1.0   SCOPE AND APPLICATION

The objective of this Standard Operating Procedure (SOP) is to present an overview of the Microsensor P200 dual channel microchip gas chromatograph (GC), including analytical capabilities, operating methods and technical limitations. This microchip gas chromatograph, complete with high resolution capillary columns, is linked to a Macintosh personal computer, allowing rapid field analysis of environmental samples.

Only vapor phase samples (i.e., soil gas samples, ambient air samples, Tedlar gas sampling bags and purgeables from soil and water samples) can be analyzed through the activation of the instrument's internal sampling pump.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0   METHOD SUMMARY

The P200 is a portable, high speed gas chromatograph that samples air, separates volatile components and then identifies and calculates the concentrations of these compounds. A Macintosh computer system connected to this instrument processes sample data and identifies selected compounds found in the air samples.

The P200 unit contains two high resolution capillary columns. A simple menu driven software package controls the unit and allows the operator to adjust all method parameters. After the correct parameters are set, a vapor sample is drawn into a sampling loop via an internal vacuum pump and is injected onto the GC columns. The sample gas is then analyzed on one or both capillary columns; and specific volatile compounds are separated, detected and identified by the computer software package (M2001 v 2.2). Examples of typical P200 GC set-up parameters appear in Appendices A and B.

Calibration standards must be analyzed prior to field sampling. Once the calibration has been validated using the Macintosh software library, sample analyses can proceed. The Macintosh calibration library currently contains only target compounds historically encountered in U.S. EPA/ERT field work.

3.0   SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Since the P200 is involved with vapor phase samples, all samples are either collected directly or stored in Tedlar gas sampling bags, as per ERT SOP #2102, Tedlar Bag Sampling.

The actual introduction of any calibration or sample gas into the instrument is done by attaching a 2 cc Becton-Dickenson glass syringe, via Teflon Luer lock valve, directly to the sampling inlet port in the front of the unit; alternatively, a Tedlar bag can be attached directly to the injection port.

4.0   INTERFERENCES AND POTENTIAL PROBLEMS

Even considering the powerful analytical capabilities in the P200 design configuration, the instrument has several inherent limitations that impact on its ability to be used during responses to chemical releases. These limitations are as follows: commercial versions of the device can detect only preselected compounds that can be identified by parameters stored within its

internal library; large quantities of other vapors could be present, and may seriously interfere with the analysis using the P200. The presence of many sample components (>30 component peaks) may confuse the identification routine of the M2001 software and yield ambiguous results. Also, large quantities of sample vapors may overload the capillary columns and the retention times of the preselected compounds may fall outside of expected retention time windows. Finally, commercial versions of the device yield detection limits which are too high (by factors of ten to one hundred) for determining levels of hazardous chemicals at or below threshold limit value (TLV) concentrations. For typical soil gas analysis, parts per billion volume (ppbv) will be required, and the P200 cannot detect levels that low. To compensate, a sample pre-concentrator is used to concentrate the compounds of interest prior to injection. A separate SOP has been established for the use of this pre-concentrator (ERT SOP# 1714, Processing Air Samples with the Portable Sample Concentrator).

5.0   EQUIPMENT/APPARATUS

In addition to the basic Microsensor P200 unit, there are several pieces of equipment required for its operation. These include a 12-volt power supply, a Macintosh computer system (SE30) and a RS232DB9-DB9 cable. The P200 GC can also operate using a IBM type PC using EZ Chrom and windows 3.0 software packages. Use of the P200 GC using the EZ Chrom system is not covered in this SOP. Diagrams of the P200 GC hardware configuration and instrument control panels appear on Figures 1 and 2 of Appendix C.

6.0   REAGENTS

The P200 utilizes high purity (99.995% or above) helium as a carrier gas. The P200 accepts the carrier directly at pressures up to 2000 psig. A single stage or high pressure two stage regulator can therefore be used. The capillary column head pressure can be adjusted via two regulators found on the back of the instrument.

Gas standards are purchased as certified mixtures at fairly high concentrations (i.e., 1-100 parts-per-milli volume (ppmv) from Scott Gas or Matheson. These concentrations are for subsequent dilution to various concentrations that enable construction of a standard

calibration curve, as well as for calibrating the M2001 libraries.

