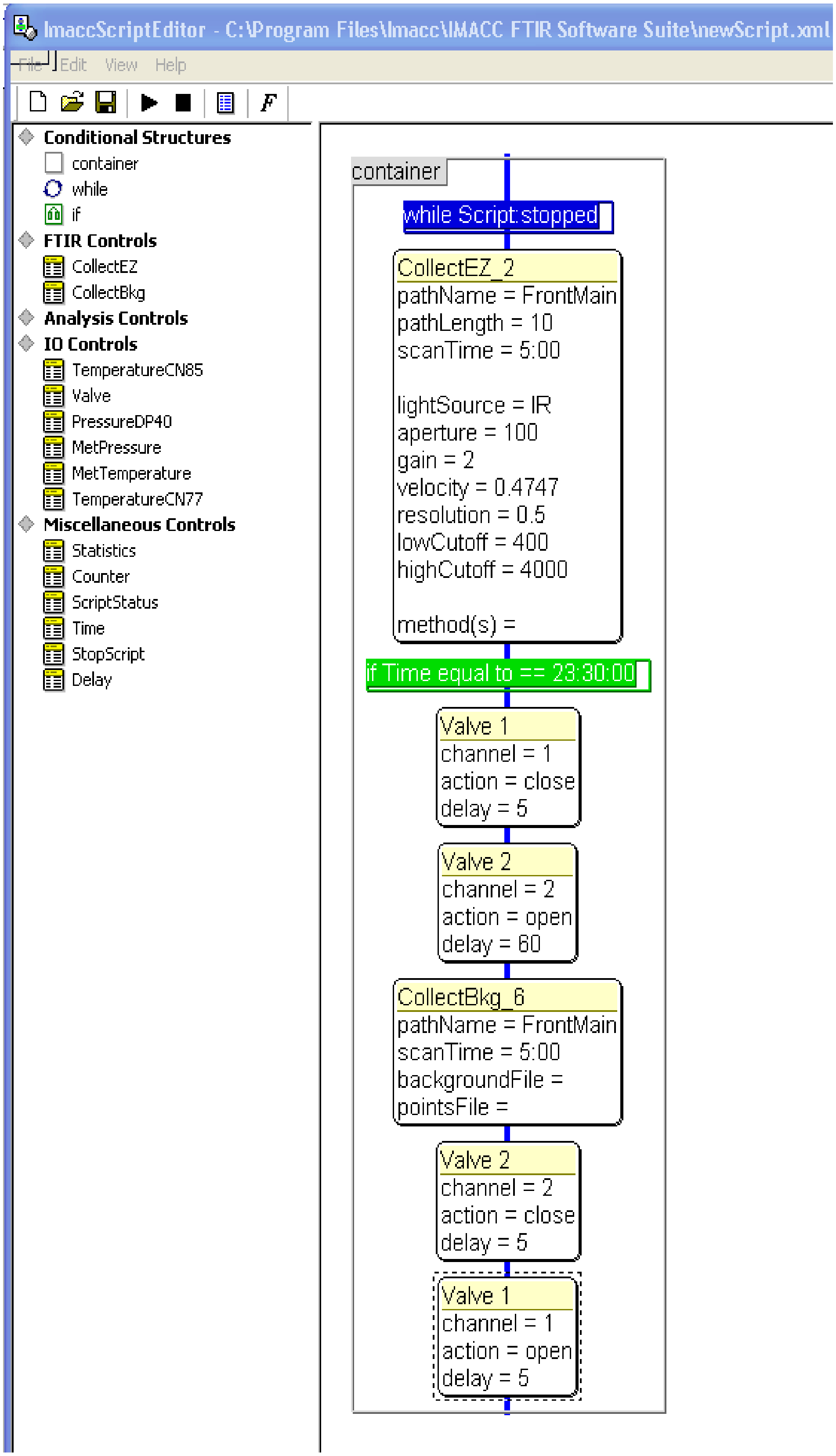


Figure 8 Imacc script set up to collect, perform valve control, and collect background.

