

D'APPOLONIA

CONSULTING ENGINEERS, INC.

November 30, 1982

HS-12

P2

John E. Glenn, Ph.D., Chief
Materials Program Section No. 2
Division of Engineering and Technical Programs
U. S. Nuclear Regulatory Commission
631 Park Avenue
King of Prussia, Pennsylvania 19406

Material By-Product License Application
Submittal of Additional Information
Re: Control No. 12435

Dear Dr. Glenn:

In response to your letter of November 8, 1982, D'Appolonia is pleased to provide the required additional information necessary for your office to complete processing of our Material By-Products License application.

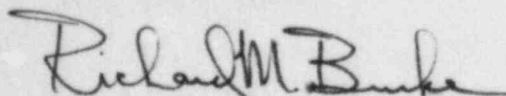
We have enclosed the following additional information:

- Table 1 lists the names of manufacturers, model numbers of detectors, cells and names of manufacturers for each chromatograph equipped with election capture detectors.
- A separate enclosure provides information regarding specific manufacturers recommended procedures for cleaning detector cells. These procedures are strictly followed in our laboratory. Cells would be returned to the manufacturer for changing detector foils as per the manufacturer's shipping instructions.

Please note that the Hewlett-Packard sealed source detector (Part No. 19303) was incorrectly identified in our original application as Part No. 19313. However, as per your request, we are enclosing the technical specifications for the detector.

We look forward to your review of this additional information. Should you have any further questions, please contact me.

Very truly yours,


Richard M. Burke, Director
Environmental Laboratory

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O F F I C E S I N M A J O R C I T I E S

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NUCLEAR REGULATORY COMMISSION
LICENSE RENEWAL APPLICATION
SUPPLEMENTAL INFORMATION

Gas Chromatograph:

Manufacturer	Perkin-Elmer	Hewlett-Packard
Model No.	Sigma 1	5880

Detector Source:

Element	Ni-63	Ni-63
Physical Form	Metal Foil	Sealed Coated Cell
Manufacturer	New England Nuclear	Hewlett-Packard
Model No.	NER-002	19303 ⁽¹⁾

Source Housing Device:

Manufacturer	Perkin-Elmer	Hewlett-Packard
Model No.	009-0282	1930-60550

⁽¹⁾ Technical Specifications regarding this source are enclosed as part of this submittal. Note that this detector cell was previously identified a Hewlett-Packard part 19313 in our original application.

- 8) Pull off the electrical connections for the ignition plug on the detector body, and partially unscrew the plug. Invert the body and the glass heat shield will slide out from the top of the body. Clean the glass with chromic acid. Replacement heat shields (Part No. 045-1417) are available if required.
- 9) Reassemble the detector following the above procedure in the reverse order.

E) ECD Cleaning and Maintenance Procedure

1) Bake-Out Procedure

It is often possible to decontaminate the ECD by purging it with carrier gas using a flow rate of 50 to 100 cm³/min. and raising the temperature to 400°C. These conditions should then be maintained until the detector is clean. Periodically, plot a graph of standing current setting versus frequency, as described in the operating instructions (Section 6L). The plot should improve as cleaning progresses, but complete cleaning may require baking for several hours or even days, depending on the type and extent of contamination.

Important: Never attempt to flush the detector cell assembly with acidic solvents, as this will result in the removal of radioactive material from the cell.

2) Wipe Test

The United States Nuclear Regulatory Commission requires that the detector be wipe tested at least once every six months, and that a record of the results of these tests be maintained for NRC inspection. The purpose of the wipe test is to ensure that removable radioactive contamination on the external parts of the cell remains at a safe level.

Important: Until the results of the wipe test are known, assume that the cell is contaminated and handle it only with suitable protection. All equipment coming into contact with the cell should be considered contaminated and handled accordingly.

It is strongly recommended that the user become familiar with the NRC regulations covering the use of nickel-63, as well as any other national, state or local requirements.

