Nicolet

May 8, 1987

U. S. Nuclear Regulatory Commission
Region III
Material Licensing Section
799 Roosevelt Road
Glen Ellyn, IL 60137

Dear Sirs:

In our letter of May 5th, which was an application for general and specitic licenses to distribute and possess Nickel 63 as part of Model 9630 Gas Chromatographs, we failed to enclose the document referenced as Attachment F. The ECD section of the GC manual is hereby submitted as an enclosure to this letter.

We regret any delay or confusion this omission has caused.

Sincerely,

Dale J. Thanig
Quality Assurance Manager

DJT:sts

Attachment F - Section from Nicolet ECD Manual

Nicolet Analytical Instruments

5225 Verona Road
P.O. Box 4508
Madison, WI 53711-0508
Telephone 608 271-3333

ATTACHMENT F

ELECTRON CAPTURE DETECTOR

Installation and Operation

April 1987

Address Comments To:

Technical Publications Department
NICOLET ANALYTICAL INSTRUMENTS
5225-1 Verona Road
P.O. Box 4508
Madison, WI 53711-0508

INTRODUCTION

This manual describes how to install and operate the Nicolet Model N4756846 Electron Capture Detector (ECD). It includes radiation safety requirements for installing, operating, maintaining and, if necessary, shipping the detector.

<<<< WARNING >>>>

The Nicolet Electron Capture Detector is designed to be issued to general licensees as defined by U.S. Government regulations and therefore cannot be dismantled without a special tool. The Nuclear Regulatory Commission (NRC) prohibits anyone without a license from dismantling the detector, breaking the seal, cleaning it internally by flushing with a solvent, or heating the detector to temperatures above 400°C.

<<<< WARNING >>>>

Avoid touching the ECD with your bare hands. However, if this is unavoidable, wash your hands immediately after handling the device. Although radiation from nickel 63 does not readily penetrate the skin, you should take every precaution to prevent ingestion of radioactive material.

<<<< WARNING >>>>

Perform periodic leak tests to assess radioactive contamination of the ECD exterior.

GENERAL DESCRIPTION AND THEORY

The ECD is highly sensitive to compounds that have an affinity for electrons. Non-electron capturing substances produce a low response. The ECD is particularly sensitive to compounds containing halogen or oxygen atoms and to organic compounds with conjugated double bonds.

The detector consists of a chamber containing a 10 mC Ni^{63} radioactive source and a pair of electrodes. A polarizing voltage is applied across the electrodes. Nitrogen is used as the carrier gas.

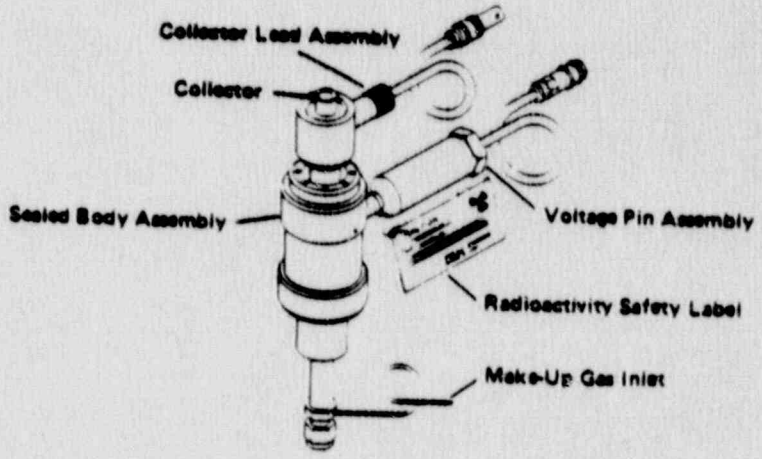
The following equation describes the ionization process that occurs in the chamber:

$$N + \beta \rightarrow N + e$$

This ionization produces a current flow between the two electrodes. If you introduce to the chamber a substance that has a high electron affinity, electrons are captured and the current flow is reduced, thus enabling the substance to be detected.

