NRC FORM 313
(10-87)
10 CFR 30, 32, 33, 34, 35 and 40

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1. THIS IS AN APPLICATION FOR *(Check appropriate item)*

☐ A. NEW LICENSE
☐ B. AMENDMENT TO LICENSE NUMBER ____________
☒ C. RENEWAL OF LICENSE NUMBER 29-11872-03

2. NAME AND MAILING ADDRESS OF APPLICANT *(Include Zip Code)*

Union Carbide Corporation
Linde Division
P.O. Box 237
Keasbey, NJ 08832

3. ADDRESS(ES) WHERE LICENSED MATERIAL WILL BE USED OR POSSESSED.

Industrial Highway, Keasbey, New Jersey

4. NAME OF PERSON TO BE CONTACTED ABOUT THIS APPLICATION: Thomas C. Ahlers

TELEPHONE NUMBER: (201) 271-2637

SUBMIT ITEMS 5 THROUGH 11 ON 8½ x 11" PAPER. THE TYPE AND SCOPE OF INFORMATION TO BE PROVIDED IS DESCRIBED IN THE LICENSE APPLICATION GUIDE.

5. RADIOACTIVE MATERIAL
   a. Element and mass number, b. chemical and/or physical form, and c. maximum amount which will be possessed at any one time.

6. PURPOSE(S) FOR WHICH LICENSED MATERIAL WILL BE USED.

7. INDIVIDUAL(S) RESPONSIBLE FOR RADIATION SAFETY PROGRAM AND THEIR TRAINING AND EXPERIENCE.

8. TRAINING FOR INDIVIDUALS WORKING IN OR FREQUENTING RESTRICTED AREAS.

9. FACILITIES AND EQUIPMENT.

10. RADIATION SAFETY PROGRAM.

11. WASTE MANAGEMENT.

12. LICENSEE FEES *(See 10 CFR 170 and Section 170.31)*
FEE CATEGORY 3P
AMOUNT ENCLOSED $120.00

13. CERTIFICATION. *(Must be completed by applicant)* THE APPLICANT UNDERSTANDS THAT ALL STATEMENTS AND REPRESENTATIONS MADE IN THIS APPLICATION ARE BINDING UPON THE APPLICANT.

THE APPLICANT AND ANY OFFICIAL EXECUTING THIS CERTIFICATION ON BEHALF OF THE APPLICANT, NAMED IN ITEM 2, CERTIFY THAT THIS APPLICATION IS PREPARED IN CONFORMITY WITH TITLE 10, CODE OF FEDERAL REGULATIONS, PARTS 30, 32, 33, 34, 35, AND 40 AND THAT ALL INFORMATION CONTAINED HEREIN, IS TRUE AND CORRECT TO THE BEST OF THEIR KNOWLEDGE AND BELIEF.

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SIGNATURE—CERTIFYING OFFICER	TYPED/PRINTED NAME	TITLE	DATE
T. C. Ahlers	Thomas C. Ahlers	Region Technical Supervisor	7-22-88

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$120	82833289	ML10 109353	8/16/88

5. Radioactive Material — No changes to License Number 29-11872-03

6. Purpose(s) for which licensed material will be used.

   A. Nickel ^{63} - Source not in use; transfer off site being investigated

   B. Hydrogen ^{3} - No changes to License Number 29-11872-03

7. Individual(s) responsible for Radiation Safety Program and their training and experience.

   Thomas C. Ahlers - No changes to License Number 29-11872-03

   Amy Ust - No changes to License Number 29-11872-03

   Elizabeth C. Hagstad - Thoroughly trained on operation and use of Hydrogen ^{3} Gas Chromatograph. Four years experience operating Hydrogen ^{3} Gas Chromatograph.

   Ronald W. Shrewsbury, Joseph F. Salfelder, Diane Pacholec, Robert Bradley and Pamela White are no longer employed at this Union Carbide Corporation location.

8. Training for individuals working in or frequently restricted areas.

   Not applicable; no restricted areas

9. Facilities and Equipment — No changes to License Number 29-11872-03

10. Radiation Safety Program — No changes to License Number 29-11872-03

11. Waste Management — No changes to License Number 29-11872-03

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OPERATING
AND
MAINTENANCE MANUAL

ATC MODEL-140 A WIDE-RANGE ELECTRON-CAPTURE DETECTOR

Analog Technology Corporation
3410 East Foothill Boulevard
Pasadena, California 91107

August, 1975

Abe Singerman
UCC - Linde S.G. Div.
Feb 26th, 1982

Model-140A Wide-Range Electron-Capture Detector

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IMPORTANT

READ BEFORE CONNECTING OR USING YOUR DETECTOR ASSEMBLY.

Each ATC Model-140A Wide-Range Electron-Capture Detector system has been carefully manufactured and tested before shipment to ensure that it will operate as specified and produce a baseline current in excess of 2×10^{-8} amperes in clean carrier gas. If factory measured baseline current levels are to be maintained and permanent damage to the detector cell is to be avoided, it is essential that the gas entering the detector be free of electrophilic compounds (other than the minute sample concentrations being measured) and substances which might react with or deposit upon the tritiated-scandium source foil. Such substances may be present in the bottled carrier gas, in the column bleed, in the septum bleed or may evolve from the walls of system tubing and fittings that are made of inappropriate materials or have been inadequately cleaned.

Section 5.0 of this manual discusses these subjects in detail and should be read and followed carefully by anyone who has not already established and demonstrated a properly operating electron-capture detector system. Please note that the Model-140A warranty does not cover repair of contaminated detector cells -no matter how short the operating time may be. It is quite possible to permanently degrade a detector cell within minutes of connection to a contaminated column or gas supply. With proper attention to these precautions, however, a detector cell should easily provide a year or more of normal operation before replacement is required.

1.0 SCOPE

This document describes the ATC Model-140A Wide-Range Electron-Capture Detector and provides sufficient information to permit its installation, operation, and maintenance by someone generally familiar with gas-chromatographic procedures and techniques.

2.0 APPLICATION

The Model-140A ECD system is intended to measure the concentration of electron-capturing chemical species in the effluent from a gas-chromatographic column. Its output signals are (a) a pulse train whose frequency is linearly related to that concentration and (b) a range-switched analog voltage suitable for driving a linear chart recorder or other analog data-acquisition equipment.

3.0 ELECTRON-CAPTURE DETECTOR OPERATING PRINCIPLES

3.1 General

An electron-capture detector is a gas-ionization chamber within which conduction electrons are produced at a known rate, usually by a radioactive source, and captured by the electronegative species within the gas sample. Electrical measurements made at the chamber's electrodes can be used to determine the free electron density and thus to infer the concentration of electron-capturing compounds.

The principal differences between the various types of available electron-capture detectors lie in the following three areas:

(1) Ionizing-source type.
(2) Geometry and design of the detector cell.
(3) Type of electrical stimulation and measurement.

Sections 3.2, 3.3, and 3.4 discuss these differences in detail.

3.2 Ionizing-Source Type

The purpose of the ionizing source is to produce ionization within the sample gas at a high and stable rate. Although gas ionization may readily be produced by electrical means, the far greater simplicity, stability, and reliability of radioactive sources have led to their use, almost exclusively, in electron-capture detectors. Furthermore, the desire for dense ionization in a small volume, with little or no penetrating radiation component, is best met by low-energy beta emitters; specifically, ^{3}H (tritium) and ^{63}Ni. Almost all of the EC detectors available use one of these isotopes.