The second package is Imacc Monitor. This is used to display data or spectra during real time acquisition or after the fact. The monitor typically opens and automatically runs a specified script when it is started. The monitor allows for custom windows to be created by the user that can graphically display spectra or interferograms, as well as plotted data and tabular lists of diagnostics or gas concentrations. It is very versatile so users can customize the display to their needs. The layout for the monitor is also saved so multiple display layouts can be set up and simply opened to configure the screen for a particular application. A typical Monitor display is shown in Figure 9. Here the top most window has buttons for loading, starting, and stopping the script. The next window has two panes. The one on the left displays gas concentration data along with the analysis (2 σ) errors. The window on the right has selected diagnostic parameters displayed. In this case these are the current system status, the percent complete for the current averaging interval, the number of scans complete, the number of “batches” or averaging intervals of data produced, the number of good scans in the last averaging interval, the number of bad scans in this interval (if any), the averaged pressure and temperature for the last completed measurement interval, and the peak interferograms voltages. The lower window was set up as a tab window with two tabs. One tab brings up plots of the gas concentrations and the tab selected here shows spectra. In this case the single beam and absorbance spectra are displayed. In all windows, a right mouse click brings up configuration menus that allow the user to select the type of plot/tabulation displayed and the parameters or data to be shown. Once the monitor is set up as desired and linked to a script, all that is required to start data acquisition, processing, and display is to start the monitor.

The final package is Imacc Quant setup. This is the package used to create new analysis routines and to test them. Once a analysis method is developed it can be used by Imacc Script or Imacc Monitor to analyze spectra. The Imacc quant software has several powerful features including spectral line shift correction and linearity correction, which were discussed above. It also allows the user to set up windows within analysis regions to avoid interferences from other compounds or to skip highly absorbing areas. The software also has dynamic reference selection. This means the software actually changes itself to keep up with process/sample changes minimizing the errors in the analysis process. Imacc

The multiple functions in Quant Setup are selected by choosing the appropriate tab in the main window. Figures 10, 11, and 12 show three of the tabs. Figure 10 is the window used to define analytes and set up references for each of them. This window is also used to define the regions and windows to be used for analysis. On the right the sample spectrum is displayed on the bottom and a chosen reference on top. The windows and regions used for analysis are then input by choosing them graphically as shown by the highlighted region for CO. Spectral residuals (indicative of errors) can also be shown in this window to assess how well the analysis is doing at matching all the compounds in the region. Figure 11 is the linearity window. Here sets of reference spectra, spanning a concentration range, are chosen to test the method for linearity and to generate a function