To obtain optimum linearity and sensitivity, keep the current flow constant by varying the frequency with which the pulsed polarizing voltage is applied to the electrodes. In some applications, however, you will get better results by applying the polarizing voltage at a constant frequency .

The ECD can be operated in either the constant current or constant pulse voltage frequency modes. When nitrogen is used as the carrier gas, the detection limit is 1×10^{-14} g lindane, and the linear range is 10^4.



Electron Capture Detector

INSTALLATION

The ECD will be shipped in a special container marked:

"CAUTION - RADIOACTIVE MATERIAL"

This container may be inside a larger box that does not have the caution label. An installation tool and two mounting screws will be shipped with the detector.

A Nicolet service representative will install the ECD on a new instrument. If you purchase an ECD as an accessory to an installed GC, you may install the detector yourself. (As a general licensee, you are permitted to install or remove the ECD as necessary.)

When the ECD is installed on a gas chromatograph (GC), a label indicating the type and amount of radioactive isotope present in the detector must be placed on the front the the GC.

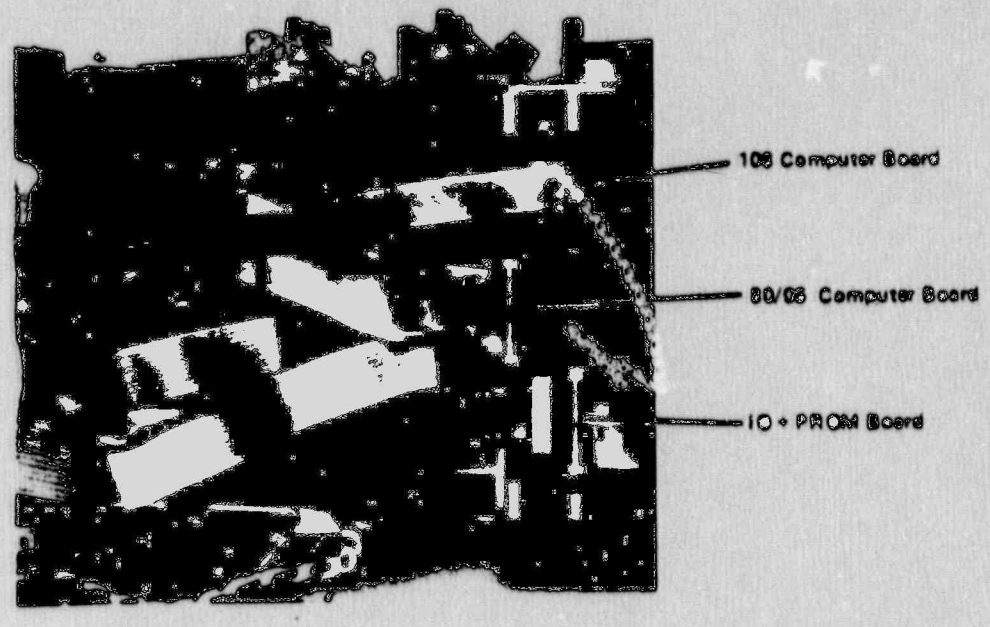
Installing the ECD

<<<< WARNING >>>>

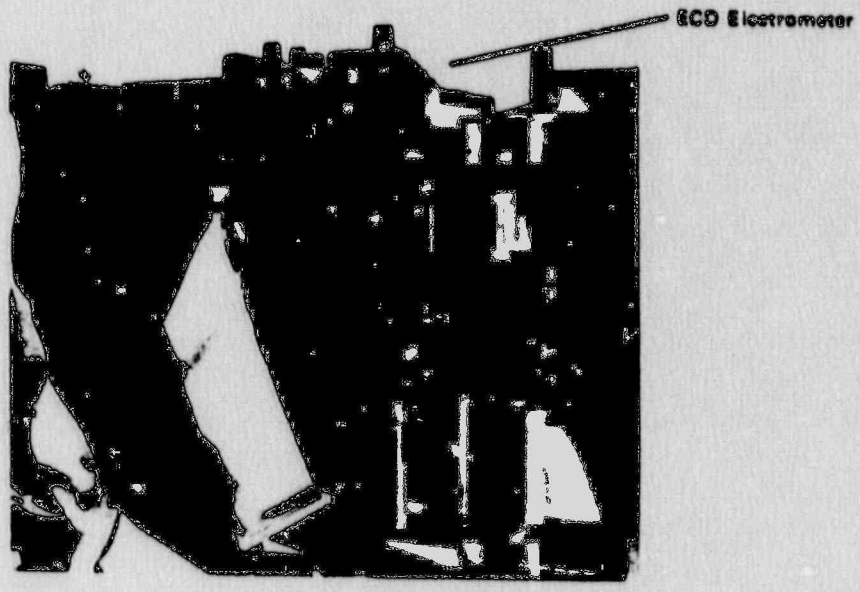
Switch off the electrical power to the instrument and unplug the unit before removing any electrical panel covers.

