SEALED SOURCE AND DEVICE FILES

Received 3/2/64 W/Letter Dated 2/28/64
Revised by
Superseded by
Reviewed by Date 3-3-64

INSTRUCTION MANUAL FOR
MICRO CROSS SECTION DETECTOR

February 26, 1964

8 pp.

I. INTRODUCTION

The micro cross section detector is useful where wide dynamic range with linear response is required. It will detect all components except the carrier gas in the system, and due to its small internal volume (78 ul) its response time is low. It is, unfortunately, not a very sensitive detector and is equivalent to a good thermal conductivity cell.

Before the introduction of closely spaced electrodes in MCS detectors, it was usual to use high energy isotopes, but with the detector described, the use of low energy (and hence safer) tritium is possible, with excellent results.

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 conditions, 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 200°C, foil life should approach 1 or 2 years with careful use.

*Reg. trademark, E. I. du Pont de Nemours & Co., Inc.

C. Tritium Loss from the Cell. The activity of the foil will gradually decrease at the rate of 40 microcuries 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 preferred carrier gas is pure hydrogen, but if this is undesirable due to safety reasons, then water saturated helium can be used -- but only with some loss in sensitivity compared to hydrogen.

V. COLUMN.

Columns to be used with MCS detectors need no special treatment other than flow conditioning for approximately one hour 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 articularly for SE-30 and similar columns as it frequently increases column efficiency.

VI. OPERATION OF THE DETECTOR.

The MCS is compatible with 400, 1400, 810 and 700 series instruments. In all cases it has a temperature control and a manual reset button with pilot light to prevent hazardous high temperatures from being reached. Voltage for operation is supplied by batteries in the electrometer (units 700, 400 and 810) or a line operated power supply (609, 1609).

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.

Control Location.

With units in the 400 and 810 series, all MCS controls are located in the main control cabinet while the 700 houses the overheat protection circuit at the left rear quarter of the oven shroud. Models 1400 require a separate "radioactive detector controller" which is fully dealt with later in the manual.

When using MCS on dual purpose units, couple the electrometer cable to the detector before attempting to operate.

VII. PRINCIPLE OF OPERATION.

The passage of radiation through the gas in the detector chamber causes the production of ion pairs which are then collected at the electrodes. The potential applied to the cell (1300 volts/cm) is sufficient to collect the ions produced without any significant loss by recombination. This ion collection causes an increase in current to flow across the cell which current is fed to an electrometer for amplification in the normal manner. Several papers are referenced which discuss the theoretical aspect more fully for those wishing to pursue this subject.

VIII. RESPONSE OF THE MCS DETECTOR.

A unique feature of this cell is its ability to detect all samples in concentrations of up to 90+% in the carrier gas with perfectly linear response. It is not temperature or flow sensitive for all practical purposes, and so lends itself to programmed temperature operation.

The response to a known concentration of any given substance can be calculated from the cross sections for consideration of the constituent atoms of the molecule with good accuracy. The validity of these calculations are discussed at length by Deal (6), Boer (5) and Otvos & Stevenson (4). Briefly, these latter authors state that the total molecular cross section for ionization by a given radiation is the simple addition of the constituent atomic cross sections of the molecule for the same radiation, and is almost completely independent of the nature of the chemical combination.

IX. CLEANING THE DETECTOR.

Loss in sensitivity will result if high boiling compounds coat the tritium foil. Running the instrument overnight 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 or tissue moistened 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.

Radioactive Detector Controller.

This unit is required when an EC or MCS detector is used with a 609 or 1609 system and contains all equipment necessary for operation of the detector. The controller contains all equipment to operate both EC and MCS detectors and the pulser and its control system will not be used for MCS operation. This will save time and trouble if, at a later date, an EC detector is purchased for operation from this controller.

It contains a regulated supply for the detector heating system which is controlled by the front panel potentiometer. The safety circuit and manual reset button have already been discussed and are located in this detector controller.

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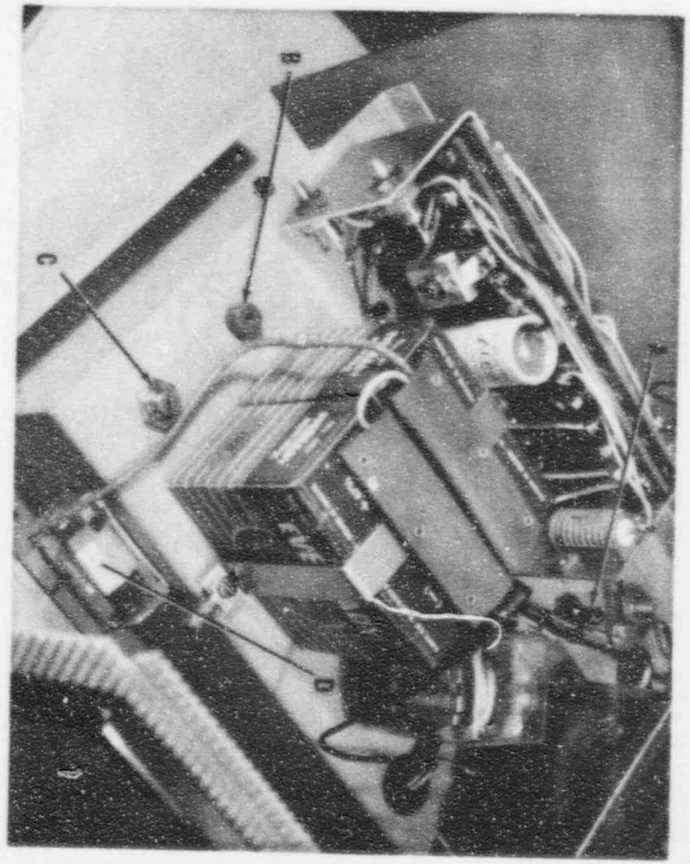

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A. POTENTIOMETER TO ADJUST VPR (DETECTOR HEATER OUTPUT) PAGE 3

B. Not applicable for MCS Detector

C. Not applicable for MCS Detector

D. Not applicable for MCS Detector