

Operation of the Kratos Profile Mass Spectrometers

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Introduction

The data system controlling the mass spectrometer features a graphical interface. You will rapidly become more adept at controlling the instrument as you learn to use the various mouse buttons and to manipulate the windows on the screen. Refer to Appendix A of this document or to the Kratos manual for basic instructions on using the graphical interface. New users are encouraged to learn by using the data system, particularly in operations involving data manipulation and analysis.

It is possible that hazardous conditions or damage to the instrument could ensue from improper control parameters. You should not change settings in the vacuum or source control systems until you have received additional instruction except as specifically delineated in the procedures below. Similarly, parameters can be set in the inlet and data acquisition control windows that are outside the working range of the instrument so care should be taken that reasonable values are used in these areas as well.

Warning and error messages will appear on occasion and in general they should be acknowledged and operation will continue. Often a second or third attempt at a given operation will be successful. Numerous frequent warnings, even from different subsystems, are an indication that something really is wrong. If warnings or errors occur at such a frequency so as to prevent effective operation of the instrument, the problem should be isolated and remedied.

The procedures outlined below are intended to allow the inexperienced user to obtain spectra. As you gain experience with the instrument you should expect to develop procedures that you feel more comfortable with. The multi-windowed interface allows you to monitor and control many operations at the same time. As you learn the effect of each of these parameters on your analysis, you will be able to manipulate the various windows and control systems at your discretion.

When you are done acquiring data, make sure that the instrument is set to standby and that the reference flow is off.

Initial inspection

- 1. Source vacuum** Check that the ion gauge filament, visible through the glass cover of the source, through the vacuum port toward the back of the instrument, is illuminated. The O-ring under the glass cover should be compressed.
- 2. Heated zones** Check that the glass cover of the source is warm to the touch and that the reference inlet (rectangular assembly extending from the front of the source) is hot to the touch. The GC inlet and transfer line to the source should be hot.
- 3. Carrier gas** The primary gauge on the helium regulator on the tank should show between 100 and 2500 psi. The secondary gauge should read approximately 30 psi. The column head pressure gauge on the lower left of the GC should read 8.6 psi (60 kPa). The split flow should be between 10 and 50 mL/min.

Accessing the data system

When the instrument is not in use, the screen should be black with the "game of life" screen saver program running.

Press any key or mouse button and the graphical interface to the Kratos Mach3 operating system will appear. Enter your name, advisor and budget number in the *Login* window.

The *Console* and the *Status and error report* windows are required for the operation of the instrument. The *Console* window should appear closed as a gray shell icon in the upper left corner of the screen next to the clock. If it is not there, hold the right button down anywhere over the gray Ks forming the screen background, move the mouse to the arrow pointing to the right from the word *Shells*, then release the button over the word *Console*. The *Status and error report* window normally appears in the lower right of the screen or possibly iconized as *System Status* in the upper left. To start this window, hold the right button down anywhere over the screen background, move the mouse to the right arrow after the *System restart* selection, then let the button up over *Status and error report*.

Data acquisition

- 1. Check disk space.** Select the *Machine Control* icon (4th from the right on the top on the screen) with the left mouse button then press the right button over the *COMPUTER* subwindow in the lower right of the *Instrument setup and status* window that subsequently appears. The green "free" slice of the upper disk space allocation chart in the *Computer status* window is the space available for data. Approximately 100 Mbytes are available after all of the data has been removed from the disk. If less than 5 Mbytes are available you should free some space before attempting data acquisition. Refer to Appendix B for the procedure for archiving or removing data files from the hard disk. Select *Quit* in the *Computer status* window with the left mouse button, then press it again to confirm that you want to quit. Quit the *Instrument setup and status* window the same way.
- 2. Set instrument to operate.** Select the word *Operate* in the *Status and error report* window in the lower right corner of the screen with the left mouse button. Within 10 seconds the background behind the word should turn red. The square red indicator lamp over the lock switch by the tape drive next to your left knee should illuminate after a short period of time.
- 3. Set data acquisition parameters.**
 - a) Open Data Acquire.** Press the left mouse button over the *Data Acquire* icon (3rd from the right on the top).
 - b) Set Source to Mass Spec.**
 - c) Set Sectors to Scan.** If you wish to carry out metastable or selected ion monitoring consult the Kratos manual or get assistance.
 - d) Set the Setup Scan parameters.** Press the left button over the *Setup Scan* control.
 - i) Set Mass Range scanned.** Reasonable default ranges are 12-600 for GC injections and 12-1200 for the direct probe. The high end should be 50 to 100 amu above the highest mass you expect to see. You may wish to increase the low mass to eliminate water and air. Smaller ranges will allow more scans per unit time and increase chromatographic resolution for GC runs. Be sure to press the return key after entering the range.
 - ii) Set Scan Speed.** Reasonable defaults are 0.5 seconds/decade for GC and 1.0 second/decade for probe. Slowing the scan speed will increase sensitivity. Remember to press the return key after entering the scan speed.
 - iii) Set Maximum Mass to 1200.**

- iv) **Set Scan Using to DAC.**
- v) **Set Magnet Cycles to 0.**
- vi) **Quit the Setup Scan window.** Press the left button over the *Quit* control then press it again to confirm.
- e) **Set Solvent delay in Scan Sequence Chart Maker window.** Open the *Scan Sequence Chart Maker* window by selecting the *Acquisition Sequence* control in the *Data Acquisition* window. The *Solvent delay* should be 0:00 for probe analyses and 2:00 to 3:00 for GC injections depending on solvent and initial oven temperature. To change the value, double click with the left mouse button over the *Solvent delay* parameter in the subwindow on the right. The *Properties: Initial conditions* window should appear. If this window does not appear after several attempts of double clicking on *Solvent delay*, try holding the right button down over the *Properties®* control near the top of the *Scan Sequence Chart Maker* window and releasing it over the *Selection...* option.

Once the *Properties: Initial conditions* window appears, enter the appropriate value for the *Solvent delay* and then select *Apply*. Verify that the new solvent delay has been copied into the *Scan Sequence Chart Maker* window, then press *Dismiss*. Quit the *Scan Sequence Chart Maker* window, confirming that you wish to save the edits that you have made.