The ECD is used in conjunction with the ECD electrometer and the range amplifier. To fit these, it is necessary to remove the retaining screw on the hinged electronics module and to swing this gate open to allow access to the electronics unit and the power supply unit. Remove the perforated covers from both of these sections.

To install the ECD electrometer and the range amplifier it is necessary to disconnect the ribbon cables linking the temperature ADC board to the middle connector of the 80/05 Computer board and the VDU connection from the rear of the electronics unit to the top connector on the 108 computer board. Reconnect these ribbon cables after the electrometer is installed.



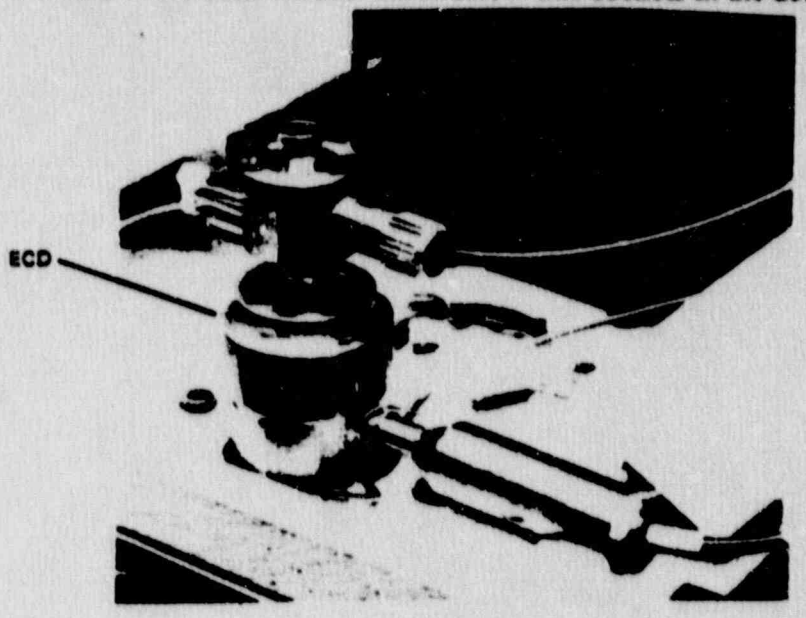
Install the ECD electrometer in either of the two slots labeled DET A or DET B on the mother board. Push the electrometer firmly into position to ensure a good electrical contact. Normally, if it is to be used with a detector mounted in the front position, the electrometer is slid into the front (DET A) slot.



Slide the range amplifier into the slot adjacent to the detector electrometer. Push it firmly into position.

Replace the ribbon connectors and the perforated covers.

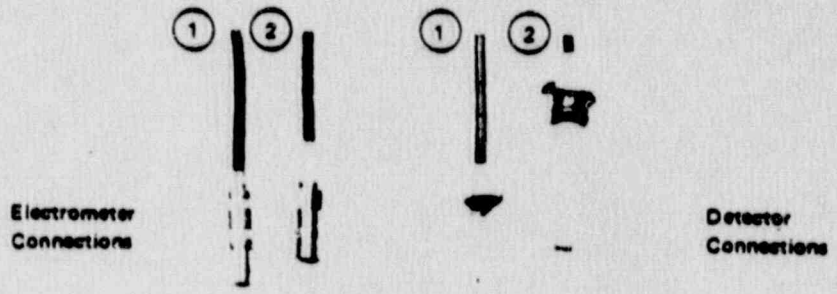
The detector is installed in the standard ionization detector oven. Place the detector in the recess in the detector oven block from above. Orient the detector so that the make-up gas inlet pipe fits into the groove cut into the recess. The detector is held in position by two screws which are located in lugs in the base of the detector and which screw into sockets in the detector oven.



