

**Miniature GC For In-Situ Monitoring Of VOC's
Within A Cone Penetrometer**

**Final Report
July 1994 - May 1996**

RECEIVED
SEP 16 1996
OSTI

May 31, 1996

Work Performed Under Contract No.: DE-AC21-94MC31187

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Microsensor Systems, Inc.
62 Corporate Court
Bowling Green, Kentucky 42103

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED
OK

Disclaimer

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

**Miniature GC For In-Situ Monitoring Of VOC's Within A Cone
Penetrometer**

**Final Report
July 1994 - May 1996**

Work Performed Under Contract No.: DE-AC21-94MC31187

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Microsensor Systems, Inc.
62 Corporate Court
Bowling Green, Kentucky 42103

May 31, 1996

CONTENTS

	page
EXECUTIVE SUMMARY	1
1.0 INTRODUCTION	2
1.1 Program Objective	2
1.2 Success Criteria	3
1.3 Background Information	4
1.3.1 General Information	4
1.3.2 Projected Cost	6
1.3.3 Projected Performance	6
1.3.4 Waste Applicability	7
1.3.5 Other Potential Applications	7
2.0 GENERAL SYSTEM CONCEPT	8
2.1 Customizing the Cone-GC	9
2.2 User Interaction with the On-board Computer	10
2.3 General Operating Principles	11
3.0 SYSTEM HARDWARE DESCRIPTION	15
3.1 Power Supply	15
3.2 Carrier Gas Generation	15
3.3 Sample Injection Valve	16
3.4 GC Column / Oven Module	16
3.5 Oven/Pump/Valve Control Electronics	18
3.6 Solid State Detector	19
3.7 Microcomputer Module	20
3.8 Cone-GC Probe Package.....	20
4.0 SYSTEM SOFTWARE DESCRIPTION	21
4.1 General Software Operation	21
4.2 Signal Processing and Data Analysis Algorithm	22
5.0 SYSTEM OPERATIONAL CONSIDERATIONS	24
5.1 Calibration	25
5.2 Maintenance Interval	26
5.3 Power and Environmental Requirements	26
5.4 Input/Output Signals	27

6.0	EXPERIMENTAL	28
6.1	Cone-GC System Test and Evaluation Procedures	28
7.0	RESULTS	29
7.1	Chromatographic Performance	29
7.2	Halogen Specific Detector Performance	31
7.3	Hydrocarbon Detector Performance	32
8.0	CONCLUSIONS and RECOMMENDATIONS	46
10.0	APPENDICES	
	APPENDIX A: Cone-GC Mechanical Drawings	
	APPENDIX B: Cone-GC Electrical Drawings	

LIST OF FIGURES

- Figure 1. Cone-GC System Concept
- Figure 2. Cone-GC Probe System Diagram
- Figure 3. Cone-GC Injection Valve Configuration
- Figure 4. Cone-GC Control Module System Diagram
- Figure 5. Cone-GC System Schedule
- Figure 6. Cone-GC chromatographic resolution demonstrated in BTEX analysis.
- Figure 7. Chromatogram of methylene chloride.
- Figure 8. Chromatogram of chloroform.
- Figure 9. Chromatogram of carbon tetrachloride.
- Figure 10. Chromatogram of trichloroethylene.
- Figure 11. Chromatogram of tetrachloroethylene.
- Figure 12. Chromatogram of a mixture of chloroform, carbon tetrachloride, and trichloroethylene.
- Figure 13. Superposition of the chromatograms of five chlorinated test compounds.
- Figure 14. Relative sensitivity of halogen-selective detector to CCl_4 (100 ppm), vs. BTEX (10 ppm each).
- Figure 15. Calibration curve for trichlorotrifluoroethane, (FREON-113) using Cone-GC with halogen-selective detector.
- Figure 16. Calibration curve for carbon tetrachloride using Cone-GC with halogen-selective detector.
- Figure 17. Calibration curve for benzene vapor using Cone-GC with a metal-oxide-semiconductor (MOS) detector.
- Figure 18. Demonstration of the Cone-GC chromatographic reproducibility with six consecutive, superimposed chromatograms obtained from a sample containing 10.4 ppmv of carbon tetrachloride vapor.

LIST OF TABLES

- Table 1. GC Retention Times of Chlorinated Solvents.
- Table 2. Observed Sensitivity to Various Vapors.

MINIATURE GC FOR IN-SITU MONITORING OF VOC'S WITHIN A CONE PENETROMETER

EXECUTIVE SUMMARY

A miniature, low cost, gas chromatograph (GC) has been developed specifically to fit within a one inch (inside diameter) Cone Penetrometer for the real-time, in-situ characterization of the volatile organic vapors within the soil profile. Measuring environmental soil contaminants from within the cone penetrometer is expected to provide rapid and accurate site characterization data at a significantly reduced cost.

This novel, fully automatic "Cone-GC" has a rugged solid-state sensor that allows the use of ambient air as the carrier gas, thereby eliminating the need for compressed cylinders of gas or other consumables to perform the analyses. The system has a 0.98 inch diameter by 36 inch long GC probe assembly that can fit inside a conventional cone penetrometer, a 100 feet long connecting cable assembly, and a power supply/data acquisition assembly that allows user interaction with the GC probe. System operation is very easy. A user must simply turn on the power switch and press a button to perform a measurement.

The demonstration system fabricated during this project was able to automatically detect and monitor a variety of halogenated VOC's at low part-per-million concentration levels with typical analysis times of less than five minutes. Special emphasis was placed on selectively monitoring chlorinated solvents that constitute a significant environmental remediation problem at many DoE sites. A novel halocarbon selective, solid-state, electrochemical diode detector used in the Cone-GC performed very well in these laboratory evaluations.

Overall, the sensitivity, selectivity, and ease-of-use of the "Cone-GC" system should make it very attractive for environmental site characterization and monitoring. Continued effort will be required to actually install the "Cone-GC" into a commercial cone penetrometer system and to develop methods and protocols for its use in site characterization applications.

1.0 INTRODUCTION

The "Cone-GC" was developed in response to a need for down hole, in-situ characterization of volatile organics within the soil profile, in the vadose zone, or a water headspace. A design based on the use of a miniature gas chromatograph was selected since it was believed that such an instrument would be adaptable to a broad range of analytes and could be used in complex, real-world situations where the environmental contaminants to be monitored may exist in complex mixtures with other vapors. The Cone-GC is versatile and will also fit within many other soil probes, hole liners, and minimally intrusive emplacement systems where small size in addition to high performance are required. The Cone-GC was designed to allow environmental specialists for the first time to obtain immediate, in-situ chemical measurements in a soil probe and to make real-time, on-site decisions that will greatly reduce the time (and cost) of site characterization and remediation. It will no longer be necessary to collect samples (using long sampling lines that may become contaminated), send them to an off-site laboratory for analysis, and then wait hours or days for results.

1.1 Program Objective

The primary objective of this program was to develop a miniature gas chromatograph with a solid-state detector, that could fit within a 1" I.D. Cone Penetrometer. The focus of the "Cone-GC" program was to develop a self-contained gas chromatograph probe, that required only a small compressed air hose and electrical connections to a Control Module, to operate. The Control Module contains a microcomputer and a small carrier gas compressor to interface with the Cone-GC. The computer automatically controls instrument operations, collects and analyzes data, stores results in onboard memory, and presents the data to the operator in a user-selected format. The results can be sent to a printer or PC. The goal was to produce a very small, portable, rugged, low cost, computer controlled GC system that could provide in-situ identification and quantification of specific VOC's, with "one button" operation and a short analysis time.

1.2 Success Criteria

Several important things need to be accomplished for the Cone-GC to be able to meet proposed objectives. These included:

1. Development of a gas chromatographic column fabricated in a helical coil less than 1" in diameter.
2. Miniaturization of other GC system components such as injection valves, detectors, and support electronics.
3. Packaging of the GC components, vapor transfer lines, fittings, etc. into a unit that will fit within the cone penetrometer 1" diameter limit.
4. Detection and quantification of typical organic vapors with the completed system in a laboratory environment.
5. Development of control, data acquisition, and signal processing software to permit fully automatic operation.

All of these objectives have been realized in the Cone-GC program.

Specific performance goals proposed for the Cone-GC included:

- Detect a broad range of the common soil organics (i.e., benzene, toluene, TCE, etc.)
- Detection limit for most volatile or semi-volatile organics of 0.5 ppm or less
- Measurement time of 6 minutes or less
- Onboard computer memory to log up to 8 hours of continuous measurements
- Capable of either single measurements on demand, or continuous and automatic operation for extended periods of time (e.g., weeks) with no routine maintenance
- Have an RS 232 serial data port to provide the option for either on-site or remote data reporting.

1.3 Background Information

The nature of the problem to be solved, anticipated performance capabilities of the system and other potential applications of the Cone-GC are outlined below.

1.3.1 General Information

Environmental waste site characterization and remediation efforts demand extensive information about the concentration of specific chemical species at the site. The objective of this project was to develop a gas chromatograph that could be used within a cone penetrometer for accessing and quantifying organic contamination at DOE environmental clean-up sites. The Cone Penetrometer Gas Chromatograph is constructed in the shape of a cylinder whose maximum outside diameter is less than one inch, thereby allowing it to be incorporated as part of the piezocone assembly which is inserted into the soil. This instrument provides the capability for the rapid assessment of vadose zone contamination allowing data to be collected directly at the emission source. By taking an in-situ measurement the Cone-GC can establish a stratigraphic contamination emission profile with no sample handling. The Cone-GC offers the unique capability of being able to go to the sample collection zone instead of the sample being collected and brought back up to a instrument on the surface. This will avoid the need for long sample collection lines, pumps and syringes, providing a convenient means for in-situ measurements. An additional use of the Cone-GC would be in perimeter monitoring of a hazardous waste area to gauge the potential migration of volatile organic contamination across the monitored zone.

The Cone-GC is completely self sufficient instrument requiring only electrical power to operate. As figure 1 illustrates the Cone-GC is split into two components; the Control Module (located above ground) and the GC sample probe assembly that resides inside the cone penetrometer rod that travels down into the earth. The cable connection between the GC piezocone and the Control Module provides a power, compressed air, and data transmission conduit.

DoE CONE PENETROMETER GC CONCEPT SKETCH

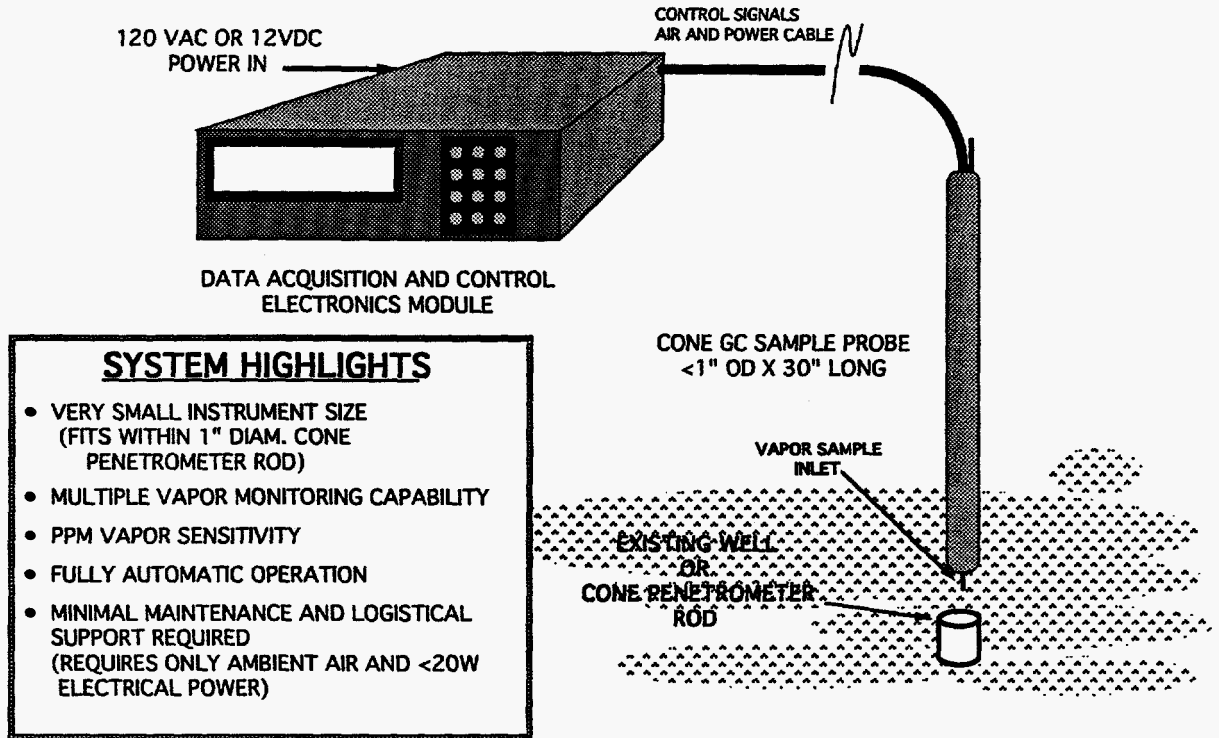


Figure 1. Cone-GC System Concept

The Cone-GC is designed to minimize the need for typical support overhead associated with gas chromatography. The instrument uses a miniature temperature controlled packed-column for sample analysis. Sample injection is performed by an electrically controlled gas sampling valve and the entire instrument operation is controlled by a microcomputer using a menu driven interface program. The Cone-GC can accept different detector options that allow additional analysis flexibility and compound detection selectivity. A halogen specific detector was developed for the Cone-GC that is well suited to the monitoring needs of many DoE waste sites. Alternatively, the instrument can be equipped with a general purpose hydrocarbon detector that is not especially sensitive to halogenated compounds. These detectors in combination with the specific column selection offer ways to improve instrument selectivity. The Cone-GC is designed to provide ppm detection levels of multicomponent samples. The typical analysis time is usually less than 10 minutes. The instrument interface has been designed to be successfully run by a field technician having little or no training in the operation of a conventional gas chromatograph.

1.3.2 Projected Cost

The commercial purchase cost of the complete Cone-GC is anticipated to be about \$10K. This price includes both the GC probe and the Controller Module. A replacement probe assembly is expected to cost about \$4K in small quantities. These costs are budgetary estimates.

1.3.3 Projected Performance

The Cone-GC is designed to detect and identify ppm levels of chlorinated organics or hydrocarbons at DOE environmental clean up sites. Sampling and analysis times of less than ten minutes will allow accurate, in-situ measurements to be made without the uncertainty associated with vapor sampling through long, unheated transfer lines, or the uncertainty associated with sample composition changes that occur when analysis is delayed.

1.3.4 Waste Applicability

Depending on the particular detector and chromatographic column installed, the Cone-GC is capable of both general hydrocarbon analysis and chlorinated compound analysis. Particular emphasis in this program was placed on development of an instrument that could do a good job analyzing and monitoring chlorinated and per-chlorinated hydrocarbons typically found at DoE sites.

1.3.5 Potential Commercial Applications

The Cone-GC can do many jobs. Its small size, ease of use, high sensitivity, high selectivity, and adaptability make it very attractive for most routine environmental monitoring applications. With no modification, the Cone-GC can also be used for monitoring a wide variety of toxic vapors in the workplace.

2.0 GENERAL SYSTEM CONCEPT

The Cone-GC offers new capability for the detection and monitoring of environment pollutants. Based on the well established technique of gas chromatography, the fully computerized Cone-GC permits even relatively inexperienced users to quickly obtain high quality results. Modular in concept, the Cone-GC portable gas chromatograph combines a temperature controlled analytical "column", an automatic sample collection/injection scheme, a low-maintenance solid-state detector, and an ambient air carrier gas generator to provide dependable results from an instrument that is exceptionally compact and easy-to-use. The system is designed to be versatile. The Cone-GC can be operated on 120 VAC or 12 VDC power, either locally or remotely via the RS232 serial port. A built-in relay switch that can activate at a user selected threshold concentration permits the instrument to function as a dedicated process controller. Experienced users can readily modify the operational configuration of the instrument to perform custom analyses.

The Cone-GC has many distinguishing features, such as a menu driven computer program to guide the user through instrument operations and the use of scrubbed ambient air as the carrier gas, which eliminates the need for cylinders of compressed gas. It was designed for reliable, consistent performance over a wide range of operating conditions and can easily detect many organic vapors at concentrations substantially below currently established OSHA and EPA action levels.

The Cone-GC uses a modular design to simplify modification and maintenance of the instrument. There are two modules that can be changed to optimize the instrument for a particular vapor analyte. These include the GC column/oven module, and the detector module.

In its simplest configuration, the Cone-GC can be used as a low resolution, general purpose, portable gas chromatograph. Raw chromatograms can be viewed on an optional serial printer or computer. By comparing chromatograms of known standards with chromatograms of field samples, an experienced chromatographer can easily obtain accurate quantitative and qualitative analytical results.

The real power of the Cone-GC lies in its ability to perform fully automated analyses for users with little prior training. The Cone-GC can be set up and calibrated at the factory to be a turn-key organic vapor monitor for a variety of environmentally important vapors. The user has no flow rates to adjust, no compressed gas cylinders to connect, no

temperatures to set, and no gauges or dials to watch. One simply plugs in the machine to a power source and turns it on. Reliable analyses of ambient air can be obtained in about 5 minutes by merely pressing a button and reading the results on the front panel display. The instrument can be instructed to monitor ambient air continuously, and to store the results in its internal, battery-backed 8 hour data log memory or, report them to a printer or personal computer by way of its built-in RS232 serial port.

2.1 Customizing the Cone-GC

Many different requirements exist for monitoring organic vapors. Each application often demands an instrument having a unique range of sensitivity, and adequate selectivity to discriminate against the unique set of other organic vapors that may coexist at the measurement location. The Cone-GC affords a great deal of flexibility due to its modular design. The selectivity can be "tuned" for specific applications by adjusting the chromatographic separation conditions (e.g., column packing, temperature, and flow rate) or by using a different detector. Thus, customizing the Cone-GC for various applications involves the selection of appropriate hardware combined with calibration of the instrument for the analyte of interest. Experienced chromatographers will find this to be a straightforward task. Less experienced users can select dedicated analyzer configurations that are pre-set at the factory to perform a particular analysis.

Setting up the Cone-GC as a dedicated vapor monitor for up to four organic vapors requires the installation of the appropriate GC column and detector module, along with flow rate adjustment and recalibration of the complete system (usually with a minimum of three concentrations of each vapor to be monitored). These calibration data (i.e., known concentration and observed peak height and retention time) must then be entered into the Cone-GC. System operating schedule parameters (e.g., sampling and injecting durations) may also be changed. Modification of the system schedule, the calibration tables, and peak picker information can be easily accomplished via a computer terminal connected to the RS232 port.

2.2 User Interaction with the On-board Computer

The complex internal operations of the Cone-GC are controlled by a powerful onboard microcomputer that supports a simple menu driven user interface. All interaction with the instrument is via the front panel keypad and display or a computer terminal connected to the RS232 port. The CONE-GC's intuitive dialog structure offers abundant prompting and error checking of user input. As a result, the need for frequent references to the operating manual has been substantially eliminated.

While the Cone-GC can be used as a stand alone instrument by simply reading the results of each analysis on the front panel display, it is often desirable to obtain a hardcopy output of each chromatogram run. Hardcopies can be made with an optional, battery powered, serial printer connected to the RS232 port. Results can also be sent from this port to most personal computers where chromatograms and reports can be displayed and stored on disk memory.

2.3 General Operating Principles

A system diagram of the basic Cone-GC Organic Vapor Monitor is shown in figures 2 and 3. Air samples are drawn into the Cone-GC sample loop automatically by the internal Cone-GC sample pump. The chromatographic analysis is initiated when the sample trapped by the sample loop is injected into the GC Column by energizing the 8-port rotary injection valve illustrated in figure 5. Once injected, the sample is swept into the column by clean air carrier gas that is derived from an ambient air pump and activated carbon scrubber located in the Cone-GC Control Module. A mixture of chemical compounds injected into the column can be separated into its individual components by virtue of the fact that different vapor molecules often exhibit different retention times on the column. Typically, compounds that have a higher boiling point and polarity will travel through the column more slowly than compounds having a lower boiling point and polarity. As each compound elutes from the column it is sensed by the detector which produces a frequency signal that is proportional to the concentration of the vapor passing by the detector. Thus, a characteristic chromatogram can be obtained for each vapor mixture that ideally contains a "peak" for each constituent whose height is related to the amount (i.e., concentration) of analyte present, and whose time from the injection point (i.e., retention time) is an indicator of the analyte's identity. Sample collection, injection, detector data acquisition, and data analysis are all controlled by the Cone-GC internal microcomputer and a user-settable system schedule. A typical system operating schedule is shown in figure 4. Once the chromatogram is completed, the microcomputer determines retention times and baseline corrected heights for all peaks. Peak heights can be determined in as many as four user definable windows and converted to concentrations by using the calibration tables stored in memory for each window. Upon power-up, the system energizes the oven and checks the carrier pump and solid-state detector for proper operation. During warm-up, the user is free to modify system parameters and to generate reports of data stored in the non-volatile data log memory. The stored data can be reported in a variety of formats selected by the user from the menu.