- f) **Set Data Collection to Nominal, Exact or Raw as required for your analysis.** Open the *Data collection* window then set the *Data collected as* parameter according to the considerations discussed below.. Accept the defaults for the other parameters or consult the Kratos manual to customize the data collection for your needs. Quit the *Data Collection* window after selecting the desired data collection mode.
 - i) **Nominal data** can be manipulated very easily using the data browser and is the easiest to use for routine work. The disadvantage of acquiring nominal data is that results are reported only to the nearest mass unit so a slight shift in the calibration combined with a significant mass defect may cause the reported mass to be off by one mass unit.
 - ii) **Exact data** will be reported to fractional mass units, up to 5 decimal places. This does not necessarily mean that the data is accurate to that extent but at least the user is in a position to evaluate the fractional mass data and account for it as mass defect, shift in the calibration, etc. The disadvantage of collecting exact mass data is that the browser cannot be used for analysis. Selection, averaging, subtraction and display of spectra and chromatograms must all be carried out using separate programs. Exact mass data may be converted to nominal data after the acquisition is complete for easier data manipulation and analysis.
 - iii) **Raw data** collection is used when accurate high resolution mass determinations are sought. The data is centroided and scans are calibrated manually after they have been acquired. Electropray spectra are usually acquired as raw data.
- g) **Open the Source tuning window** by selecting *Setup source* in the *Data Acquisition* window.

Do not change anything in the Source tuning window except as noted below. Hazardous conditions or damage to the instrument could result from improper choice of parameters in the source window.

 - i) **Turn the filament on** if it is not already on. The filament may be observed through the glass cover to the source housing.
 - ii) **Set the gain to between 360 and 450.** If the gain is between these values do not change the setting. Under no circumstances should the gain ever exceed 600.
 - iii) **Close the window, do not quit it.** Position the mouse over the window and press the *Open* key on the left of the keyboard, or hold the right button down over the green bar on the top

of the window and release the button over the word *Close*. Closing a window keeps the program in memory for quick access later.

- h) **Open the *Reports* window.** Reposition and resize the reports window to a convenient area of the screen. The *Parameters* control in the *Real Time Reports* window allows you to set up the real time display to show chromatograms, spectra, statistics or profiles. The default setting is to display the total ion chromatogram and the current spectrum.

4. Calibration

- a) **Open the *Reference Inlet* window.** The menu control after the word *Inlet* in the *Data Acquisition* window controls which inlet window will be opened when you select *Setup Inlet*. If the *Reference* inlet is not currently selected, hold the right mouse button down over the circular arrows after the word *Inlet*, position the pointer over the word *Reference*, and release the button. Press the left button over the *Setup Inlet* control. Move the resulting window to a convenient position on the screen.
- b) **Turn on the reference flow.** Press the left button over the flow control in the *Reference Inlet* window. If the instrument has not been used in a while, it is possible that air may have leaked into the calibration vial and that the pressure surge upon opening the vial to the source is enough to trip off the vacuum system. If this happens the instrument will reset itself after about a minute. If the ion gauge filament is glowing, the system is properly under vacuum and ready to acquire data.
- c) **Select *Calibrate experiment* in the *Data acquisition* window.**
- d) **Adjust the gain to give total ion current between 3×10^7 and 10^9 .** Monitor the TIC in the *Real Time Reports* window. The value may drop over time as air is removed from the calibration vial. If the 100% value is greater than 10^9 or less than 3×10^7 , open the *Source tuning* window by pressing the left button over its icon, then adjust the gain as necessary to bring the TIC into this range.

If the *Real Time Reports* window does not display the TIC, select *Parameters*, then verify that *Chromatograms* is checked after *Displayed Reports*:. Select *Chromatograms* after *Show parameters* for then verify that *Display TIC* is checked. Select *Done* when you are finished with this window.

- e) Stop the acquisition after about 20 seconds. Press the left button over the *STOP* control in the *Data acquisition* window. Press the button again to confirm that you want to terminate the run. The blue *Calibration* window will appear after a short wait and display the profile of the last scan acquired. This profile is the centroided raw data before masses have been assigned. The horizontal axis is logarithmic with respect to mass with higher masses to the left and lower masses to the right. The numbers by the peaks are a consecutive numbering of the all peaks exceeding the threshold.
- f) **Select *calibrate* in the *Calibration* window.** When you select *calibrate* in this window, the program attempts to assign masses to the peaks using the scan speed and spectra of the calibration material stored in a file. The resulting report will show the acquired spectrum on the top, the reference spectrum from the file in the center, and the calibration curve of scan speed (seconds/decade) vs. mass on the bottom. The peaks in the acquired spectrum that have been matched to the reference spectrum will be displayed in color on the top spectrum. Ideally, the calibration should extend above the highest mass that you expect to encounter in your samples, and the colored peaks in the acquired spectrum should be the highest ones, i.e., the reference spectrum should be matched to strong peaks in the acquired spectrum rather than to noise. The bottom trace should be relatively smooth and not deviate more than a few percent from the expected scan speed.

If a satisfactory calibration is not obtained, several techniques can be used to improve it.

- i) **Edit the calibration.** Select the *edit* control in the *Calibration* window. The *Calibration editor* window will appear. To manually assign a peak, enter the peak number and if necessary the appropriate reference mass, then select *Assign*. The calibration window will be updated each time an additional peak is assigned.
- ii) **Verify that PFK is selected as the calibration compound.** Hold down the right mouse button over the *parameters*[®] control then release it over the word *calibration*. Verify that the *Reference* is *pfk* in the resulting *Calibration parameters* window. If you change this parameter, reject the calibration and then select *calibration* again and the program will recalibrate using the new reference file.
- iii) **Select a different scan from the calibration acquisition run.** The calibration program selects the last scan acquired to calibrate to. This will normally give the best results since the magnet has had the most time to equilibrate to its scan. You can enter an earlier scan number or use the left and right arrows in the *Calibration* window to select a different scan. After selecting a new scan, reject the calibration then select *calibrate* again.
- iv) **Change the tolerance used in the calibration procedure.** Open the *Calibration parameters* window by selecting *parameters*[®] *calibration* with the right button. You can increase or decrease the tolerance from the default value. You may also change the units - ppm (parts per million) is a relative measure so the tolerance will increase as the mass increases; mmu (milli mass units) is an absolute measure and remains the same across the entire calibration. 500 mmu = 0.5 amu = 5000 ppm at 100 amu. After changing the tolerance, reject the calibration and press *calibrate* again.
- v) **Repeat the run using a different mass range, scan speed or gain setting.** Press *reject*, quit the *Calibration* window, change parameters and try again.
- g) Accept the calibration and quit the calibration window. Select *accept* in the *Calibration* window then quit the window.
- h) **Turn off the reference flow and quit the *Reference inlet* window.**