Perform the wipe test as follows:

- 1) Switch the Analyzer off and allow the detector to cool.
- 2) Expose the detector by pulling the detector cover forward and downward. The cover may be detached completely if required.
- 3) Pull off the collector housing (Figure 10-5).

Warning: <u>DO NOT DISMANTLE THE CELL.</u>

- 4) Refer to the instructions included with the wipe test kit (Part No. 009-1667) supplied with the detector, and wipe the external surfaces of the cell, as shown in Figure 10-5. Once the wipe test paper has been moistened and any part of the cell has been wiped, do not re-moisten the paper. Also, do not allow any of the wipe test solution to enter the cell.
- 5) Put the paper in the container provided in the wipe test kit. Include a data sheet stating that the wipe test was performed on a Perkin-Elmer electron capture detector cell, Part No. 330-0119, and give the date of the test. Return the container to either:

Nuclear Radiation Dev. Corp.
2937 Alt. Blvd.
Grand Island, N.Y.

or

Nuclear Sources and Service
5711 Etheridge
Houston, Texas 77017

Note: The sensitivity of the wipe test is 0.0001 microcuries.

Request that a new wipe test kit be forwarded with the test results.

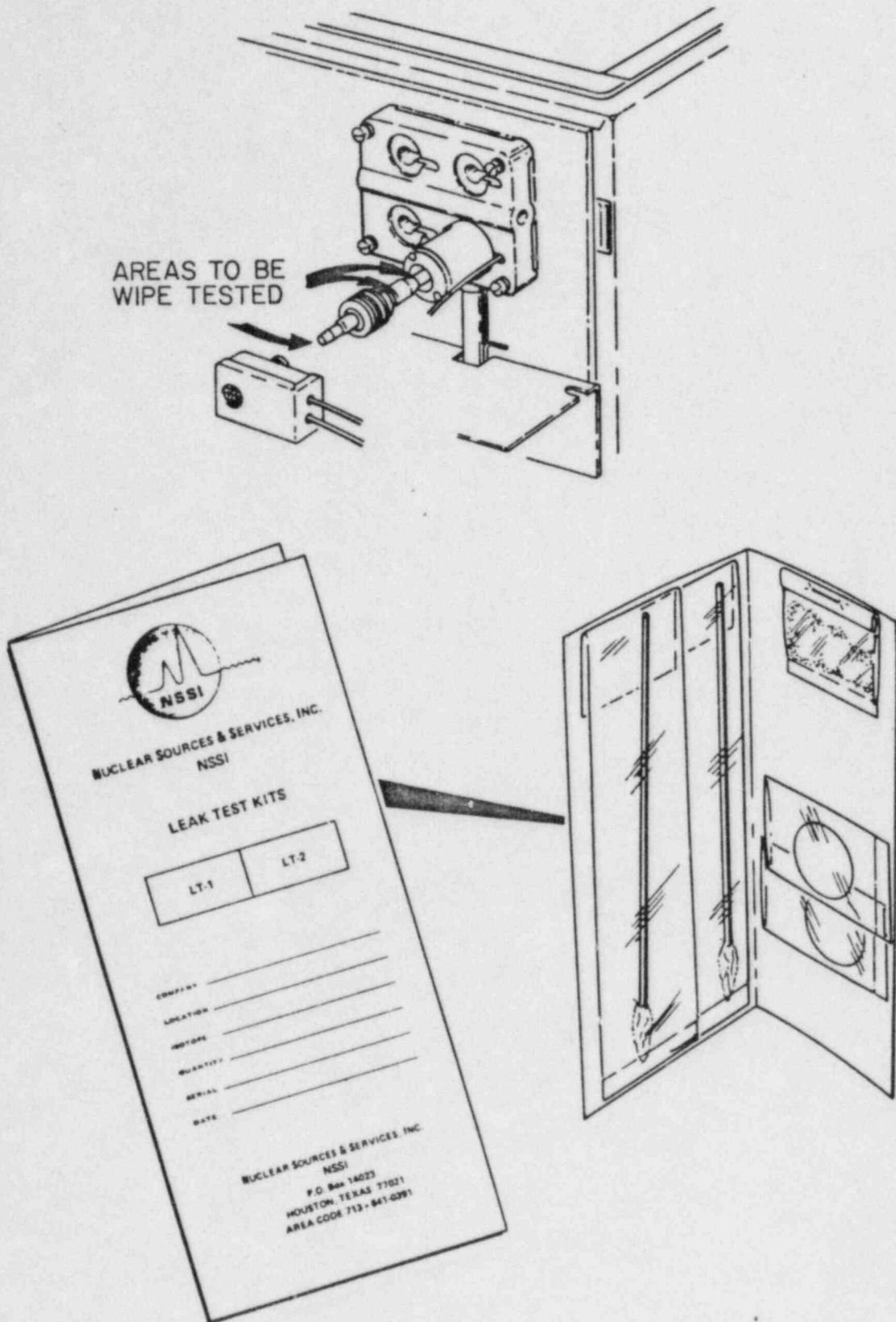
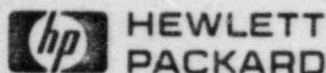