3.2.1 Tritiated-Titanium Source. Tritium holds several major advantages of Nickel-63 as an ECD source material. Its betas have much lower energies (18 keV maximum compared with 61 keV), so that all the ionization is contained within 2 mm of the source-foil surface, and a very small detector volume is possible. Furthermore, tritium sources can be prepared with much higher specific activities than Nickel-63, leading to larger baseline currents and consequently greater sensitivity and wider dynamic range. Finally, the radiological health aspects of tritium are not nearly as severe as for Nickel-63, which tends to be retained indefinitely within the body, if ingested. The ECD source must be in solid form, however, so that tritium source technology is largely concerned with finding a mechanism for binding large numbers of hydrogen atoms on or very near the surface of a solid. Until very recently, the best solution involved adsorbing the tritium gas onto a hot titanium-plated metal surface. This technique produces high-intensity (0.25 curies/cm²) sources which will maintain the tritium surface bond at temperatures up to approximately 220 °C. Unfortunately, it is often desirable to operate GC detectors at higher temperatures than this, particularly for cleaning and bakeout, and tritiated titanium is relegated to those special applications where its restricted temperature limit is not important.

3.2.2 Nickel-63 Sources. Detector operation in excess of 220 °C is the one advantage which made Nickel-63 the most widely used ECD source during the time when tritiated titanium was the only other reasonable alternative. Nickel is a metal which can be plated on other metals and maintain source integrity over the full temperature range required for ECD operation and bakeout. As described before, however, the higher-energy betas it produces require a larger detector volume than is desirable, and its low specific activity restricts source activities to a few tens of millicuries. Detectors which use Nickel-63, therefore, achieve high-temperature operation at the expense of baseline current, dynamic range, sensitivity, and response time. Coupled with the more severe restrictions on handling and distributing Nickel-63, these drawbacks have spurred the development of a higher-temperature tritium source foil.

3.2.3 Tritiated-Scandium Sources. Tritiated scandium combines all of the best features of tritium with a maximum temperature limit of 325 °C, which is quite adequate for essentially all ECD applications. Source activities are available that approach one curie/cm², producing a higher specific activity and thus a higher baseline current than any other beta source available for ECD use. The ATC Model-140A Wide-Range Electron-Capture Detector employs a 1-curie tritiated-scandium source with a surface area of 1.6 cm², and achieves a baseline current of at least 2×10^{-8} amperes in a detector volume of only 180 microliters.

3.3 Geometry and Design of the Detector Cell

A good ECD design must maximize the cell's baseline current, minimize its sample volume, and maintain appropriate electrical and mechanical properties over the desired temperature range—from room ambient up to at least 300 °C. As discussed above in 3.2, the choice of source material plays a key role in that design. Other pertinent design factors will now be considered.

3.3.1 Detector Cell Geometry. The requirements for large baseline current and small detector cell volume are somewhat contradictory and require a good compromise in chamber shape and dimensions. However, a cell geometry which maximizes the ratio of source area to cell volume will always be superior to a geometry that causes this ratio to be small. The ATC Model 140A detector design uses coaxial cylindrical electrodes with the source foil used as the outer electrode (cathode). The effective sample volume (180 microliters) is the gas volume contained within this cylinder, since its radius is such as to provide ionizing radiation throughout essentially all of the interelectrode space.

3.3.2 Carrier-Gas Flow Considerations. If the ECD output signal is to accurately reflect peaks separated by the column, it is important that the detector flow characteristics be carefully controlled. Flow must be diffuse rather than laminar, and there must be rapid sweep-out so that readings are not contaminated by remnants of earlier sample concentrations. The symmetrical peak shapes produced by the ATC Model-140A ECD and the rapid recovery it provides following large peaks are results of a unique design that eliminates trapped gas volume, thoroughly mixes the gas at the sample-volume entrance, and minimizes gas volume throughout the detector.

3.4 Detector Electrical Operation

Although the ATC Model-140A ECD design has been optimized in all of its aspects, the principal improvement over previous designs is in its constant-current, variable-frequency mode of operation. The advantages this operating mode has over dc and fixed-frequency operation will now be described.

3.4.1 DC Operation. Electron-capture detectors of earlier design are conceptually very simple devices. The chamber electrodes are biased with a dc voltage, and the resultant direct current is measured using an electrometer amplifier. Obviously, the maximum current (baseline) occurs when only pure carrier gas is present and reductions in the current are used to determine concentrations of electronegative species. Unfortunately, detectors using this mode of operation are often unable to distinguish between electrons and other charge carriers, such as positive ions and electronegative molecules bearing captured electrons, so that the measured current is strongly dependent on carrier-gas flow rate, detector temperature, cell voltage, and other parameters that are not simply related to sample concentration. DC-operated electron-capture detectors are, therefore, characterized by drifting baselines, sensitivity variations and very limited dynamic range.

3.4.2 Fixed-Frequency Pulsed Operation. Substantial improvement over dc operation can be obtained by applying the detector bias as a sequence of short-duration pulses, durations long enough to collect the very mobile electrons, but not long enough to permit the heavier, slower ions to contribute significantly to the total integrated current. Here, as in the case of the dc-operated detector, a dc electrometer is used to measure the average current induced by the bias-voltage pulses. A properly designed, fixed-frequency pulsed ECD thus eliminates much of the drift problem associated with dc operation. Since the detector current must actually vary over the measured dynamic range, however, the linear response of a fixed-frequency system is still limited, at best, to a range of a thousand to one. Some detectors of this type provide manual frequency ranging to permit the operator to select where this limited dynamic range will lie, and some provide a form of circuitry which extends dynamic range by linearizing a basically nonlinear output signal. None, however, escape the basic linearity problem associated with any small ionization chamber device that operates over a wide range of currents.

3.4.3 Constant Current, Variable-Frequency Pulsed Operation. Before describing the operation of the **ATC Model-140A ECD system**, it will be instructive to consider the manner in which the free electron density varies with time in a pulsed ECD. If the bias pulse is "on" long enough for all electrons to be collected at the anode, then at the instant when this bias pulse goes "off", the number of free electrons in the detector is essentially zero. (We assume that the bias pulse is so short in duration that a negligible number of new electrons is produced while it is "on".) While the bias remains off, the number of electrons changes due to:

(1) electron production at a constant rate, and

(2) electron attachment at a rate proportional to the product of the number of electrons N_e and the number of electrophilic molecules N present.

That is,

$$\frac{dN_e}{dt} = k_o^2 - k^2 N N_e, \tag{1}$$

where dN_e/dt is the time rate of change of the number of free electrons present and k^o and k are constants. For the sake of clarity and simplicity we are considering that only one electron-capturing substance is present.