Liquid phase standards, if required, must be of the highest purity, such as Aldrich-Gold Label or Supelco Environmental standards kits. If air is to be used for sample/standards dilutions, it must also be ultra-high purity gas. No liquids can be introduced into the P200 system.

7.0   PROCEDURES

In the following set of procedures, all P200 software menu options will appear in brackets (e.g., [FILE]). The P200 GC must be interfaced with a computer in order to access the hardware of the unit for controlling the instrument.

The P200 GC must be calibrated in order that proper identification/quantitation occurs. A calibration file and calibration library must be resident in the P200 applications software (M2001 v2.2) in order to make a sample run. The reader should be familiar with basic Macintosh computer operations, since proper use of the Macintosh mouse to select menu options is paramount. Operating manuals, for the specific computer used, in this case the Macintosh SE30, should be read to acquaint the user with basic keyboard functions.

The M2001 v2.2 data reduction and instrument control software is relatively easy to use. Detailed descriptions of the identification, calibration and P200 GC operations procedures are provided in the M2001 v2.2 manual. The manual's table of contents is provided in Appendix B.

7.1   Step-Wise Procedures for Entering M2001 Software System

1.   Power up SE30 Macintosh computer.

2.   Double click on hard disk icon (SCS1 40MB).

3.   The menu of SCSI appears, double click on M200 icon.

4.   The menu of M200 appears, double click on M2001 icon.

5.   The top menu bar of the M2001 software will

appear; the menu selections will allow the user to select all of the functions and subsequent sub-menus to calibrate the software libraries, change P200 parameters and run sample analysis.

6.	If no library or calibration file is residence in M2001 system, the software will prompt the user to select either one or both from available file directories.

7.	Select the appropriate library (40EC, 60EC or 100EC) for the operating conditions. An error message will appear if an inappropriate library is selected, (e.g., the 100EC library is selected and the P200 GC column temperatures are set at 60EC). Select the calibration file which is appropriate to the sample matrix to be analyzed.

8.	The P200 GC operating parameters can be adjusted by selecting the [WINDOWS] options on the top M2001 menu bar, and scrolling down to select [CURRENT METHOD]. The current method window will appear. All the operating parameters can be accessed from this window.

	Changes are made by selecting an item and clicking to increase the values. To decrease a value, the "option" key on the keyboard is held down while clicking on a value. After all the parameters are entered the new method must be sent back to the P200 GC by selecting the [SET M200] button at the bottom right hand side of the window. Examples of typical operating parameters are presented in Appendix A.

9.	The P200 GC can now be used to analyze gas phase samples using the M2001 v2.2 software package.

10.	The stored library and calibration files must be updated to normalize the retention time indices (RTI) to the current operating conditions as per Section 7.2.

11.	A sample run is initiated by selecting [FILE] from the top M2001 menu bar, selecting [NEW] and [SAMPLE]. Alternatively, a sample run can be started from the keyboard by typing "command key" and "N"

simultaneously. A menu box will appear where the sample name is typed in, and directories to save the sample file can be selected.

12.	The M2001 software allows the use of automated routines to select integration parameters, display options and file actions, such as saving and closing files after each run. These routines are setup using the preferences window. This window is accessed by selecting [EDIT] from the top M2001 menu bar and scrolling down to the last option [PREFERENCES]. The system default values are adequate for most field screening requirements. Changing these values should be done only when necessary. The reader is advised to read the M2001 v 2.2 manual for more background on the automated procedures before modifying the default values.

## 7.2	Step-Wise Procedures for Creating Calibration Library Files

1.	The correct RTI to normalize the calibration library must first be established using an homologous series of compounds, such as C_4 to C_8, n-alkanes.

2.	Attach an n-alkane standard of known component concentrations to the P200 injection port, and select [FILE], [NEW] and [SAMPLE] from the top M2001 menu bar to run the standard. Name the file and save it to an appropriate directory.

3.	The software will display the chromatograms for column A and column B.

4.	At the end of the P200 run, an inventory page will appear which will display the identified compounds and their concentrations.

5.	If these values are acceptable, no further calibration procedures for the n-alkanes are required. If not, which is the typical case, a new calibration will be required.

