SEALED SOURCE AND DEVICE FILES

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INSTRUCTION MANUAL

FOR

ELECTRON CAPTURE DETECTOR

10 pp

I. INTRODUCTION.

The electron capture (E.C.) detector is a very specialized measuring device due to its broad spectrum of response to various organic compounds. While it is extremely sensitive to certain substances, it is completely unresponsive to gross quantities of most organic compounds. Obviously, the unique characteristics of this detector make possible a number of applications which could not be accomplished with any other gas chromatography detector. Some useful references to publications on electron capture detectors and analyses performed with them are listed in the attached bibliography.

II. PHYSICAL DESCRIPTION.

The detector contains a stainless steel foil coated with a thin layer of titanium. Approximately 200 millicuries of tritium, adsorbed on the foil as titanium tritide, supplies energy to the detector in the form of B radiation. The radioactive source and electrodes are housed in a stainless steel and 25% glass filled Teflon* cylinder. The detector is sealed within a massive aluminum heat sink for operation at elevated temperatures. In compliance with AEC regulations, the detector must not be removed from the aluminum block under any circumstances.

The block also contains two 85 watt cartridge heaters, a thermocouple and a thermal switch which prevents overheating. These components are easily removed if their replacement should be required.

III. RADIOACTIVE SOURCE.

As previously mentioned, the source contains approximately 200 millicuries of tritium. The radiation energy emitted from the detector is very low (0.018 M.E.V.) and requires no special shielding other than the materials normally used to enclose the foil. However, the total amount of radioactive material present is potentially hazardous if handled improperly and therefore, the detector should be treated with the care normally due to any radioactive source. The following points should be strictly observed in order to insure safe operation and long and efficient service.

A. Excessive heat will vaporize small amounts of tritium which can present a serious safety hazard to laboratory personnel. In order to prevent any possibility of this occurring, an overheat circuit will shut off the power to the detector heaters if the temperature exceeds 225°C. Under normal conditons, overheating will not occur because the heating circuit is balanced against thermal losses so that full power to the heaters is required to reach a block temperature of 220°C. For this reason, it is imperative to replace defective parts with original equipment only.

B. Life of Foil. The average foil life varies with operating conditions and is reduced by continuous use at high temperatures. It is desirable to operate below 180°C when possible, not only to prolong foil life, but also to reduce the possibility of contaminating the laboratory atmosphere. Even at 200 to 220°C, foil life should approach 1 or 2 years with careful use.

* Reg. Trademark E. I. duPont deNemours & Company, Inc.

C. Tritium Loss from the Cell. The activity of the foil will gradually decrease at the rate of 40 microcuri... per day due to the natural radioactive decay at room temperature. With a new foil, tritium loss increases to .38 millicuries at 200°C and 1.3 millicuries at 225°C.

SINCE VAPORIZATION OF TRITIUM DOES OCCUR, IT IS RECOMMENDED THAT THE FOLLOWING PRECAUTIONS BE TAKEN:

1. IF THE DETECTOR IS OPERATED ABOVE 150°C, THE GAS SHOULD BE VENTED FROM THE LABORATORY THROUGH A RUBBER TUBE CONNECTED TO THE CELL OUTLET. THE VENT (EG. FUME HOOD) SHOULD NOT EXHAUST NEAR AN AIR INTAKE AND, FOR MAXIMUM SAFETY, THE TUBING SHOULD EXTEND TO THE TOP OF THE FUME HOOD. IF THE EFFLUENT GAS IS NOT PIPED OUTSIDE THE LABORATORY, A MINIMUM FLOW OF 120 CUBIC METERS OF AIR PER 24 HOURS SHOULD BE PASSED INTO THE LABORATORY AND EXHAUSTED FROM THE BUILDING.
2. IF THE DETECTOR IS OPERATED BELOW 150°C, THE CELL EFFLUENT CAN ENTER THE LABORATORY AIR WITHOUT HAZARD.

D. Health Hazards of Tritium. Due to its low energy, the hazard of exposure to tritium radiation is negligible. Penetration of these rays through body tissue is only 1-2 mm which is insufficient to cause damage. The detector body and heat sink completely absorb the radiation before it can reach the operator.