The solution of (1) which satisfies the appropriate initial condition (at $t = 0$, $N_e = 0$) is:

$$N_e = \frac{k_o^2}{k^2 N}\left(1 - e^{-k^2 N t}\right) \tag{2}$$

As would be expected, equation (2) shows that N_e begins at zero and increases exponentially toward an equilibrium value of k^{o2}/k^2N. If we apply a brief charge-collection pulse periodically with a frequency f, each such pulse will collect a charge increment ΔQ, where:

$$\Delta Q = \frac{k_o^2 q}{k^2 N}\left(1 - e^{-\frac{k^2 N}{f}}\right), \tag{3}$$

Where q is the electronic charge, and average current is

$$I = \frac{k_o^2 q}{k^2}\left(\frac{f}{N}\right)\left[1 - e^{-k^2\left(\frac{N}{f}\right)}\right] \tag{4}$$

Note that the average current collected by this periodic collection pulse depends only on the ratio f/N. Everything else in equation (4) is constant. If, therefore, we hold the detector current constant, the system frequency will vary linearly with sample concentration N. The ATC Model-140A ECD system does just that, employing a variable-frequency detetector-bias pulser within a feedback loop which maintains constant detector current.

Figure 3-1 is a simplified block diagram of the ECD system.

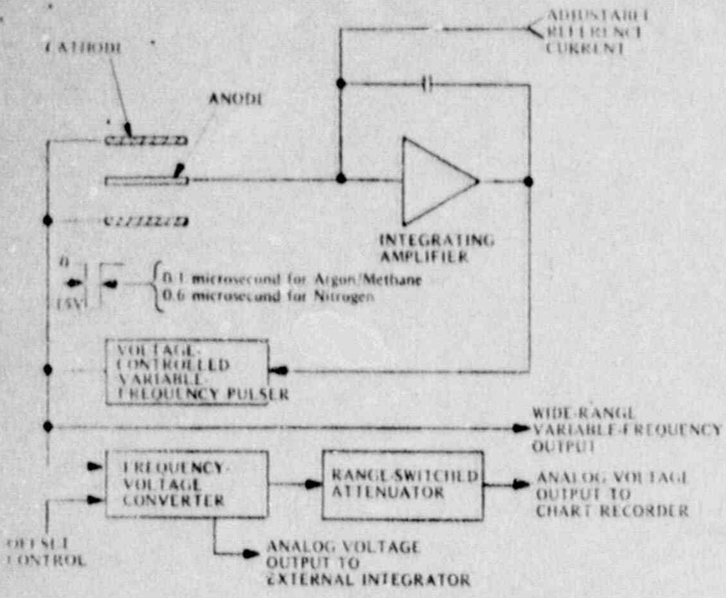

Figure 3-1

**Simplified System Block Diagram–
ATC Model-140A Wide-Range Electron-Capture Detector**

VERY important

The system operates as follows. If the average detector output current is different from the operator-controlled reference current, the resulting non-zero input current to the integrating amplifier will cause the amplifier output voltage to change, thus changing the frequency of the detector cathode bias pulses. The feedback loop forces a change in frequency that will produce a different detector current, thus reducing the net amplifier input current to zero. The action of the overall servo loop therefore forces the detector current to equal the reference current by varying the frequency in response to changes in the sample concentration. As required by equation (4), the observed change in frequency varies linearly with sample concentration over an extremely wide operating range compared to that of variable current electron-capture detectors. The low-frequency limit of operation is set by requiring that the pulse period be short compared to the time constants for electron loss mechanisms that compete with the desired sample absorption, while the high-frequency limit is determined by requiring the collection pulse width to be wide enough to collect all the electrons present, yet small compared to the pulse period. In the ATC Model-140A LCD system, this frequency range runs from a 5-kHz baseline to 5-MHz maximum. Frequency changes of ±5 Hz can be seen and measured with this system, so that the maximum linear dynamic range covers six decades, or a million-to-1. Some electronegative substances of common interest are restricted, by their chemical properties, to a 10^5-to-1 linear dynamic range, yet the ATC Model-140A LCD provides much broader dynamic range for these species than that afforded by available dc or fixed-frequency systems or by other "constant-current" systems of more restricted frequency range.

4.0 INSTALLATION

The Model-140A LCD system consists of two separate units, the Detector Assembly and the Control Unit, which, in use, are interconnected by three electrical cables.

4.1 Installing the Detector Assembly

> **CAUTION:** Before connecting the detector to the GC column, read and observe the carrier-gas cleanliness precautions contained in Section 5.2. Contamination of the detector cell is *not* covered by the Model-140A warranty.

The Detector Assembly normally mounts on an outside wall of the GC column oven with its inlet tubing extending into the oven through a suitable access hole, as shown in Figure 4-1.

* Suggestion: Are we using the ECD correctly?

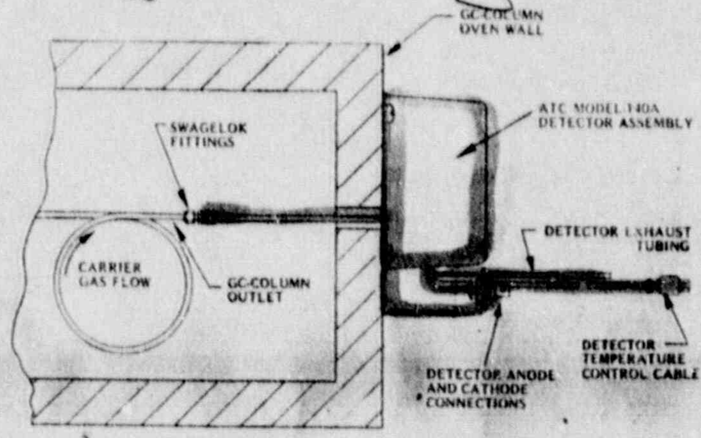

Figure 4-1

* THIS SECTION DOES NOT APPLY TO HOW WE ARE USING the instrument at Kewaunee!

Typical Attachment of ATC Model-140A ECD to a Gas-Chromatographic Column

The Detector Assembly inlet tubing must be connected to the outlet end of the GC column in a manner that will permit unobstructed gas flow without leakage over the full column temperature range. For metal column tubing, Swagelok-type fittings are usually recommended for making this connection. The Detector Assembly inlet tubing is 1/8" O.D. stainless-steel tubing. If the GC column is also 1/8" O.D. stainless steel, the connection may be made using two 1/8" Swagelok nut/ferrule sets and a single 1/8" Swagelok union. Stainless steel is the preferred Swagelok fitting material. Connection to other sizes of metal column tubing or tubing made of other materials will require modification of this procedure to suit the materials and sizes encountered. The Detector Assembly inlet and outlet tubing may be bent or cut, as necessary, to fit the particular installation, assuming that proper tools and techniques are used to avoid kinking.

The Detector Assembly should be securely attached to the outside of the oven wall using sheet metal screws through the flange mounting holes provided. Figure 4-2 shows these mounting dimensions. The hole through which the inlet tubing enters the oven should be completely covered by the Detector Assembly housing to minimize oven heat loss and temperature nonuniformities in the detector inlet tubing. The detector exhaust tubing should be connected to a fume hood or exhausted to the outdoors, using the length of flexible plastic tubing provided with the detector, or through suitable substitute tubing.