5. Acquiring sample data

- a) **Set up the inlet system.** Set up either the probe or the GC as appropriate for your analysis.
 - i) **Direct probe**
 - (a) **Open the Probe control window.** Select *Probe* as the inlet system by holding the right button down over the circular arrows after the word *Inlet* in the *Data acquisition* window and then releasing the button over the word *Probe*. Select *Setup inlet*. Move the resulting *Probe control - Standard probe* window to an unused area on the screen if necessary.
 - (b) **Verify that the *Probe Power* is off and that the *Demanded Temperature* is set to 0.**
 - (c) **Wait until the *Current temperature* is less than 60° C** before removing the probe from the source.
 - ii) **GC**
 - (a) **Open the GC control window.** Select *GC* as the inlet system by holding the right button down over the circular arrows after the word *Inlet* in the *Data acquisition* window then releasing the button over the word *GC*. Select *Setup inlet*.
 - (b) **Edit the program in the GC control window to fit your analysis.** Be sure to press the return key after entering new values. Initial oven temperature is set in the column of

initial temperatures to the right. Start time, ramp rates, final temperatures and hold times are set in the table in the center of the window. Additional ramps may be added or deleted by changing the number in the *Ramps* control. Split or splitless injections may be selected using the Injection control and the split time entered as appropriate. The *Injector*, *Interface* and *Re-entrant* temperatures should normally be left between 180 and 280. The *Initialize* control sends the program to the GC. The *Start program* control starts the GC program independently of MS acquisition. The *Abort* control will ramp the oven ballistically from its current temperature to the final temperature, hold it there for the designated hold time, then return it to its initial temperature. The *Condition* control will implement the indicated *Condition* program starting at the current oven temperature. *Parameters* opens a window which allows adjustment of the equilibration tolerances and times for the four heated zones. *Displays* provides a graphical display of the current temperatures and the temperature program.

- (c) **Select *Displays*[®] Zone temperatures.** Hold the right button down over the *Displays*[®] control and then release it over *Zone temperatures*. Move the resulting zone temperatures display window to a free area of the screen if necessary.
 - (d) **Initialize the GC.** Press the left mouse button over *Initialize* in the *GC control* window. This sends the program to the GC so it can come to equilibrium at its initial temperatures.
 - (e) **Close the *GC Control* window.** Do not quit it. The zone temperatures display will remain open.
 - (f) **Wait for the GC to equilibrate.** The letter *R* will appear in each of the four subwindows of the zone temperatures display. When all of the zones are ready each *R* will turn green.
- b) **Introduce the sample and start data acquisition.** Follow the procedure for the probe or the GC as appropriate for your analysis.
- i) **Direct probe**
 - (a) **Be sure the probe temperature is below 60 °C** before removing the probe from the source.
 - (b) **Open the *Source tuning* window and turn off the filament.** The pressure surge into the source when the probe is introduced will shorten the life of the filament if it is energized.
 - (c) **Remove the probe from the source:**
 - (1) Close the toggle valve to the inlet roughing pump on the line leading down from the probe guide assembly.
 - (2) Withdraw the probe slowly and firmly until the notched flange on the handle just meets the pin on the probe alignment guide. Do not withdraw the probe beyond this point since the alignment guide is not fixed and will be drawn out with the probe.
 - (3) Close the main probe isolation valve by moving the handle to the right until it is perpendicular to the probe. Be sure that the probe is withdrawn to the stop before closing the valve - closing the valve onto the glass tip of the probe will result in very expensive damage.
 - (4) Withdraw the probe completely.

- (d) **Put capillary tube containing your sample into the end of the probe.** Put a small amount of your sample (if you can see it, it is more than enough) into a capillary tube, tap the tube so the sample falls to the closed end, break off about 1.5 cm of the tube from the end containing the sample, then place this tube into the end of the probe with the open end facing out. The tube should be straight as it extends from the probe. Avoid touching the tip of the probe with your fingers.
- (e) **Partially insert the probe into the source:**
- (1) Place the end of the probe into the probe inlet assembly until the flange is even with the end of the probe alignment guide. Open the toggle valve to the inlet roughing pump, then after about 15 seconds slowly insert the probe until the flange meets the first stop on the probe alignment guide. You should be able to feel the probe being pulled in by the vacuum.
 - (2) Open the main probe isolation valve very slowly. Observe the ion gauge filament through the back of the source chamber. The filament will brighten when the valve first opens and the pressure surge enters the source chamber. Stop moving the valve at this point and allow the pressure to drop. Move the valve in small increments until the ion gauge filament no longer brightens, then open the valve until the handle is aligned with the probe guide.
 - (3) Guide the probe into the source chamber until the tip of the probe is about 5 cm from the source. Place a stop between the flange on the probe handle and the source chamber to keep the source vacuum from drawing the probe further into the source.
- (f) **Turn on the source filament with the control in the *Source tuning* window.**
- (g) **Press *GO* to start acquiring data.** Press the left mouse button over the *GO* control in the *Data Acquisition* window. The total ion current and the spectra should be displayed in the *Real Time Reports* window; use the *Parameters* control to reset them if they are not. You should see peaks for air (28, 32, 14 and 16), water (18) and any background contamination at this point.
- (h) **Remove the stop and guide the probe up to the re-entrant on the source.** The capillary tube should extend into the hole in the re-entrant.
- (i) **Heat the probe as required for your sample.** Observe the TIC and spectra in the *Real Time Reports* window. Generally you will see an increase in the TIC as the probe approaches the source and a spectra of your sample will appear. If you are not observing the parent ion peak, heating of the probe will be required.
- To heat the probe:
- (1) **Turn on the *Probe power* in the *Probe control* window.**
 - (2) **Increase the probe temperature in increments of 25° to 50 °.** The *Demanded Temperature* can be changed by entering a new number or by using the slider control. Allow the temperature to equilibrate after each increment. **Do not exceed 300°C on the probe.** Monitor the spectra and TIC in the *Real Time Reports* window.
- (j) **Adjust the gain as necessary to keep the TIC from saturating.** The TIC trace will saturate between 2 and 4 billion counts and the trace will drop down. Use the gain control to lower the total ion count if the TIC is approaching these levels. The abundances will be distorted on spectra that are nearly saturated since the more abundant ions will be clipped. For this reason, total ion counts below 1 billion are

preferable, particularly if you are observing isotope ratios or doing other work where relative abundances are important.