HOWEVER, A HEALTH HAZARD DOES EXIST IF SUFFICIENT TRITIUM IS VAPORIZED FROM THE DETECTOR AND CONTAMINATES THE LABORATORY AIR. ABSORPTION CAN OCCUR THROUGH THE LUNGS AND THE 1 TO 2 MM PENETRATION IS SUFFICIENT TO CAUSE DAMAGE WHEN THE ISOTOPE IS CIRCULATED THROUGH THE BODY FLUID SYSTEM. FOR THIS REASON, THE PRECAUTIONS LISTED UNDER "C" ABOVE SHOULD BE STRICTLY OBSERVED TO INSURE THE SAFETY OF PERSONNEL IN THE AREA.

IV. CARRIER GAS REQUIREMENTS.

The following dry gases may be used.

Type of Circuit	Carrier Gas	Purge Gas
Pulse	Argon + 5% CH_4	Same
Pulse	Argon + 1% CO_2	Same
Pulse	Argon + 1% H_2	Same
Pulse	Helium	Argon + 10% CH_4*

*Best results are obtained with 1 part helium to 3 parts argon-methane.

V. COLUMN.

Columns to be used with the E.C. detector should not be prepared with electron capturing solvents or liquid phases due to the extremely high sensitivity of the detector toward these substances. Even when using low vapor pressure, liquid phases such as silicone rubbers and greases, it is important to condition the column for approximately one hour with flow at a temperature of 20-30°C higher than normal operating temperature. This must be done with the column exit disconnected from the detector to prevent liquid phase from condensing on the inside of the detector and causing noise or sensitivity loss.

This flow conditioning should be done even if the column has been conditioned at high temperature without flow, which technique is recommended particularly for SE-30 and similar columns as it frequently increases column efficiency.

As a starting point for pesticide analysis, we would suggest a 2% SE-30 coating on "Diatoport S" 80-100 mesh. This is a siliconized packing and, with this low liquid loading, analysis can be run as low as 160-180°C while eluting D.D.T. in a reasonable time (± 15-20 minutes with normal flows). We normally "no flow" condition these columns at 325°C for 1 hour and then flow condition at 210-220°C for 1 hour before use (as described above).

VI. OPERATION OF THE DETECTOR.

The E. C. is compatible with 400, 1400, 810 and 700 series instruments. In all cases, it has a pulse interval control; a temperature control and a manual reset button with pilot light to prevent hazardous high temperatures from being reached.

APPENDIX I. TEMPERATURE LIMITATION OF THE DETECTOR.

Two independent systems are incorporated to prevent the temperature of the detector exceeding the safe maximum of 225°C. In the event of one of the systems failing, the second will take over and prevent temperature overshoot.

These systems are:

A. Matching of the heaters to the load so that maximum power applied by the panel control will not allow the temperature to rise above 225°C.

B. The overheat protection circuit (O.P.C.) will operate if the temperature of the detector exceeds the set point of the thermal switch which is inside the detector heat sink. The closing of the switch operates a relay and removes all voltage from the detector heater. This relay remains energized, even though the system cools and the thermal switch opens, until a manual reset button is operated. Voltage can then be applied once more to the heaters. If the temperature control requires calibration--which should rarely occur--this is performed with the internal calibrate potentiometer, setting it to prevent the O.P.C. from being activated with full power applied by the front panel.

Pulse Control Circuit.

One control is used for the pulse system which applies voltage to the detector for short (3/4 micro sec) periods with an interval between pulses determined by the position of "pulse interval" control. Higher sensitivities are obtained with 150 micro sec. rate (lower frequency), while the pulse width remains at 3/4 micro sec.

When the unit will not be used for several hours, it is advantageous to position the pulse interval switch to off (no warm up time required when switching back on).

With units in the 400 and 810 series, all EC controls are located in the main control cabinets. 700 series instruments house these controls at the left rear quarter of the oven shroud. Models 1400 require a separate "Radioactive Detector Controller" to allow operation of the controller. This is fully dealt with later in the manual. When using E.C. on dual purpose units, couple the electrometer cable to the E.C. cathode before attempting to operate the detector.

VII. PRINCIPLE OF OPERATION.

The tritium source emits B-rays (electrons) which travel to the anode of the cell thus producing a current flow of about 10^{-9} amps. G.C. peaks having an affinity for electrons reduce the current flow as they pass through the detector by "capturing" a fraction of the electrons present. This reduction in current represents the detector signal which is amplified by the electrometer. The number of electrons captured depends primarily on the degree of attraction the compound has for the electrons; but also on the concentration of the compound in the carrier gas, the temperature of the detector, the carrier gas flow rate and the potential across the cell.