If the detector mounting plate interferes with installation in the desired location, it may be removed by disconnecting the two BNC receptacles and removing the four flathead screws which attach the plate to the Detector Assembly housing. After removing this mounting plate, be sure to replace the four screws to hold the housing cover in place. With the mounting plate removed, the anode and cathode BNC receptacles will be free to move about and will tend to rest against the hot Detector Assembly housing. In order to hold the temperature of these receptacles to a suitable level, it may be necessary to insert a small piece of thermal insulating material between the receptacles and the housing

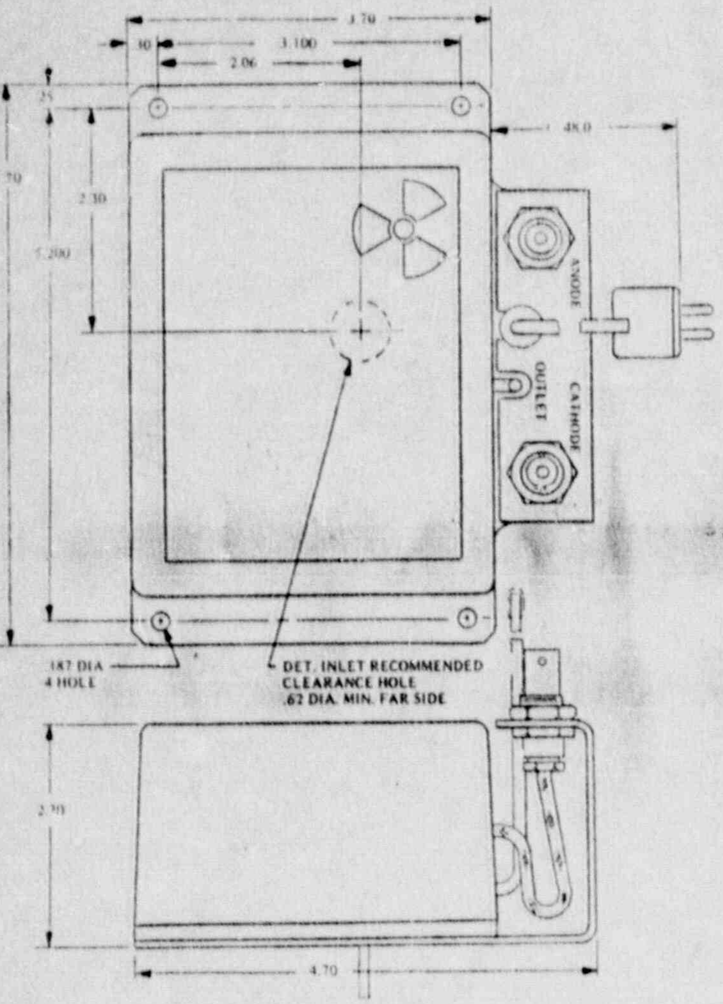

Figure 4-2

ATC Model-140A WRECD Mounting Dimensions

4.2 Installing the Control Unit

The Control Unit may be placed or mounted in any location within approximately five feet (cable length) of the Detector Assembly. There are no special orientation requirements, except that the front-panel controls must be accessible for operation.

4.3 Making the System Interconnections

Three cables connect the Detector Assembly to the Control Unit for system operation. The two coaxial connectors are labeled "anode" and "cathode", and must be connected to the correspondingly labeled BNC receptacles on the Control Unit rear apron using the BNC-to-BNC coaxial cables provided. The third cable provides for the detector-block temperature control. It terminates in the four-prong plug which must be inserted in socket J5 on the Control Unit rear apron. J4 on the rear apron is the analog voltage output jack. The output from J4 is between 0 and +10V and it must therefore be attenuated with either an ATC 140A-3 or 140A-4 attenuator to drive a chart recorder; see 4.5 below for more details.

4.4 System Power Input

The Model-140A system operates directly from a 110-V 60-Hz power source, generating its own dc-operating potentials, detector-bias pulses, and temperature-control output. Simply connect the Control Unit line cord to a source of 110-V, 60-Hz power. A Control Unit front-panel lamp will automatically indicate the presence of suitable input power. For operation on 220 volts, 50/60 Hz, it will be necessary to use a step-down transformer with an output rating of at least 150 volt amperes. If a suitable transformer is not available locally, one can be purchased directly from Analog Technology Corporation. With full heater power output, the complete system draws approximately 70 watts. In stand-by condition, with the detector temperature controller "off", the power drain is negligible.

4.5 Using the Analog Voltage Output

BNC receptacle J4 on the Control Unit rear apron provides a range-switched linear voltage output for driving a linear chart recorder or other analog voltage monitoring instrument. The output voltage range is 0 to +10 volts at receptacle J4 and may be attenuated to 0 to 1 volt, 0 to 10 millivolts or 0 to 1 millivolt using the appropriate ATC analog output attenuator. One such attenuator (0 to 1 millivolt unless ordered otherwise) is supplied with each Model 140A, and additional attenuators may be purchased directly from ATC. The input resistance of any monitoring instrument should be at least 1000 ohms for output ranges of 0 to 10 millivolts and smaller and should be at least 10,000 ohms for ranges larger than 0 to 10 millivolts.

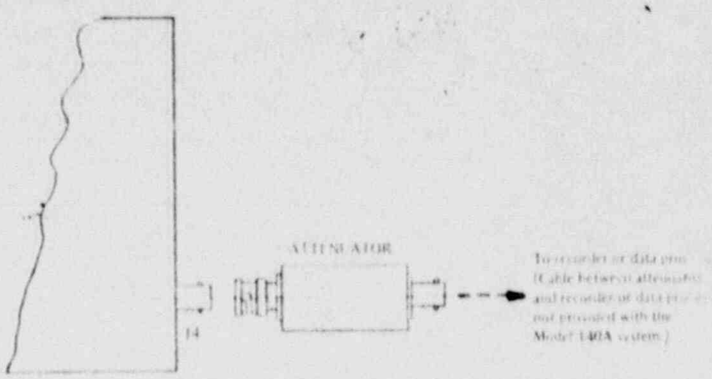

Figure 4-3

4.6 Using the Frequency Output

BNC receptacle J3 on the Control Unit rear apron provides a wide range frequency or pulse-train output for direct output monitoring over the full Model-140A dynamic range. To use this feature, connect this output to the input of any standard pulse-counter instrument, using a BNC-to-BNC coaxial cable. The output pulses have a positive 5-V amplitude, and can drive a 10-kΩ or larger load impedance. The normal output-frequency range is from 5 kHz to 5 MHz.

Although many Model-140A users will use only the analog recorder output signal, the direct frequency output is potentially far more powerful for acquisition of wide-dynamic-range chromatographic data. The change in frequency at receptacle J3 is directly proportional to sample concentration over a dynamic range of up to one-million-to-one and can be measured digitally over that range without need for any range-changing, either manual or automatic. Furthermore, the signal is already in digital form, making subsequent data handling more convenient and accurate.

In order to make use of this unique frequency-output feature from the Model-140A Wide-Range ECD and from the Model-150 Wide-Range Digital Electrometer, ATC has developed the Model-160 Chromatographic Memory and Display instrument, which accepts this signal directly and permits acquisition, manipulation, and storage of chromatographic data over a full 10-million-to-one linear dynamic range. With suitable interface modules, the frequency output can also be used to provide a direct digital input to a computer system or other data-handling equipment. The ATC engineering staff is available for consultation on all such potential applications and welcomes your inquiry.

5.0 CARRIER-GAS CONSIDERATIONS

5.1 Carrier-Gas Selection

The Model 140A may be used, without modification, with either argon/methane or pure nitrogen carrier gases. The higher atomic number of argon (hence greater electron density) results in a larger baseline current than is obtained using nitrogen and, with the nominally 5% methane additive, the electron drift velocity is significantly increased over that observed in nitrogen. For these reasons, argon plus 5% methane is the preferred and recommended carrier gas for use in the Model 140A, and it is for this reason that the detector system, when delivered, is normally set for use with argon/methane.