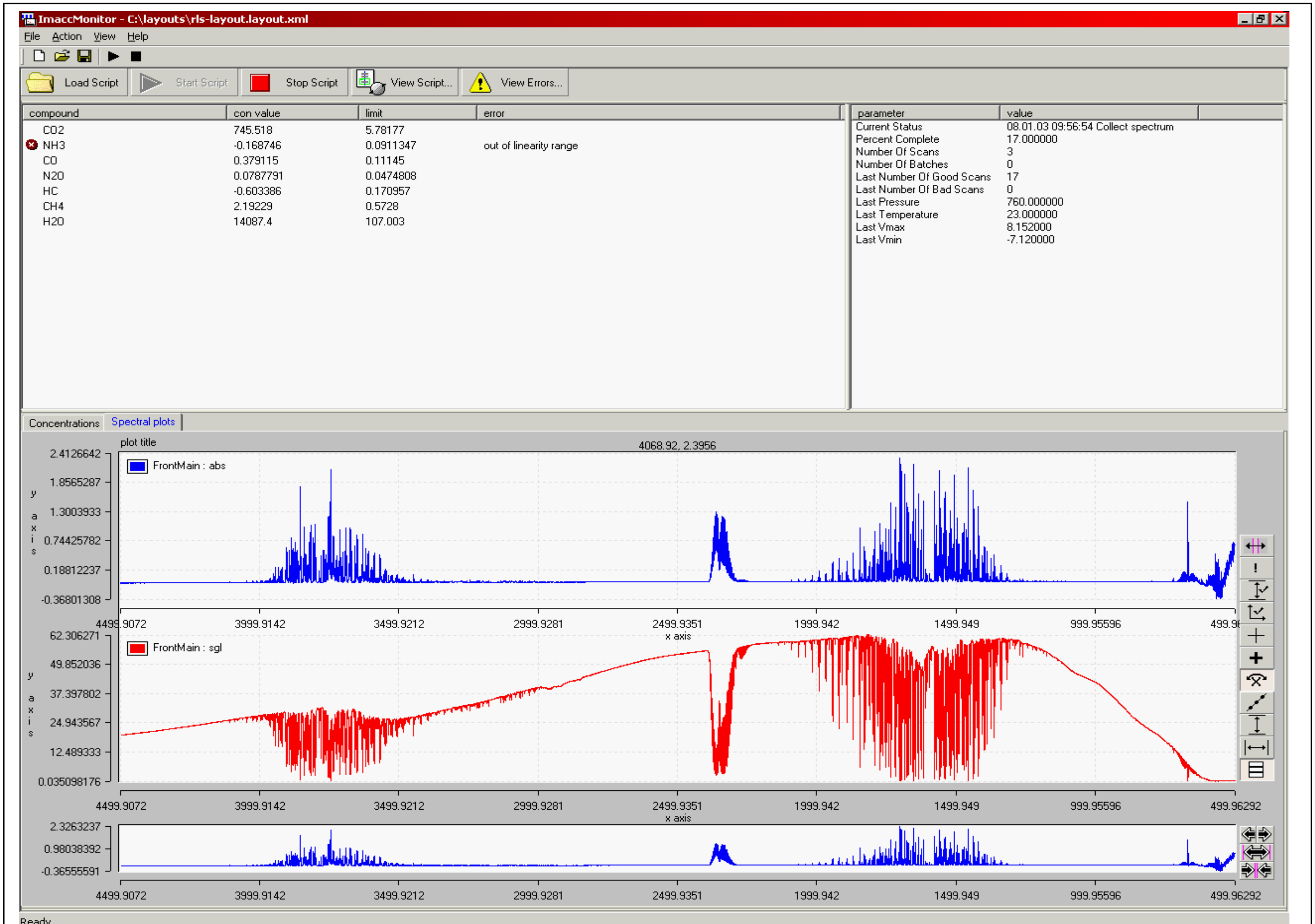


Figure 9 Typical layout created by the Imacc Monitor program for display of tabular and graphical data from the FTIR