Figure 10-5. The ECD Wipe Test



INSTRUCTIONS FOR ACQUIRING, OR AMENDING A RADIOACTIVE BYPRODUCT MATERIALS LICENSE FOR HEWLETT-PACKARD DETECTORS WITH RADIOACTIVE SOURCES. Applicable to either Federal (Nuclear Regulatory Commission) or Agreement State Licensing Requirements.

These instructions are provided as a guide to filing your license application or amendment in the proper form. The model number of the detector assembly is particularly important, because the licensing agency has Hewlett-Packard's drawing on the file under this number. Refer to your application form, the accompanying instructions, and the following:

NOTE: These instructions are provided as guidelines based on present requirements. In as much as regulations are periodically revised, the latest revision of Title 10, CFR, the Federal Register, and NRC Form 313 and Regulatory Guide 10.7 should be consulted.

A. Important — your license status

1. If you are presently holding a license covering Hewlett-Packard detectors, your license must be current; and it must specifically apply to the type, amount, and form being ordered (see Paragraph B and C).
2. If you are presently holding a license not covering HP detectors, an amendment to your present license is required and an application must be completed. We suggest that your Radiation Safety Officer or appropriate personnel complete the form (see Paragraph B and C).
3. If you are not licensed, you must apply for a byproducts material license (see Paragraph B and C).

B. General Instructions for Licensing

For your convenience, we have outlined the following step-by-step procedure for completing the application form. The numbers refer to NRC 313.1 (1/79) and most other forms.

1-7. Self-explanatory

8. Licensed Material

a. For Hewlett-Packard 5700 Series of Gas Chromatographs	Ni63	Plated Part	Hewlett-Packard Electron Capture Detector #18713A	_____ dets., 15 mCi per detector
b. For Hewlett-Packard 5840 Series of Gas Chromatographs	Ni63	Plated Part	Hewlett-Packard Electron Capture Detector #18803-60520	_____ dets., 15 mCi per detector
c. For Hewlett-Packard 5880 Series of Gas Chromatographs	Ni63	Plated Part	Hewlett-Packard Electron Capture Detector #19303	_____ dets., 15 mCi per detector

NOTE: a, b, and c above. Some Agreement States may request the radioactive source number. If this is the case, use "15 millicuries Ni63 plated part HP No. 18713-80020 for 8a and b; use HP No. 19303-80010 for 8c."

d. Repaired/Rebuilt Detector for Obsolete Hewlett-Packard Gas Chromatographs.