6.	Before starting a new calibration, select the upper left hand box (the go away box) to display the peaks for channels A and B. Record the run times (RTs), in seconds, for

the compounds you are going to calibrate on columns A and B. It is imperative that these RTs be correct. It may be necessary to analyze each component individually to establish the correct RTs for columns A and B target compounds. Once the RTs are established, proceed with building a new calibration file. The peaks for channels A and B can also be accessed by selecting [WINDOWS] from the top M2001 menu bar and scrolling down to peak A or peak B.

7. From the top M2001 menu bar, (Section 7.1, Step 5), select [FILE], [NEW] and [CALIBRATION].

8. The multi-point calibration window will appear with its own menu to the right of the window. The file name used in step #2 (Section 7.2) will be highlighted, and appears in the "file name" menu to the left of the window. Other available files can also be selected from this window. Select the sample file you are going to use for the calibration. Calibrations can only be performed on open active files. Select [SPECIFY STANDARDS] for the menu.

9. The calibration editor window will appear with windows and dialog boxes for the current library compounds and the sample peaks listed for columns A and B.

10. Scroll down to the bottom library window, to select the first compound you are going to calibrate. Type in the concentration and the appropriate units in the bottom dialog boxes. The selected compound will be highlighted.

11. Select the RT on column A and click the mouse once. The selected compound and concentration from step #10 above will be displayed next to the RT. Do the same for column B.

12. Continue steps 10 and 11 until all the compounds you are going to calibrate are selected. Select [OK] on the bottom right hand box when done.

13. The calibration window (step #8) will reappear. Select [CALIBRATE], the second box on the right hand side, to run the software calibration. A small watch will appear while the program is running.

14. Select [PLOT QUALITATION], the third box on the right hand side, to plot the regression of the RTIs for the n-alkanes in the calibration standard. The qualitative calibration window will be displayed with a plot of RT vs RTI for columns A and B. Check to see if the lines are straight and parallel to one another. If this is not the case, a mistake may have been made during the calibration procedure. Redo the calibration and make sure the proper RTs and RTIs have been used. When completed select [OK] at the bottom of the window. The multi-point calibration window will reappear.

15. Select [PLOT QUANTITATION] from the multi-calibration window. The quantitative calibration window will be displayed. The first compound used in the calibration procedure will be plotted for columns A and B. In the upper right side of the window, two radio buttons are present. Make sure that the [LINEAR-ZERO] button is highlighted. This indicates that a single concentration point calibration, per compound, is being used. This is sufficient for most field screening needs. Check to make sure the correct compound is present and the concentration is accurate.

16. Select [NEXT] at the bottom of the window to move to the next compound in the calibration mode. After viewing all the compounds select [OK] when done. The calibration procedure is completed.

17. The multi-point calibration window will reappear, select [OK] at the bottom right hand of the window. This will end the calibration process.

18. A file window will appear to save the calibration file just created. Type an appropriate name, in the correct directory, and select [SAVE], to save the calibration. This will archive the new calibration and establish it as the new current operating calibration for the M2001 software.

19. The P200 calibration library is now normalized to a series of n-alkanes. This establishes the correct RTIs to be used for the identification routines of the M2001

software. The detector responses to n-alkanes is significantly different from the response of typical chlorinated organic and aromatic compounds. As such, a new (second) calibration must be run using a gas standard containing miscellaneous target compound; this is to derive detector response for the target compounds of interest. Two calibrations are required to correctly identify and quantify miscellaneous organic compounds. The first calibration based on a homologous series establishes the correct RTI used to identify compounds. The second calibration, using target compounds of interest, establishes the correct detector response factors used to quantify detected compounds. The calibration of the target compounds is identical to the previous procedures, steps #6 to #18.

20.   At present, three calibration libraries exist at operating temperatures of 40, 60 and 100EC. These libraries should be adequate for most field screening purposes and should not be modified.

        Creating new libraries or modifying existing libraries, requires that some information on the compound(s) be known. The exact RTI and the standard deviation of the RT (in seconds) on a population of consecutive runs (n≥7) must be determined. The exact chemical name and chemical abstract number (CASRN) is also recommended. Step-Wise procedure for creating/modifying calibration libraries are presented in Section 7.3.

7.3   Step-Wise Procedures for Creating/Modifying M2001 v2.2 Calibration Libraries

1.   To create a new calibration library select [FILE] from the top M2001 menu bar, [NEW] and [LIBRARY]. The library parameters dialogue window will appear. The current column temperature(s) will be displayed. If this is the correct temperature for the library, select the [OK] button. If not, type in the correct temperature and select [OK].