Connect the make-up gas inlet pipe to a molecular sieve trap carrying a supply of nitrogen from the pneumatics compartment.

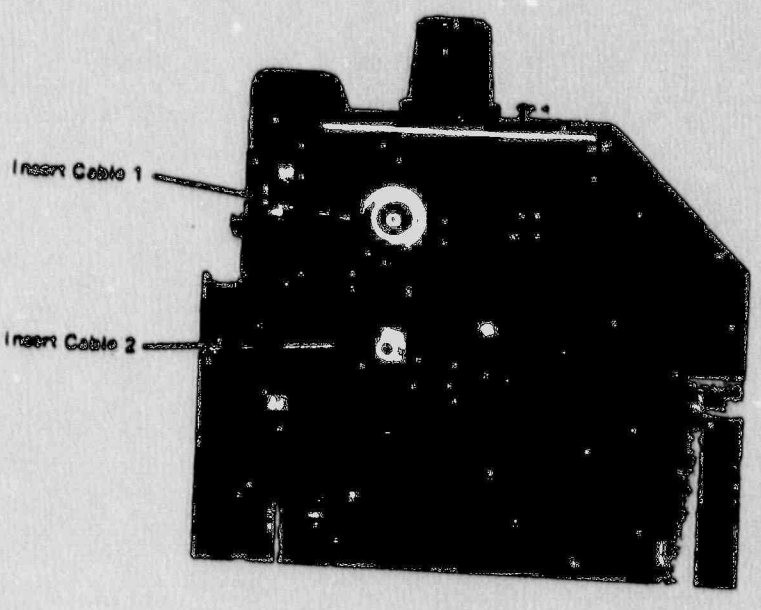
Two electrical connections must be made between the ECD and its electrometer:

1. Collector electrode connection to electrometer input.
2. Polarizing voltage connection to ECD jet



Locate the lead with a coaxial plug at one end and a spring-loaded pin at the other end. Use this lead to make the collector electrode connection. Screw the end of the lead with the spring-loaded pin into the top side arm on the detector. Plug the other end of the lead into the top socket on the ECD electrometer.

Locate the lead with the gold-colored coaxial jack. Use this lead to make the polarizing voltage connection. Plug the gold-colored coaxial jack into the lower socket on the electrometer. Screw the other end of the lead into the lower of the two detector side arms.



STORING THE ECD

When the ECD is not installed on a gas chromatograph, place the detector in the original or an equivalent container along with the installation tool and mounting screws. Stored the container in a locked cabinet in a designated area that bears the label "CAUTION - RADIOACTIVE MATERIAL."

VENTING EXHAUST GASES FROM THE ECD

<<<< WARNING >>>>

Since the ECD effluent may contain radioactive material, it must be vented to a fume hood. In the U.S.A., detector venting must conform to title 10 of the code of Federal Regulations (CFR) part 20 or with the regulations of a state with which the National Regulatory Commission (NRC) has entered into agreement. Outside the U.S.A., the appropriate agency should be contacted to determine the equivalent regulations.

Vent the exhaust gases from the ECD to a fume hood or other suitable exhaust system. This prevents minute radioactive particles from being carried by the eluting gas and spread randomly outside the cell body. It also prevents toxic fumes from accumulating in the immediate vicinity of the ECD.

To vent the exhaust gases, attach one end of a length of 1/4 inch plastic or metal tubing to the exit tube of the ECD. Route the other end of the tubing into an exhaust system.

OPERATION

ECD Electrometer

The ECD electrometer provides signal attenuation but no range amplification (similar to the TCD). The output attenuation is selected from the keyboard with settings from 1 to 2048 in binary numbers.

The zero control on the electrometer enables the detector standing current signal to be adjusted so that the recorder/data system can be zeroed. Only one zero control knob is required.