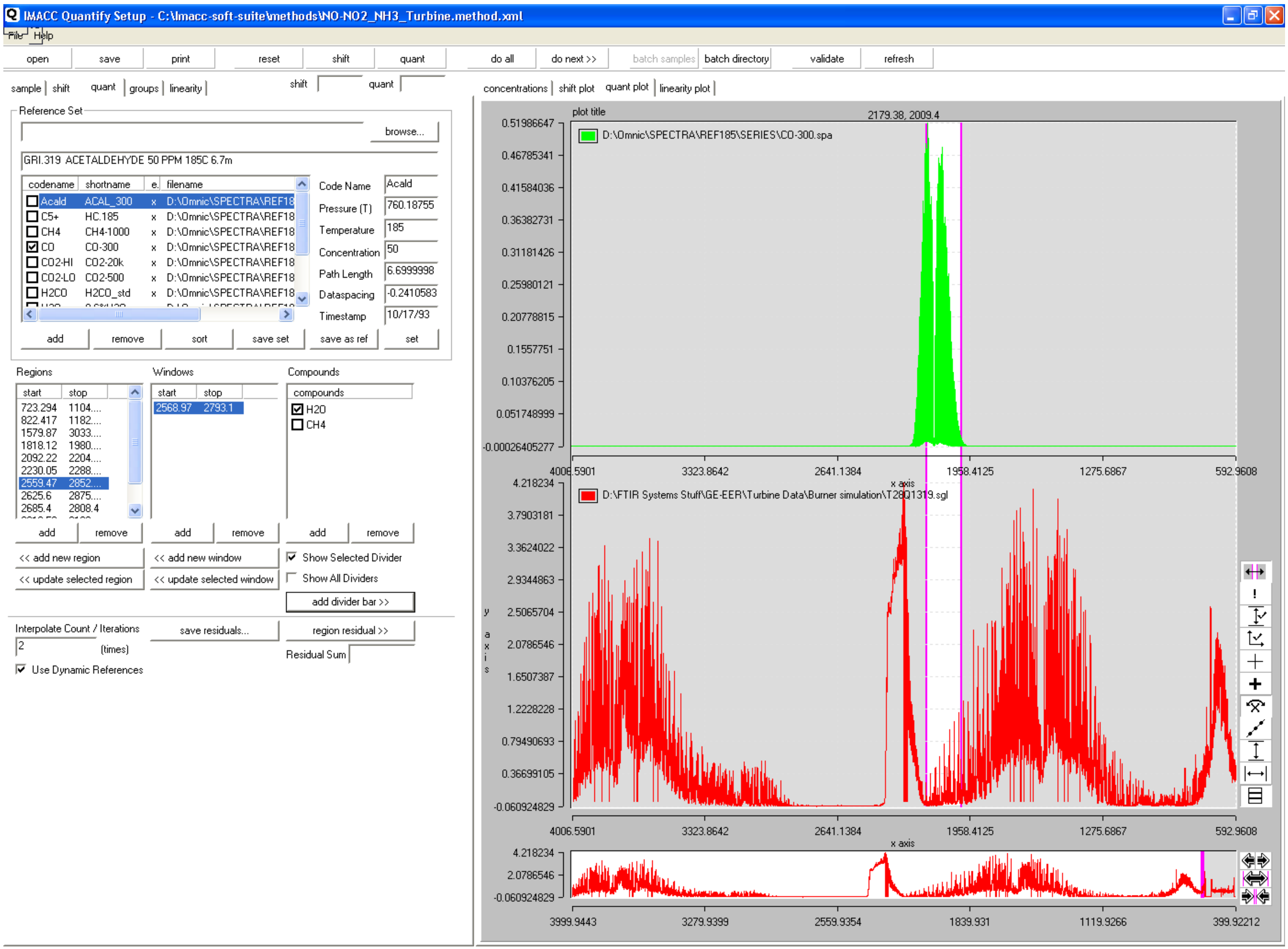


Figure 10 Imacc Quant Setup showing the window used to select analysis regions and windows within regions.