(k) **Stop the run when satisfactory spectra have been obtained.**

- (1) **Withdraw the probe about 5 cm from the source and insert the stop to prevent it from being drawn back into the source.**
- (2) **Stop data acquisition.** Press the left mouse button over the *STOP* button in the *Data Acquisition* window then press it again to confirm that you want to stop the acquisition.
- (3) **Cool down the probe.** Turn off the *Probe power* in the *Probe control* window and set the *Demanded Temperature* to 0°. When the probe temperature has dropped below 250° the probe cooling air valve on the green strut on the back left corner of the instrument may be opened to speed up the cooling of the probe. **Do not withdraw the probe from the source from the source chamber until it is cooler than 60°.** Make sure that the probe cooling air valve is shut after the probe has cooled.

ii) **GC**

- (a) **Make sure that the GC inlet system is selected.** The GC program will not start if the GC inlet system is not selected in the *Data Acquisition* window.
 - (b) **Wait until all GC temperatures are ready.** The four subwindows in the zone temperatures display window should each have a green *R*.
 - (c) **Load the syringe.** 1.0 µL of sample is normally the amount used.
 - (d) **Press GO and inject after the countdown.** Press the left mouse button over the word *GO* in the *Data Acquisition* window. The data system will take several seconds to establish communication with the GC and start acquiring data. When it is ready it will count down from 5 to 0, beeping with each count. Inject the sample into the injection port on top of the GC when the count reaches 0 after the long beep.
 - (e) **Observe the TIC and spectra in the Real Time Reports window.** After the solvent delay period has elapsed the TIC and the mass spec. scans will appear in the reports window. If they do not appear after the solvent delay period, check the *Parameters* control in the *Real Time Reports* window to see if the window is set up correctly. You may analyze previously acquired data or do anything else you like with the data system while the acquisition is progressing.
 - (f) **To stop the run before the GC program is complete:**
 - (1) **Press STOP.** Press the left mouse button over the word *STOP* in the *Data Acquisition* window.
 - (2) **Terminate the GC program.** Open the *GC control* window. Use *Abort*, *Condition* or *Initialize* in the *GC control* window as necessary. Bear in mind that any compounds in the GC column need to elute before the next run can begin.
- c) **Repeat the acquisition procedure as necessary for subsequent samples.** Allow the probe to cool or the GC to equilibrate at its initial temperature before proceeding with the next sample.

If you wish to look at the data from a run that has just been acquired while collecting data from the next run, select *Export* in the *Data Acquisition* window to transfer the data to one of the data analysis programs.

You may check the calibration during an acquisition by opening the reference flow and observing the peaks from the calibration compound. A listing of the peaks in the PFK spectrum is kept with the instrument. The calibration should last for many hours but if you have reason to suspect that the calibration has shifted, repeat the calibration procedure.

6. Return the instrument to its standby condition after the last acquisition is complete.

- a) **Return the inlet systems to standby conditions.** The probe should be cool and partially withdrawn from the source with the stop in place. Do not leave the capillary tube containing your sample in the probe. The GC should be left with the oven less than 250° and all of the other heated zones between 180° and 200°. The split flow on the GC should be between 10 and 30 mL/min.
- b) **Verify that the reference flow is off.**
- c) **Quit all windows associated with data acquisition.** This includes *Source tuning*, *Real Time Reports*, *GC control*, *Probe control* and *Data Acquisition* windows. Select *Export* in the *Data Acquisition* window before you quit it if you want to analyze your data immediately.
- d) **Set the instrument to *Standby*.** Press the left mouse button over the word *Standby* in the *Status and error report* window in the lower right of the screen.

Data Analysis

Programs for the analysis of data are started from icons located along the left side of the screen. All of these programs have a vast array of features that are best explored on your own with the help of the Kratos documentation. A cursory overview of some of the more commonly used programs will be presented below.

Multiple copies of each of the data analysis programs may be run at the same time but this should be avoided unless it is serving some purpose. Every time you select the *Browser* icon, for instance, another copy of the browser program will start, taking up space in the computer memory and slowing down the operation of the data system. A program that has been closed (iconized) is still resident in memory - you must **quit** a program when you are done with it. To move from the analysis of one acquisition to the next, do not just close the program and start a new copy; either quit the program and start a new copy, or import the next acquisition into the analysis program that is currently open. Calling up a new data file will reset a program to its default set of parameters.

Selecting any of the data analysis programs with the **right** mouse button will start the program in the **import** mode: the last data to be exported will automatically be loaded when the program opens.

Printed output from the *Browser* and *Raw Data* program will go to the dot matrix printer. Output from all other programs will be sent to the laser printer.

1. **Browser** The *Multi-view nominal data browser* is the most versatile program for manipulating data after it has been acquired. The major disadvantage of this program is that it can only be used with nominal data. The operation of the program is intuitive so it is recommended that you learn to use the program by experimenting with it. Note that mass and scan ranges can be entered either with the keyboard using the control parameters at the top of the window, or graphically using the mouse. Start the program by clicking on its icon with the left button.
 - a) **Load data file into browser.** Two methods may be used.
 - i) **Use *Import* if you are analyzing data that you have just acquired.** Press *Export* in the *Data Acquisition* window to load the data file just acquired, or currently being acquired, onto the data transfer clipboard, then press *Import* in the browser window to load the file into that program.