For various reasons, including the relatively high cost of argon, the ready availability of nitrogen and the desire to avoid gas mixtures in some applications, many ECD users prefer to operate with nitrogen carrier gas, accepting the reductions in baseline current and electron mobility that result. To accommodate these users, a switch has been provided on the Model 140A, which permits selection of a detector collection pulse width that is appropriate for either argon/methane (0.1 microsecond) or nitrogen (0.6 microsecond). New instruments are normally delivered with this switch set to "argon/methane". Since this is not an operator control, the switch is located within the Control Unit housing, on the cover of the main electronic module (Assembly 10). Removal of the two recessed screws at the Control Unit rear panel will permit this assembly to be removed from its housing, exposing the carrier gas selection switch. Be sure to replace these screws upon reassembly so that the Control Unit cannot accidentally slide from the housing during subsequent handling.

Attempts to use nitrogen carrier gas while this switch is set to "argon/methane" will result in severely degraded performance, since that pulse width is inadequate to collect more than a small fraction of the free electrons in the detector. The instrument may, however, be operated on argon/methane with the switch set in the "nitrogen" position. In that case, the collection pulse width is greater than necessary and the maximum linear operating frequency (and hence the system dynamic range) is needlessly restricted. Apart from that restriction, satisfactory operation will be obtained over the resulting smaller dynamic range.

Although other gases may possibly be useful, or even provide significant advantages in certain applications, ATC has not investigated the use of carrier gases other than argon/methane and nitrogen, and therefore cannot recommend any others. A principal requirement for any substitute carrier gas is that it be inert with respect to the detector materials (including the tritiated-scandium radiation source) as well as the sample and column materials. In particular, hydrogen, even in fractional amounts, is known to produce excessive source bleed and should never be used in the ATC Model 140A.

5.2 Carrier-Gas Purity

The extreme sensitivity and susceptibility of electron-capture detectors to damage by the accumulation of internal deposits require that the gas passing through the detector cell be free of certain contaminants. Common electrophilic materials, such as oxygen, freon, and others, can, if present, substantially reduce the detector's baseline current and interfere with measurement of trace sample concentrations. Materials which tend to condense, settle, polymerize, etc. on the radiation source foil also produce immediate baseline current reduction which is, furthermore, often irreversible, requiring foil cleaning or replacement to effect recovery.

It is not feasible to compile a complete listing of all the substances to be avoided in the ECD carrier gas, particularly in the case of compounds which may become deposited on the source foil. Clearly electrophilic materials (oxygen, freon, carbon tetrachloride, chloroform, etc.) must certainly be avoided. Often, however, the offending substance is present in such small concentrations as to defy reasonable attempts at qualitative analysis and, in fact, only becomes apparent in its effect on the ECD. The ultimate definition of "clean carrier gas" is, therefore, gas which permits attainment of satisfactory baseline current. If a new Model 140A ECD produces much smaller values of baseline current than obtained during acceptance testing at ATC, it should be assumed that the difference is due to contaminants in the gas flowing through the detector cell.

The possible sources of such contamination are many and, in fact, several may be present in the same system at once. The most likely sources and means for reducing their effect are the following:

(1) Impure bottled gas. It is recommended that a quality grade of carrier gas be used at all times, either argon plus 5% methane or nitrogen. The major gas suppliers offer an electron-capture detector grade of both gases which is particularly low in oxygen content and should give good results in a clean system. Since the suppliers do not normally test their gases using an ECD, however, it is quite possible that even these gases may be too contaminated for ECD use. For this reason, the ATC Quality Assurance

Department has found it necessary to employ the following procedure in procuring argon/methane carrier gas for test-bench use:

(a) The gas is always bought from the same source.

(b) A new batch of gas is procured at least one month before the on-hand supply of proven gas is exhausted.

(c) The bottles used for ATC's carrier gas are specially marked and are never used for any other purpose.

(d) When a new batch of gas is received, each bottle is briefly connected to a Model 140A to determine the baseline current it can produce. Only after it is determined to be satisfactory for ECD use is each bottle placed in stock for future use.

(2) Contaminated Regulators, Valves, Fittings, and Tubing—Standards of cleanliness that are suitable for many GC applications may be totally inadequate for sensitive ECD work. Insofar as possible, all surfaces that contact the gas stream should be glass or metallic, preferably stainless steel, and should be thoroughly cleaned and baked before use. Although regulator diaphragms made of neoprene and other elastomers may be used successfully, they are somewhat more difficult to clean initially than are all-metal regulators. The commercial "ultra-pure" grade of regulator will usually provide adequate service. In particular, avoid the use of valves in which the gas stream is exposed to any packing or lubricating material.

New regulators, valves, and fittings are often cleaned at the factory using FREON fluids. The FREONS are all highly electrophilic materials, traces of which will severely reduce the detector baseline. It is usually possible to order new regulators with a special final acetone cleaning, and this is a desirable option. Older regulators, which have been in use for some time in nitrogen, argon or other non-electrophilic gas service, may require no additional cleaning.

All parts of a new carrier-gas transport system should be cleaned before assembly and then baked after assembly with clean carrier gas flowing through the system. The following cleaning sequence is generally adequate:

(a) Scrub the parts (fittings, tubing, etc.) in detergent-water solution, following with ultrasonic agitation.

(b) Rinse in clean water.

(c) Rinse in clean methanol.

(d) Rinse in clean acetone.

(e) Blow dry with dry, clean nitrogen gas.

(f) Store in clean container until assembly.

(g) After assembly, flow clean carrier gas through the entire system while heating the outside surfaces with a heat gun or torch. Do not heat any high-pressure portions of the system nor any components beyond their maximum operating temperature limits.

After cleaning the carrier-gas transport system, it should be kept clean by using only clean carrier gas and by avoiding direct exposure to the atmosphere when it is not in use.

(3) Inadequate Filtration—Since even the best-quality carrier gas seems to contain some water vapor and other potentially bothersome impurities, a simple oxygen/water trap or filter cartridge should be located near the beginning of the low-pressure line. Suitable commercial filters are available from chromatography supply houses. Our experience at ATC has been that a 100 cm^3 molecular sieve filter can absorb the water from 1000 ft^3 or more of carrier gas before it needs regeneration. Such regeneration merely involves heating the outer surface of the filter canister to drive off the adsorbed water while carrier gas flow is maintained. Since the effluent carrier gas is highly contaminated during this procedure, it should be vented directly to the ambient rather than through the column and detector. Regeneration of the filter material may also be necessary if ambient air is allowed to enter the system during periods of no carrier-gas flow.

(4) Excessive Septum Bleed—Often new septa, particularly in flash vaporization inlets, will bleed electrophilic vapors in quantity sufficient to reduce an otherwise acceptably high ECD baseline. This can be countered by selecting a "low-bleed" septum, operating it at the lowest temperature consistent with chromatographic requirements and conditioning each new septum for several hours at a temperature somewhat higher than its intended operating temperature.

(5) Improperly Cleaned GC Column Tubing—Occasionally a column will bleed much more than would be expected from its stationary phase because of dirty column tubing. The column material must be cleaned as well as the carrier-gas transport tubing before packing the column.