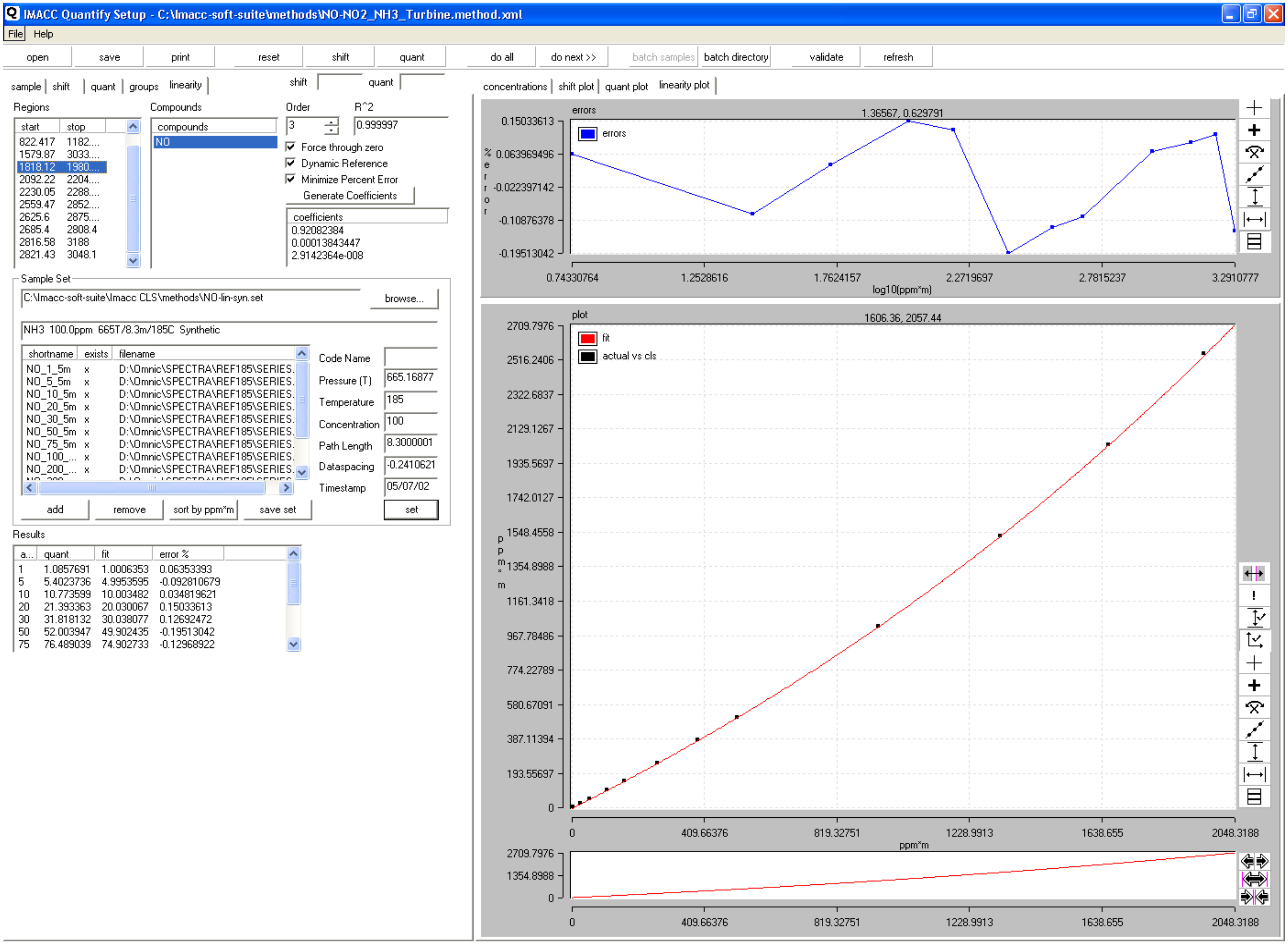


Figure 11 Imacc Quant Setup showing the screen used for linearity corrections of individual compounds. Shown is the linearity for NO from zero to 500 ppm in a 5m cell. The upper plot on the right is the percent error which ranges from - 0.19% to 0.15% over the