VIII. RESPONSE OF THE E. C. DETECTORS.

As previously mentioned, the E.C. detector has a very broad range of response to various types of compounds. Compounds containing halogens, particularly iodine, are extremely sensitive as are those containing heavy metals such as lead. Also compounds having carbonyl groups or those having conjugated systems of unsaturation respond well. On the other hand, almost all hydrocarbons possess essentially no electron affinity and therefore, produce little signal even when present in gross amounts.

IX. CLEANING THE DETECTOR.

Loss in sensitivity will result if high boiling compounds coat the tritium foil. Running the instrument over-night at 200+°C with carrier gas flowing through the unit will usually clear most contaminants from the cell. Chemically cleaning the detector is accomplished first by cooling the block to room temperature, removing from the instrument and standing the block with the exit tube facing downwards into a 250 ml beaker. A funnel is connected to the inlet tube and about 50 mls of methanol is poured through the cell. After draining is complete, the cell is dried with a clean dry gas and replaced on the unit. The methanol used to clean the cell is poured down the drain and flushed with a copious

amount of water as it will contain approximately 5×10^{-3} microcuries of tritium.

X. TROUBLESHOOTING.

A. Noise Originating in the Cell. Dirt on the teflon feed through the cell exit. This is removed by wiping with a lint free cloth of tissue mositened with a solvent such as n-hexane. It is suggested this be done at room temperature due to the flamability of hexane.

B. Condensation of Solvent in the Teflon Exhaust Tube. Remove this tube and wash with a volatile solvent such as n-hexane.

C. Noise Originating in the Electrometer. This should be corrected by following instructions in the instrument manual for the control console.

D. Overloading the Detector. It is possible to overload the cell with a large sample having a high electron affinity and this may prevent normal operation of the cell for up to several days. Only dilute solutions of these substances, (eg. highly halogenated compounds) should be analyzed. A range of 1×10^{-5} gms. to 1×10^{-7} is considered normal.

When a compound of unknown electron affinity is used, it is wiser to initially use more dilute solutions and then increase concentrations to the required working range. If the cell is advertently overloaded, the time required for stabilization can be reduced by increasing purge gas flow and raising the detector temperature.

Care of Syringes and Sampling Handling Techniques.

The field of pesticide residue analysis is one where housekeeping is of greatest importance. This is due partly to the very low levels of component being analyzed and the nature of the extraction solution in which the component is usually dissolved. The following suggestions may help in obtaining meaningful results from residue analysis.

1. Always use A.C.S. or better grade hexane when preparing extracts and standards taking care not to contaminate the stock hexane by returning unused hexane to the stock bottle.

2. As a stopper, do not use rubber or similar substances as solvents can dissolve electron capturing compounds from these. An aluminum foil capped screwed glass phial is preferred, as wax coated tops can cause trouble in this sphere.

3. Syringes, although prone to contamination, are still convenient to use and are preferred by many workers. The ground glass cyclinders readily absorb chlorinated pesticides and can cause confusing results without special precautions.

4. Ideally, a separate syringe should be kept for each concentration of each compound, particularly when frequent injections must be made of different compounds. This is not always practical due to cost and availability.

5. As an alternative to 4, the next best solution would be to use the same syringe for a series of different concentrations of the same compound, starting with the most dilute and working towards the most concentrated.

6. If the same syringe must be used for many different compounds, it is essential to insure complete absence of the last used sample before proceeding with further analysis.

7. The syringe should be washed by drawing up to 5 ml of solvent for the contaminant through the body of the syringe, washing the plunger carefully and drying each completely if the solvent is foreign to the system to be used.

8. Before injection of the new sample, a blank should always be run, using the solvent used in the new system. Comparisons of the chromatogram so obtained with one from a newer syringe will indicate if further cleaning is required. Repeat step 7 until results indicate no contamination.

9. Step 7 and 8 should be performed when using a syringe for 2 different concentrations of the same compound if the new sample is more dilute than the old.

10. If strongly electron capturing substances must be used to clean syringes completely, all trace of it must be removed by following steps 7 and 8 before using.

Radioactive Detector Controller.

This unit is required when an E.C. or M.C.S. detector is used with a 609 or 1609 system and contains all equipment necessary for operation of the detector.

Preliminary Adjustment of the Unit.