2.   The file dialogue window will appear. Type the appropriate library name, choose the proper directory, and select [SAVE] to name and send the currently empty library to the system.

3.   Select [WINDOWS] from the top M2001 menu bar, scroll down to the new library and select.

4.   A blank library window will appear. Double click anywhere on the window to start the editing process.

5.   The library editor window will appear. Type in the appropriate values for sample name, CASRN (chemical abstracts register number), RTI and RT standard deviation.

6.   Select the [CHANGE] button on the bottom menu of the window.

7.   Continue adding compounds to the library by selecting [NEXT], typing the library value, and selecting [CHANGE] until all the compounds of interest are entered. Select [DONE] to end the editing process.

8.   The library window will reappear. Check to see if the correct values were entered. If not, the library will have to be modified.

9.   Modifying the calibration library must be done on a currently active library window. This is achieved by opening a library, selecting [FILE], [OPEN] and [CALIBRATION] or selecting a library from [WINDOWS] on the top menu bar.

10.   Once the proper library window is active, the item to be modified is selected by double clicking on the item.

11.   The library editor window appears and the current values are displayed.

12.   The entire entry can be deleted by selecting the [DELETE] and [DONE] buttons.

13.   Library values (i.e., RTI, name) can be modified by typing in the correct value, and selecting [CHANGE] and [DONE]. The library window will reappear with the modified values displayed.

14. New compounds can be added to existing libraries by scrolling down to an empty line in the library directory and double clicking.

15. A blank library editor window will appear. Values can be typed and [CHANGE], [DONE] selected to send the new values to the library.

16. The library window will reappear with the new value added, in the proper ascending RTI order.

17. Succeeding and previous libraries entries can be accessed by using the [<<PREV] (previous) and [NEXT>>] buttons on the bottom of the library editor window.

18. No entry is permanent unless the [CHANGE], [DONE] buttons are used.

8.0 CALCULATIONS

The M2001 software identifies the various components in the gas phase samples and calculates their respective concentration. A single point calibration has been found to be adequate for most field screening purposes. The software does allow for multi-point calibration curves to calculate sample concentrations. The response factors used to calculate concentrations are listed in the text files of the current M2001 calibration file. It is advisable to check one or two samples for the proper calculation of the sample concentrations. Multiply the sample peak areas by their respective response factors to see if the M2001 software is yielding the proper concentration values. Regardless of the calibration procedure used, the M2001 internally calculates concentration.An external calibration can be performed by analyzing three or more standard concentrations. A straight line equation in the form of Y = (m)(x) + b (where: x = concentration, Y = area counts, m = slope and b = the y-intercept) is fit to the standard's raw data. The unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of erroneous detector response. Alternatively, sample concentration can be calculated as below:

$$Sample \ Conc. \ \cdot \ \frac{(standard \ conc.)(sample \ area)}{(standard \ area)}$$

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following quality assurance/quality control procedures must be followed:

1. A calibration must be run daily.

2. Duplicates of a standard, preferably close to sample results, should be run every ten samples to ensure constant detector response. Ten percent (10%) of the total sample population should have duplicates run. The duplicate response should be within 10-20% of each other in terms of area counts and retention time values.

3. Matrix spikes, or spiking samples with known levels of standards, must be run along with the samples, and should bracket the levels found in the field samples.

4. The same Tedlar bag may be analyzed by other field instrumentation (i.e., Photovac, OVA, Sentex, etc.) and /or collected onto tubes for GC/MS confirmation. If Tedlar bags are used to prepare standards, the time of preparation should be noted.

5. During sample analysis, one of the standards should be periodically re-analyzed to ascertain if any sample loss occurs in the bag over time.

6. A Performance Evaluation (PE) sample is typically sent along to determine if any loss or contamination occurs from transit or handling during sampling.

7. A trip blank of zero air is also sent along and is analyzed at the end of the sampling run to determine if any contamination of the Tedlar bags occurred during transit. Tedlar bag sampling should follow the ERT/REAC SOP #2102, Tedlar Bag Sampling.