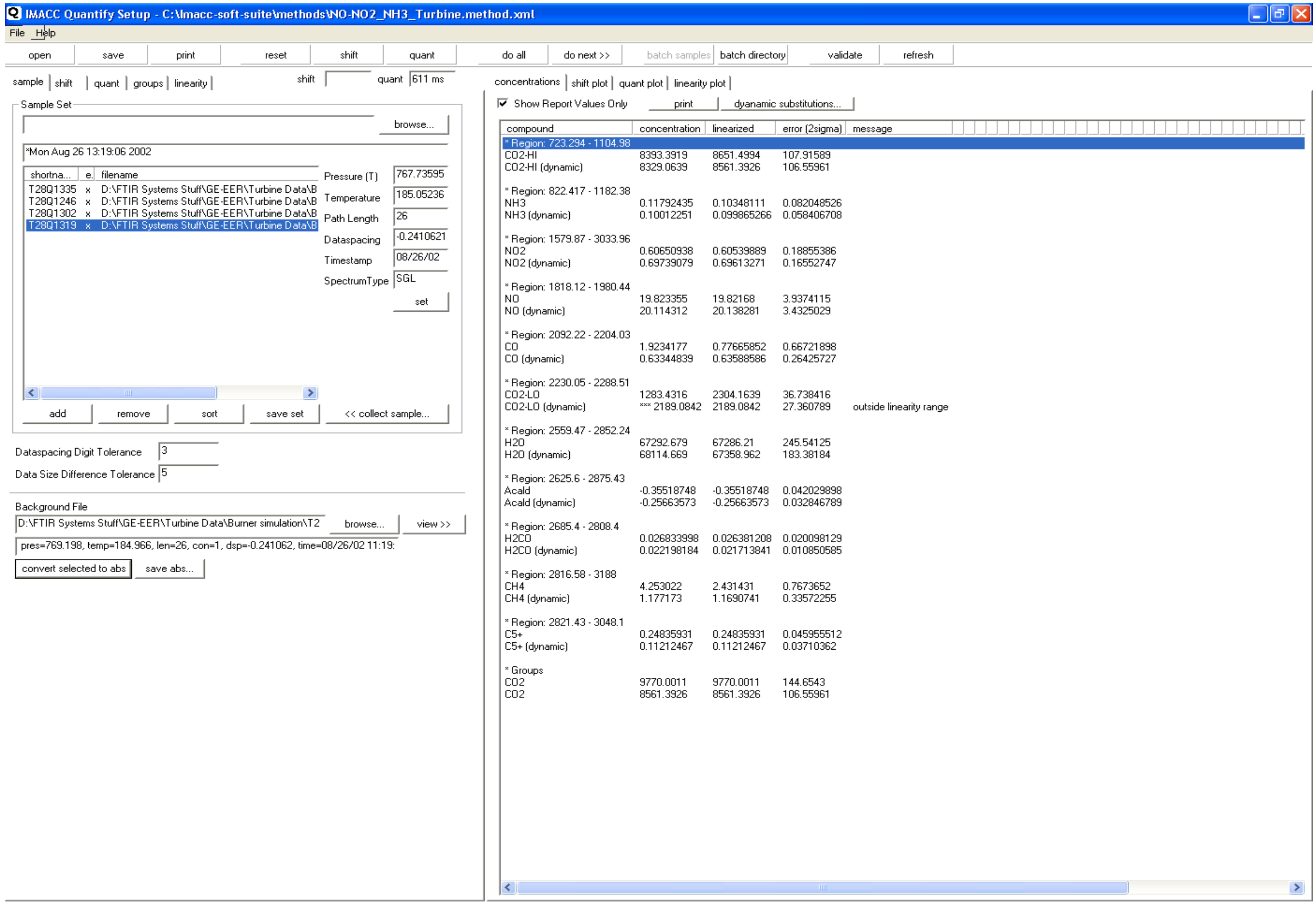


Figure 12 Imacc Quant Setup screen showing quantitations of sample spectra. There are two lines of numbers for each compound because dynamic references selection is being utilized so the software adjusts all references to match the observed concentrations and then reanalyzes the spectrum.

correcting for any non-linearity found. In this window the linearity fit is shown in the bottom plot while the percent error is shown for each spectrum above it. In this case, the maximum errors are from –0.19% to +0.15%. Without this correction the errors could be factors of 3 to 5 (300% to 500%). The final window, shown in Figure 12, is the concentrations window. Here the concentrations of all analyzed compounds are shown for the sample spectrum chosen on the left. This allows the user to step through a full set of spectra and see the concentrations, errors, and dynamically chosen references. This window is used while making small changes to regions, windows, or interfering compounds to optimize the analysis method.

Graphical Display Software

There are two ways to display graphical data from the Imacc FTIR. The first is using the Monitor which can display data in real time or can be used to display concentration data from files. A separate package that is small and can reside on a number of computers is ConcView. This package imports file data and allows for several types of plots to be displayed as shown in Figures 13 through 18. ConcView is windows based so a number of windows can be opened simultaneously to look at the same or different data in different views. As shown in the Figures, ConcView can produce:

- Time series plots of gas concentration as a function of time of day (including 95% confidence limits if desired);
- Bar charts showing a bar for each monitored compound with its height being the percentage of a user selected alarm threshold;
- Correlation plots showing the correlation between the concentrations of two different gases, a helpful tool in process analysis;
- "Pollution-rose[tm]" plots (useful for open path systems) showing the concentration of each gas as a function of wind direction. This plot allows the user to identify the direction from which the pollutant came.
- Spreadsheet pages, giving tabular results of concentrations and errors for all gases monitored throughout the test.

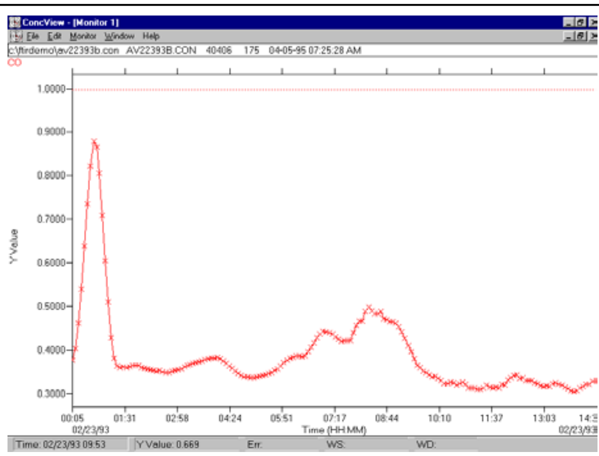
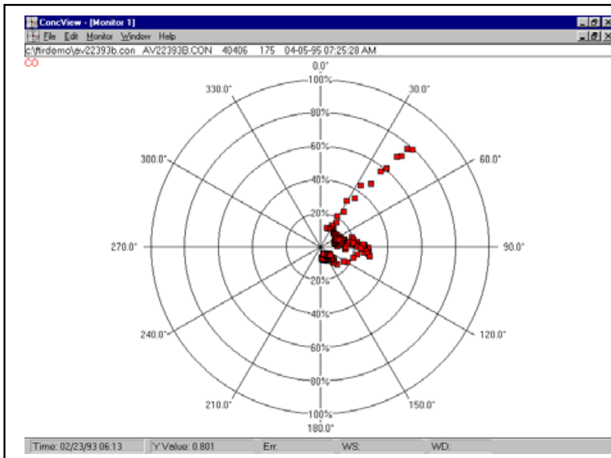