- ii) **Enter the file name as the *Name:* parameter in the browser.** Previously acquired data may be loaded by entering the name of the data file into the browser window. Note that the name will appear as something like test0267 in the *Data Acquisition* window but as test#267 in the browser. Both refer to the same data.
- b) **Tile layout** The browser displays the total ion chromatogram across the top of the window. The bottom portion of the window may be divided into smaller tiles with the layout controls *Across* and *Down* located at the top center of the window. By default, the top tile may not be manipulated. To change the data displayed in the top tile or to eliminate this tile, hold the right mouse button down over the *layout®* control, move to the right side of the *Top Graph®* selection at the bottom of the menu, then select *Hide top graph* or *Enable changes to top graph* as desired.
- c) **Types of tiles** Select from *Graph* control at upper left corner of browser window.
- i) **TIC** The total ion current will be displayed in the selected tile for the scan range selected.
 - ii) **Spectra** Selected scans will be displayed. If more than one scan is selected, several spectra will be displayed stacked on each other in the tile unless enough scans are selected to make such a display unreasonable, in which case the block format will be used.
 - iii) **Chromatograms** Selected ion chromatograms will be displayed of the ions entered at the prompts *Masses: A* through *H*.
 - iv) **Block** The mass range and scan range will form an X-Y plot with the intensity indicated by the color of a given point on the grid.
 - v) **Isometric** A three dimensional plot of mass range, scan range, and intensity will be displayed.
- d) **Graphical manipulation of data**
- i) **Selecting tiles** Press the right mouse button over a tile to select that tile for subsequent activity. A small hook will appear while the button is pressed and the border of the tile will become bold. Any manipulation as to selection of tile type, spectra, averaging, etc. will be applied to the tile with the bold border.
 - ii) **Cloning tiles** It is sometimes desirable to create a copy of the contents of one tile into another tile so that subsequent data manipulation may be carried out. To accomplish this, hold down the right mouse button over the source tile, move the mouse to the target tile, then release the button.
 - iii) **Graphical manipulation of data ranges** Data may be selected and manipulated using the mouse. The left mouse button is used for data manipulation. Dragging the mouse with the left button depressed over a range of mass or scans will enter that range into the currently active tile. Use the *all* selections after the mass or scan range listings at the top of the window to expand a range back out. The graphical manipulation of data is largely intuitive so it is recommended that you learn this operation by experimenting with the program.
- e) **Mathematical manipulation of data**
- i) **Averaging** To average mass spectra over a range of scans, select the desired range into a tile then check the *Average spectra* box with the left mouse button. To undo the averaging, click on the box again.
 - ii) **Subtraction** To subtract a background from a spectrum displayed in a tile, press the left mouse button over the *subtract* control near the top of the browser window, then either

select a range of spectra to average and subtract using the mouse on the TIC, or enter the range from the keyboard at the appropriate prompt. To undo a subtraction, select *subtract* and then select *cancel*.

- f) **Printing** The only printing option available from the browser is to print the entire graphical portion of the window by selecting *dump@ mono dump* with the right mouse button.
 - g) **Exporting data** Selecting *export* with the left mouse button will allow transfer of the contents of a given file to another program such as *Mass List* to tabulate a spectrum or *Spectra* to provide annotated hardcopy. It is not possible to export averaged data from a run that is still being acquired.
2. **Mass List** This program provides for the tabulation and printing of spectra. Start the program by clicking on its icon with the left mouse button.
- a) **Load data** Data is loaded into the program either by importing it from another program or by entering the run name as the *Name:* parameter in the *Mass List* program.
 - b) **Set filters**
 - i) **Mass range** The report will only list those peaks within the mass range entered. Be sure to press the return key after entering the mass range. Select *all* to list all peaks present in a given scan.
 - ii) **Int low limit %** The report will only list those peaks with intensities equal to or above the entered value. Intensity is expressed as percentage of the base peak (highest peak in the scan).
 - iii) **Filters** This controls allows you to filter out peaks in the reference compound, saturated, unresolved, doubly charged or non-high resolution peaks. It allows setting an upper and lower limit on the intensities and selecting different schemes for normalizing the spectra. Consult the Kratos documentation for further details.
 - c) **Select Report** To display the mass list press the left mouse button over the *Report* control. You may use the *Next Page*, *Next Line*, *Last Page* and *Last Line* controls to page through the list.
 - d) **Select Print to print mass list.** The output from this program will go to the laser printer.
3. **Library search** The *Library search* program compares a spectrum to a library of over 100,000 spectra and tabulates the best matches.
- a) **Open the Library program** Select the *Library* icon with the left mouse button
 - b) **Load data file into Library search window.** Data may be loaded by specifying the run as the *Name:* parameter in the top left corner of the window, or by importing a run or spectrum using the *Import* feature.
 - c) **Select Scan and Mass range** Enter the desired scan number and mass range in the appropriate fields. If you are trying to match an averaged or background subtracted spectrum exported from the browser program, these entries will not be necessary.
 - d) **Select PBM Search to start library search.** A listing of the top matches will appear as well as a plot of the sample and library entry spectra. The listing includes library serial number, match reliability scores, molecular weight, chemical formula and the name of the compound.

4. **Atomic Composition** The *Atomic Compositions* program will calculate the molecular weight and natural isotopic distribution for a compound, given the molecular formula. It will also list possible molecular formulas for a given molecular weight given ranges for the various constituent atoms.
 - a) **Open the *Atomic Compositions* program** Select the *Atom* icon with the left mouse button
 - i) **Isotopic distribution**
 - (a) **Select *Graphical or Tabular distribution as the Tile type***. The *Tile type* parameter is in the upper left corner of the window. *Graphical distribution* will generate a mass spectrum plot of the parent ion isotope distribution for the indicated molecular formula. The *Tabular distribution* option will generate a tabular listing of the spectrum.
 - (b) **Select *Display selections to enter the molecular formula***. Enter the number of each of the elements listed as it would appear in the molecular formula. To add elements to the list, choose *Select element* in the parent window, select the desired elements from the periodic table by clicking on them with the left mouse button then clicking again to confirm the choice. To remove an element from the list, check the box to the left of the element, then select *Remove item*. To keep an element on the list but not include it in the molecular formula, enter 0 (zero) as the number for that element.
 - (c) **Select *Plot distributions or Tabular report in the parent window to display the plot or table***.
 - (d) **Adjust the *Abundance cutoff: parameter as desired to report lower abundances***. If you enter an abundance cutoff of zero for a complicated molecule, the program may run for a very long time calculating all of the possibilities, so only enter a value only as low as you really need.
 - ii) **Formula Search** This feature is useful in high resolution exact mass determinations. Consult the Kratos documentation or get help if you wish to use this feature.

For all other data analysis programs consult the Kratos documentation or get assistance.