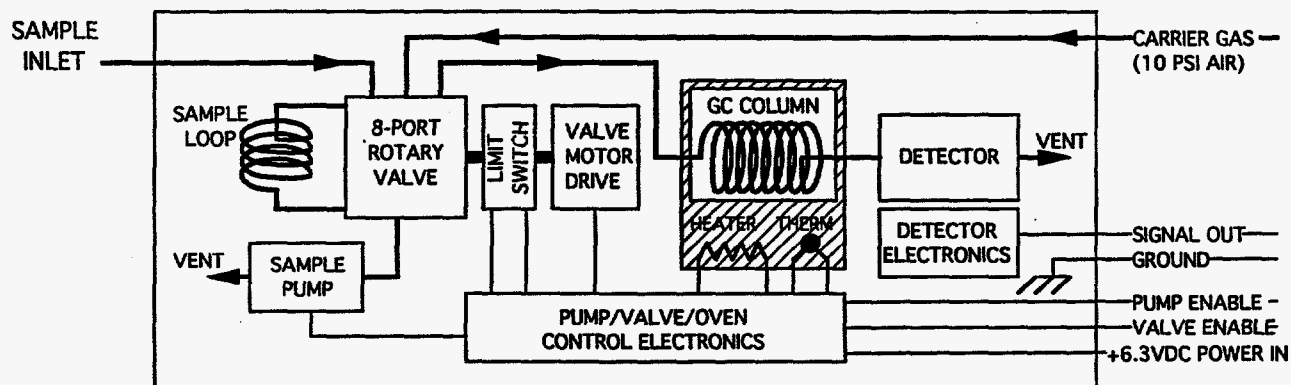


Figure 2. Cone-GC Probe System Diagram

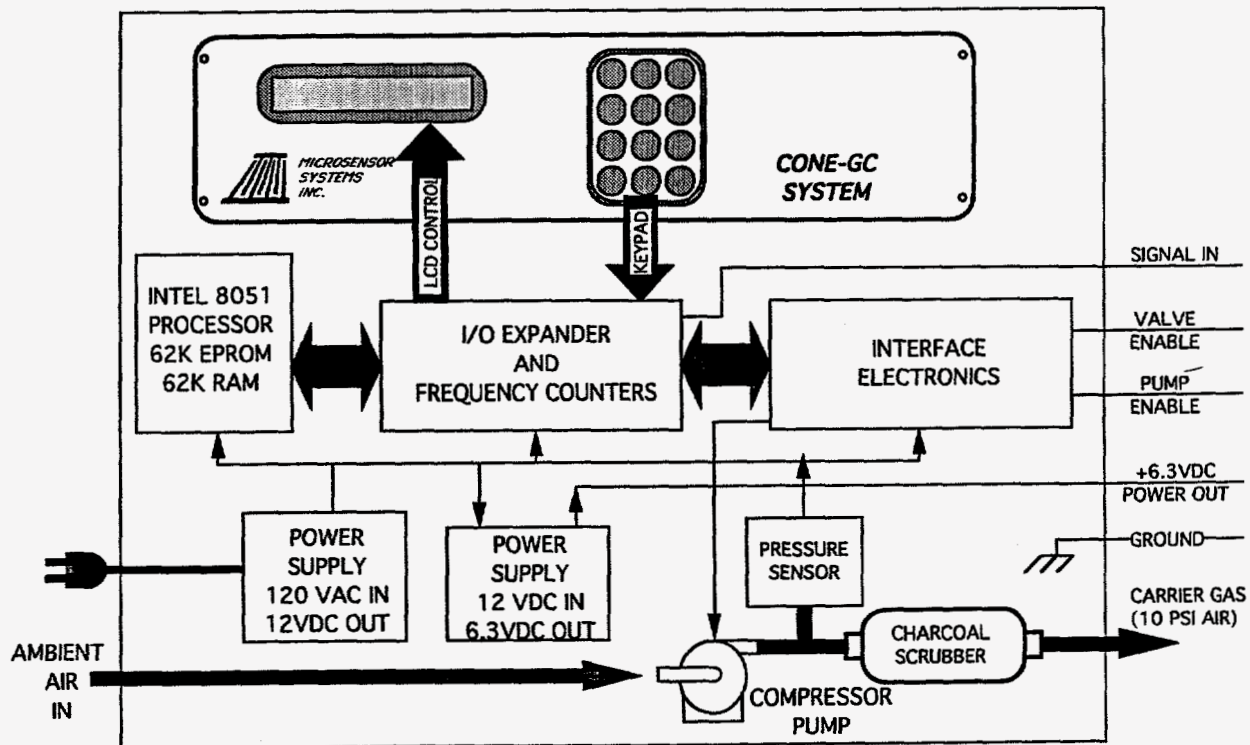


Figure 3. Cone-GC Control Module System Diagram

TYPICAL SYSTEM SCHEDULE FOR CONE-GC CHROMATOGRAPHIC ANALYSIS

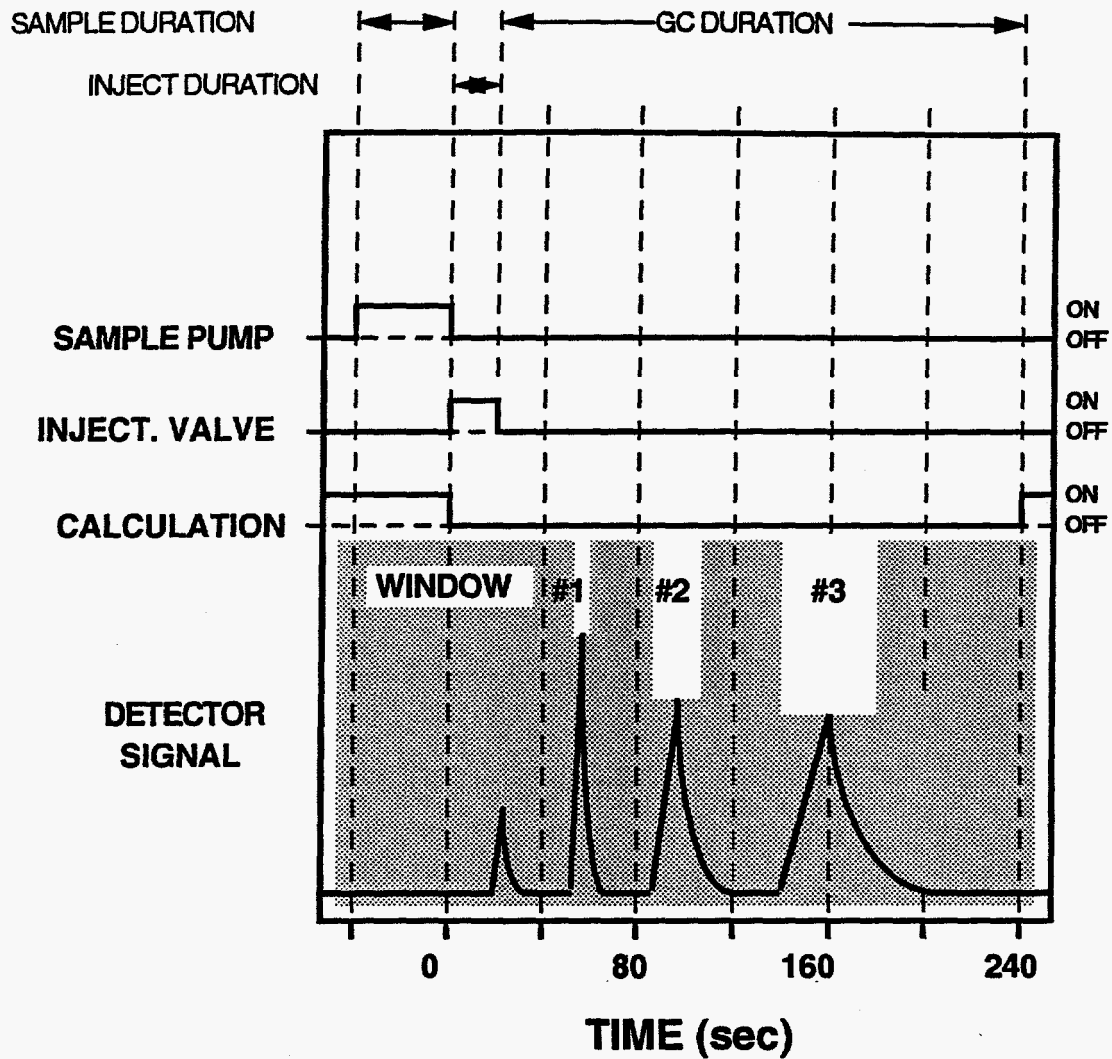


Figure 4. Cone-GC System Schedule

3.0 SYSTEM HARDWARE DESCRIPTION

The Cone-GC Control Module consists of the following items:

- Power Supply
- Microcomputer and Interface Electronics
- Carrier Gas Compressor
- LCD Display, Keypad, and Chassis

The Cone-GC Probe assembly consists of the following items:

- GC column and oven
- Sample injection valve
- Sample pump
- Sample loop and (optional) Sample Concentrator
- Power control electronics
- Solid-state detector and electronics
- 0.987 inch outside diameter tubular brass housing
- Multi-conductor signal/control cable with 1/8 inch compressed air hose

3.1 Power Supply

The internal electronics of the Cone-GC are designed to operate on 12 VDC. A self-contained power supply converts 120 VAC (50 - 60 Hz) line power to the required 12 VDC and 6.3 VDC. The Cone-GC will also operate on external battery power. Average power consumption of the instrument (after oven warm-up) is approximately 10 watts.

3.2 Carrier Gas Generation

The carrier gas for the Cone-GC is generated from ambient air using a small compressor pump and an activated carbon scrubber to remove impurities. The pressure in the scrubber tank is held constant by means of a silicon chip pressure sensor and a feedback control loop to the pump motor. The carrier gas pump adds air to the scrubber tank as required to maintain a pressure set by an internal potentiometer on the Cone-GC electronics pc board. The maximum carrier gas pressure achievable with this scheme is approximately 10 psi. Ordinarily, this pressure is able to generate flow rates of up to 10 sccm on 1/16 inch OD packed columns that are less than 4 feet long.

3.3 Sample Injection Valve

The Cone-GC collects ambient air samples automatically. In a typical automatic sampling mode, the on-board sample pump pulls ambient vapors into a sample loop of fixed volume for subsequent injection into the GC column. The Cone-GC sample loop consists of a 16 inch long piece of 1/16 inch OD x 0.040 inch ID nickel tubing, helically wound on a 5/8 inch diameter mandril. The loop has an internal volume of 0.33 cc. Vapor samples are pumped into this loop under vacuum from the sampling pump. Samples stored in this loop are injected into the GC column for subsequent separation and detection. With the sample loop, an extended sampling line can be added to the gas inlet fitting to permit sample collection from distances up to 10 feet away.

The Cone-GC can be modified with the addition of a sample concentration module for applications requiring very high sensitivity. Sample enrichment is accomplished by pulling ambient air through the absorbent trap at a high collection rate (e.g., 200 sccm). The adsorbent tube preferentially traps the organic vapors of interest, while permitting the very low boiling compounds (and water vapor) to escape. The adsorbent tube is then heated to an elevated temperature (e.g., 140°C for Tenax GC™) to vaporize the trapped organics which are then injected into the GC column at a much lower rate of flow (e.g. 5 sccm). The concentration enrichment factor is determined by the ratio of the volume of air sampled to the volume of carrier gas required to desorb the analyte of interest.

3.4 GC Column / Oven Module

The Cone-GC Organic Vapor Monitors use isothermal (e.g., 50°C) 1/16" diameter packed chromatographic columns. Each GC Column / Oven Module contains a column mounted on an aluminum thermal mass with an integral heating element and temperature sensor. The entire assembly is surrounded with a fiberglass insulating layer. The column packing, and the temperature of the GC oven are parameters that can be varied to optimize the separation and identification of a specific organic vapor. Power and material stability considerations limit the temperature of the module to the range from ambient to 100°C. The packed column provides satisfactory resolution for monitoring many typical organic vapor mixtures. The column length is limited to about 3 feet due to space limitations within the oven, and the 10 psi maximum carrier gas pump pressure.

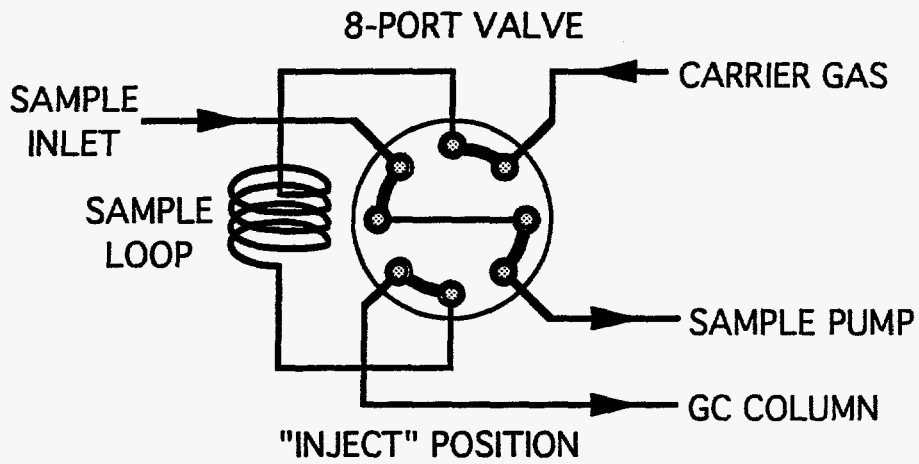
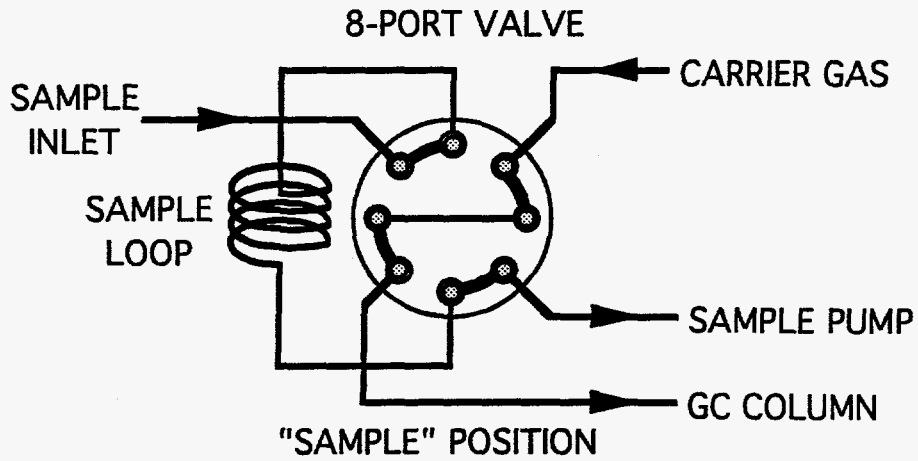


Figure 5. Cone-GC Injection Valve Configuration

3.5 Oven/Pump/Valve Control Electronics

Electronic circuitry is required to control then GC column oven temperature, and to activate the injection valve and sample pump. A schematic for this subsystem is provided in the Appendix.

GC Column temperature control is accomplished using a closed-loop, On/Off type controller. Oven temperature is sensed with a small thermistor whose voltage signal is compared to that of a setpoint potentiometer. If the temperature is low then an analog comparator circuit turns on power to a resistive heating element in the GC column mandril. When the temperature rises, the thermistor signal varies proportionally until it matches that of the setpoint pot and the comparator turns the heat off. This simple control system allows the temperature of the column to be maintained within about ± 0.8 °C of the desired setpoint temperature.

Sample pump operation is controlled using a programmable voltage regulator integrated circuit (MAXIM MAX667). When enabled, this device supplies a constant 5 volts DC to the sample pump, regardless of power supply voltage variations.

Valve control is the most complex operation in the Cone-GC Probe since the travel of the valve rotor must be limited (using limit switches) and the control must be "smart" (i.e., the circuit must restore the valve to the correct position even after power has been interrupted. A system of digital logic using flip-flops, an exclusive-OR gate, and a mechanical relay is used to accomplish the valve control task.

3.6 Solid State Detector

The Cone-GC has been developed primarily to detect chlorinated hydrocarbons typically found at DoE remediation sites. For this application a solid-state electrochemical diode (INFICON D-TEK P/N 703-020-G1) is used. This device consists of a tiny bead of a high temperature solid state electrolyte deposited onto a noble metal filament. The device is mounted in a small 3-pin, TO-39 style, transistor header. A current passing through the filament heats the bead to several hundred degrees centigrade. A small bias potential is applied to a small electrode attached to the bead. Chlorocarbon vapors that contact the bead surface decompose to release chlorine ions that are transported through the bead and cause an observed increase in conductivity. The selectivity for halogens, and chlorine in particular, is extremely high. The beads typically offer an operating life of about 100 hours before requiring replacement.

The detector supporting electronics has a high efficiency, step-down voltage regulator to provide the 1.2VDC @ 0.4 A required by the filament from the 6.3VDC Cone-GC Probe power supply. The detector electronics also have a regulated ± 5 VDC power supply, a precision current-to-voltage converter, and a precision voltage-to-frequency converter to provide a noise-immune signal to the remotely located Control Module. The signal is in the form of a TTL logic compatible frequency that is proportional to the observed bead conductance. The response of this detector is non-linear with respect to vapor concentration.

Alternatively the Cone-GC can be equipped with a Metal-Oxide-Semiconductor (MOS) gas sensor. This device exhibits some selectivity to typical hydrocarbons (both aromatic and aliphatic) and common solvents (e.g., alcohols, ketones, etc.). They do not respond at all to fully chlorinated compounds like carbon tetrachloride, and they respond only weakly to many other chlorinated hydrocarbons. The detector has a ceramic tube coated with tin oxide that is heated with a filament to several hundred degrees centigrade. These solid-state detectors have proven to be extremely robust. They require no maintenance of any kind. Like the electrochemical diode, the detector has a non-linear response to vapor concentration which necessitates calibration of the detector at several concentrations over its operating range if optimal accuracy is required. The non-linear response offers the advantage of expanding the dynamic range of vapor concentrations that can be detected.

3.7 Microcomputer Module

The internal Cone-GC microcomputer module consists of a pair of printed circuit boards. One board (located on the bottom) contains a fast CMOS 8051 series microprocessor, 64K of EPROM, 64K battery backed RAM, battery backed clock/calendar chip, serial I/O ports, keypad input port, and 32 bits of digital I/O, regulated 5 volt power supplies, and a multichannel digital frequency counter to read the detector signal. The second pc board (on the top) contains circuitry to control the carrier gas and sample pumps, fan, valves, heaters, alarm relay and LCD display. The batteries in the clock calendar and RAM memories have a lifetime in excess of 5 years before replacement should be necessary. These battery backed devices permit the Cone-GC to retain logged data and important system parameters even when the instrument is not turned on.

Schematics for the Microcomputer electronics are provided in the Appendix

3.8 Cone-GC Probe Package

The Cone-GC Probe resides in a custom-drawn 0.987 inch OD brass tube that provides a rigid mechanical shell for the instrument as well as electrical shielding. The forward end of the probe is equipped with a sintered metal vapor inlet to reduce the possibility of dirt and grime from entering the gas chromatograph. The aft end of the probe has a twist-lock electrical connector and a swagelok fitting to allow connection to the 100 feet long umbilical cable that provides the probe assembly with electrical power and compressed air carrier gas.

4.0 SYSTEM SOFTWARE WARE DESCRIPTION

4.1 General Software Operation

The operating system software resident in the microcomputer module largely defines the operational characteristics and capabilities of the Cone-GC series. The version of the system software is briefly displayed on the front panel LCD a few seconds after power is turned on.

All interaction with the Cone-GC is conducted by menu selections. The MAIN menu provides the option to RUN, CALIBRATE, REPORT, or MODIFY the instrument. The user can escape from any of these modes by pressing the "0" key.

In RUN mode the user can select the type of run (i.e., MANUAL, or AUTORUN), the type of report (i.e., real time chromatogram, plotted chromatogram, calculated results, etc.) and destination of the data report (i.e., LCD, printer, or personal computer). Once the measurement configuration is defined, the Cone-GC automatically performs the analysis and displays the results.

CALIBRATE mode offers users the option to perform a single point span adjustment of the Cone-GC internal calibration curve. Ordinarily, the Cone-GC contains an internal 5 point calibration curve for each analyte. These points can be automatically fitted with a second order polynomial expression. To recalibrate the Cone-GC in the field it is merely necessary to connect a standard vapor of known concentration, enter that concentration into the keypad and start the CALIB process. After the chromatogram is complete, the user is given the YES/NO option of updating the "R" (Response) factor for each vapor.

REPORT mode offers reports of raw data, results stored in the 8 hour data log, chromatogram plots, and system parameters lists.

MODIFY mode affords total flexibility for the user to change the operational characteristics of the Cone-GC. Modifying OPTIONS allows the user to select alarm threshold concentrations. Modifying the SCHEDULE causes a menu prompt to send or receive the schedule parameters (i.e., "CLONE" the parameters) from an attached personal computer. The schedule parameters control of the pump, valve, and heater timing that define the chromatographic analysis. Modifying the CALIB TABLE permits up to six calibration points to be stored (i.e., Cone-GC signal value, and

vapor concentration producing it) for up to 4 analyte vapors. The CALIB TABLE also contains the "R" factor from the most recent calibration run and permits the user to select either a LINEAR or POLYNOMIAL calibration curve look-up. Modifying the PEAK PICKER allows the user to define up to four retention time "windows" where particular vapors are expected to elute. The PEAK PICKER also allows the user to specify a region over which the computer will integrate the entire chromatogram to produce a total signal value.