Figure 13 Time series plot showing the gas concentration versus time of day.

Figure 15 Bar chart showing concentrations as a percentage of individual alarm thresholds.



relationship between two gas concentrations.

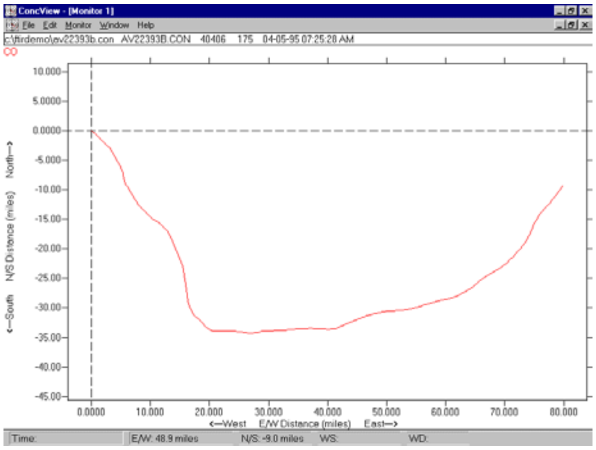
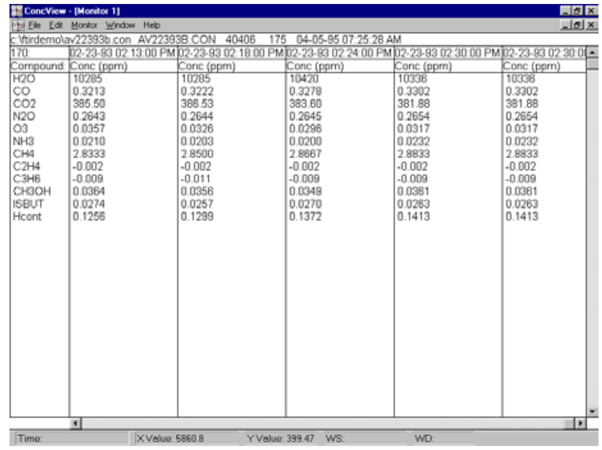


Figure 17 Ground plot showing the path followed by the wind prior to getting to the FTIR



170:	02-23-93 02:13:00 PM	02-23-93 02:18:00 PM	02-23-93 02:24:00 PM	02-23-93 02:30:00 PM	02-23-93 02:30:0
Compound	Conc (ppm)	Conc (ppm)	Conc (ppm)	Conc (ppm)	Conc (ppm)
H2O	10285	10285	10420	10336	10336
CO	0.3213	0.3222	0.3278	0.3302	0.3302
CO2	385.50	386.53	383.60	381.88	381.88
N2O	0.2643	0.2644	0.2645	0.2654	0.2654
O3	0.0357	0.0326	0.0296	0.0317	0.0317
NH3	0.0210	0.0203	0.0200	0.0232	0.0232
CH4	2.8333	2.8500	2.8667	2.8833	2.8833
C2H4	-0.002	-0.002	-0.002	-0.002	-0.002
C3H6	-0.009	-0.011	-0.009	-0.009	-0.009
CH3OH	0.0364	0.0356	0.0349	0.0361	0.0361
ISBUT	0.0274	0.0257	0.0270	0.0263	0.0263
Hcont	0.1256	0.1299	0.1372	0.1413	0.1413

Figure 18 Tabular display of gas concentrations for each compound versus time and date.

This software can view data from archival files and these can be grouped any way one chooses, multiple files of data can be simultaneously displayed – color coded as desired. This allows data to be colored by source or date and easily compared with data from other facilities or measurements. As many windows as desired can be opened simultaneously.