Saving your data and logging off

1. **Return the instrument to standby conditions.** Verify that the reference flow is off and that the instrument is set to *Standby* in the *Status and error report* window. The square red lamp over the tape drive should be off. Quit all windows except *Status and error report*, *Console* and the *Clock*.
2. **Select the *Mach3* icon with the red K in the upper left corner of the screen.** This will bring up the *log off* window. A tabulation of your time and charges will be presented.
3. **Enter a comment in the *comments* field if you want to leave a message for the facility administrator.**
4. **Select *no charge* if you should not be billed for this session.** Leave a comment explaining why.
5. **Save your data** if you want to refer to it in the future. By default, all data is saved temporarily in the directory */usr/kratos/mach3/data/archive* on each instrument. When the disk is full, everything is deleted from this directory, normally about once every week or so. If you want a permanent copy of your data you must save it to the external 1.7 Gbyte drive using the following procedure:
 - a) **Select *save data in the log off window***. A list of the runs acquired while you were logged on will appear.
 - b) **Select the runs you want to save.** Click on the run names with the left mouse button to highlight the runs that you want to save. A tabulation of disk space and file size will be updated

in the lower left corner of the window. You may select a different directory by clicking on the *source directory* control.

- c) **Designate the target directory.** If you have already established a directory, select *target directory* with the left mouse button then select your directory from the list. If not, enter a directory name in the target directory field. The name should start with a capital letter and not contain any spaces or other illegal characters.
 - d) **Select *transfer files & then exit*.** Your data will be written to the target directory on the external hard drive, you will be logged off, and the screen saver will appear.
6. **Select *done to exit to the screen saver*** if you are not saving any data.

Windows

The programs that control the mass spectrometer and provide for analysis of the data all run in windows. Windows may be manipulated on the screen using the mouse or the keyboard. There are a number of operations that are common to all windows.

Open & Close When a program is selected by pressing the left mouse button over its icon on the top or left side of the screen, it will open a window somewhere on the screen. Closing a window will shrink its display to an icon on the right side of the screen but keep the program in memory where it can be accessed more quickly than starting it for the first time. To open a closed or iconized window, position the mouse pointer over the icon and either press the left mouse button, hold down the right mouse button and select *Open*, or press the *Open* key on the left side of the keyboard. To close an open window, either position the mouse over the window and press the *Open* key, or hold down the right mouse button over the border of the window and select *Close*. Closing a window is a different operation than quitting it. When a window is quit it is removed from memory; to restart it you need to click on the icons on the left side or top of the screen

Front and Back When a program is started up its window will overlay all of the other windows on the screen. To move a window to the front either press the left mouse button over the border of the window (when the circle pointer is visible), hold down the right button over the border of the window and select *Front*, or position the mouse anywhere over the window and press the *Front* key on the left side of the keyboard. To move a window behind the other windows on the screen either position the mouse over the window and press the *Front* key or hold the right button down over the border of the window and select *Back*.

Moving To move a window, position the mouse over the border so that the circle cursor appears, hold down the middle mouse button and drag the window to its new position. If the window is grabbed near a corner it can be moved in two dimensions; if it is grabbed near the center of an edge it will move in one dimension only.

Resizing The size of a window can be changed by holding down the *Control* key, positioning the pointer on the corner that you want to move, holding down the middle mouse button and dragging the corner to its new position. Alternatively you may hold down the right button over the border of the window, select *Resize* from the resulting menu, then use the left or middle button to move one of the corners as indicated by the message that will appear on the screen.

Quit To quit a window use the *Quit* panel control in the window. This operation will remove the window from memory.

The mouse

The mouse used with this instrument is an optical mouse - it operates by reflecting the light from an LED off of the grid on the mouse pad. Turn the mouse over and look at the bottom. It must be used with the mouse pad provided and the mouse pad must be oriented more or less square with the bench. If the pointer seems to be moving in funny directions, check that the mouse pad is in a reasonable orientation.

The **pointer** associated with the mouse can have 5 different shapes.

1. The arrow is the most common shape and is used to select controls, icons, data ranges, etc.

2. The circle appears when the mouse is on the border of a window. When the circle appears you can execute some window function such as moving a window or bringing it to the front or the back.
3. The hourglass indicates that the program associated with the window that the mouse is over is executing a lengthy process and not accepting input.
4. The caret indicates that a text or numeric entry is expected.
5. The fish hook is used to indicate which tile is selected in multi-tile programs such as the browser.

The mouse may be used either by depressing one of the buttons and releasing it, similar to a key on the keyboard, or by holding down a button, moving the mouse, and then releasing it. The latter operation, sometime referred to as dragging the mouse is used in moving and resizing windows and in making menu selections.

The **left mouse button** is used to open a window from an icon or to initiate whatever action is designated by a control on the screen (e.g. ,the *Operate* control). Data ranges can be selected by dragging the mouse with the left button down when you are working with graphical representations of data.

The **right mouse button** is used to invoke and select from a menu. Menus can be invoked from four places:

1. If a control consists of text followed by an arrow pointing to the right it is a menu. To use controls of this type, hold down the right mouse button over the control until the menu appears, move the cursor to the desired selection and then let up the button. Releasing the button when no selections are highlighted will erase the menu without selecting anything.
2. Controls consisting of a description followed by two circular arrows pointing at each other followed by more text are menu controls that can be operated by either the right or the left button. Pressing the right button anywhere over the control will display a menu from which a selection can be made as described above. Pressing the left button over the control will select the next consecutive item in the menu list. Using the left button with these types of controls is convenient when there are only two menu choices, e.g. *on* and *off* in which case the left button will toggle the control on and off.
3. When the mouse is positioned over the border of a window or on the colored title bar, the cursor changes into a small circle. Pressing the right button at this point will invoke a menu associated with the designated window that allows you manipulate the window as discussed above. With minor exceptions, the menu window for all windows are the same. Pressing the right button over an icon will also access the window menu.
4. If the right button is pressed over the screen background filled with gray Ks, the system menu will be displayed. This menu allows the user access to features provided by the SunView operating environment. Some of the selections in this menu have submenus designated by an arrow pointing to the right. To access the submenu, move the pointer to the arrow with the right button depressed, then release the button over the desired submenu selection.

The right button is also used in the browser program to select a tile.

The **middle mouse button** is used to move a window by positioning the cursor on the border or title bar then dragging the window with the middle button. If the window is grabbed near a corner it can be moved in two dimensions; if it is grabbed near the center of an edge it will move in one dimension only.

The middle mouse button will also display help on a given control or parameter when pressed over that control. To clear the help window, select *done* from its window menu (hold down the right button over the border and select *done*).

Entering text or numbers.