(6) Inadequate Column Conditioning—Even with the best of care in column tubing cleaning and support and stationary phase selection, a new column will usually bleed electrophilic compounds at a rate which will produce considerable ECD baseline reduction. After operating the column with clean carrier gas flowing for several hours at temperatures 10 or 20 °C higher than the intended normal operating temperature, this initial bleed will usually be reduced to acceptable levels. This conditioning should be accomplished with the column effluent exhausting directly to the ambient, *not* through the ECD or other detectors.

(7) Leaks—Since oxygen is an electrophilic gas, it is important that the system be tight against leaks that could permit air to enter the detector's carrier-gas stream. The effect of leaks is more pronounced in the lower-pressure portions of the system upstream of the detector, such as the detector inlet connection, but may become evident even at the septum and other high-pressure fittings that are subject to leaking. Leaks at low-pressure joints are rather difficult to find, so the detector outlet tubing is usually capped and the entire system is pressurized to 20 - 50 psig for locating such leaks. If the system will not hold pressure when sealed off, a leak exists and may be located using a leak-detecting liquid.

5.3 Carrier-Gas System Operation

Figure 5.3-1 shows a simple carrier-gas supply system adequate for most GC-ECD applications. This sketch was prepared under the assumption that a suitable constant-flow control is contained in the GC instrument. All components should be selected and cleaned as described above in 5.1 and 5.2.

5.3.1 Initial Operation. Connect the system as shown. Be sure there are no leaks. With the vent valve and shut-off valve closed and the regulator control fully counterclockwise (low output pressure), open the gas bottle valve. Next, turn the regulator control clockwise until the desired GC inlet pressure is attained. Do not exceed 100 psig! Open the vent valve slightly to flush air from the line for a minute. Finally, close the vent valve and open the shut-off valve. Adjust the pressure regulator as needed.

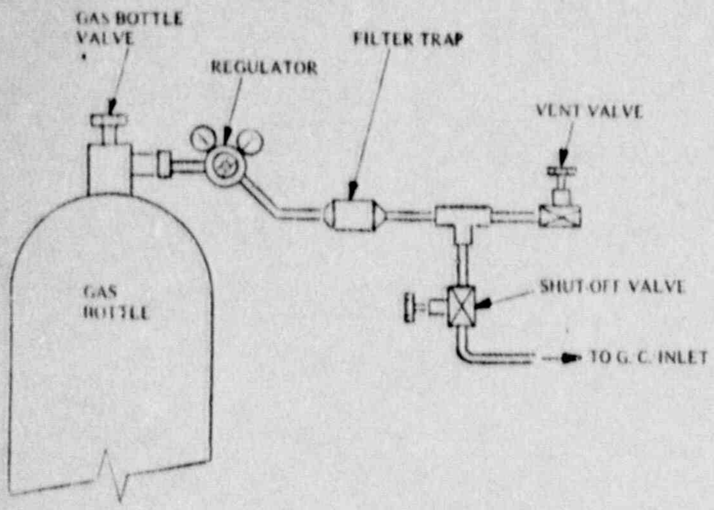

Figure 5.3-1

Typical Carrier-Gas Supply System

5.3.2 Shutting Down the Carrier-Gas Flow. Close the shut-off valve and the gas bottle valve and set the regulator control fully counterclockwise. This will keep the line and filter pressurized with carrier gas until the next usage, keeping it clean.

5.3.3 Restarting the Carrier-Gas Flow. Open the gas bottle valve, set the regulator to the desired pressure, and open the shut-off valve.

5.3.4 Changing Gas Bottles on an Operating System. Close the shut-off valve and the gas bottle valve and set the regulator control fully counterclockwise. Disconnect the regulator from the old bottle and reconnect it to the new bottle. Open the gas bottle valve. Next turn the regulator control clockwise until the desired pressure is attained. Open the shut-off valve.

5.3.5 Reconditioning the Filter. With the system in operation at just a few pounds of regulated pressure, close the shut-off valve and open the vent valve slightly to flush the filter and line directly to ambient. Beginning a few inches downstream of the regulator, heat the line with a heat gun or, very carefully, with a torch, moving the position of heat application slowly in the direction of the gas flow. When the filter is reached, heat it thoroughly all around its outer surfaces for at least 5 minutes. Be careful not to exceed the maximum safe operating temperature of the filter canister or any other line fittings. Continue heating the line downstream of the filter until the vent valve is reached. Allow the line and filter to cool while still flushing, then close the vent valve and re-open the shut-off valve for continued operation.

With carrier gas of the quality used at ATC, a small (100 cm^3) molecular sieve filter will require reconditioning after passage of 1000 to 1500 ft^3 of carrier-gas through it.

5.3.6 Carrier-Gas Flow Rate. Normal flow rates for operation of the Model-140A Wide-Range ECD Range from 30 to 100 cm^3 per minute. Lower flow rates will result in substantially reduced baselines, while higher flow rates are needlessly wasteful of carrier gas. At flow rates below 60 to 70 cm^3/minute the baseline varies with changing flow rate, so that a stable flow control is required to minimize drifting of the instrument's baseline. **At 80 cm^3/minute and higher, the baseline is nearly independent of flow rate.** Very important

6.0 SYSTEM OPERATION

6.1 Temperature Control

The Control Unit front panel includes a detector temperature control knob which also operates a heater power "on-off" switch at an extreme counterclockwise position. The Model-140A ECD system can be operated with no heater power, if desired, by placing this knob in the "off" position. Generally, however, the detector should be operated at a somewhat higher temperature than the GC column being monitored to avoid condensation of column-effluent materials in the detector. The actual temperatures used, of course, will depend upon the type of column being used, the species being resolved, and other factors established by chromatographic considerations. The Model-140A temperature-control range is from +160 °C to +320 °C, and any temperature in this range is stable within 0.1 °C. The indicator lamp in the lower-left portion of the Control Unit front panel is illuminated to indicate a heater-power "on" condition. When the set-point temperature is reached, this lamp will cycle on and off with approximately a one-half second period, indicating a properly operating temperature controller.

> CAUTION: In order to avoid condensation and possible contamination of the detector, always heat the detector before heating the column oven and ensure that the detector temperature is at least 10 °C above the column temperature at all times.

6.2 Offset Control

The single-turn front-panel control labeled "offset" permits shifting the position of the recorder output trace without changing the system sensitivity. The normal position of this control is centered (knob arrow aligned with panel mark) so that moderate baseline shifts in either direction may be offset, keeping the recorder trace on scale. The maximum range of the offset control is plus and minus full scale at the recorder output with the attenuator control in the 2^2 (times 4) position. This control (1) has no effect on the frequency output.

6.3 Recorder Output Attenuation Control

← Zeroing Recorder and instruments!

A sixteen-position front-panel switch establishes the relative sensitivity of the recorder output voltage to concentration of the electron-capturing species present in the detector. **It has no effect** (2) **on the frequency output.** The most sensitive position is fully clockwise, while the fully counterclockwise position, labeled "∞", is used to provide a zero-voltage output for setting the recorder zero level. The switch position labeling is in powers of two indicating that each step produces a factor-of-two change in system sensitivity. Thus, in order of increasing attenuation (or decreasing sensitivity), $2^0 = 1$, $2^1 = 2$, $2^2 = 4$, etc. up through $2^{15} = 32{,}768$. The wide dynamic range of the Model 140A produces the need for this high attenuation capability.