4.2 Signal Processing and Data Analysis Algorithm

During the chromatographic "run", the Cone-GC microcomputer controlled frequency counter measures the signal from the detector once every second and stores the results in an internal data array that is not saved after each run or power-off. Upon completion of the run, these data are automatically analyzed to determine the retention times and baseline corrected peak heights for each peak in the chromatogram. The user can specify a retention time at which this general peak search will begin. This feature permits early, irrelevant peaks to be completely ignored, thereby reducing computation time. Baseline correction is accomplished by determining the start and end point of the peak. The computer calculates the baseline signal values corresponding to a straight line between these two points. The peak height is then calculated by subtracting the calculated baseline signal at the time corresponding to the peak maximum from the value of the detector signal at the peak maximum. The peak start point is defined when the first derivative is greater than zero and the second derivative is greater than or equal to zero for 2 consecutive points. The peak end point is defined either as the point that is six times the distance from the start point to the peak maximum, or as the point at which another start point is detected. The peak maximum is determined when the first derivative is less than zero for three consecutive points.

Once all the peak heights and retention times have been determined, then a search is made to see if any of these peaks appear in the windows defined by the PEAK PICKER parameters. A measured peak that is closest to the specified peak location and within $\pm 1/2$ of the window width will be classified as the vapor specified for the window.

Determination of a vapor concentration is made by using the calibration table data stored for its window. The calibration method can be selected based on either a linear or polynomial curve fit. The linear calibration

method is the simplest, requiring only one point in the calibration table. Zero concentration at zero peak height is assumed. The method is useful when it is only necessary to determine whether or not a particular vapor concentration threshold has been exceeded. The quantitative accuracy of the linear calibration method at concentrations significantly different (e.g., ten times higher or lower) from the calibration concentration is very poor owing to the non-linearity of the detector response. When highly accurate concentration values are required over a wide range of concentrations, the polynomial calibration method is most desirable. This method requires a minimum of three calibration points that bracket the range over which the highly accurate results are required. With the polynomial calibration method, zero concentration at zero peak height is not assumed. The Cone-GC will accept up to six calibration points for the curve. These calibration data (i.e., concentrations and corresponding peak heights) are converted to natural logarithm form and fitted to a second order polynomial using a least squares criterion. The actual second order polynomial calibration curve equation for each window is available in the system parameter listing. Measured peak heights are adjusted by dividing them by the "R" factor found by the most recent calibration. The adjusted peak height value is used by the polynomial equation to obtain the natural log of the vapor concentration.

Summary data from each chromatogram is stored in non-volatile, battery backed memory. Up to 80 summaries (i.e., about 8 hours of continuous measurements) can be stored before old results will be replaced by new results in the FIFO buffer. The data summary includes the date, time, vapor peak height, retention time, and concentration. Concentrations are calculated from stored peak height data at the time a report is generated.

5.0 SYSTEM OPERATIONAL CONSIDERATIONS

5.1 Calibration

Field calibration of the Cone-GC is basically a single point span adjustment to correct for variations in the apparent instrument sensitivity. A standard sample is supplied to the instrument, a chromatogram is run, and the peak height of each standard is measured. If the observed standard peak height is different from a peak height predicted theoretically from the stored, multi-point calibration curve and polynomial equation, then a correction to the "R" response factor is made. The "R" factor is used to scale subsequent measurements to produce the correct concentration reading.

The recommended Cone-GC calibration interval depends on the application. For routine environmental surveys or process control applications, a weekly calibration should suffice. Calibration is required to verify the proper operation of the entire instrument and to correct for variations in the sensitivity of the instrument caused by small variations in such things as detector sensitivity and sample collection efficiency.

Calibration of the Cone-GC must be made using the same method by which the sample will be introduced. Normally, calibration standards are selected at concentrations in the range of those for which the highest accuracy is desired. Gas calibration standards can be obtained from several vendors as trace (e.g., 1 ppm concentration by volume) additives to cylinders of compressed air or nitrogen. A sample bag is connected to the calibration gas standard and partially filled with the standard. The bag should then be emptied and refilled to guarantee that the bag has been purged of residual air diluent. The bag containing the calibration standard should then be promptly connected to the Cone-GC inlet.

5.2 Maintenance Interval

The only item in the Cone-GC system that needs scheduled replacement is the activated charcoal in the carrier gas scrubber tank. The recommended replacement interval is 500 hours of instrument operation depending on the cleanliness of the ambient air. (The appearance of large *negative* peaks in the chromatogram is indicative of contaminated charcoal.) The elapsed time of scrubber use can be obtained from the system parameter menu. Scrubber life can be extended considerably by using an external charcoal filter attached to the ambient air carrier gas inlet located on the rear panel of the Cone-GC Control Module. The activated charcoal can be replaced by removing the Cone-GC cover (4 screws) and removing the 4 screws on the scrubber tank holder. Disconnecting the Swagelok® fittings on each end of the stainless steel scrubber tank permits it to be removed completely from the instrument. After unscrewing one of the pipe fittings from the end of the scrubber tank, the charcoal can be poured out and replaced with fresh, activated charcoal. A wad of glass wool helps to retain the charcoal bed before replacing the pipe fitting and re-installing the tank into the Cone-GC. The duty cycle of the carrier gas pump should be noted before and after charcoal scrubber replacement as a diagnostic test for leaks. It is a good practice to check the retention times of a standard vapor to verify that the carrier gas flow rate has not been altered by the charcoal replacement.

The carrier gas pump used in the Cone-GC typically runs for 5000 hours before replacement is needed. The sample pump (which is located in the Cone-GC Probe) is used so sporadically that it seldom needs replacement. Infrequently, leaks might develop in the carrier pump diaphragm that can be fixed by simply re-tightening the screws on the pump diaphragm retainer.

The chromatographic column and oven is very stable, requiring no scheduled maintenance. Column replacement is appropriate if there is obvious degradation of the resolution of the instrument.

The solid state detector has proved to be quite reliable. No scheduled maintenance is required for the MOS detector. The electrochemical diode halocarbon detector requires replacement after about 100 hours of use.

The microcomputer uses battery backed RAM memory chips and a battery backed clock/calendar chip that contain lithium batteries with a five year rated operating life. Battery replacement will be needed after five years.

5.3 Power and Environmental Requirements

The Cone-GC requires either 120 VAC 50-60 Hz line power or 12 VDC power. A 2 amp fuse for the 120 VAC power supply is located near the power switch on the rear power entry module. A 4 amp fuse for the 12 VDC power supply is located in the cigarette lighter plug of the 12 VDC power cable. Both power sources are connected to their respective receptacles on the Cone-GC rear panel. A rear panel switch is used to select the power source. Power requirements are greatest (e.g., 15 watts) on power-up when the oven is cold. After about 10 minutes, when the oven is hot, the system power requirements drop to an average of about 7 watts.

The Cone-GC is designed to function in non-condensing atmospheres over the temperature range of +5°C to +40°C. Operation at ambient pressures in the range of 500 to 1500 torr is possible if the Cone-GC is calibrated at the operating pressure.

The chassis of the Cone-GC Control Module and Probe, while quite rugged, are not waterproof and operation away from sources of mud and water is recommended. The Cone-GC Control Module and Probe assembly can be operated in either a vertical or horizontal orientation.

The Cone-GC operates on low voltages and low powers but it is not certified for intrinsic safety and should not be used in explosive atmospheres.

5.4 Input/Output Signals

The Cone-GC offers three forms of I/O. All results, instrument status information, and menu selections can be viewed and accessed using the front panel LCD display and keypad. For dedicated applications, an internal, normally open, SPST relay can be programmed to close if a desired concentration threshold has been exceeded. This is especially useful for process control, and occupational safety applications. Access to the relay is provided through two pin jacks on the rear panel. Finally, a DB-25 RS232C connector on the rear panel offers full access to the control and reporting features of the Cone-GC. The RS232C data stream operates at 2400 baud with 8 data bits, 1 stop bit, and no parity. Signal lines for TxD (pin #3), RxD (pin #2), and ground (pin #7) are all active. Connection of the Cone-GC serial data port to a personal computer normally requires a null modem cable. To control the Cone-GC remotely using a personal computer it is simply necessary to send the "@" symbol to logon and the "&" symbol to logoff. Under remote control, both the front panel keypad and the remote computer can be used for input. When using external devices connected to the RS232 port it is advisable to first power up and initialize the external peripheral (i.e., pc or printer) before turning on or connecting the Cone-GC. This guarantees that the serial I/O port of the peripheral device will be initialized correctly.

6.0 EXPERIMENTAL

6.1 Cone-GC System Test and Evaluation Procedures

The Cone-GC systems were assembled and operationally bench tested prior to performing the overall system test.

Preliminary studies were conducted to establish an operating temperature for the GC Column, carrier gas flow rate, and injection timing that would allow adequate, but rapid separation of a representative set of chlorinated and aromatic hydrocarbons.

The initial system evaluation was carried out in the laboratory using the following compounds:

- benzene
- toluene
- ethylbenzene
- o-xylene
- methylene chloride
- chloroform
- carbon tetrachloride
- trichloroethylene
- tetrachloroethylene
- freon-113.

The aromatic hydrocarbon test vapors were delivered from compressed standard gas cylinders at a nominal concentration of 10 ppm. These vapors were transferred to ambient pressure gas sample bags for automatic sampling by the GC for analysis. The sample bags were attached to the GC sample inlet and the onboard pump pulled a vapor sample into the Cone-GC sample loop. Vapor dilutions were made to determine detector sensitivities, detection limits, and non-linearity.

The halogenated hydrocarbon vapors were prepared by injecting a small amount of headspace vapor (from reagent bottles containing the material) into a clean tedlar bag and back-filling the bag with zero air.

Certified standard compressed gas cylinders of dilute freon-113 in air (1,512 ppmv) and carbon tetrachloride in air (10.4 ppmv) were used to establish the quantitative sensitivity, detection limit, and non-linearity of the halocarbon gc detector. A certified standard compressed gas cylinder of dilute benzene, toluene, ethylbenzene, and o-xylene (BTEX) in air was used to characterize the MOS detector.

7.0 RESULTS

7.1 Chromatographic Performance

The Cone-GC chromatographic performance met the design specifications for moderate-resolution chromatography. Figure 6 shows the resolution of the components of a BTEX calibration gas mixture using a metal oxide semiconductor detector. The mixture contained benzene, toluene, ethylbenzene, and o-xylene, at a concentration of 10 ppmv each in air. The chromatogram illustrated in Figure 6 shows fairly good peak symmetry and adequate resolution of the four compounds to permit reasonable quantitation accuracy. Figure 6 also illustrates the graphical output plotting capability of the Cone-GC system. This plot was obtained directly from the Cone-GC connected to a small printer.

Chromatograms were obtained from a variety of chlorinated hydrocarbons to determine chromatographic retention times. The column was 36 inches long, 1/16 inch OD and 0.040 inch ID. It was packed with 80/100 mesh Supelcoport having a 5% loading of OV-101. Column temperature was 43°C and column flow rate was nominally 6 sccm. Typical results are shown in figures 7 - 18. Retention times are tabulated in Table 1. The results show that the retention time differences are significant enough to permit the Cone-GC to measure the concentrations of individual chlorinated solvents that may be present in a mixture.

Table 1. GC Retention Times of Chlorinated Solvents.

Compound	Retention Time (sec)
methylene chloride	52
chloroform	66
carbon tetrachloride	88
trichloroethylene	110
tetrachloroethylene	232

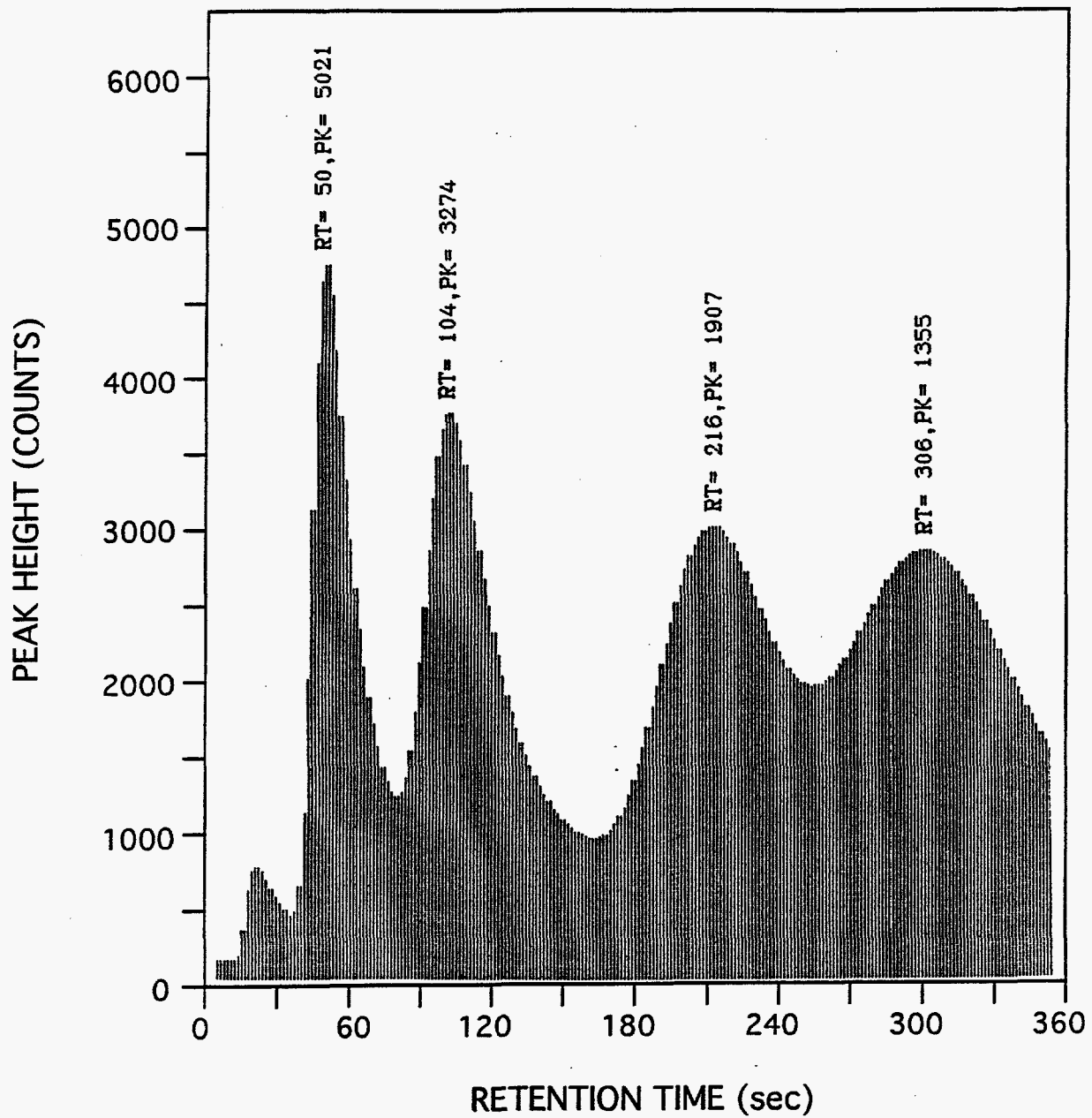


Figure 6. Cone-GC chromatographic resolution demonstrated in BTEX analysis.

7.2 Halogen Specific Detector Performance

The solid-state halogen-specific detector was shown to respond to several representative chlorinated solvents, as summarized in Table 1. Figures 7-11 show actual chromatograms of methylene chloride, chloroform, carbon tetrachloride, trichloroethylene, and tetrachloroethylene, respectively. These figures show the response of the detector to concentrations on the order of several thousand parts-per-million by volume. The detector seemed to respond rapidly to all the compounds, although tailing of the peaks was consistently observed, possibly owing to slow response of the detector. In all cases the symmetry was adequate to permit good quantitation of the peak.

Figure 12 is a chromatograph of a mixture of chloroform, carbon tetrachloride and trichloroethylene, showing that these three are easily resolvable. Figure 13 shows a superposition of the chromatograms of the five chlorinated test compounds.

The specificity of the detector was measured by analyzing a mixture of carbon tetrachloride (100 ppm), benzene (10 ppm), toluene (10 ppm), ethylbenzene (10 ppm) and xylene (10 ppm). Figure 14 shows the results of this analysis. It can be seen that the halogen-specific detector has a very low sensitivity (almost non-existent response) to the non-halogenated components of the mixture.

The quantitative sensitivity of the halogen-specific detector was determined by analysis of different concentrations of halocarbon 113 (Freon 113), using formulated dilutions of gas from a 1,512 ppm gas cylinder (certified standard cylinder from Matheson Gas Products). Figure 15 shows a calibration curve for this compound. The data indicate that a signal-to-noise of greater than 10 would be maintained down to a concentration of 3 parts-per-million. The response is quite non-linear, thus necessitating the use of polynomial calibration curves when accurate quantitation is desired.

The performance of the halogen-specific detector when challenged with carbon tetrachloride is presented in figure 16. As with freon-113, the curve is non-linear. Sensitivity was very good with a concentration of 0.1 ppm providing an easily detectable and quantifiable signal with a peak height of 114 counts and a standard deviation of ± 24 counts. The error bars associated with the data plotted in figure 16 indicate the relative standard deviation of the chromatographic peak height obtained from a set of carbon tetrachloride exposures at the same concentration. The carbon

tetrachloride standard samples were prepared by an automated vapor dilution apparatus that metered precise quantities of the carbon tetrachloride compressed gas standard into a tedlar bag that was backfilled with a precisely metered volume of zero air.

7.3 Hydrocarbon Detector Performance

Figure 6 showed the performance of a solid state, metal-oxide-semiconductor (MOS) hydrocarbon detector in the detection of benzene, toluene, ethylbenzene, and xylene (BTEX); common environmental pollutants associated with gasoline spills. The MOS device does not exhibit high sensitivity to many chlorinated solvents.

Figure 17 illustrates the calibration curve obtained from the MOS detector when used with the Cone-GC exposed to various concentrations of benzene vapor in air. The benzene vapor was obtained from certified compressed gas cylinders prepared by Scott Specialty Gas. The MOS detector also exhibits some non-linearity although not as large as the halogen specific detector. As in figure 16, the error bars associated with the data plotted in figure 17 indicate the relative standard deviation of the chromatographic peak height obtained from a set of benzene vapor exposures at the same concentration. For all but the highest concentration, the error bars fall beneath the circle used to plot the curve. The observed sensitivity of the MOS detector to benzene was good with a 0.99 ppm sample of benzene producing a peak height of 82 counts with a standard deviation of ± 4 counts.

The results of the sensitivity evaluation of the Cone-GC with both the halocarbon detector and the MOS detector are summarized and presented in Table 2.

Figure 18 demonstrates the chromatographic reproducibility of the Cone-GC with six consecutive, superimposed chromatograms obtained from a sample containing 10.4 ppmv of carbon tetrachloride vapor in room air. The retention time stability, injection volume reproducibility, and detector reproducibility are clearly very good to produce such results.

Table 2. Observed Sensitivity to Various Vapors.

Compound	Response Sensitivity (counts/ppm)	Test Conc (ppm)	Signal to Noise	Estimated Detection Limit (ppm)
Freon-113	119	3.02	10	0.6
Carbon Tet.	1,100	0.1	100	0.002
Benzene	83	0.99	10	0.2

Note: Tests were conducted using the halocarbon detector except for benzene, in which case the metal-oxide-semiconductor detector was used.

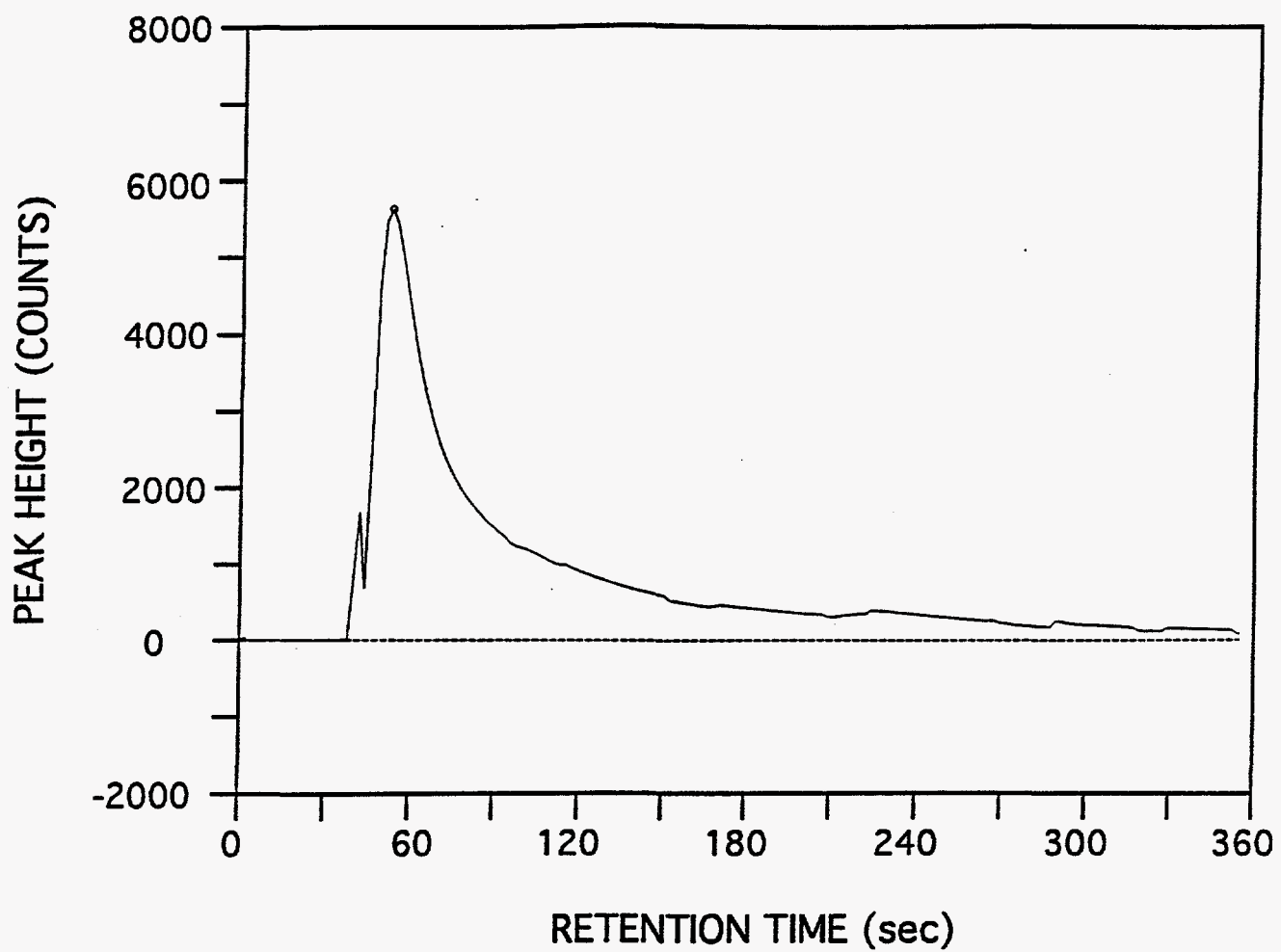


Figure 7. Chromatogram of methylene chloride.