Text entry fields are designated by a label followed by a colon. Numeric fields have a label but no colon. The mouse pointer must be positioned over a window for any field in that window to accept input. The currently active input field is indicated by a small triangular caret. The caret reverts to a gray diamond when the mouse leaves the window. To enter text or a number, press the left mouse button over the desired item. If the current entry disappears, enter the new number or text then press the return key, otherwise use the delete key to backspace over the current entry before entering the new information. The backspace key will not work as expected if you are used to a PC. If you are making a numeric entry, you must delete enough spaces to make your entry, i.e. if you want to replace 500 by 5000, you need to press the delete key 4 times so the cursor is positioned at the space before where the 5 in 500 was originally. All entries must be followed by the return key for them to become effective.

The *Copy*, *Cut* and *Paste* keys on the left side of the keyboard can be used to edit text or numeric entries or to copy entries from one window to another. To select a portion of text press the left mouse button at one end of the text to be selected and the middle mouse button at the other. The selected text will be highlighted.

All data acquired on the mass spectrometer as well as averaged and subtracted data generated by data analysis programs are saved to the `~mach3/data` directory on the hard disk in the instrument. When you log off the instrument, the files you select to save will be copied to your directory on the external hard disk, then all of the data generated during your session is moved to the `~mach3/data/archive` directory. When the internal hard disk is full, all of the files in the archive directory are deleted, leaving only copies of the selected data on the external drive. If you do not specifically select data from a given run to be saved when you log off, you will be able to retrieve it for perhaps a week or so until the internal drive is full; after that it will be removed.

Retrieving data from the external hard drive.

1. **Select the *Data Select* icon with the left mouse button.**
2. **Select the *Data Directory* control with the left mouse button.** A list of directories will appear.
3. **Select your directory from the directory list with the left mouse button.**
4. **Select the *Run Name* control.** A list of the runs in your directory will appear.
5. **Select the run you want from the list with the left mouse button.**
6. **Export the run from the *Data Select* program.**
7. **Import the run into the data analysis program that you want to use.**

The data analysis programs will continue to work from a given directory so once you have imported one run from your directory, you only need to enter the new run name in the data analysis program to retrieve another one from the same directory.

Deleting data from the *archive* directory

The space available on the internal hard disk is displayed on the title bar of the *Real Time Reports* window; it is displayed in the *computer status* component of the *Machine Control* program; and it can be obtained using the `df` command from a UNIX shell. It is important to be aware of the available disk space since the system will not warn you when the disk is full; it will just start behaving very erratically. You will not be able to acquire data and may be prevented from opening windows or doing anything at all. Generally the system administrator will delete files from the archive directory as needed to keep enough working space on the disk, with some effort taken to ensure that no one has files there that they want to keep. The following procedure may be used to remove files from the archive directory if additional space is needed.

1. **Select the *Archiver* icon with the left mouse button.**
2. **Select *Archive to tape* in the *Archiver* window.**
3. **Select the files that you want to delete.** Select the blue file folder icon labeled *data* and the select *archive* when the subdirectories of *data* are displayed. The runs will be displayed organized by run name. You may select individual runs by pressing the left mouse button over a series name to display the individual runs, or you may select an entire series of runs by holding the right mouse button down over the run name and releasing it over *Select all injections*. The middle button may be used to deselect an individual run. A tabulation of the number of files and the amount of disk space selected appears in the upper panel of the archiver window. Each run consists of five or more files

depending on whether there is averaged, subtracted or peaks data generated by the data analysis programs.

4. **When all of the runs have been selected, click on *Archive* with the left mouse button.**
5. **Select *Erase files*, then select in again to confirm that you want to delete the data from the disk.**
6. **Select *Done* and then *Quit* until the *Archiver* is removed from the screen.**

APPENDIX C

Restarting the Data System

System crashes are to be avoided since the integrity of the operating system and data files can be jeopardized when the system goes down. Every attempt should be made to resolve the problem within the Kratos Mach3 operating program, or if necessary, by exiting the SunView environment and restarting the Mach3 program. If it is absolutely necessary for the data system to go down, it is vital that it be halted gracefully or damage to the file system may result. A few selected procedures will be discussed below. If you have some familiarity with UNIX, please be judicious in how you exercise it since a little knowledge can create a lot of damage.

Don't hesitate to call or page the facility administrator if you are having problems with the instrument - telephone numbers are posted by the door of the laboratory. In most cases problems with the data system can be easily resolved over the phone.

If the display of a given window is distorted or the program in the window does not seem to be running right, quit the window and then start it up again. If a program is not accepting input it could be that it is involved in some lengthy operation.

If only part of a window appears, or if something appears wrong with more than one window, select the *Redisplay all* option from the system menu. (Hold right button down over the gray Ks background, then select *Redisplay all*.)

If messages appear in large text across the screen such as *Too large a window number (128)!* and the graphics environment starts to scroll off the screen it means you have quit the *Console* window. Select *Shells® Console* from the system menu and then *Redisplay all*.

If one window refuses to accept input or will not leave the screen after it has been quit, it is possible to stop the program from a UNIX shell. Select *Shells* from the system menu. When the UNIX shell comes up enter the command *ps*. A listing of active processes owned by the Mach3 login will be displayed. Locate the process ID number (PID) for the offending process, then enter the command *kill -9 PID* where PID is the process ID number. Exit the UNIX shell by issuing the command *exit*. Any parameter changes in the window since the last time it was opened will be lost during this process. Be sure you enter the correct PID; you could damage the system if the wrong process is killed. If the window that you terminated controlled a heated zone such as the reference inlet or the source, enter the temperature setting again.

If the entire system refuses to accept input it may be possible to kill the offending process by logging into the data system remotely. Go to the other mass spectrometer, open a UNIX shell and type *rlogin kratos* or *rlogin kratos2* depending on which instrument you are trying to revive (*kratos* is the GC-MS, *kratos2* is the one with electrospray). When you get a prompt on the remote computer start killing processes using the procedure outlined in the paragraph above starting with the program you were using when the system locked up. When you reach the program that caused the problem, all of your subsequent mouse clicks will be implemented so menus will frantically appear and disappear and windows open and close rapidly in succession. Close the remote login by typing *exit*; close the UNIX shell by typing *exit* again. If you are logging in from a computer other than one of the mass spectrometers or the Chemistry VAX, telnet to *kratos.chem* or *kratos2.chem*, log in as *mach3* using *massspec* as the password.