6.4 Baseline Adjust Control

The ten-turn front-panel control labeled "baseline adjust" is used to vary the adjustable reference current shown in Figure 3-1 and thus to set reference output levels with pure carrier gas flowing through the detector. The nominal baseline frequency setting is 5 kHz, which produces zero volts recorder output when the "offset" control is centered.

The "Baseline Adjust" setting that must be used to establish a 5-kHz or zero-volt baseline is a measure of the detector baseline current, and is therefore a useful parameter for monitoring the condition of the detector source foil, the carrier-gas purity, air leaks into the system, and all other conditions that can result in reduced baseline current. This 10-turn control has been provided with a turns-counting dial to display this parameter directly to the operator, who may use it to calculate the actual baseline current or merely to observe relative changes in detector condition from day-to-day or month-to-month. The actual baseline current, in amperes, is equal to the dial reading divided by 2×10^{10}. Thus, a typical baseline setting of 420 would represent 2.1×10^{-8} ampere of detector current. *

The "Baseline Adjust" control, which affects both the frequency and recorder outputs of the Model 140A, also affects the overall system sensitivity. It is therefore difficult to compare, quantitatively, two chromatograms made with different settings of this control unless each chromatogram is accompanied by a standard made with the same "baseline adjust" setting. For this reason, the "offset" control has been provided to permit repositioning the recorder output trace as required by changing baseline conditions without changing the "baseline adjust" setting.

6.5 Normal Operation

With the system installed and interconnected as described in Section 4, the Model 140A will operate automatically, generating a variable-frequency pulse-train output and a recorder analog output. After all elements of the chromatographic system have reached stable equilibrium temperature and carrier-gas flow conditions, the detector output will stabilize at a baseline value. With the "offset" control centered, adjust the "baseline adjust" control to produce a 5000-Hz baseline frequency or 0-volt recorder output baseline. If the recorder output is used, the "recorder output attenuation" control should be in one of the most clockwise positions (around 2^0) to achieve good sensitivity in this adjustment. After setting the baseline, set the "recorder output attenuation" control as needed to provide an adequate recorder trace of the chromatograms run. If moderate baseline changes occur due to continued detector clean-up, changes in operating conditions or other causes, use the offset control, as necessary, to reposition the recorder trace before each injection without changing sensitivity. All chromatograms made with the same "baseline adjust" setting will have the same detection sensitivity even though the offset control has been used to reposition the recorder trace between runs.

7.0 SYSTEM MAINTENANCE

7.1 Control Unit Maintenance

There are no special or unusual maintenance [illegible] the Control Unit that would not apply to any [illegible] instrument. There are no high voltages present and no components, such a[illegible]acuum tubes, that would be particularly sensitive to ordinary laboratory shock and vibration conditions. The Control Unit will operate within specifications over the ambient temperature range from 0 to +140 °F.

7.2 Detector Assembly Maintenance

The Detector Assembly is a rugged unit which is unlikely to be damaged by any conditions encountered in or around a GC instrument. The only reasonably credible mechanism by which the detector cell might be damaged is by introduction of contaminants that can condense or otherwise deposit on the source foil or insulator surfaces. For this reason, the detector should only be exposed to clean, pure carrier gas and samples that have passed through a clean column at a temperature lower than the detector temperature. Any substance that can traverse the entire column is not then likely to condense in the detector in any significant amount.

If an operating error is made and some source contamination does occur, it will be evident in a reduction of the detector baseline current. Usually this contamination can be removed and full baseline current can be restored by heating the detector to its maximum temperature limit of 320 °C for a period ranging from a few minutes to several hours. If this treatment does not adequately renew the baseline, a new detector source foil will be needed for achieving the maximum possible linear dynamic range. However, a scandium-tritide detector can operate quite successful with its baseline reduced by as much as an order of magnitude, if full dynamic range is not required. Even with such a reduction, the residual baseline current would exceed that of a new Nickel-63 source detector.

If source contamination occurs which cannot be relieved by heating the detector cell to its maximum temperature limit for a few hours, it will be necessary to replace the detector cell or, for those possessing a specific license permitting them to do so, to remove, clean and replace the source foil.

WARNING: The detector block within the detector assembly contains a one-curie tritium source that can be potentially hazardous if the mechanical integrity of the detector block is violated or if the detector temperature is permitted to exceed 325 °C. **Do not attempt to open the detector block or remove the source. Do not expose the source to temperatures exceeding 325 °C.** Operation of the detector should be immediately suspended until any necessary repairs have been made if there is any indication of possible failure of or damage to the detector block or source containment. More information regarding radiation safety is contained in Appendix A.

7.3 Troubleshooting Procedures

If, after following the procedures outlined above, the Model 140A does not appear to function properly, the [illegible] the problem should be isolated if possible prior to [illegible] ATC. The following [illegible] may be of assistance in such [illegible].

Symptom	Probable Causes
1) Heater light is "off" all the time	a) No instrument power. b) Heater cable not connected. c) Detector temperature higher than set point–wait for detector to cool. d) Temperature sensor open–check with ohmmeter (see Figure 7.3-1). e) Heater circuit open–check with ohmmeter. Should be ~ 300 ohms. f) Fuse blown–replace with 1-amp unit.
2) Heater light is "on" all the time	a) Detector temperature lower than set point–wait for detector to heat. b) Temperature sensor shorted–check with ohmmeter (see Figure 7.3-1). c) Heater open–check with ohmmeter.
3) Heater light cycles with long time period (greater than 1 sec)	Over-temperature protection thermostat is cycling–consult ATC.
4) Main power lamp does not come on.	a) No instrument power. b) Fuse blown–replace with 1-amp unit. c) Lamp blown–consult ATC.
5) Recorder off-scale negative with pure carrier gas flowing.	a) Baseline adjust set too low–increase the setting. b) Offset adjustment not at zero–readjust. c) Frequency-to-voltage converter inoperative–consult ATC.
6) Recorder off-scale positive with pure carrier-gas flowing.	a) Baseline adjust set too high–decrease the setting. b) Offset adjust not at zero–readjust. c) Anode and cathode coax cables reversed. d) Carrier-gas selector switch in wrong position (see section 5.1). e) Carrier gas impure (see section 5.2).
7) Low baseline current (requires "baseline adjust" setting lower than 100 to achieve zero-volt recorder output).	a) Carrier-gas selector switch in wrong position (see section 5.1). b) Carrier gas impure (see section 5.2).

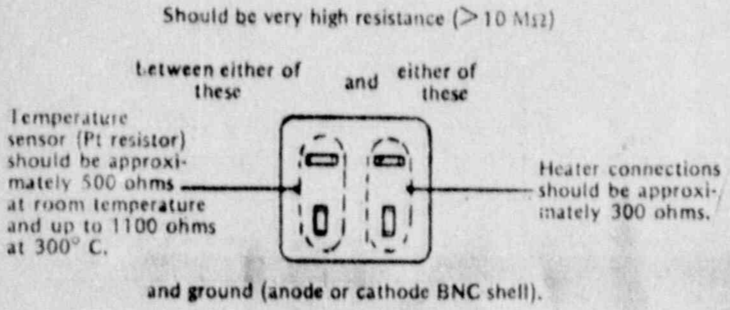

Figure 7.3-1

View of the Heater Plug, Prongs Pointing Toward Viewer

8.0 WARRANTY PROVISIONS

The Model-140A Wide-Range Electron-Capture Detector is guaranteed by Analog Technology Corporation to be free of defects in materials and workmanship when delivered and to operate as described herein for a period of one year after delivery to the original customer, except for baseline current degradation caused by a contaminated detector. ATC shall determine whether such degradation exists. If a new source foil is required, ATC will replace the entire detector for the usual exchange price then in effect. The exchange detector will also carry a new one-year warranty for failure modes other than detector contamination. All instrument returns for warranty service shall be prepaid by the user and preceded by ATC approval.