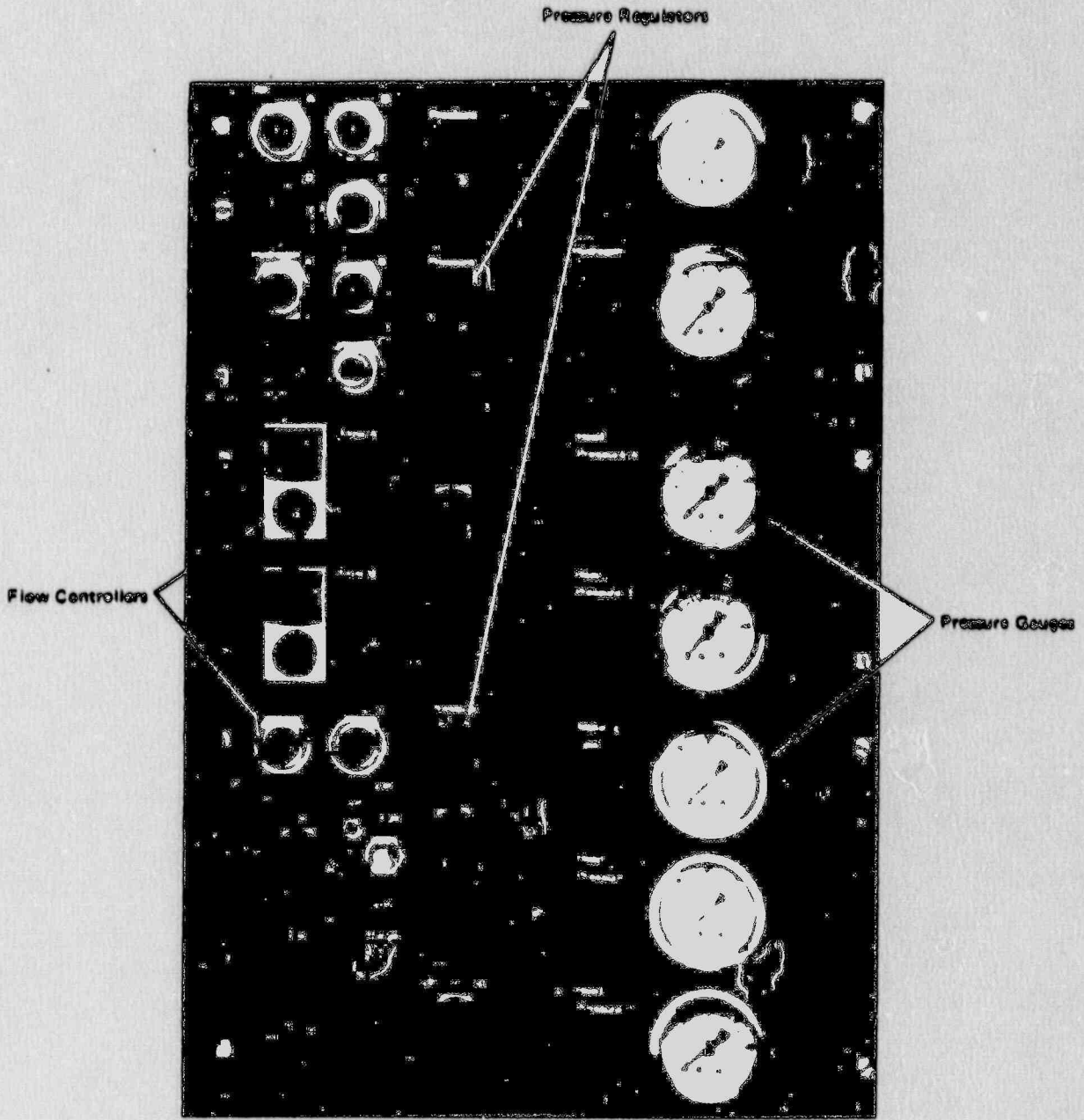
Pneumatics

<<<< WARNING >>>>

Since the ECD effluent may contain radioactive material, it must be vented to a fume hood. In the U.S.A., detector venting must conform to title 10 of the code of Federal Regulations (CFR) part 20 or with the regulations of a state with which the National Regulatory Commission (NRC) has entered into agreement. Outside the U.S.A., the appropriate agency should be contacted to determine the equivalent regulations.