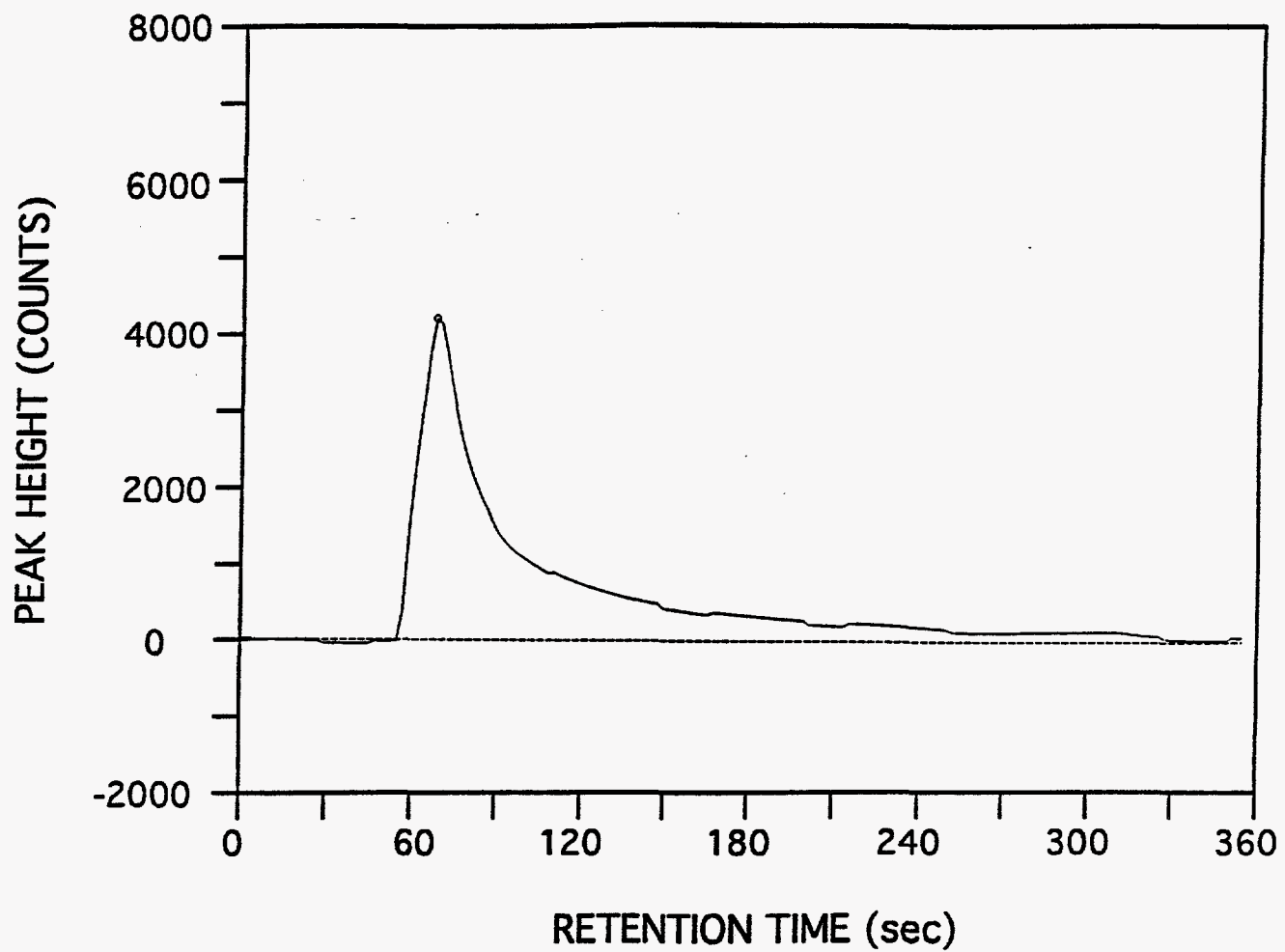


Figure 8. Chromatogram of chloroform.

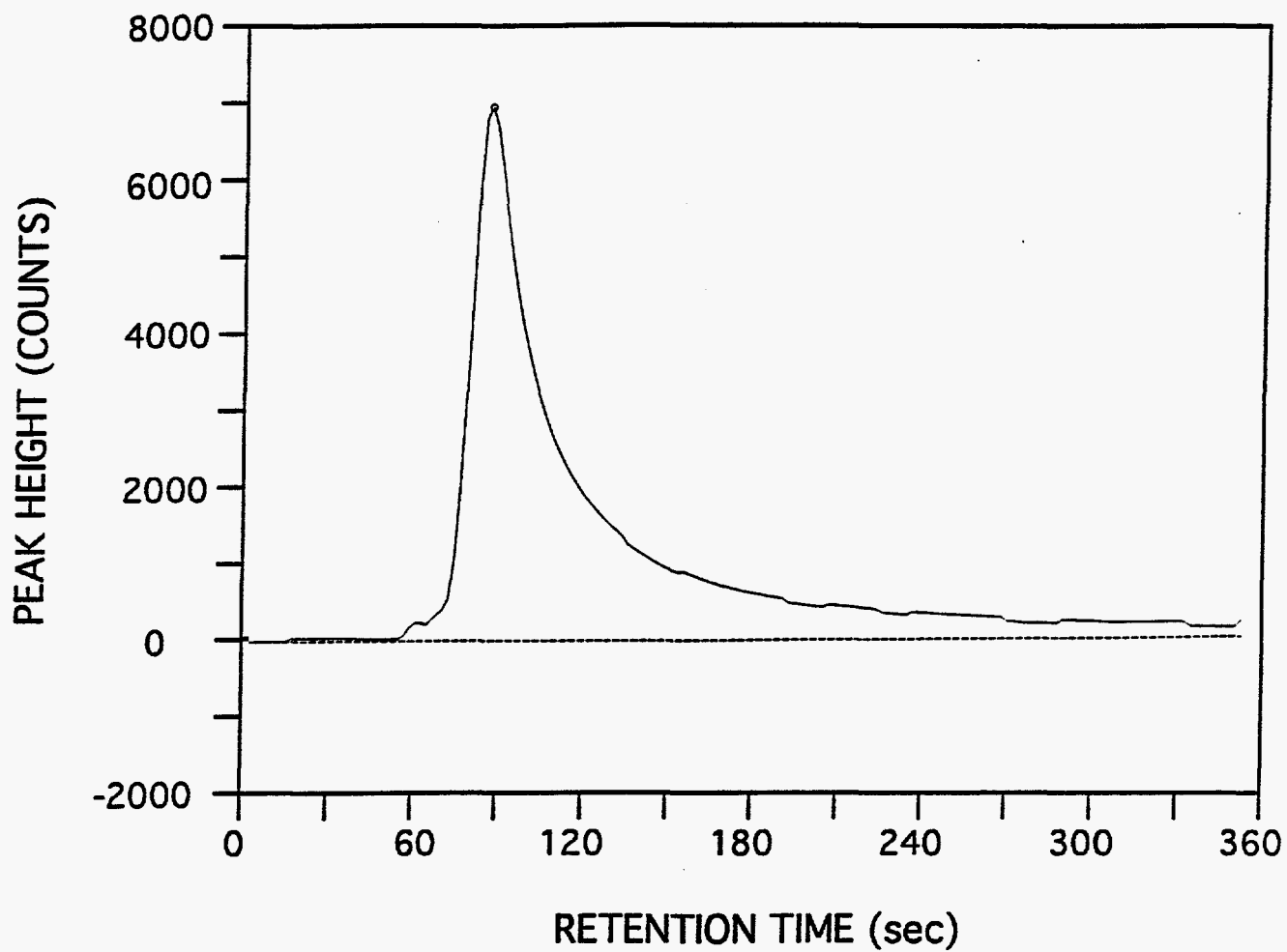


Figure 9. Chromatogram of carbon tetrachloride.

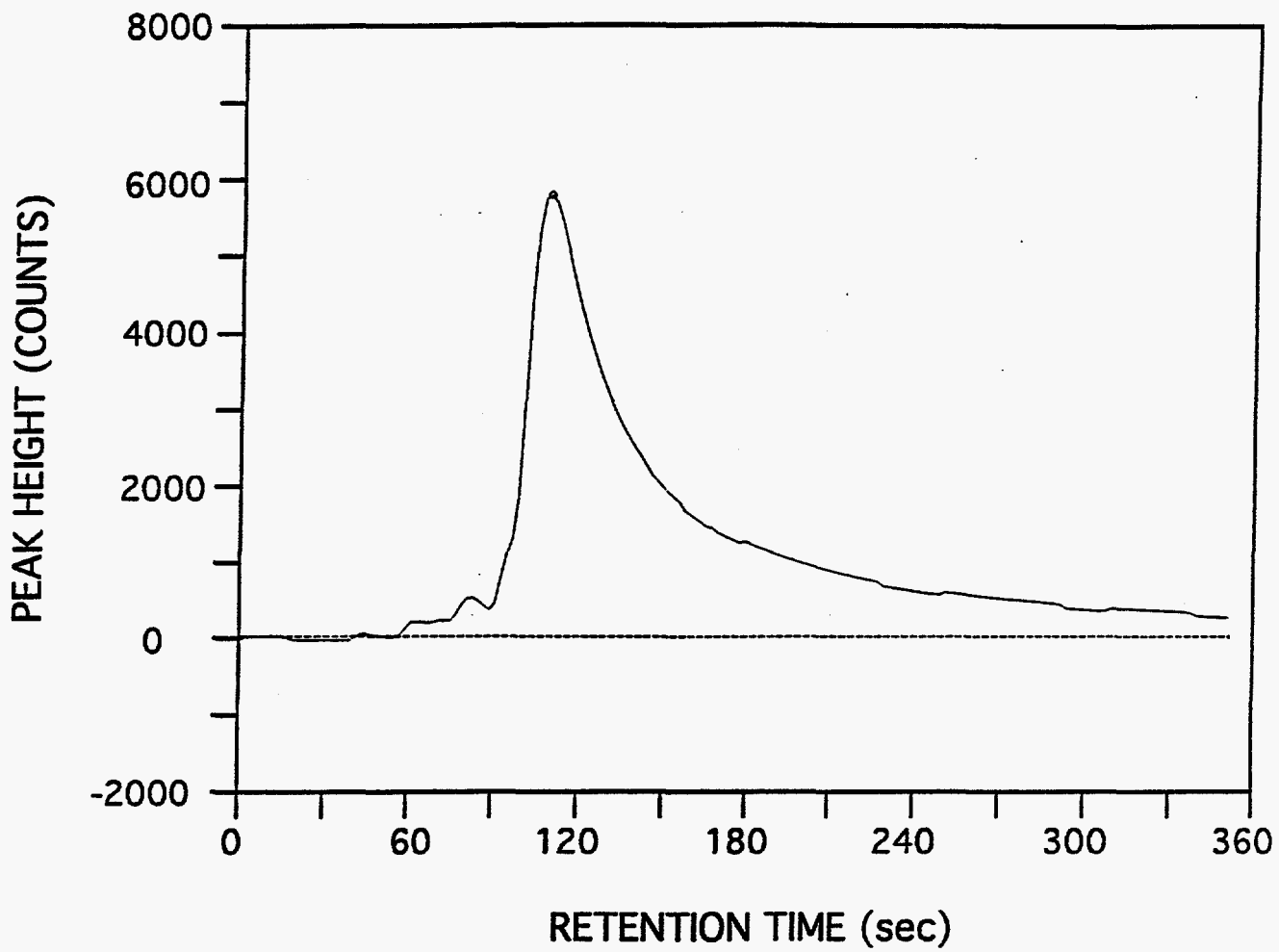


Figure 10. Chromatogram of trichloroethylene.

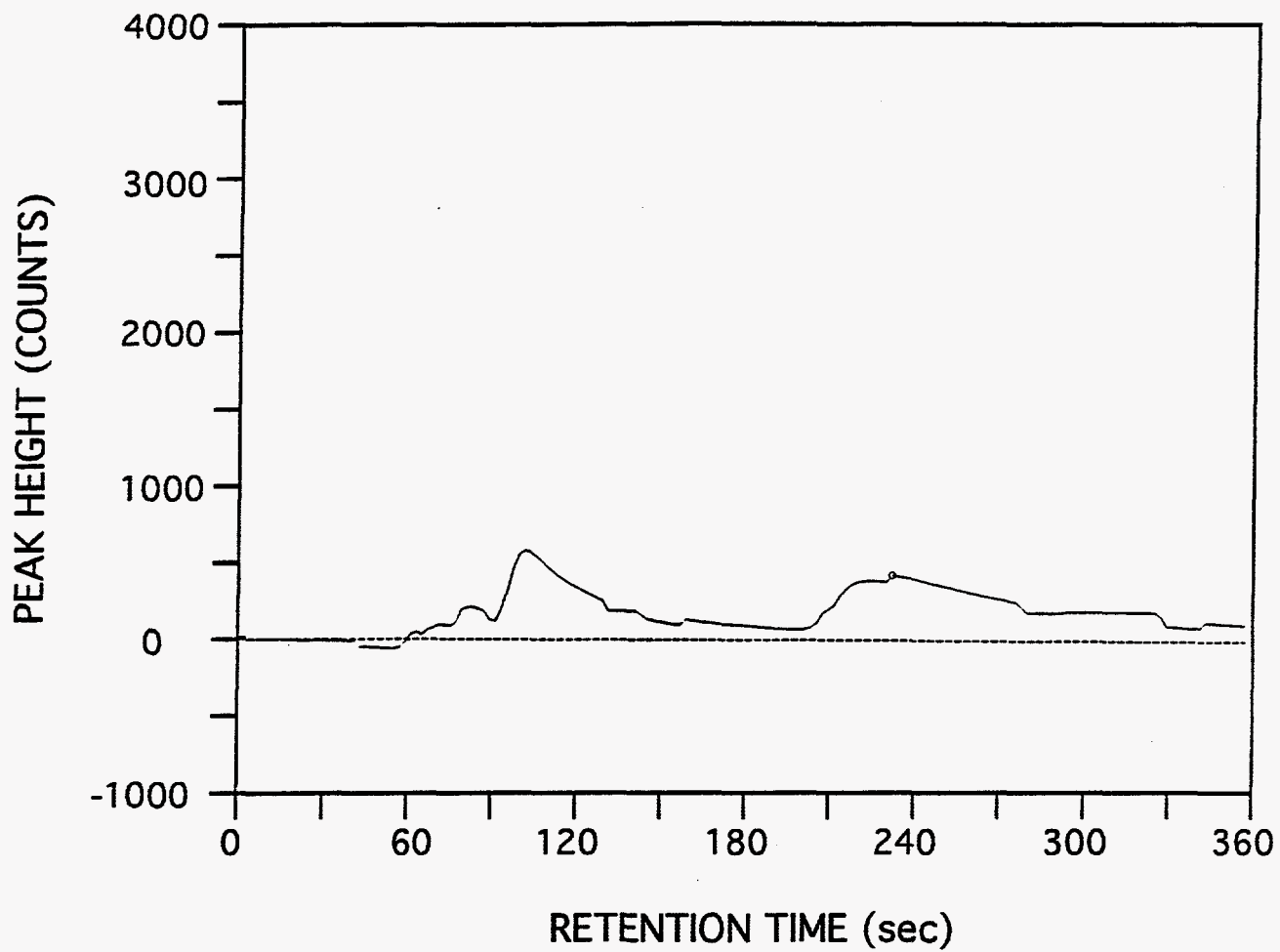


Figure 11. Chromatogram of tetrachloroethylene.

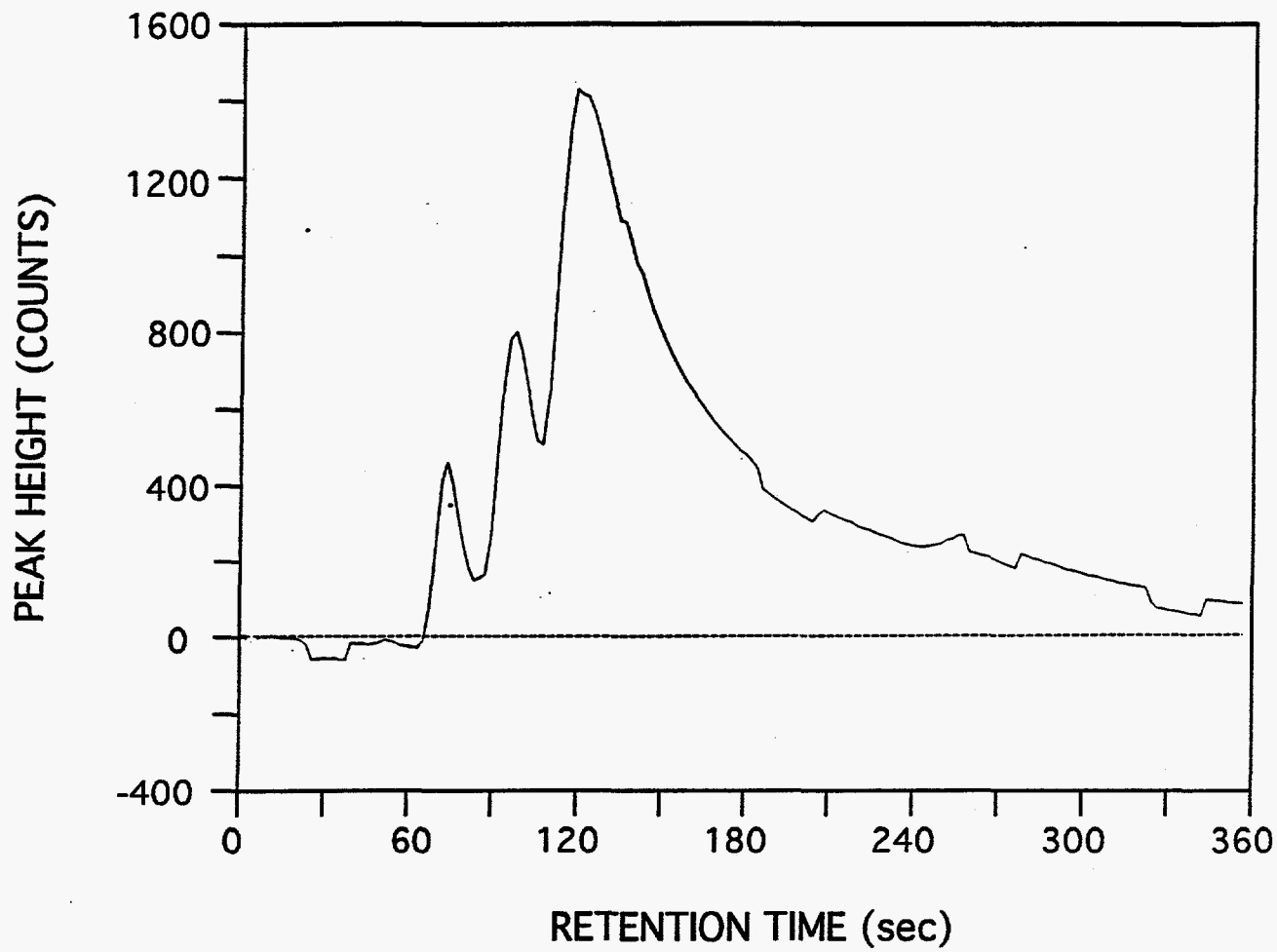


Figure 12. Chromatogram of a mixture of chloroform, carbon tetrachloride, and trichloroethylene.