10.0 DATA VALIDATION

The RTI is used for peak identifications. If peaks are eluting close to the target compounds, sample spikes, using known levels of target compounds, can be prepared to identify the absence/presence of these target compounds in the samples. Typically, only the

RTI is needed to identify the peaks of interest. The P200 GC data reduction software, M2001 v2.2, will identify compounds on both column libraries. The two column capability allows verification of the compounds identified on one column with those identified on the second column. The software will ascribe a level of confidence to the identification of the compounds which can be used to evaluate the performance of the M2001 quantification/qualitation routines.

Quantification is determined from the P200 calibration library or by solving for concentration from the straight line equation. The coefficient of variation on the straight line equation should have an R-squared, (R^2), of 0.90 or greater. Confirmation of the identity of any particular target compound may also be done by other analytical methods, typically GC/MS.

The P200 GC contains two columns and detectors with column (A) acting as a confirmation column for the other (B).

11.0   HEALTH AND SAFETY

When working with potentially hazardous materials, refer to U.S. EPA, OSHA and corporate health and safety practices. More specifically, analysis should be performed in a well ventilated room. The sample vent port on the back of the P200 should be equipped with either a carbon scrubber or a long Tygon tube to vent sample gases outside of the work area or in an approved hood. All carrier gas cylinders must be securely bolted to a table or piece of heavy furniture. When liquid reagents are used to prepare standards, the work should be performed under a vented hood with the analyst wearing safety glasses and disposable protective gloves.

12.0   REFERENCES

The below citations describe the use of the P200 GC in various field applications.

Overton, E.B., T.H. McKinney, C.F. Steele, E.S. Collard, and R.W. Sherman, "Rapid Field Analysis of Volatile Organic Compounds in Environmental Samples", Proceedings of the Third Annual Symposium on Solid Waste Testing and Quality Assurance, USEPA, Washington, DC, July, 1987.

Overton, E.B., R.W. Sherman, E.S. Collard, P. Klinkhachorn, and H.P. Dharmasena, "Current Instrumentation for Field Deployable Analysis of Organic Compounds", Proceedings of the Joint Army-Navy-NASA-Air Force Safety and Environmental Protection Subcommittee Meeting, Monterey, CA, May, 1988.

Overton, E.B., R.W. Sherman, H.P. Dharmasena and C.F. Steele, "Remote Sensing Using Gas Chromatography Via a Macintosh Interface", Proceedings of the Sixth Annual Hazardous Materials Management Conference International, Atlantic City, NJ, June, 1988.

Overton, E.B., R.W. Sherman, C.F. Steele, and H.P. Dharmasena, Correlation "Chromatography with a Portable Microchip Gas Chromatograph", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, October, 1988.

Overton, E.B., C. Steele, R. Sherman, E. Collard, S. and T. McKinney, "Field Useable Compound Specific Analytical Device", Proceedings of Safety and Environmental Protection Subcommittee of the Joint Army-Navy-NASA-Air Force Interagency Propulsion Committee, Cleveland, OH, May, 1987.

Sherman, R., T. McKinney, M. Solecki, R. Gaines, and B. Shipley, "Field Use of a Microchip Gas Chromatograph", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, October, 1988.

Sherman, R.W., M.K. Solecki, E.S. Collard, T.H. McKinney, L.H. Grande, and E.B. Overton, "Development of a Field Portable Concentrator/Purge and Trap Device for Analysis of VOC in Ambient Air and Water Samples", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, October, 1988.

Kaelin, L.P, M.F. Solecki, T.H. Pritchett and R.E. Wynnyk, "Field Evaluation of a Microchip Gas Chromatograph for the Analysis of Volatile Organics at Hazardous Waste Sites", Proceedings of the 6th Annual Waste Testing and Quality Assurance Symposium, Washington, DC, July, 1990.

APPENDIX A

Microsensor P200 Gas Chromatograph Set Up Parameters

MICROSENSOR P200 GC SET UP PARAMETERS

Typical P200 GC Operating Parameters:

Module choice	A + B
Sample time	6 Sec
External wait	off
Autorun	off
# of Autorun	3
Run interval	0 min.
CHP off set	0

	Column A	Column B
Column temperature	40 - 100EC	40 - 100EC
Run time	1 - 255 sec	1 - 255 sec
Detector filament	on	on
Detector autozero	on	on
Detector sensitivity	low - med - high	low - med - high
Inject time	100 ms	100 ms
CHP offset code	values cannot be adjusted	
Temp offset code	values cannot be adjusted	
Temp scale code	values cannot be adjusted	
Column type	other	other