The ECD requires carrier gas and make-up gas at a total flow rate of 30-40 ml/min. That is, if the column flow is 20 ml/min., additional flow of 10-20 ml/min. is required through the make-up gas inlet. Nitrogen is the required carrier gas and is active in the operation of the detector. An ultrapure grade of nitrogen (containing no oxygen) is required. Argon with 5% methane can also be used as the carrier gas.

The applicable controls in the pneumatics module are highlighted here for operation of the ECD in the Detector A position.



General Operation

<<<< WARNING >>>>

Maximum recommended ECD operating temperature is 350°C.

Install a clean column in the instrument and set the carrier gas flow rate as appropriate for the column being used. By means of the purge gas inlet on the detector, adjust the total flow of gas through the detector to 40 ml/min.

Turn on the instrument, enter the program index via the keyboard and insert a new program. The instrument begins by requesting the program title, then it asks which detector is required. The instrument senses that an ECD is installed and asks if it is required to operate in the constant current (C) or constant Frequency (F) mode:

ECD mode (C or F)

During setting up and in the majority of analyses, the answer to this question will be "C", i.e., constant current. The default value for this prompt is "C".

In the constant current mode, the pulse frequency of the detector is varied to ensure that the current flowing through the detector is constant under all conditions. This results in a wider linear range of operation than would otherwise be possible. However, in some cases optimum sensitivity is obtained when the detector is operated in the constant frequency mode.

The attenuations available are 1 to 2048, in powers of two, as entered from the keyboard.

The ECD is the most sensitive detector in common use, and as a consequence, more care and patience are required in order to obtain high quality results. Use the following rules as a guide:

1. The ECD is susceptible to small changes in temperature or flow rate and so requires a longer time for the system to stabilize than is normal for other ionization detection systems. It is common practice when using an ECD routinely to leave the instrument running continuously in order to avoid a time-consuming stabilization period at the start of each series of analyses.

2. Generally the ECD is unsuitable for temperature programmed operation, although temperature programming can be performed using capillary columns.

3. Always use dilute samples of strongly electron capturing compounds (below 10^{-9} g of each component). Never use halogenated solvents to dilute samples for ECD work.

4. Whenever possible, keep the temperature of the detector at least 50°C above the column oven temperature but less than 350°C.

5. Always maintain a flow rate through the detector of 30-40 ml/min. to ensure that the detector dead volume is adequately swept.

6. As the ECD is susceptible to contamination, it is wise to reserve syringes and columns exclusively for use with this detector.

CLEANING AND ROUTINE MAINTENANCE

Electron capture detectors require periodic cleaning. The frequency of cleaning depends on the conditions of use. A build up of the contamination will tend to reduce the linear range of the detector, giving rise to a noisy chromatogram and "double peaking." To check the state of contamination of the detector, a self-checking routine is provided. The software continually checks the standing current available and, if this is found to be inadequate, a message "ECD ERROR" is displayed on the VDU.

Oxygen is an electron capturing substance. When a new system is initially used, it takes some time for all the air in the system to be removed. For this reason, it is not unusual for the "ECD ERROR" message to appear when a new system is first used. Column bleed can also result in the detector being unusable because of an inadequate standing current.

In order to measure the standing current of the detector and thereby determine the extent to which the system is contaminated, the following procedure may be used:

1. Ensure that the ECD is operating in the constant frequency mode.
2. Using the zero control, set the baseline to 50% full scale deflection on the recorder chart.
3. Disconnect the signal lead from the electrometer.
4. The recorder pen should now move downscale. The difference between the old and new baseline positions should be noted as a percentage of the full scale deflection.

Full scale deflection on attenuation 1 represents 10^{-10} A (in the constant frequency mode).