To terminate the entire Mach3 program select *Exit SunTools* from the system menu. You will be asked if you wish to logout from UNIX. Answer *yes* to this question. The Mach3 program can be restarted by logging in as *mach3*. The password is *massspec*. Answer *yes* to invoking the Mach3 environment but *no* to downloading the MSCPU processor. Refer to the discussion below on rebooting the system for the steps that need to be taken after the software is restarted.

To shut the system down:

1. **Open a command shell**
2. **Log in as the super user**
 - a) **Enter *su***
 - b) **Enter the root password** -- It was *massspec* at the time this document was printed.
3. **Halt the system:**
 - a) **Enter *fasthalt***
 - b) **Wait for the system to respond with:**
Synching file systems... done
Halted

>
4. **Remove the data system console cover.** It is the large front panel closest to the console.
 - a) **Free the bottom of the cover by gently pulling up on it.**
 - b) **Slowly pull out on the bottom of the cover until the Velcro fasteners at the top come free.**
 - c) **Move the cover away from the instrument.**
5. **Turn off the keyed switch at the lower right of the VME bus housing.**

If the console will not except any input whatsoever so that a graceful shutdown is not possible, and if repeated attempts have been made to contact the system operator, and if the rebooting of the system is absolutely necessary since hazardous conditions may be present, and if you are fully prepared to accept all consequences of a crashed system, you may shut down the system down by observing the hard disk drive indicator lamp (the hard disk is up and to the left of the on/off switch), waiting until there has been no disk activity for 30 or 40 seconds, then turning off the switch.

Rebooting the system.

If the system has been turned off, turn the key to the on position; if you are still at the monitor prompt (>) enter *b*. The system will check its memory, load the operating system, initialize hardware and peripheral systems, and start various system daemons. If the system has crashed or if it was halted by a command other than *fasthalt*, lengthy file system checks will be carried out upon rebooting. If the system crashes during these tests or otherwise fails to get through the boot up procedure, turn the key off and start again.

When the system has booted it will display a login prompt, either *kratos login:* or *kratos2 login:*, depending on which system you are on. Login as *mach3*. The password is *massspec*. After copyright information has been displayed, you will be asked if the Mach3 Operating environment should be invoked; respond positively. The system will ask if the MSCPU processor should be downloaded. Check that the seven segment LED on the MSCPU located on the VME buss is displaying only a decimal point. If it is not, press the uppermost of the two small buttons immediately below the seven segment display and the MSCPU will reset. Proceed with the downloading only after the MSCPU has been reset as indicated by only the decimal point being displayed on the LED array. If you are just restarting the Mach3 software and the system has not been shutdown or rebooted, it is not necessary to reset or download the MSCPU and you may respond *no* to the question in this case. Do not download the MSCPU processor if it has not been reset.

Once the Mach3 environment is up there are a number of tasks to attend to:

1. Select *Standby* in the *Status and error report* window. The Inlet, HT, Vacuum and Magnet subsystems will reload at this point. The progress of this operation will be displayed in a text window in the upper right corner of the screen. If the MSCPU has not been reset this may not take long as all of the systems will be ready. Normally it might take as long as five minutes or more.
2. If the system has been down for any length of time reset the system time. Open a console shell and issue the following commands.

```
Kratos{mach3} (#51): su
```

```
Password: massspec
```

```
Kratos# date 9410241338 current date and time in format yymmddhmm
```

```
Mon Oct 24 13:38:00 PDT 1994
```

```
Kratos# exit
```

```
Kratos# Kratos{mach3} (#52): exit
```

3. Reset temperatures of heated zones. Type in the numbers again. The system will have forgotten even though it displays the numbers in the appropriate windows.
 - a) Set reference inlet temperature to 180° C.
 - b) Set the source temperature to 330. This should heat the source to about 200° C.
 - c) Select *Initialize* in the *GC control* window.
4. Adjust the zero offset of the DAC and check that the threshold is appropriate:
 - a) Set the instrument to *Operate* in the *Error and status report* window.
 - b) Select *Start scope* from the *Source tuning* window.
 - c) Enter *0.1 V* as the *Scale* value in the *Scope Display* window, or select approximately this value by dragging with the left mouse button down vertically from 100 mV to below zero on the graph.
 - d) Turn the filament *off* using the control in the *Source tuning* window.
 - e) Adjust the *Set zero* value in the *Scope Display* window so that the baseline appearing on the scope is between 1 and 5 mV.
 - f) Set the *mass* value in the *Scope Display* window to a strong PFK peak such as *181 amu*. Select the *max* panel control after the *sweep* parameter near the center of the top of the *Scope Display*.
 - g) Turn *on* the filament.
 - h) Turn on the *Reference flow* in the *Reference Inlet* window. Open this window if necessary from the *Data Acquisition* window.
 - i) Use the *Scale* panel button in the *Scope Display* window to display the entire reference peak. Adjust the *Gain* if necessary in the *Source tuning* window to keep the peak less than 10 V.
 - j) Use the left mouse button to select the voltage range to observe only the base of the peak.
 - k) The threshold value is indicated by the dotted line across the scope. Adjust the threshold if necessary so that the valleys between peaks are completely below the threshold. The real peaks are spaced about 1 amu apart; everything else is noise.
 - l) Quit the *Scope display* window.
5. Proceed with your analysis, or turn off the *Reference flow* and reset the instrument to *standby*.

Appendix D

Electrospray Ionization

1. Start the syringe pump:

- a) **Turn on the syringe pump power.** A small rocker switch is located on the right rear corner of the pump.
- b) **Start the pump.** Press the red *start/stop* button - the *run* light should come on.

The flow is normally set to 4 $\mu\text{L}/\text{min}$ although flows up to 20 $\mu\text{L}/\text{min}$ may be used. To change the flow rate hold down the *set* button and press the *rate* button, then enter the new flow rate followed by *enter*. It is okay to change the flow rate while the pump is running.

2. Set the source heater to 100° C.

Use the *TEMPERATURE* knob near the center of the electrospray control panel. The red heater demand lamp will illuminate until the temperature approaches the setpoint.

3. Turn on the N₂ blanket gas:

- a) **Open the cylinder valve** on the Nitrogen tank on the wall behind the instrument.
- b) **Open the toggle valve** on the lower right of the electrospray control panel.

4.

End of File

October 25, 1994, 11:27 AM