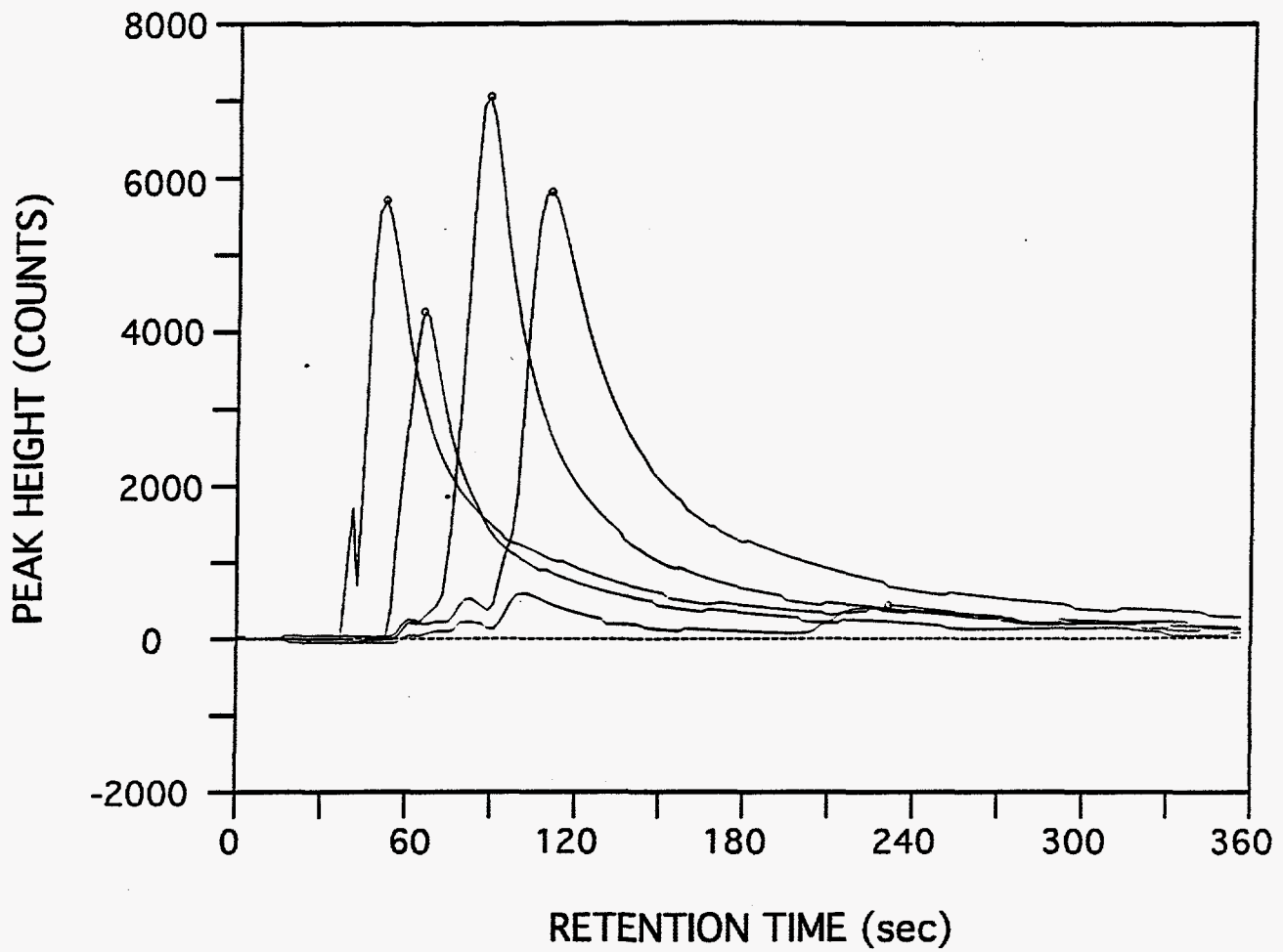


Figure 13. Superposition of the chromatograms of five chlorinated test compounds.

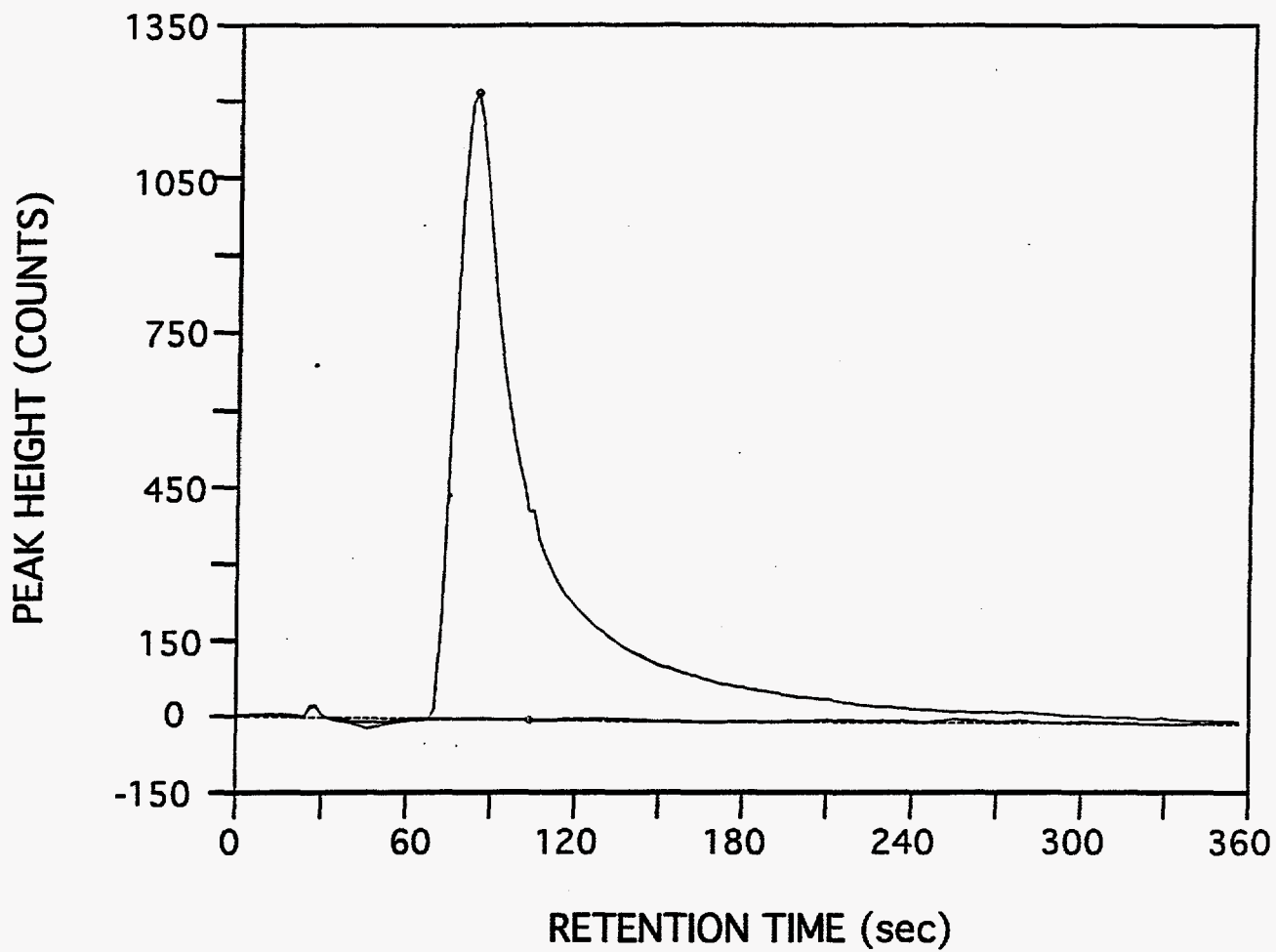


Figure 14. Relative sensitivity of halogen-selective detector to CCl_4 (100 ppm), vs. BTEX (10 ppm each).

CONE-GC HALOCARBON DETECTOR RESPONSE TO FREON-113

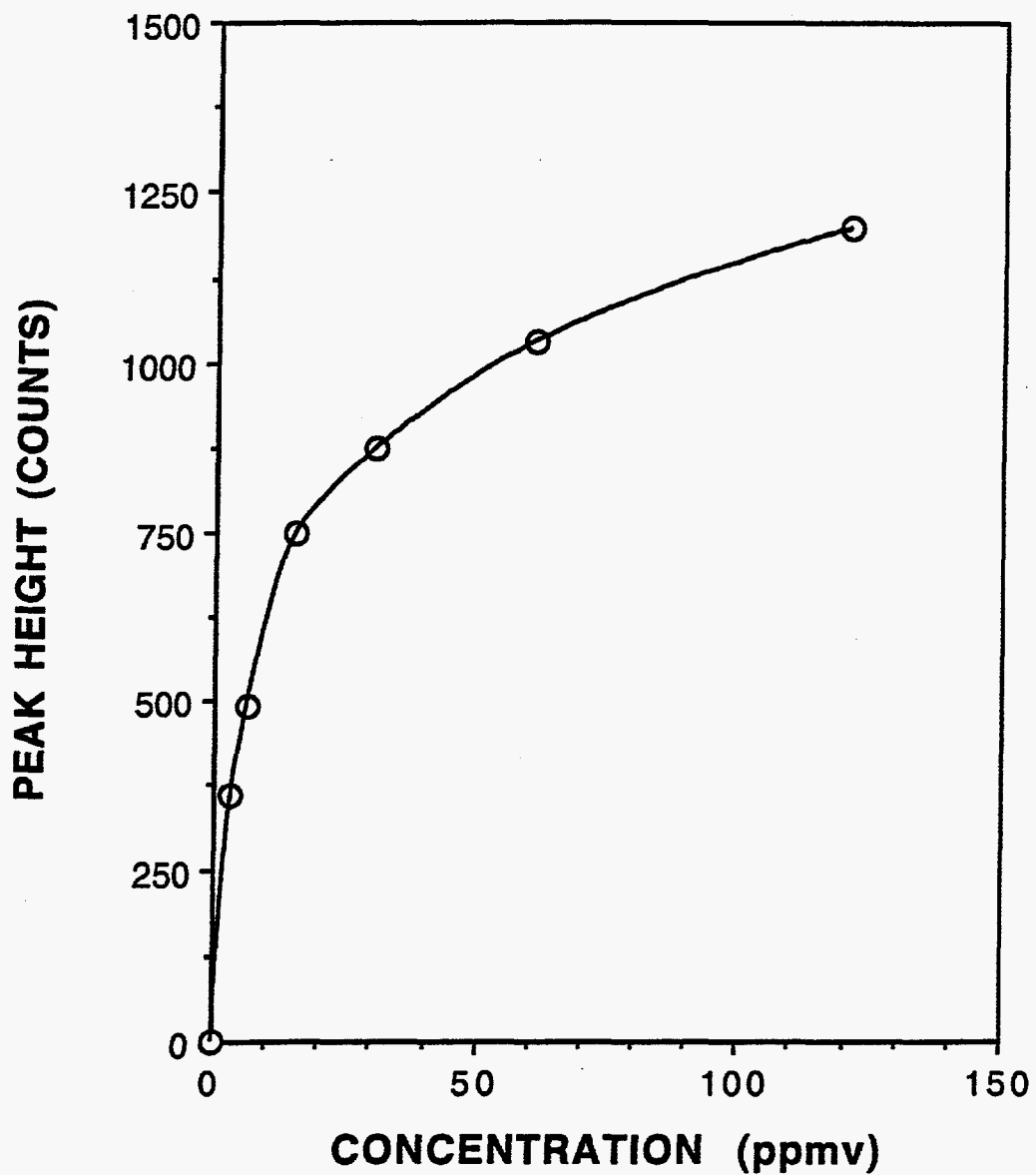


Figure 15. Calibration curve for trichlorotrifluoroethane, (FREON-113) using Cone-GC with halogen-selective detector.

**CONE-GC HALOCARBON DETECTOR
RESPONSE TO CARBON TETRACHLORIDE**

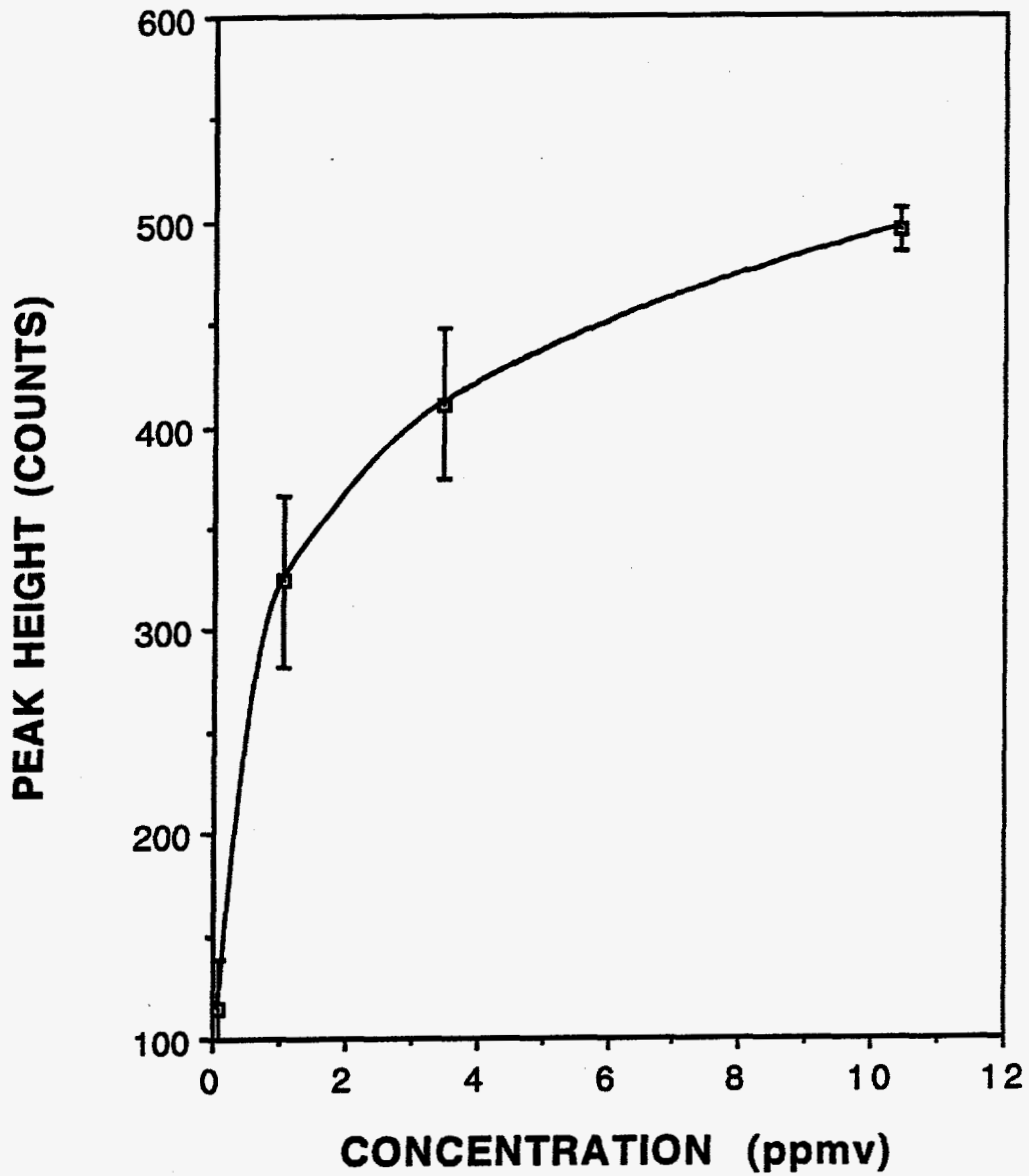


Figure 16. Calibration curve for carbon tetrachloride using Cone-GC with halogen-selective detector.

CONE-GC MOS DETECTOR RESPONSE TO BENZENE

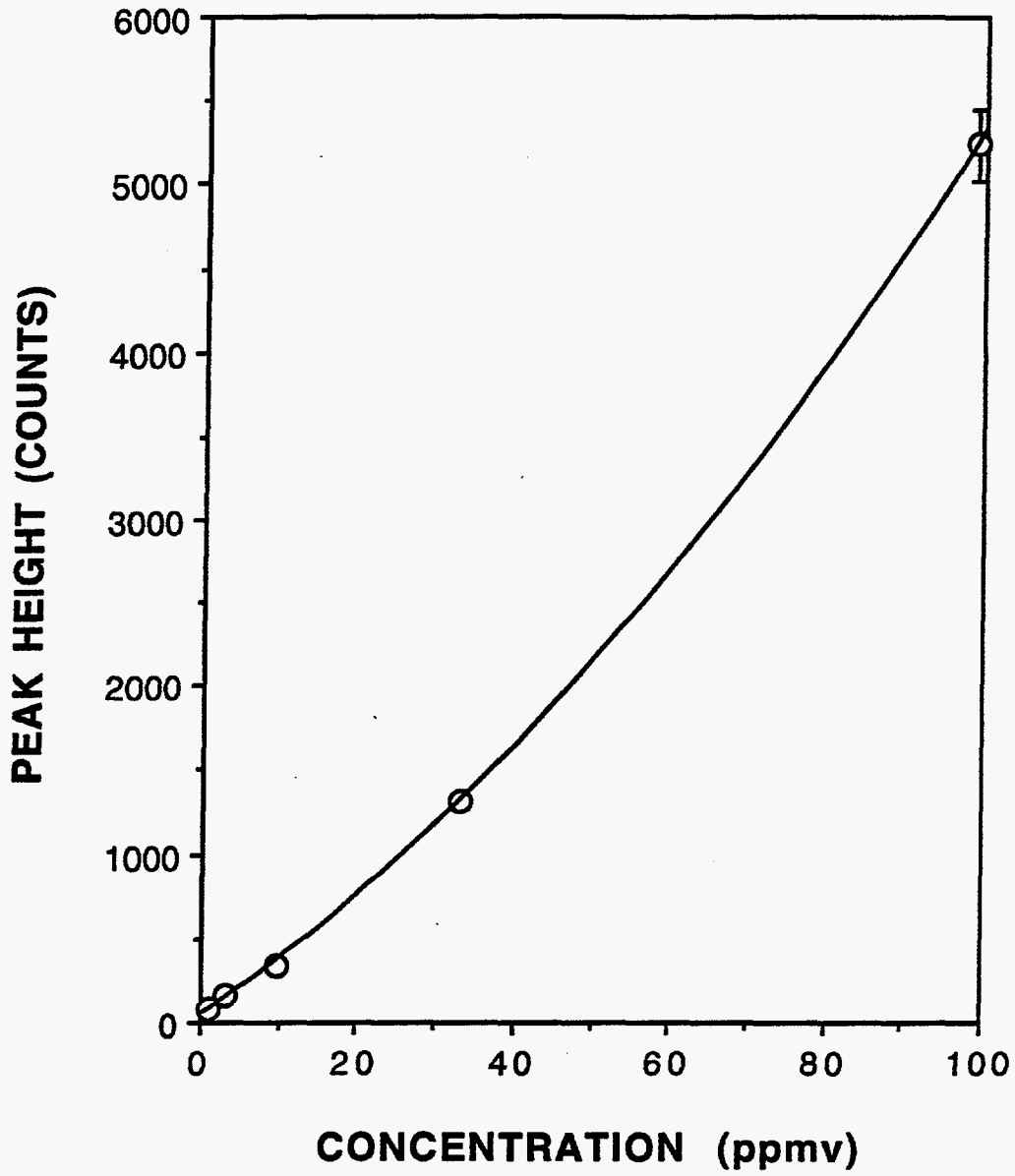


Figure 17. Calibration curve for benzene vapor using Cone-GC with a metal-oxide-semiconductor (MOS) detector.

**EXAMPLE OF CHROMATOGRAM REPRODUCIBILITY
(6 CONSECUTIVE RUNS OF 10.4 ppm CCl₄)**

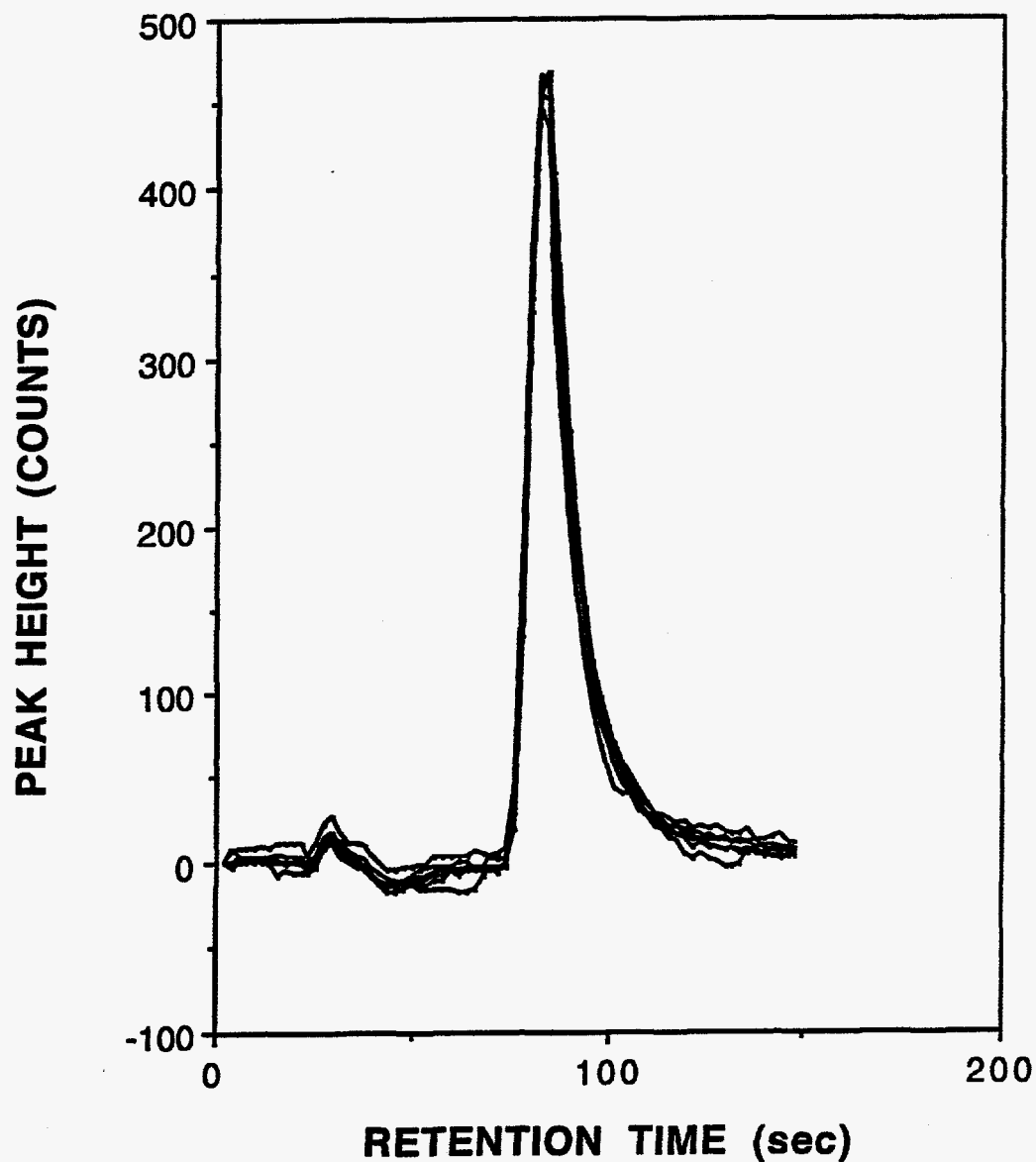


Figure 18. Demonstration of the Cone-GC chromatographic reproducibility with six consecutive, superimposed chromatograms obtained from a sample containing 10.4 ppmv of carbon tetrachloride vapor.