(1) For Hewlett-Packard 5830 Series of Gas Chromatographs	Ni63	Plated Part	Hewlett-Packard Electron Capture Detector #18803-60520	dets., 15 mCi per detector
(2) For Hewlett-Packard Models 400, 402, 402B, 700, 810, 5750A/B/G, 76720A, or 7610				
use: (a) Ni63	(Nuclear Radiation Development Foil N-1002)	HP High Temp. EC Detector No. 2-6195		dets., 2 mCi per detector
or (b) H3	(Titanium Tritide Foil US Radium LAB 508-1 or LAB 508-3)	HP Electron Capture Det. No. 2-2837		dets., 200 mCi per detector
or (c) H3	(Titanium Tritide Foil US Radium LAB 508-1 or LAB 508-3)	HP Microcross- section Det. No. 2-2830		dets., 200 mCi per detector

NOTE: Rebuilt detectors will be refoiled with the U.S. Radium LAB 508-3 rather than the LAB 508-1, an obsolete number.

We suggest that you request permission to hold an amount sufficient to cover at least two detectors. This will give you the opportunity to order an additional detector without an amendment to the licenses.

9. Storage of Sealed Sources. You should include the Hewlett-Packard Gas Chromatograph in which the electron capture detector will be used. For example, Hewlett-Packard Series 5880 Gas Chromatograph.
10. a. This question generally is not applicable if the detector is not to be disassembled and/or cleaned.
b. If the detector is to be disassembled and/or cleaned by the user rather than Hewlett-Packard, read title 10 CFR part 20, the manufacturer's instructions, and consult with your radiation protection officer.
11. Self explanatory.
12. This question generally is not applicable.
13. You should include a statement that the detector effluent gas must be piped into a fume hood or otherwise vented in compliance with the latest revision of 10 CFR Part 20.
14. State the following: "Return detectors to supplier, Hewlett-Packard Company, Route 41, Avondale, Pennsylvania 19311." If cell disassembly (for detector numbers 18713A, 18803A, 19303, and 18803-60520 only) or washing is to be performed by the user rather than Hewlett-Packard, so indicate, and also state that the user shall read and adhere to the manufacturer's cleaning and waste disposal procedure and comply with 10 CFR Part 20.

NOTE: Customer disassembly of/or refoiling of detector number 2-6195, 2-2837, and 2-2830 is not permitted.
15. Read 10 CFR Part 20 so that you are aware of the requirements and outline an appropriate program for maintaining compliance with these requirements. For Nickel 63 detectors, also state: "Perform leak test with Hewlett-Packard No. 18713-60050 leak test kit at six month intervals."
- 16-18. Self explanatory.

C. License Verification

We require a signed license verification (Form 5950-8209, latest revision) in our files before we can ship you a new detector containing a radioactive source or before we can return a repaired detector to you.

We hope that the above comments will be of value to you in obtaining a Radioactive Materials License for your particular circumstances. Please contact us if we may be of assistance to you.

2. Turn off the carrier gas supply.
3. Disconnect the columns at the detector.
4. Disconnect the detector tubes at the detector solenoid valve. The detector solenoid valve is located behind the flow panel. To gain access to the tubes, remove the hinged flowpanel cover, remove the two Poxidriv® flow panel retaining screws, and swing open the flow panel. The solenoid valve will be noticed as the panel is opened.
5. Using a syringe (e.g., 5mm gas tight syringe, part no. 9301-0045) and lengths of tubing, force solvent through the detector. All internal passages are interconnected and solvent should be forced through them all. Remove the muffler wire and wipe it clean.

Use a suitable solvent to dissolve the samples that were analyzed. A general purpose solvent that is sometimes effective consists of equal parts of Acetone, Methanol, and Hexane.

6. When finished, reconnect the detector tubes to the solenoid as originally installed. (Front detector tube to solenoid front-center fitting; rear detector tube to solenoid short side port fitting.)
7. Replace the muffler wire.
8. Reconnect the columns.
9. Service the "S" Moisture Traps and associated Chemical Filters. (See FLOW SYSTEMS in this Section for details.)