APPENDIX
RADIATION SAFETY

General Description

The following comments are not intended to negate or modify the appropriate government regulations covering radiation sources. The discussion herein is provided to acquaint the user with the radiation safety considerations associated with the ATC Model-140 ECD system. The comments also serve to indicate the responsibilities of personnel and organizations which use this instrument.

Within the detector assembly of the Model 140 is a scandium-coated stainless-steel foil which has been impregnated with tritium. This radioactive isotope, ^{3}H, decays with a half life of 12.26 years by the emission of beta particles to a stable nuclide of helium, ^{3}He. These beta particles are emitted with a maximum energy of 0.018 MeV and are absorbed by less than 1 mg-cm^{-2} of aluminum. Thus, there is no discernible radiation from the tritium external to the detector chamber and no hazard so long as the chamber integrity is not violated.

Temperature Limit

Scandium tritide is chemically stable at temperatures up to at least 325°C, which exceeds the maximum operating temperature of the Model-140 ECD. The instrument's temperature-control circuitry is designed to limit the maximum source temperature to less than 320°C, and a bimetallic thermostat is provided for back-up over-temperature protection.

It is very important that no attempt be made to defeat these temperature limit controls or otherwise heat the detector beyond 320°C. Such modifications would be in violation of applicable government regulations and might permit the release of radioactive material into the ambient air.

Assurance of Source Containment

The Model-140 ECD detector block is designed to resist attempts to open it using ordinary tools and nondestructive methods. Following assembly and initial testing, drive pins are inserted to prevent rotating the threaded plugs which would otherwise provide access to the source foil. This design feature ensures that destructive, clearly detectable means are necessary to gain access to the scandium-tritide source foil.

Any attempt to open the detector block forcibly is prohibited without prior written and specific approval from the cognizant government agency responsible for radiation safety enforcement.

Registration is Required

Registration with the State Health Department is required in states which regulate use of radioactive material under agreement with the U. S. Nuclear Regulatory Commission and for your convenience has been accomplished by Analog Technology Corporation (a copy of this registration form has been provided to you for your information). As a general licensee you are required to comply with radiation control regulations in force in your state. A copy of the regulations currently in effect in California and the name, address, and telephone number of your local radiation control agency are appended.

Your Local Radiation Control Agency is:

Excerpts from

CALIFORNIA
RADIATION CONTROL REGULATIONS

Title 17, Section 30192

(e) (1) A general license is hereby issued to acquire and use radioactive material when contained in devices designed and manufactured for the purpose of detecting, measuring, gauging or controlling thickness, density, level, interface location, radiation, leakage, or qualitative or quantitative chemical composition, or for producing light or an ionized atmosphere, when such devices are manufactured pursuant to a specific license authorizing distribution to general licensees; provided that each such device:

(A) is labeled in accordance with the provisions of the specific license which authorizes distribution of the devices;

(B) bears a label containing the following or a substantially similar statement:

"The receipt, possession, use and transfer of this device, Model ________, Serial No. ________, are subject to a general license or equivalent and the regulations of the US AEC or of a State with which the AEC has entered into an agreement for the exercise of regulatory authority. Removal of this label is prohibited.

CAUTION—RADIOACTIVE MATERIAL

________________________________"

(Name of Supplier)

The model, serial number and name of supplier may be omitted from this label provided they are elsewhere specified in labeling affixed to the device; and

(C) when specified by the label on the device, is installed on the premises of the general licensee by a person having a specific license which authorizes installation of such devices.

(2) Persons who possess a device pursuant to the general license contained in Section 30192(e)(1) shall, with respect thereto, be exempt from the requirements of Group 3 of this subchapter except for Sections 30253, 30254, 30293(a)(2), 30294, and 30295, but shall comply with all of the following:

(A) Within 30 days of the receipt of any such device register with the department and within 30 days of transfer of any such device notify the department, in accordance with the provisions of Group 1 of this subchapter (Registration of Radiation Sources).

(B) Shall not transfer, abandon or dispose of the device, except by transfer to a person holding a specific license to receive such device.

(C) Assure that all labels affixed to the devices at the time of receipt and bearing the statement, "Removal of this label is prohibited" are maintained thereon, and comply with all instructions contained in such labels.

(D) Have the device tested for leakage of radioactive material and proper operation of the on-off mechanism and indicator, if any, at the time of installation of the device or of replacement of the radioactive material on the premises of the general licensee and thereafter at no longer than six-month intervals or at such longer intervals as may be specified in the specific license which authorizes distribution of the device to general licensees, except that any such intervals shall not exceed three years unless specifically approved by the department. Devices containing only krypton in gaseous form need not be tested for leakage and devices containing only tritium need not be tested for any purpose.

(E) Have the tests required by Section 30192 (e)(2)(D) and all other services involving the radioactive material, its shielding and containment, performed by a person holding an appropriate specific license therefor.

(F) Maintain records of all tests performed on the devices as required under this section, including the dates and results of the tests and the names and addresses of the persons conducting the tests.

(G) Upon the occurrence of a failure of or damage to or any indication of a possible failure of or damage to, the shielding or containment of the radioactive material or the on-off mechanism or indicator, immediately suspend operation of the device until it has been repaired by or disposed of to a person holding an appropriate specific license therefor.

(H) Within 30 days after the occurrence of a failure of or damage to the shielding or containment of the radioactive material or the on-off mechanism or indicator or upon the detection of 0.005 microcurie or more of removable radioactive material, furnish to the department a complete description of the device (manufacturer, type, serial number) and a brief description of the event and the remedial action taken.

BETWEEN:

LICENSE FEE MANAGEMENT BRANCH, ARM
AND
REGIONAL LICENSING SECTIONS

(FOR LFMS USE)
INFORMATION FROM LTS

PROGRAM CODE: 03123
STATUS CODE: 2
FEE CATEGORY: 3P
EXP. DATE: 19880930
FEE COMMENTS:

LICENSE FEE TRANSMITTAL

A. REGION I

1. APPLICATION ATTACHED

APPLICANT/LICENSEE:	UNION CARBIDE CORP.
RECEIVED DATE:	880803
DOCKET NO:	3020793
CONTROL NO.:	109359
LICENSE NO.:	29-11872-03
ACTION TYPE:	RENEWAL

2. FEE ATTACHED
AMOUNT: $120
CHECK NO.: 82833289

3. COMMENTS

SIGNED DT
DATE 8/8/88

B. LICENSE FEE MANAGEMENT BRANCH (CHECK WHEN MILESTONE 03 IS ENTERED /✓/)

1. FEE CATEGORY AND AMOUNT: 3P $120

2. CORRECT FEE PAID. APPLICATION MAY BE PROCESSED FOR:
AMENDMENT ✓
RENEWAL
LICENSE

3. OTHER

SIGNED S. Kimberley
DATE 8/16/88