8.0 CONCLUSIONS and RECOMMENDATIONS

The data obtained in this project demonstrates that all of the proposed program objectives for the Cone-GC were met. Specifically, a miniaturized GC with all data acquisition and control software was successfully designed, fabricated, and tested in the laboratory. The instrument had an outside diameter of 0.99 inches thereby making it able to fit inside a one inch diameter tube typically available in cone penetrometer systems. In this regard, the Cone-GC significantly surpassed the originally proposed 1.25 inch diameter goal. The smaller size of the instrument means that it can be used at lower cost in a wider variety of cone penetrometers and in a wider variety of applications.

The Cone-GC has met all of the proposed performance objectives. The system has been demonstrated to be able to resolve and quantify mixtures of low concentrations (e.g., ppm) of environmentally relevant pollutants (e.g., BTEX, and various chlorinated solvents) in less than five minutes. Most attention in the performance evaluation was focussed on chlorinated solvents that pose a major problem at many DoE remediation sites. Carbon tetrachloride is commonly found at DoE sites and cannot be monitored with simple field portable instrumentation (e.g., electrochemical sensors, PIDs, and FIDs). The Cone-GC has demonstrated excellent performance in the detection of carbon tetrachloride with detection levels well below 1 ppm and analysis times of less than 3 minutes. We believe that few other instruments can match the Cone-GC for its versatility, selectivity, and sensitivity in monitoring this difficult analyte.

All of the proposed Cone-GC performance features have been realized in this project. The instrument has a full-featured software package that permits stand-alone operation and offers data logging, RS232 output, a variety of report options, and easy calibration, even with non-linear calibration curves. The system functions from either 120VAC or 12VDC power supplies with minimal set-up and maintenance.

The only shortcomings of the instrument are associated with the (intended) low chromatographic resolution of the instrument which allows fast analysis times but limits its ability to deal with complex analyte mixtures, and the somewhat limited life of the inexpensive halocarbon detector which will require periodic replacement after every 100 hours of use. Nevertheless, the incredibly compact size, modest power requirements, ease-of-use, and high performance of the Cone-GC should make it the method of choice for a broad range of site characterization problems.

Field testing of the Cone-GC in an actual cone penetrometer will be the logical next step for this system. During the course of this project we have discussed the Cone-GC system with representatives of cone penetrometer producers (ARA and Hogentogler) and have attended cone penetrometer field exercises in order to understand the operational environment for the Cone-GC.

Continued effort will be required to actually install the "Cone-GC" into a commercial cone penetrometer system and to develop methods and protocols for its use in site characterization applications. Such a site demonstration will be essential for ironing out unexpected difficulties and providing a clear example of its utility for site characterization applications.

APPENDIX A

Cone-GC Mechanical Drawings

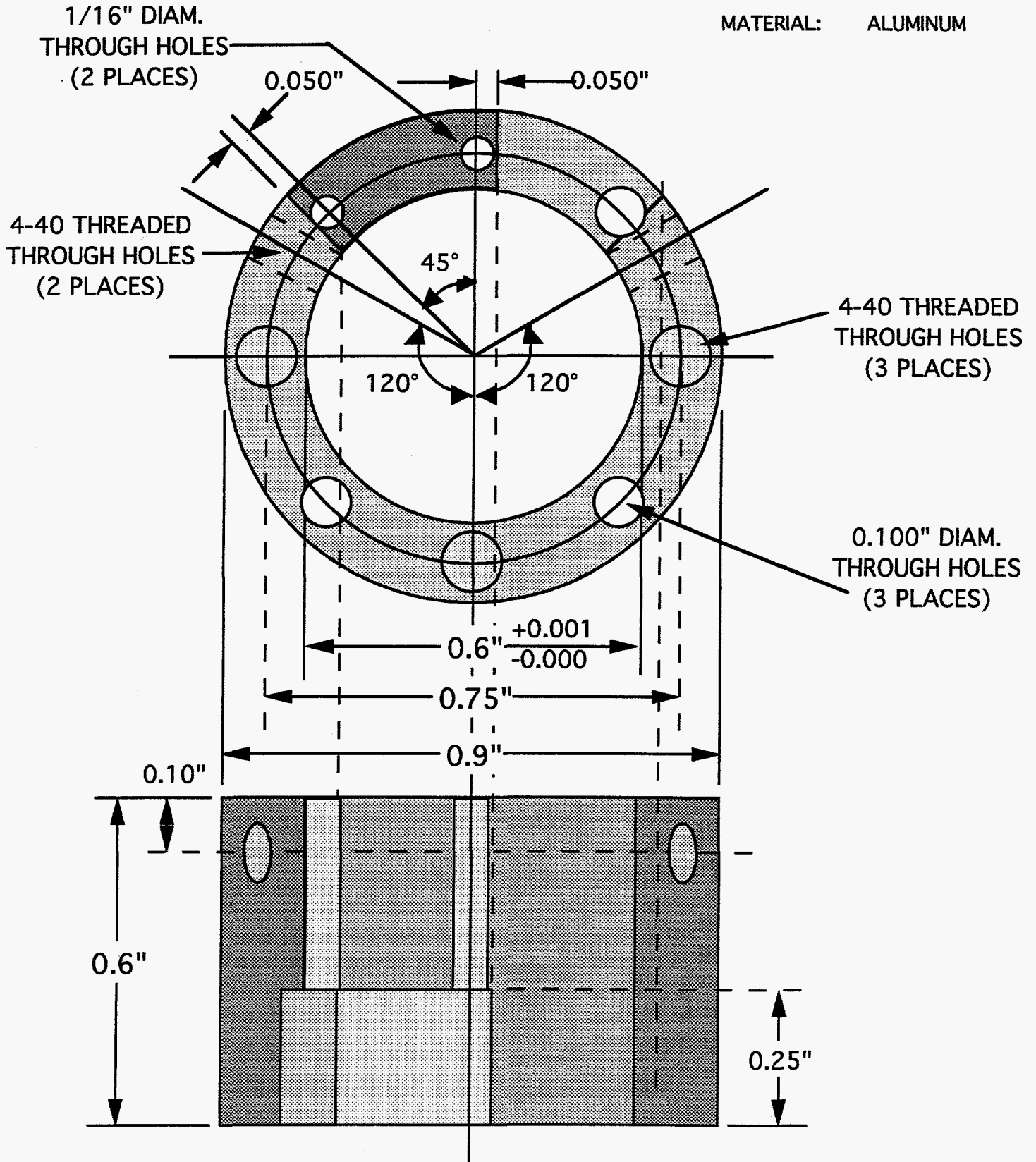
MICROSENSOR SYSTEMS, INC.

DATE: 16 May 1995
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-01
CAGE CODE: OGLR6
REVISION: 1

MINI GC VALVE-SHELL

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: ALUMINUM



MICROSENSOR SYSTEMS, INC.

DATE: 8 Sept. 1994

MINI GC VALVE-ROTOR

DRAWN BY: H. Wohltjen

DRAWING NO.: 9120-02

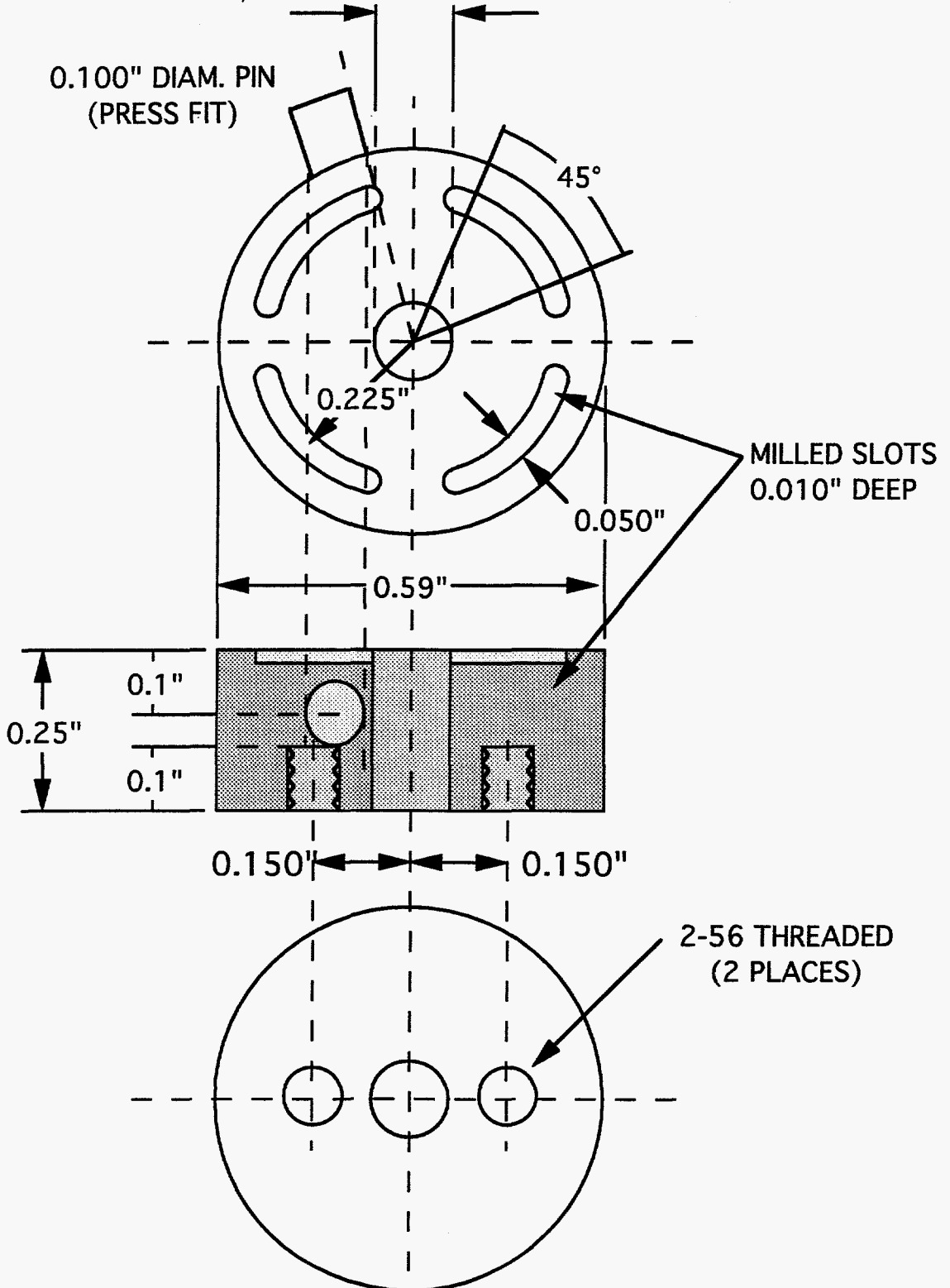
CAGE CODE: OGLR6

REVISION: 0

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: GLASS FILLED TEFLON

3mm



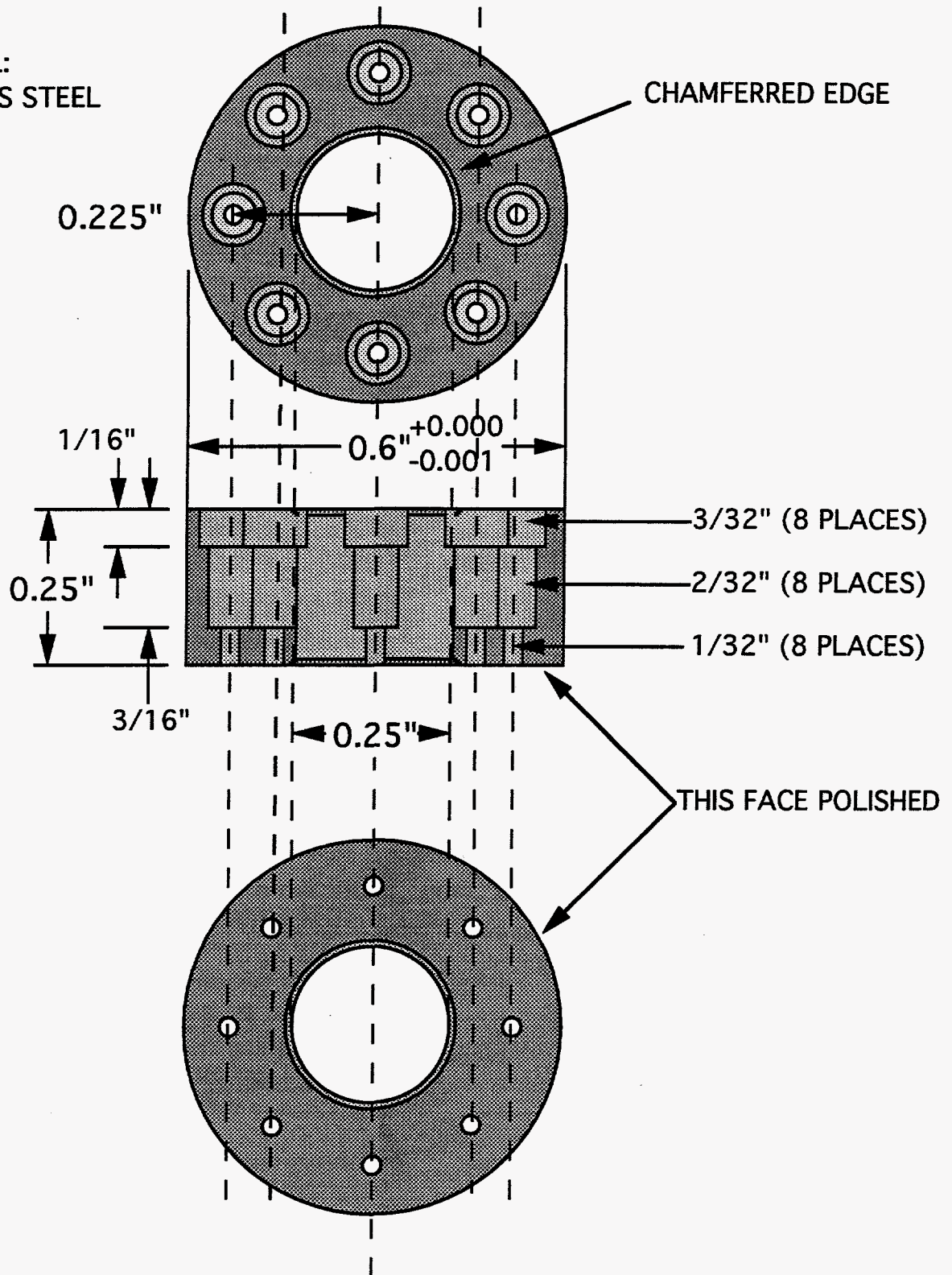
MICROSENSOR SYSTEMS, INC.

DATE: 8 Sept. 1994
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-03
CAGE CODE: OGLR6
REVISION: 0

MINI GC VALVE-PORT PLATE

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL:
STAINLESS STEEL



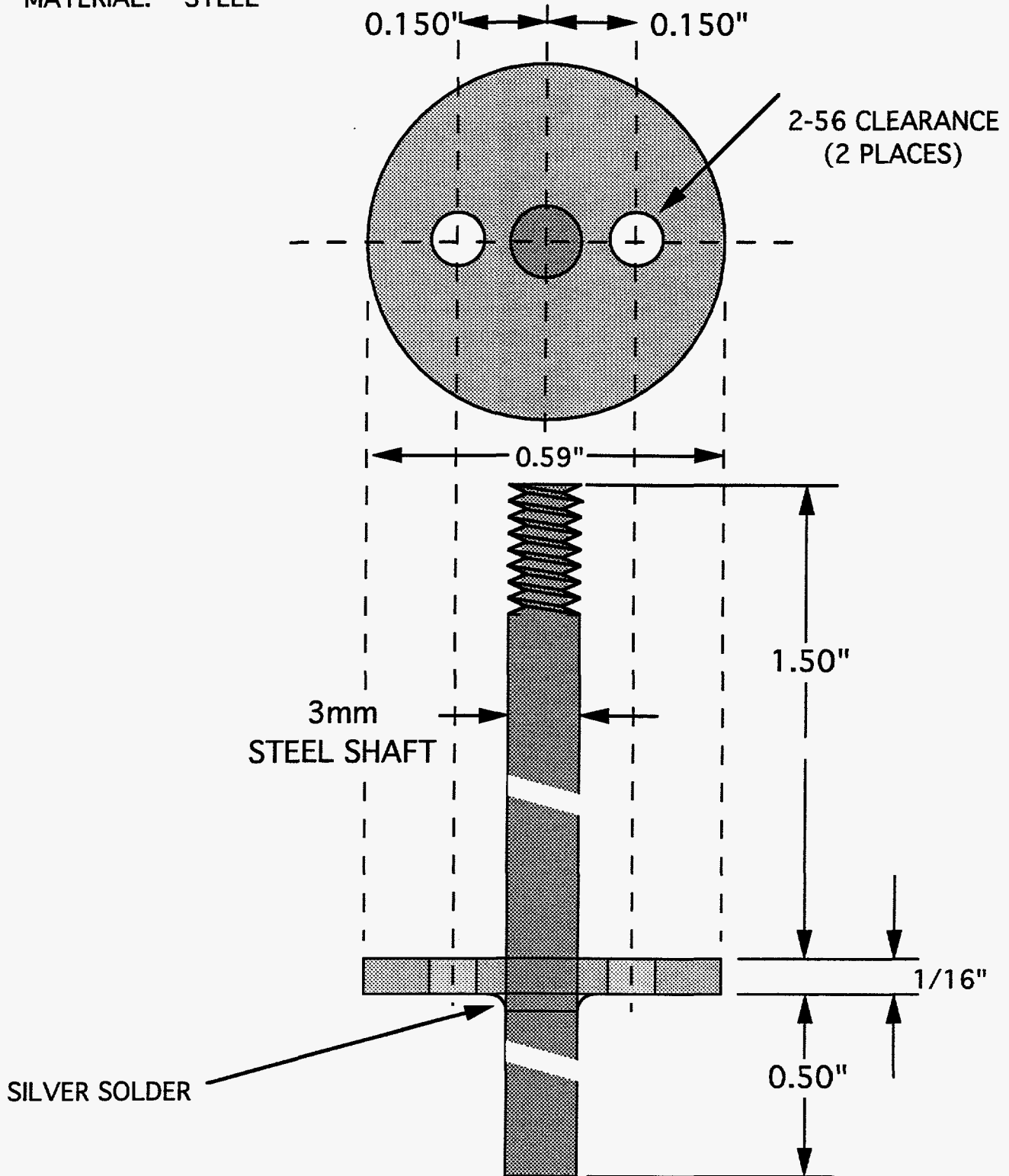
MICROSENSOR SYSTEMS, INC.

DATE: 1 Dec., 1994
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-04
CAGE CODE: 0GLR6
REVISION: 0

MINI GC VALVE-ROTOR SUPPORT

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: STEEL



MICROSENSOR SYSTEMS, INC.