Before operation, it is essential to adjust the unit to exactly match the electrometers with which it will be used. This is done by following the following steps:

1. Remove top cover from unit by removing holding screws and and sliding cover upwards and off unit.

2. Obtain an 11 meg ohm impedence vacuum tube voltmeter and adjust zero, set range at 0-150 + volts D.C., (a 20,000 ohms/volt multester is not suitable for this adjustment).

3. Adjust the electrometer to sensitivity of 100 X Infinity, pulser off, connect the V.T.V.M. probe to the electrometer inner connector and the other lead to the test point on mid-left chassis (see diagram).

4. Adjust potentiometer (left chassis) until voltage indicated by V.T.V.M. approaches zero, switch scales and readjust potentiometer until voltage indicates 0.2--0.25v.

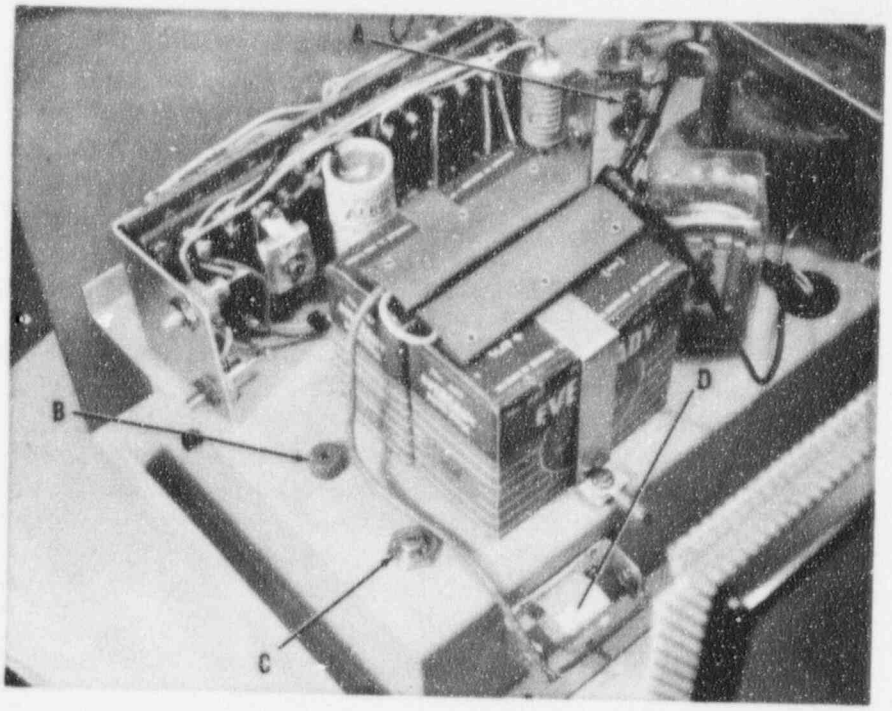
5. It is important that the correct polarity is observed (cable positive with respect to test point) and although any voltage between 0.05 and 0.25 is acceptable, the lower value will require more frequent adjustment of the unit.

6. In some instances, it will not be possible to obtain the correct polarity by the above method. In such case, an Everready #504 battery, 15v is required and is installed in the small battery clip on the front of the inner chassis (observe polarity) only after removing the jumper wire connecting the two terminals. The adjustment can now be made as detailed.

7. It is necessary to check this potential approximately every 3-4 weeks to insure polarity has not reversed and also as a check on the batteries. Life of batteries should be almost shelf life due to the low drain of 1.3 microamps.

8. When purchasing new batteries, it is desirable to insure that they are in good condition as if they have been stored for long periods (particularly in hot weather), their performance could be poor and require more frequent re-adjustment. This unit can now be attached to the detector by the 8 pin plug and cable provided and the line cord powered. After switching on the master power switch, the detector can be heated to the desired temperature. Pulse controls and temperature protection circuit, both in this unit, are discussed previously.

   It is important when changing from flame detection to E.C. detection that the recorder leads are also reversed. This is required because signals are in the reverse direction with the two different detectors and although it is possible to run the E.C. with a base line at "90%" of the recorder scale, attenuation of the peaks is somewhat untidy.



A POTENTIOMETER TO ADJUST VPR (DETECTOR HEATER OUTPUT). PAGE 3.

B TEST POINT TO CONNECT TO VTVM. PAGE 6.

C POTENTIOMETER TO ADJUST TEST POINT POTENTIAL. PAGE 6.

D BATTERY HOLDER. PAGE 6. INSERT BATTERY WITH + TERMINAL ON THE RIGHT FACING THE FRONT PANEL.

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