Nicolet Software

The Imacc software packages are supplemented by Nicolet's Omnic® software. Omnic® is a laboratory based package allowing for direct, manual collection and analysis of individual spectra. The user can also manually view and manipulate data incorporating a multitude of spectral processing features. With Omnic one can

- Fourier process interferograms,
- Generate absorbance spectra,
- Scale spectra,
- Add, subtract and perform other mathematical operations on spectra,
- Search libraries to identify features in spectra,
- Perform special functions like baseline correction or spectral shifting of spectra, and
- Quantify spectra using the CLS, PLS or other quant algorithms.

Omnic is very powerful and allows the user to validate data taken in the field as well as manually manipulate and analyze data.

Acceptance Testing

Imacc believes an acceptance test should be formally agreed upon between the Client and Imacc to establish performance specifications for the system prior to delivery. Imacc will *guarantee that the instrument meets or exceeds all specifications* and we will demonstrate this at Imacc's facilities in

Austin, Texas prior to delivery of the system. We usually have a preliminary conference call to agree on specifications for the instrument shortly after a PO is received by Imacc. Based upon this, Imacc will generate a formal acceptance test plan for approval. At a minimum, we use an acceptance test that will include demonstration of the following performance parameters:

- Instrument signal-to-noise ratio;
- Instrument spectral resolution;
- Minimum detectable gas concentrations for key compounds agreed upon, and
- Linearity and Interference corrections for typical operational conditions (to the extent possible with available spectra/cal gases).

Imacc has, in its FTIR spectral library, calibrated references for all major analytes and most of the possible interfering compounds of significance in industrial facilities. Three libraries are provided with the instrument without charge: one at ambient temperature containing approximately 200 compounds, one at 100°C containing about 25 compounds, and one at 185°C containing roughly 100 compounds. The ambient temperature library contains most compounds of concern to industrial and urban monitoring. The elevated temperature libraries contain mostly compounds of interest in *industrial process or stack monitoring*. Although it is not anticipated, if any compounds not in the library are found to be significant during testing, references can be generated by Imacc at minimal cost. We have computational codes that allow for generation of highly accurate synthetic spectra for all normal atmospheric gases. These can be used to provide very low or very high concentration references not generally available in standard libraries.

Training

Classes are usually provided as part of the overall training program. This training prepares operators for using and maintaining the system and, if desired, can also cover development of analysis procedures for the instrument. These latter classes allow the user to write analysis methods for non-standard measurement scenarios as they arise. We usually recommend that at least one person in the organization take “analysis development” training. This will provide the company with the capability to develop specialized analysis routines for future field tests if needed. However, Imacc can also contract for the development of methods on an as needed basis.

Training is usually conducted at our facilities in Austin, Texas at the time of acceptance testing. It usually consists of about three days of training which includes: basic principles of operation of FTIR systems, analysis procedures, hardware alignment and care, and software operation. If desired, this training can be done elsewhere at the time of system delivery, this would add only appropriate travel costs to the overall bid.

Because the system can support modem link capability for remote access, we can operate the system remotely as well as diagnose problems and suggest corrective action remotely

Guarantees

Imacc warranties the FTIR system to be free of defects in materials or workmanship for a period of one year from the time of delivery. This covers any equipment returned to our facility for repair or replacement. Most repairs can be handled through component replacement by on-site personnel. However, Imacc has also trained personnel in who can perform maintenance as required. Parts for the FTIR itself can be obtained through Imacc. If components are not in stock on site, we can typically ship within 24 hours. If a modem link is available into the instrument, Imacc can run and diagnose the systems via telephone link. This allows us to diagnose a problem anywhere in the world and suggest corrective action. If a problem arises that cannot be resolved by on-site personnel, or our field staff, the unit would need to be returned to our facilities in Austin, Texas for warranty repair. If necessary for a critical ongoing program, Imacc can arrange to fly in personnel to make emergency repairs. This would normally be handled on a time and materials basis.

Imacc passes through all warrantees received from our vendors for *stand-alone* components in our instruments. We cannot provide any warrantee other than that provided us for these components. This applies to items like the computer, individual PC-cards, disk drives, monitors, modems, etc. which are warranted by the original manufacturer and not by Imacc. However, Imacc will interface with third parties to obtain a repair or replacement for these items. However, we can not assume full responsibility for the warranty of these items.