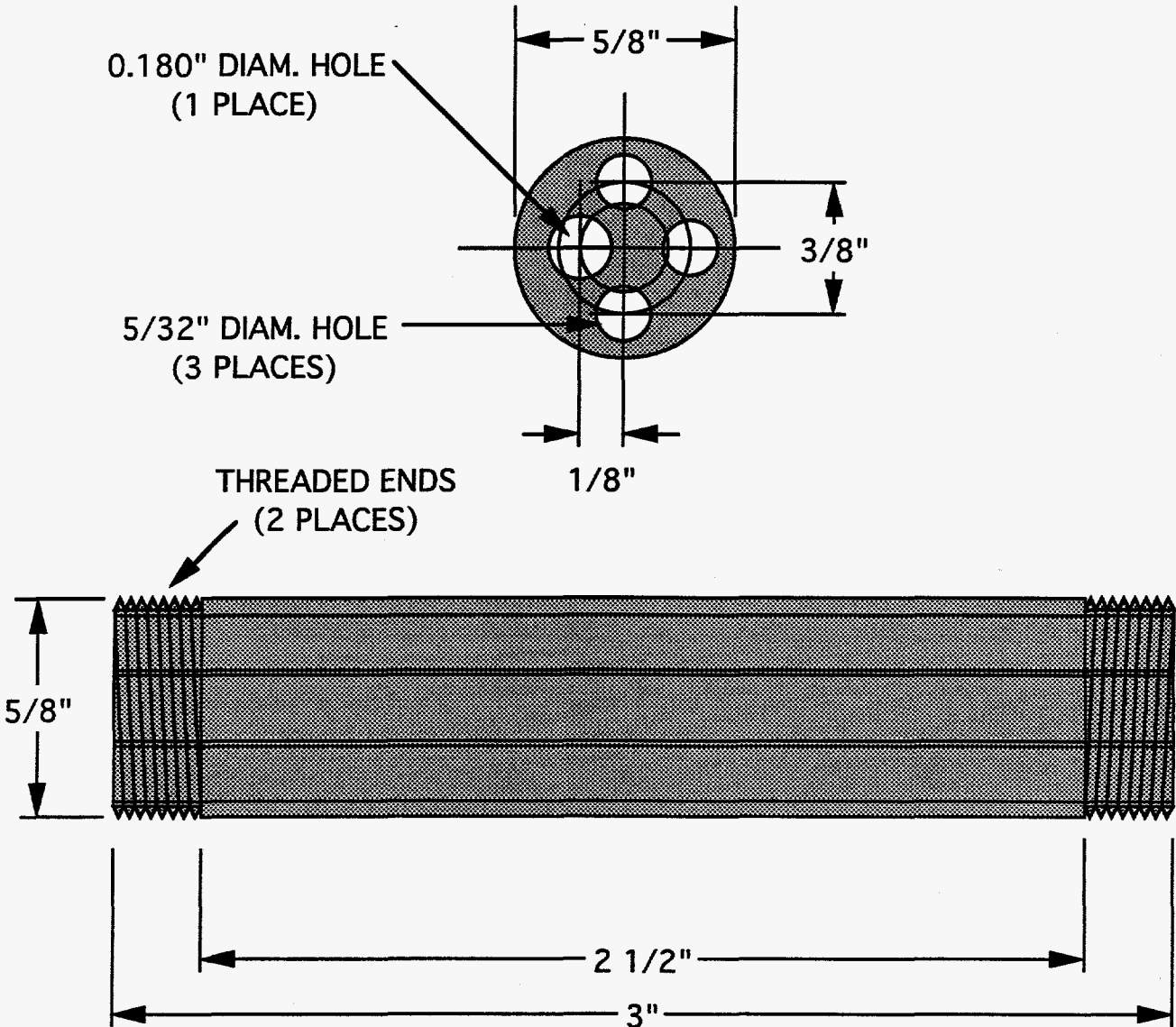
DATE: 13 March 1995
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-05
CAGE CODE: 0GLR6
REVISION: 0

MINI GC COLUMN MANDRIL

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: ALUMINUM
FINISH: BLACK ANODIZED

ALL DIMENSIONS ± 0.02 "



MICROSENSOR SYSTEMS, INC.

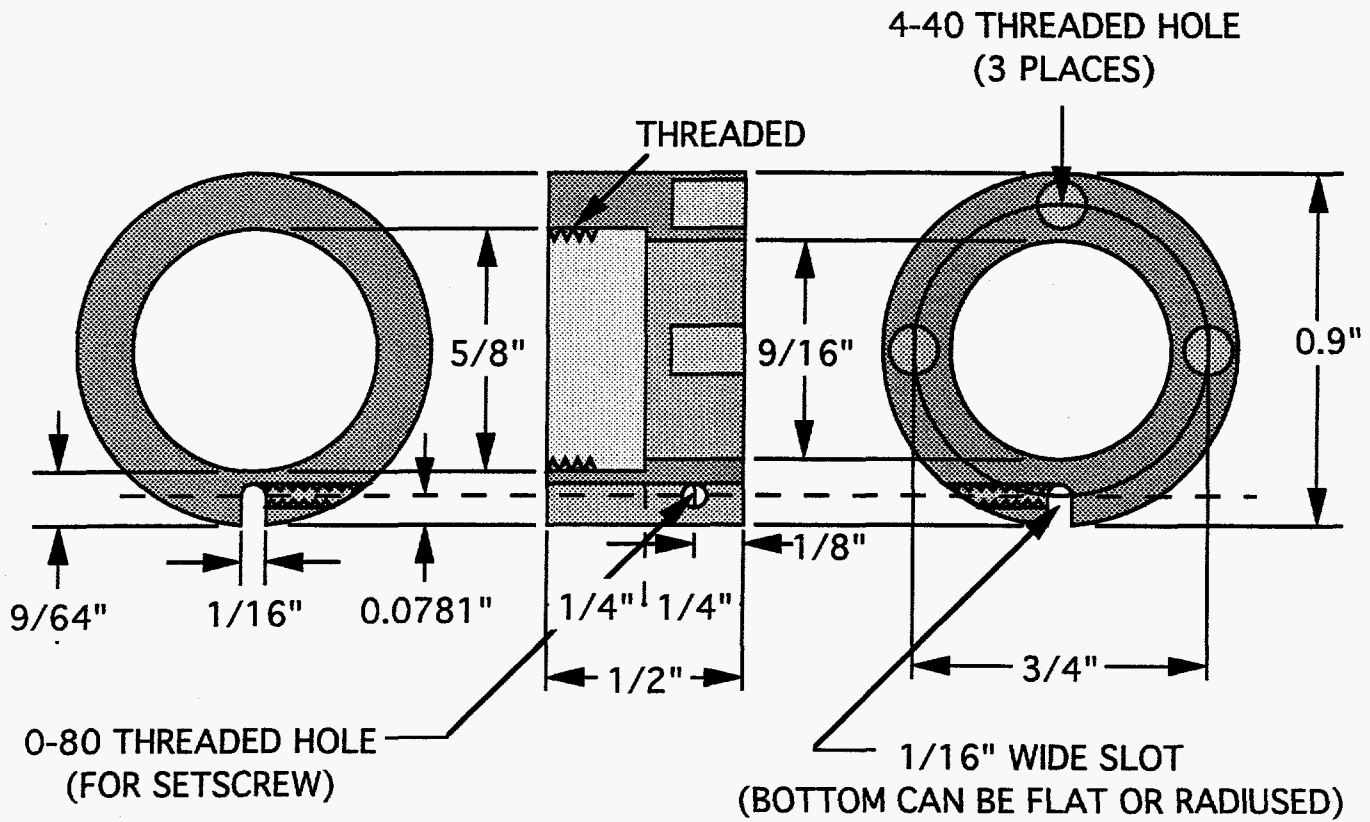
MINI GC COLUMN MANDRIL
END CAPS

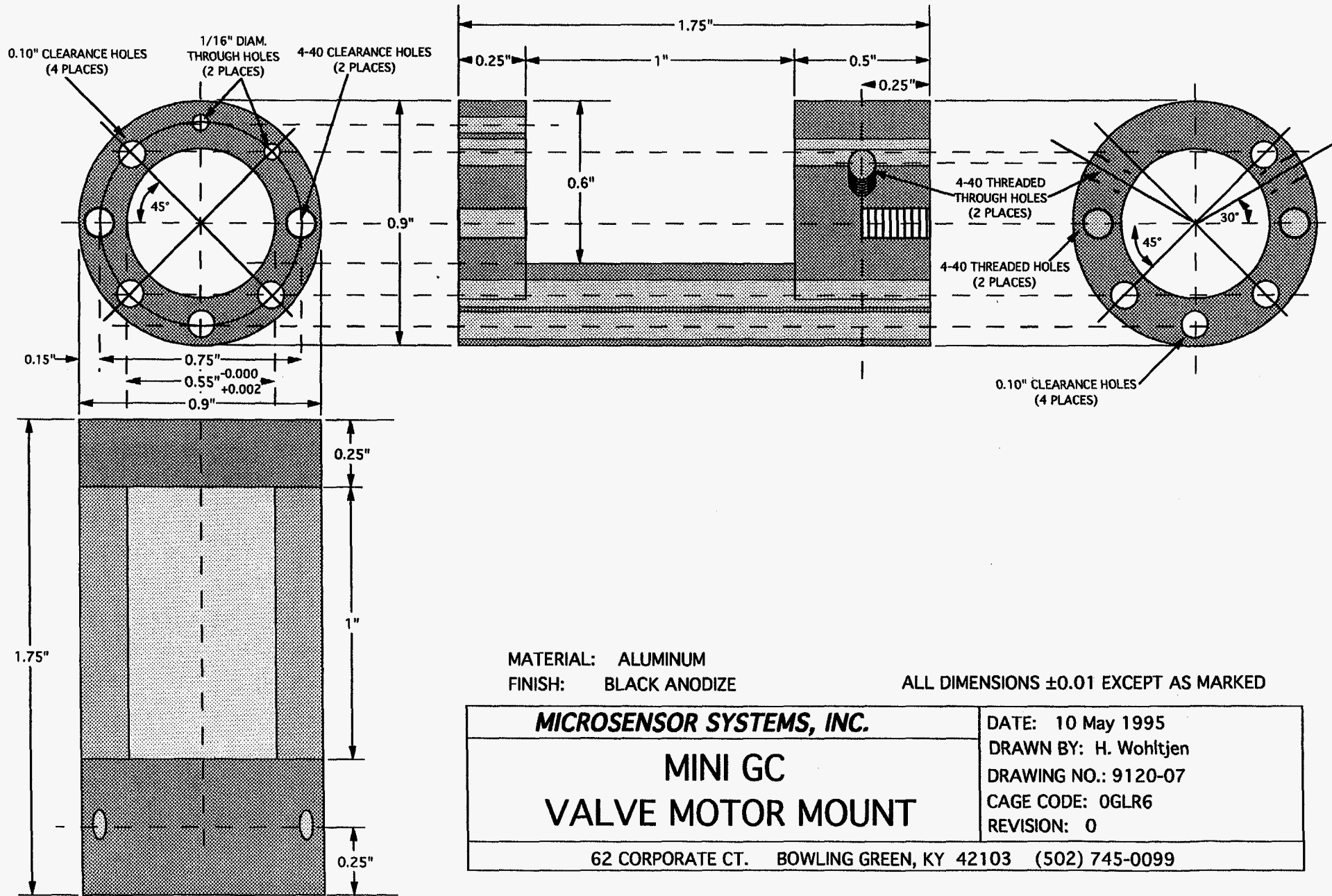
DATE: 13 March 1995
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-06
CAGE CODE: 0GLR6
REVISION: 0

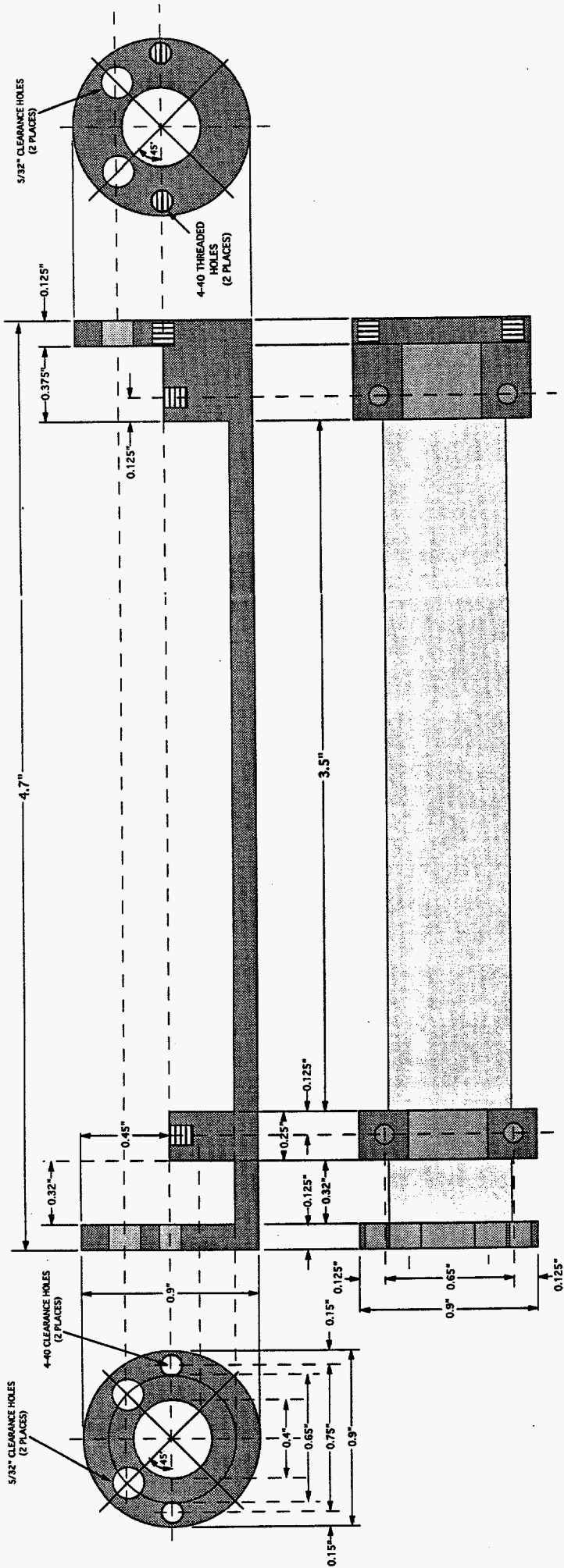
62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: NYLON OR DELRIN

ALL DIMENSIONS ± 0.02 "

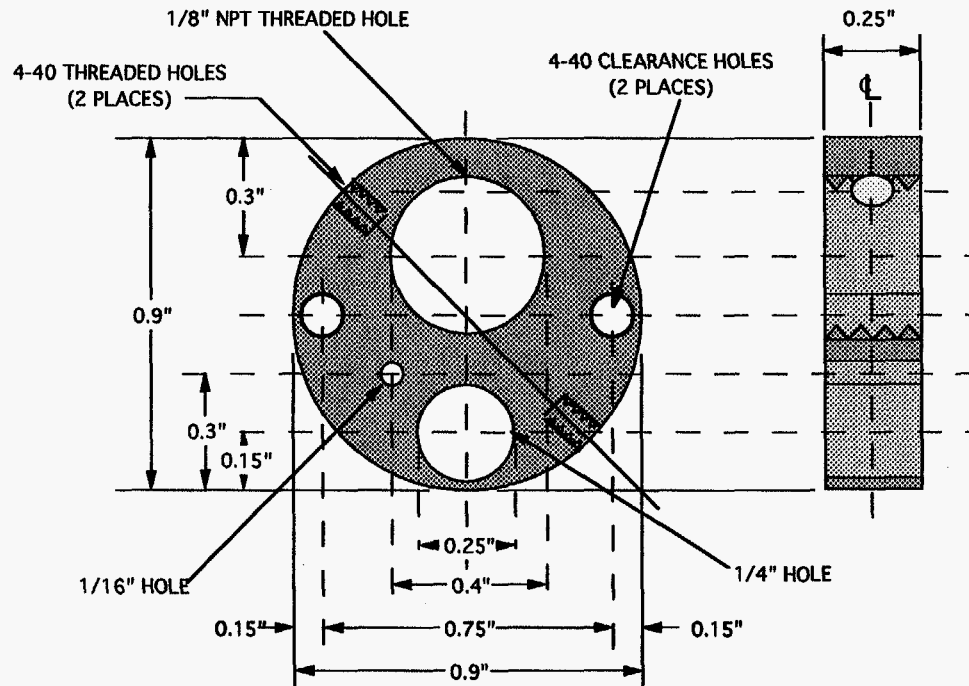






MATERIAL: ALUMINUM
 FINISH: BLACK ANODIZE
 ALL DIMENSIONS ±0.01 EXCEPT AS MARKED

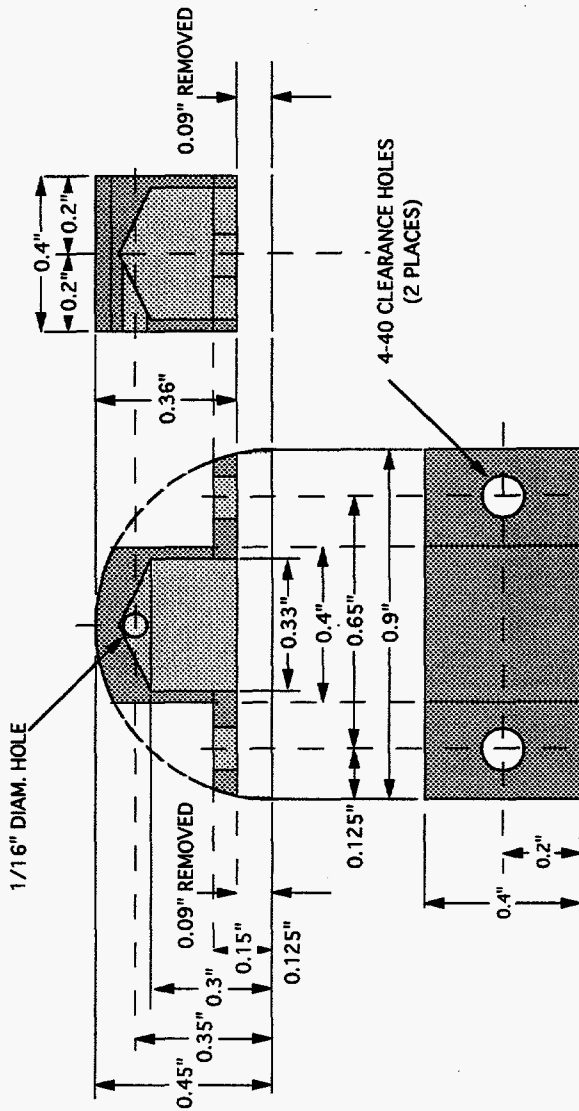
MICROSENSOR SYSTEMS, INC.	
MINI GC	
PC BOARD MOUNT	
DATE: 10 May 1995	REVISION: 0
DRAWN BY: H. Wohltjen	CAGE CODE: OGLR6
DRAWING NO.: 9120-08	62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099



MATERIAL: ALUMINUM
 FINISH: BLACK ANODIZE

ALL DIMENSIONS ±0.01 EXCEPT AS MARKED

MICROSENSOR SYSTEMS, INC.	DATE: 10 May 1995
MINI GC REAR BULKHEAD	DRAWN BY: H. Wohltjen
	DRAWING NO.: 9120-09
	CAGE CODE: OGLR6
	REVISION: 0
62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099	



MATERIAL: BRASS ALL DIMENSIONS ± 0.01 EXCEPT AS MARKED

MATERIAL: BRASS

MICROSENSOR SYSTEMS, INC.		DATE: 10 May 1995
MINI GC		DRAWN BY: H. Wohltjen
SENSOR MOUNT		DRAWING NO.: 9120-10
		CAGE CODE: 0GLR6
		REVISION: 0
62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099		

MICROSENSOR SYSTEMS, INC.

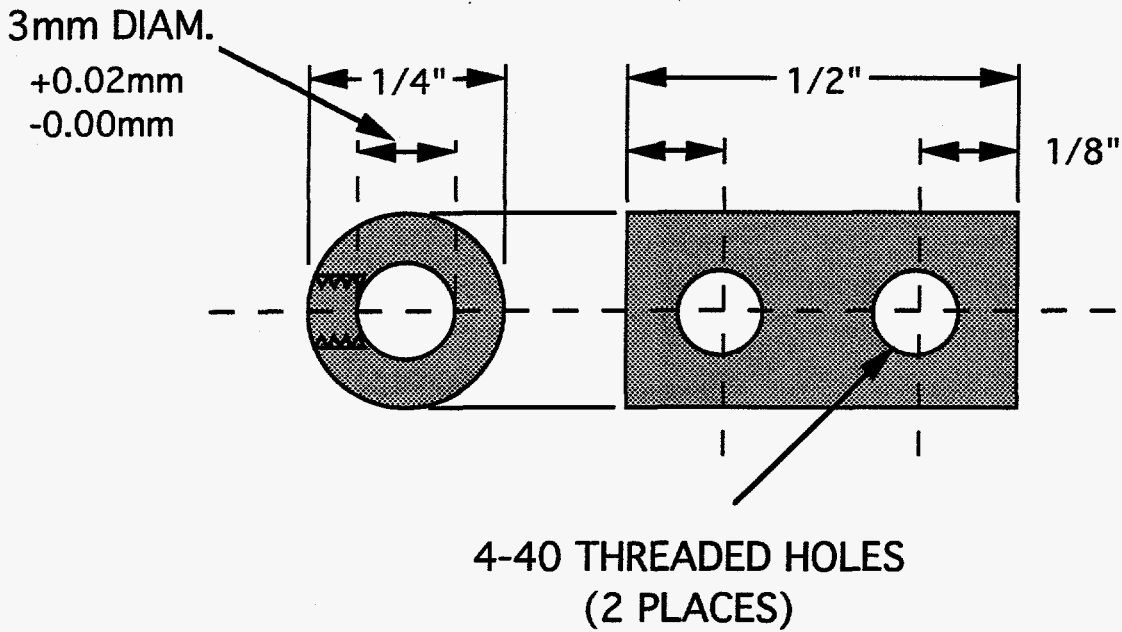
**MINI GC
VALVE/MOTOR SHAFT COUPLER**

DATE: 16 May 1995
DRAWN BY: H. Wohltjen
DRAWING NO.: 9120-11
CAGE CODE: OGLR6
REVISION: 0

62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099

MATERIAL: ALUMINUM
FINISH: NATURAL

ALL DIMENSIONS ± 0.02 "
(EXCEPT AS NOTED)



APPENDIX B

Cone-GC Electrical Drawings

MICROSENSOR SYSTEMS, INC.