The standing current may be calculated as follows:

Standing current = % change in pen deflection x attenuation x 10^{-10} A

Removing contamination

Most contamination can be removed by purging the detector system. This is carried out by installing an empty glass column in the instrument and passing a flow of clean carrier gas through the detector. The detector should be heated to 350°C and left to purge overnight, or preferably for several days. If a pen recorder is connected and left to run on a slow chart speed, the progress of the purging process can be monitored; a falling base line indicates an improving situation.

Cleaning the Collector Electrode

The collector electrode may be cleaned by unscrewing the collector assembly from the body of the detector and cleaning the surface of the collector gently with very fine grade emery paper. The detector should not be further dismantled.

<<<< WARNING >>>>

Only trained personnel who are licensed by the NRC, agreement state, or other appropriate regulatory agency to handle radioactive material are authorized to disassemble and/or clean an ECD.

<<<< WARNING >>>>

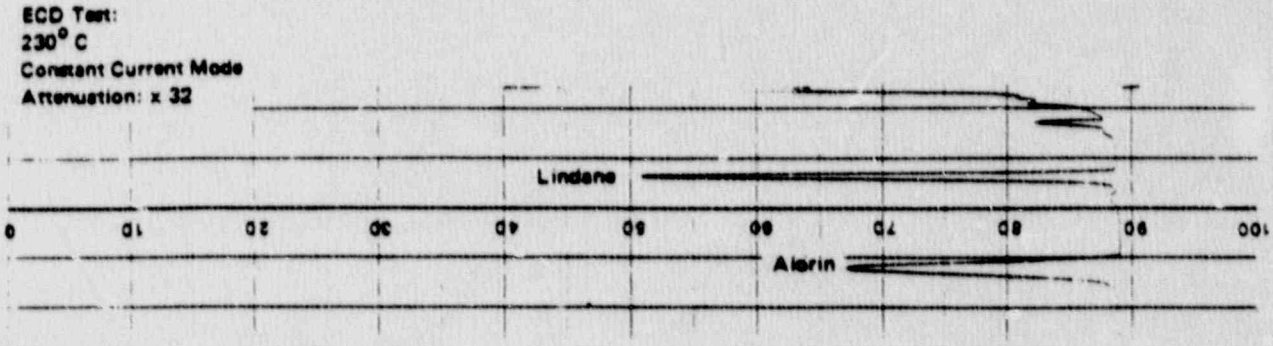
Wipe tests must be performed periodically to assess radioactive contamination of the detector exterior.

Should purging fail to clean a contaminated detector, return the detector to Nicolet for cleaning .

TROUBLESHOOTING

When chromatographic performance is questionable, a standard evaluation procedure is a best approach to identifying the problem. The ECD can be tested using the installation test procedure and standard test mixture.

Install the 6 ft x 2 mm ID column packed with 3% SE-30 on Chrom W (80/100 mesh). Set the nitrogen carrier gas at 30 ml/min. and the make-up gas at 1-5 ml/min. Ultrapure, oxygen-free gas must be used. Set the column temperature to 200°C, the injector temperature to 200°C and the detector temperature to 200°C. Inject 2 microliters of ECD test standard containing 33 pg/microliter lindane and 33 pg/microliter aldrin in iso-octane. The detector should be operated in the constant current mode with an attenuation of 256. Examine peak shape and response.



Noise

Measure the noise in %fsd over a 10 minute interval. If the noise exceeds 2%fsd at maximum sensitivity, check electrical cables and connections and check for leaks or sources of oxygen in the gases. If the detector needs cleaning, raise the detector temperature to 400°C with gas flowing and bake the detector overnight to remove any contamination. Let the detector restabilize at 200°C and remeasure the noise.

RADIATION SAFETY

What to do If the ECD is Damaged

If you receive a damaged ECD or if the detector is damaged during use, stop using the device immediately and notify the Radiation Safety Officer at Nicolet Analytical Instruments, 5225-1 Verona Road, Madison, WI 53711. Telephone: (608) 271-3333.