APPENDIX B

Microsensor P200 M2001 v2.2 Manual

CONTENTS

10/8/90

M2001 v 2.2

APPENDIX B (Cont'd)

Microsensor P200 M2001 v2.2 Manual (Cont'd)

APPENDIX C

Figures

FIGURE 1. P200 GC Hardware Configuration
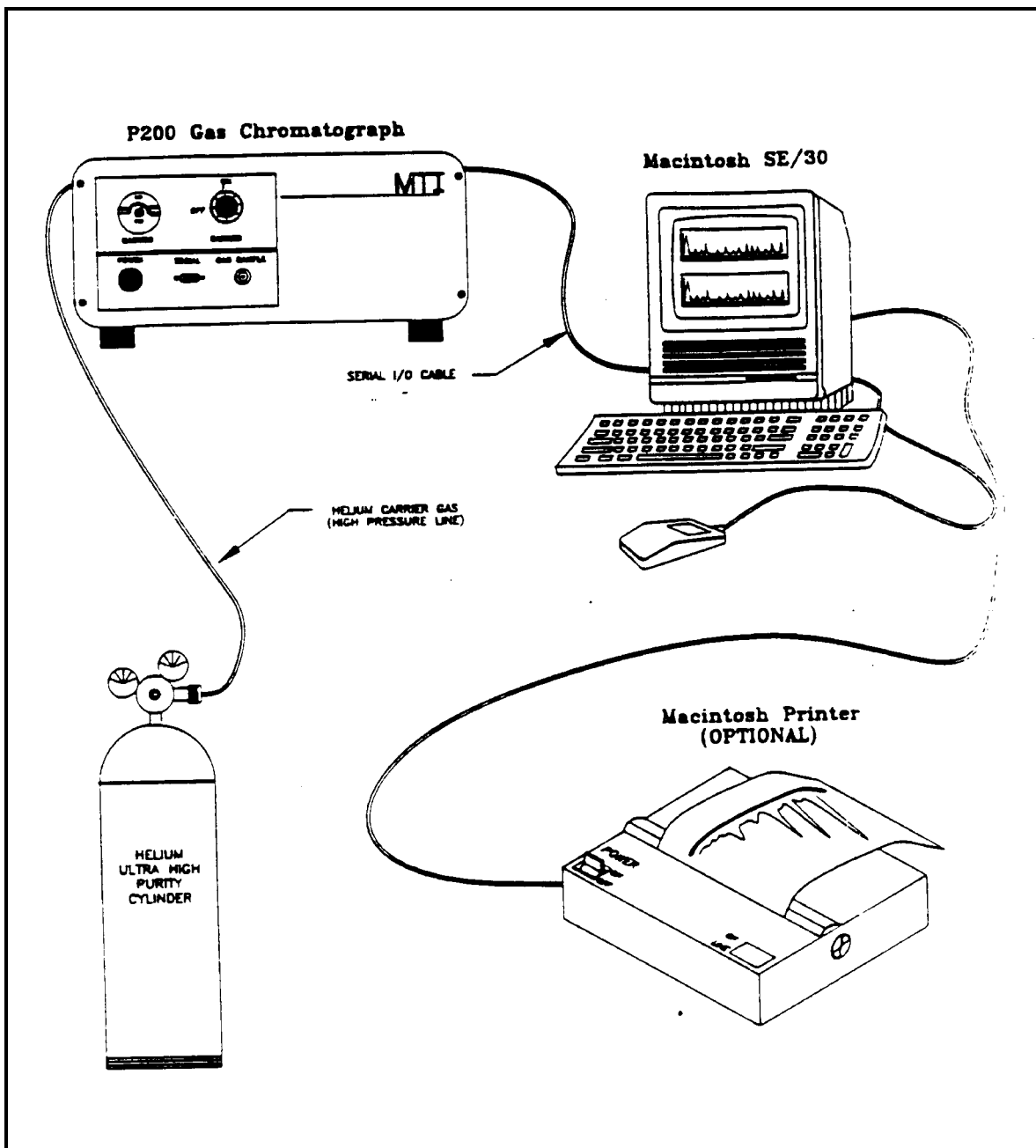
APPENDIX C (Cont'd)

Figures

FIGURE 2. P200 GC Instrument Control Panels