DOE CONE GC INJECTOR VALVE CONTROLLER SCHEMATIC

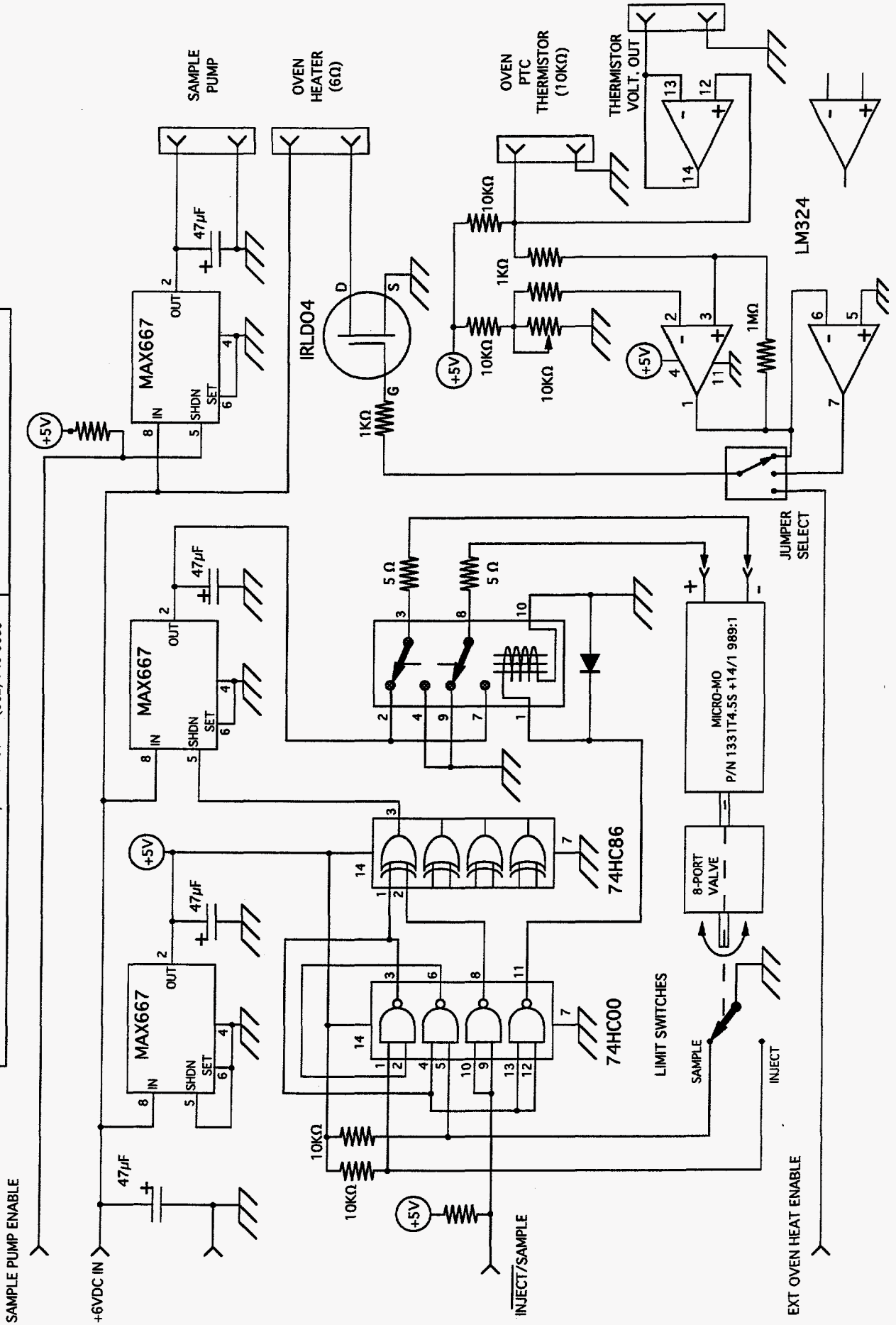
DATE: 9 October 1995

DRAWN BY: H. Wohltjen

DRAWING NO.: 9120-20

REVISION: 0

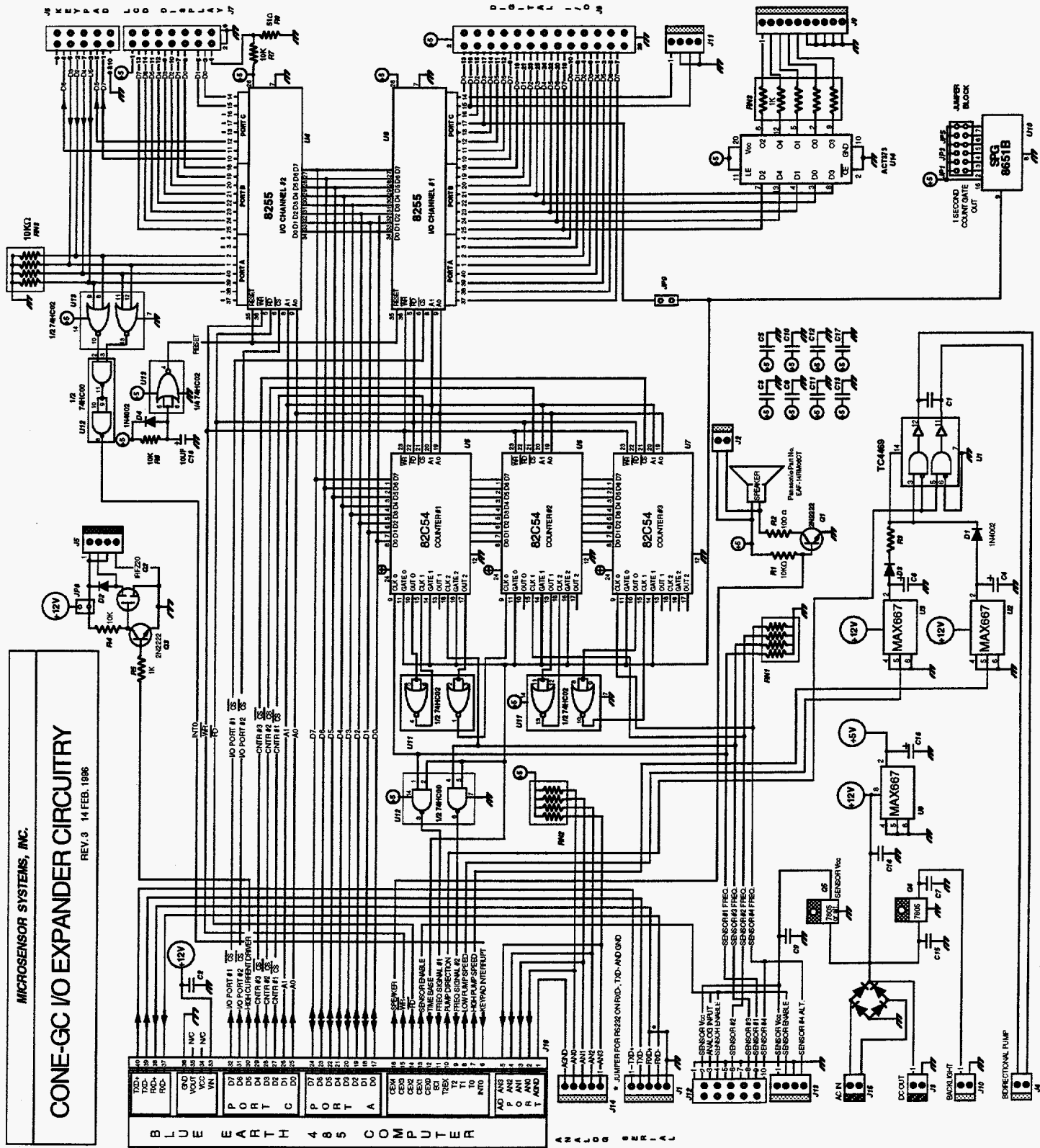
62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099



MICROSENSOR SYSTEMS, INC.

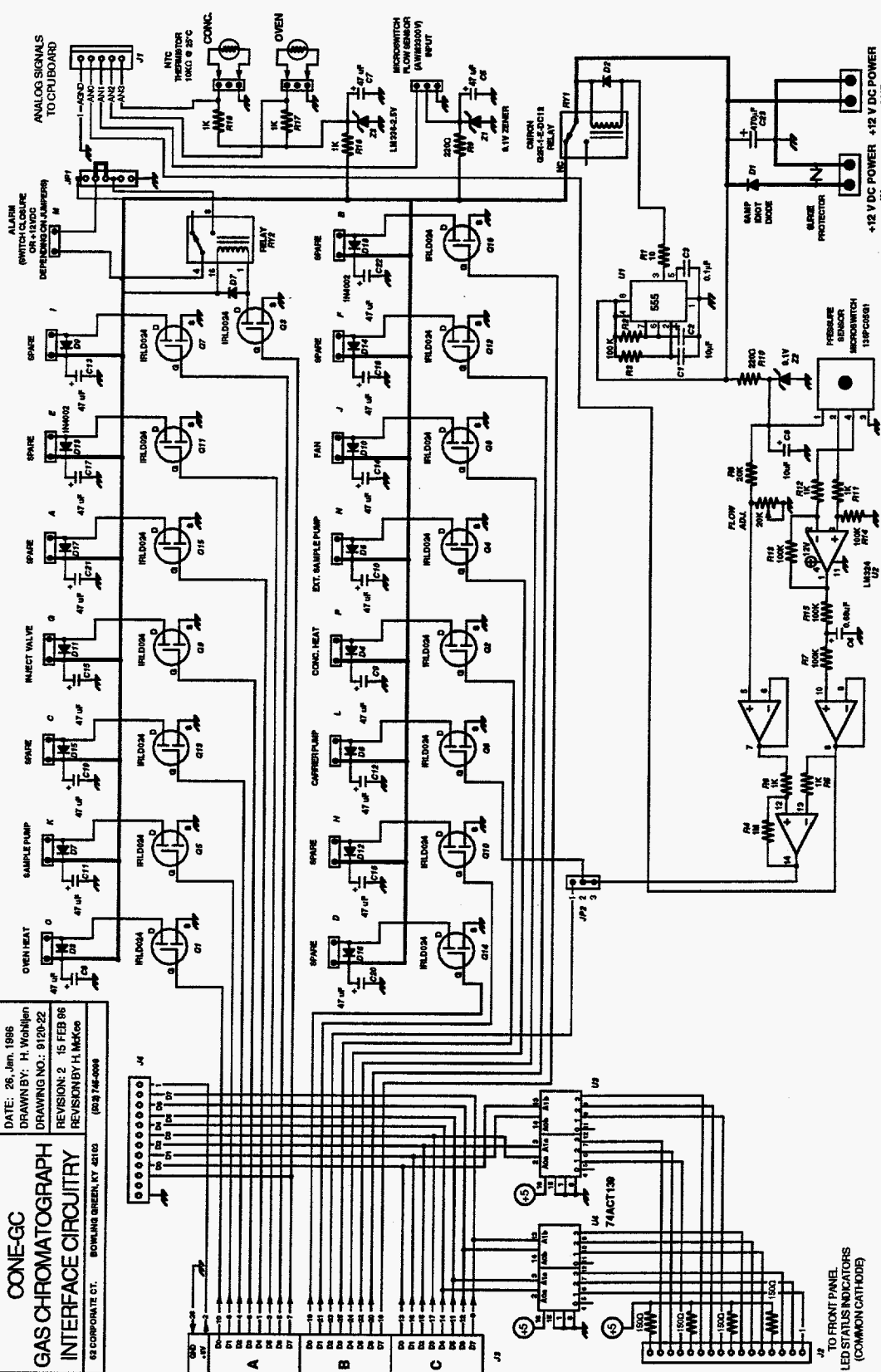
CONE-GC I/O EXPANDER CIRCUITRY

REV. 3 14 FEB. 1986



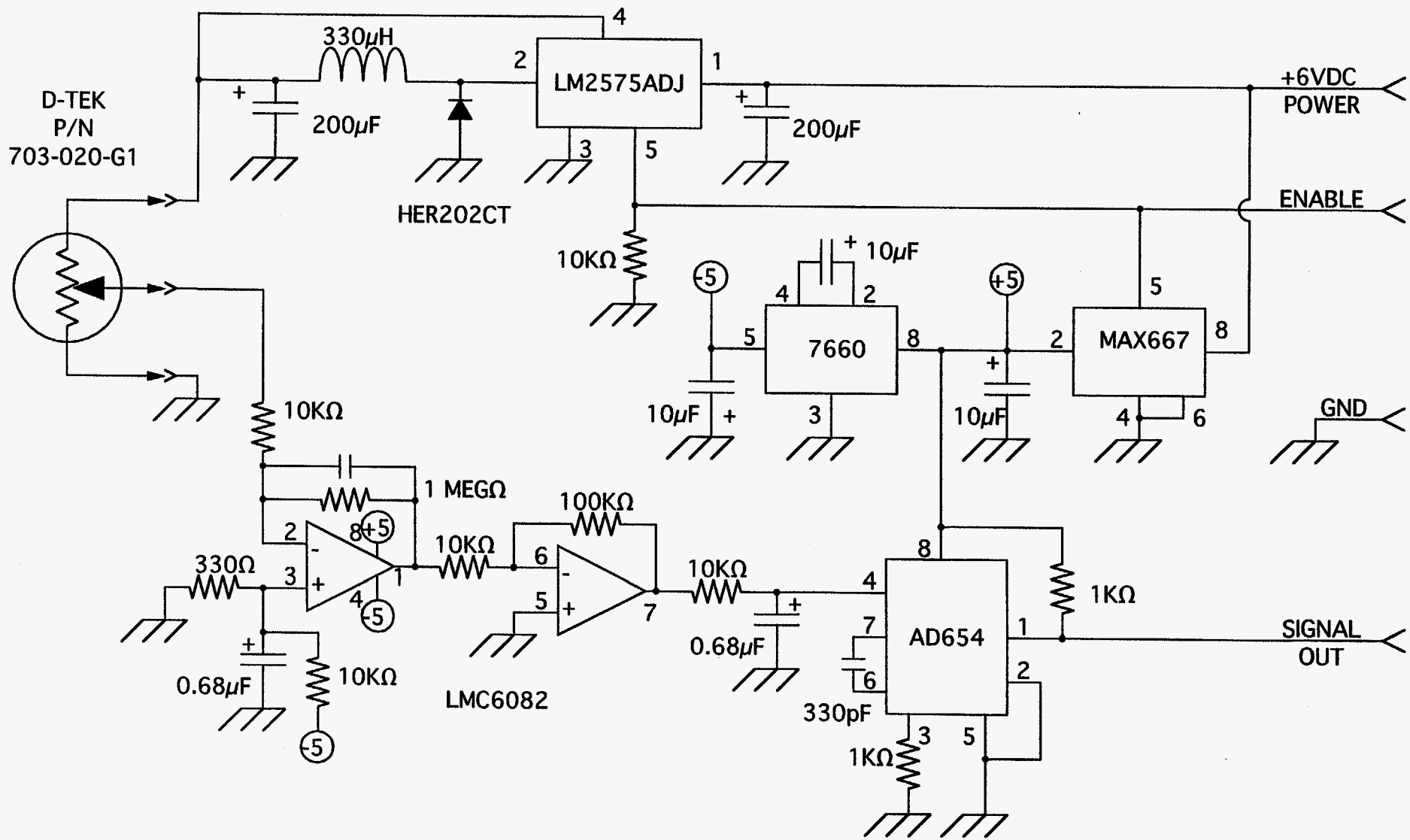
MICROSENSOR SYSTEMS, INC.
CONE-GC
GAS CHROMATOGRAPH
INTERFACE CIRCUITRY
 61 CORPORATE CT. BOWLING GREEN, KY 42103 (606) 746-0098

DATE: 26 Jan, 1986
 DRAWING BY: H. Wahljen
 DRAWING NO.: 9120-22
 REVISION: 2 15 FEB 86
 REVISION BY: H. McKee



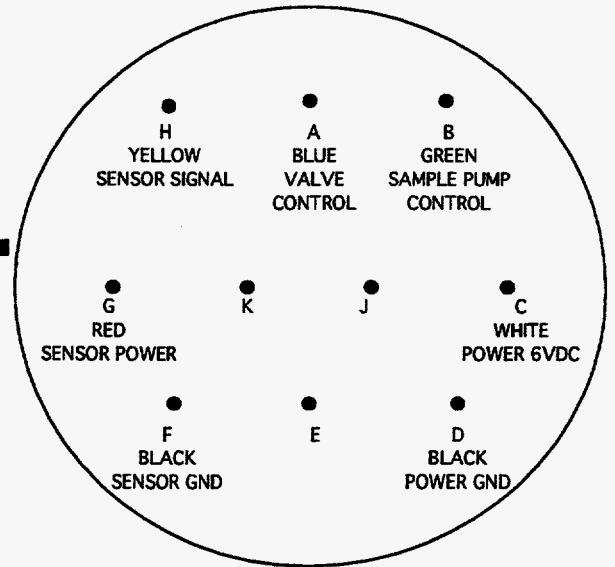
TO FRONT PANEL
 LED STATUS INDICATORS
 (COMMON CATHODE)

MICROSENSOR SYSTEMS, INC.		DATE: 9 October 1995
DOE CONE GC DETECTOR ELECTRONICS SCHEMATIC		DRAWN BY: H. Wohltjen
62 CORPORATE CT. BOWLING GREEN, KY 42103 (502) 745-0099		DRAWING NO.: 9120-21
		REVISION: 0

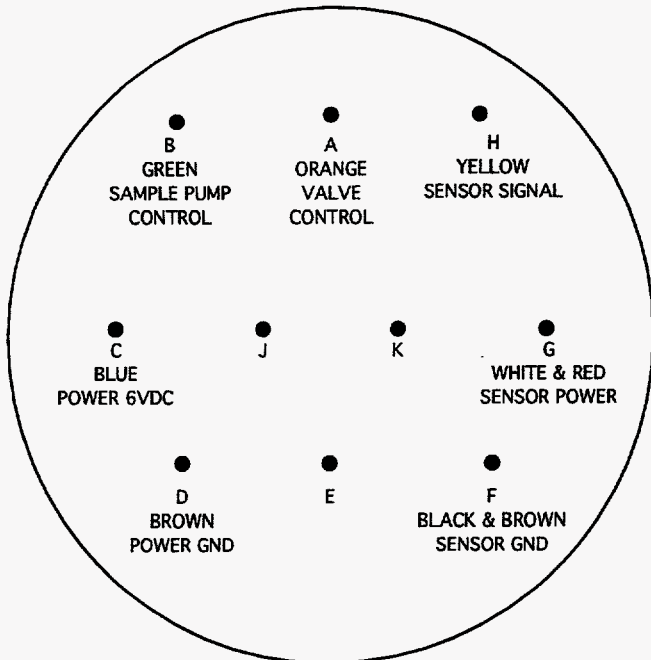


CONE PENETROMETER INTERFACE CABLE WIRING CONFIGURATION

CONE PENETROMETER BULKHEAD CONNECTOR

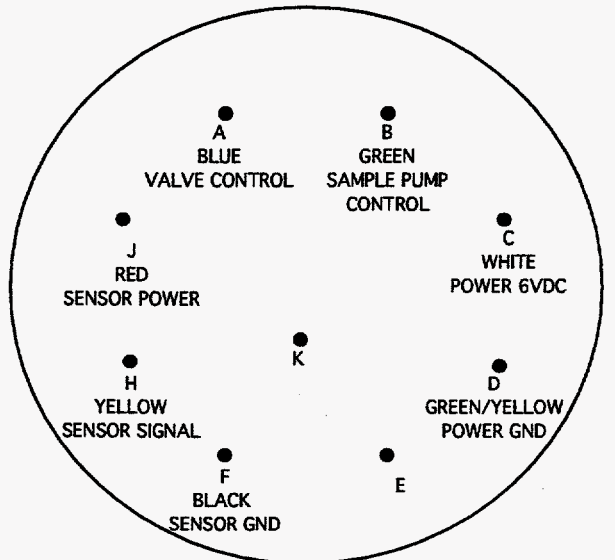


MS3112E12-10S BULKHEAD SOCKET,
VIEW FROM SOLDER TERMINALS.
CONNECTOR LINKS TO CONTROL UNIT CIRCUITS .



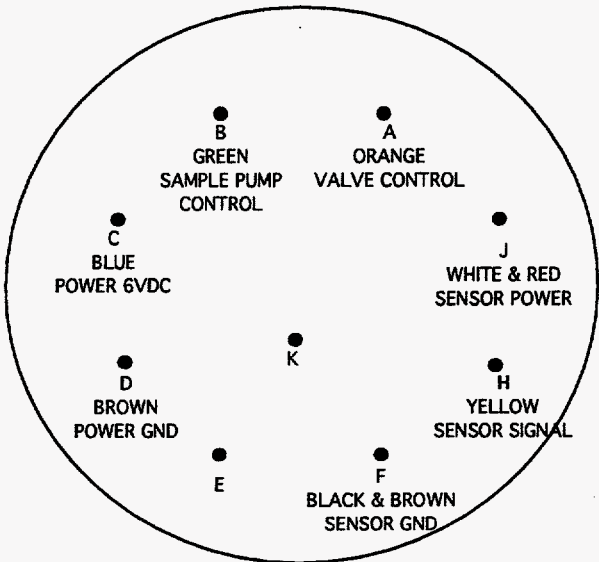
MS3116F12-10P MALE PLUG, VIEW FROM SOLDER TERMINALS. SOLDER
CONNECTOR TO A 100' LENGTH OF 22 GA 8-CONDUCTOR, SHIELDED
CABLE, WITH MAIN POWER TO 14-2 CORD.

CONE PENETROMETER REMOTE HEAD CABLE RECEPTACLE



AMPHENOL 165-14 CABLE RECEPTACLE, VIEW FROM SOLDER
TERMINALS . CONNECTOR LINKS TO REMOTE HEAD CIRCUITS

CONE PENETROMETER REMOTE HEAD CABLE PLUG



AMPHENOL 165-15-1002 CABLE PLUG, VIEW FROM SOLDER
TERMINALS . SOLDER CONNECTOR TO REMOTE END OF
100' LENGTH OF 22 GA 8-CONDUCTOR, SHIELDED
CABLE, WITH MAIN POWER TO 14-2 CORD.