Leak test the damaged detector as soon as possible (refer to the section titled "Radioactive Contamination Leak Test" for instructions). Then remove the ECD from the instrument, if installed, being careful not to touch the detector with your bare hands. Place the ECD in a clean, plastic bag along with the installation tool, mounting screws, and any other item that may have touched the detector during this operation. Place the bag and its contents along with the leak test kit and Shipper's Certificate in the original container and return it to Nicolet. (Refer to the section titled "Shipping the ECD" for shipping requirements.)

Radioactive Contamination Leak Test

U.S. government regulations require that ECD's containing Nickel-63 as an ionization source must be given a leak test every six months to insure that the total removable radioactivity on the exterior surfaces of the detector and housing is within the maximum permissible level of 0.005 microCurie. It is the responsibility of the user or general licensee to ensure that the wipe tests are satisfactorily completed and to keep appropriate records of the results of such tests.

Each Nicolet ECD is shipped with a Leak Test Kit for Nickel 63 supplied by National Leak Test Center, P.O. Box 486, North Tonawanda, NY 14120. Phone: 716-693-0550. Each shipment also includes a Leak Test Certificate indicating the date and result of the most recent leak test. The first leak test to be performed by the user will be due six months from the date on the certificate. Follow the directions provided in the kit. Send the leak test sample to National Leak Test Center or any other registered laboratory for analysis.

Radioactive Foil Replacement

The sensitivity of the ECD gradually diminishes with time. This loss of sensitivity is due to contamination or oxidation of the radioactive source. It usually means that the foil may have to be cleaned or replaced.

If you notice a consistent sensitivity loss, contact Nicolet service personnel. If necessary, ship the detector to Nicolet Analytical Instruments, 5225-1 Verona Rd., Madison, WI 53711 for service. Refer to the section titled "Shipping the ECD" for details on how to package and ship the detector. **DO NOT** attempt to repair the foil yourself.

Shipping the ECD

When shipping radioactive material, you must comply with Department of Transportation regulations as stated in paragraph 173.391 of Title 49 of the Code of Federal Regulations and Part 2, Section 4.2 of the International Air Transport Regulations. According to these regulations, Nickel 63 ECDs, such as the one offered by Nicolet, may be shipped without the label "RADIOACTIVE" on the outer container if all of the following conditions are met:

1. The materials are packaged in strong, tight packages such that there will be no leakage of radioactive materials under conditions normally incident to transportation.

2. The package must be such that the radiation dose rate at any point of the external surface of the package does not exceed 0.5 millirem per hour.

3. There must be no significant removable radioactive surface contamination on the exterior of the package (less than 2,200 disintegrations/min per 100 cm^2).

4. The outside of the inner container must bear the marking "Radioactive."

The outer container must be large enough to allow at least a 4 inch space around the inner package. Shock absorbing material must be placed between the inner and outer containers so that the inner box doesn't shift during transport. The outer container must be made of new, rigid packing material such as heavy corrugated cardboard.

Shipments of Nickel 63 ECDs to Nicolet must be accompanied by a Shipper's Certificate (shown on the following page). Fill out the Shippers Certificate completely and place it in an envelope attached to the outer container.

SHIPPER'S CERTIFICATE

CONTENTS: ONE "RADIOACTIVE DEVICE n.o.s."

Isotope - Nickel 63 - Transportation Group IV - Solid Form

Amount - 10 milliCuries

NO RADIOACTIVE MATERIAL LABEL REQUIRED ON OUTER CONTAINER

The contents of this package are hereby certified to be properly classified, packaged, marked and labelled and are in suitable condition for shipment according to regulations of the Department of Transportation and IATA Restricted Articles Regulations. Furthermore, this shipment is approved for passenger-carrying aircraft.

NAME AND ADDRESS OF SHIPPER

_____

_____

_____

SHIPPING DATE: _____

BY: _____

TITLE: _____

PHONE: _____

REASON FOR RETURN:(if applicable)

_____ Chemical Cleaning

_____ Refoil

_____ Detector Repair

_____ Decontamination

_____ Disposal

SEND TO:
Nicolet Analytical Inst.
5225 Verona Rd.
Madison, WI 53711
Attn: Radiation Safety Officer

COMMENTS:

_____

_____

Note: Attach this form in an envelope to the outside of the outer container and supply 2 copies to shipper.