NOTE

Cartridge replacement is described in Section D, TCD Corrective Maintenance of this manual.

EC DETECTOR MAINTENANCE

The radioactive Electron Capture Detector (ECD) has been designed to provide the degree of performance required for the 5880 Gas Chromatograph. However, as with any complex product, variations from its optimum performance can be expected when parts are contaminated or simply wear out. Routine cleaning and the replacement of items of an expendable nature are the responsibility of the user.

Wipe (Radioactive Leak) Test

In the United States, the Nuclear Regulatory Commission (or State agency in "Agreement States") requires Hewlett-Packard Ni63 Electron Capture Detectors to be tested for radioactive leakage and/or contamination at intervals not to exceed six (6) months. It is essential that this test be performed in order to assure regulatory compliance. Owners in other countries should check local and national regulations for equivalent requirements. The user's first leak test should be performed no later than six months from the date on the HP wipe test certificate which accompanies the detector. This test certificate and all subsequent leak test records must be retained as evidence of regulatory compliance.

Quick Frequency Test

Under existing operating conditions, at least 2 hours after the last sample was injected, key:

```
PLOT SIGNAL A (B, C or D) ENTER
LIST ZERO ENTER
STOP PLOT ENTER.
```

If the resulting number printed on the terminal exceeds 1000 indicating the background level of the signal, investigate the possibilities listed below:

- Bad gas
- Insufficient conditioning
- Contaminated detector
- Column bleed
- Septum bleed
- Moisture (from saturated traps)
- Bleed from injector deposits
- Leaks
- Anode insulation leakage

NOTE

Starting from a cold detector and column, a 24 hour wait may be required until the baseline is completely stabilized for high sensitivity operation. Condition a new septum in the unused injection port for several hours with 1-5 ml/min carrier flow before installing. Only low bleed septa should be used.

Carrier Gas Evaluation

This test should be performed each time the carrier is replaced.

With the instrument energized, gases properly connected, and oven at ambient temperature, remove the column.

With the oven door left open, key the detector temperature to any value over 100 °C. Example:

```
DET TEMP 1 (or 2) 100 ENTER
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While the detector is heating:

1. Using the second stage control, reduce the carrier gas regulator pressure to zero.
2. Disconnect the carrier gas line at the fitting on the back of the instrument and reconnect it to the detector inlet fitting using a Vespel® ferrule (and an adapter fitting, if needed). If possible, include the moisture trap in the line connecting the carrier supply to the detector.

CAUTION

A metal ferrule used at the detector fitting has a tendency to seize and may damage the fitting upon removal.

3. Set carrier gas pressure to 6.9 kPa (1 psi) and check that there is flow through the detector.

4. Key in: ATTN 2t () 8 ENTER
% OFFSET 0 ENTER
PLOT SIGNAL A (B, C, OR D) ENTER
ZERO OFF ENTER

The resulting plot should be on scale within 15 minutes (may have some downward drift).

If the evaluation does not meet this criterion, check the trap by connecting the carrier line directly to the detector thereby removing the trap from the line and run test again.

Key in: STOP PLOT ENTER

Pneumatic Leak Test

Establish injection port, column oven and detector temperatures at or near ambient. Attach a plugged silicone rubber tube (part no. 5060-9055) to the detector vent. With a column firmly installed, set the carrier pressure at the two stage regulator on the gas cylinder to 276 kPa (40 psi). Open any flow controllers and regulators in the instrument that might retard gas flow to the detector. Turn the second stage regulator valve (generally a tee handle) until it's completely loose and allow the pressure to equilibrate. Note the pressure reading. There should be less than 13.8 kPa (2 psi) change during the next 5 minutes.

If the entire system fails this test, leak test the unheated fittings with a liquid soap for indication of gas bubbles. If this fails to help, using the process of elimination, recheck parts of the flow system until the leak is discovered and corrected. See FLOW SYSTEMS LEAK TESTS in this system.

Thermal Cleaning

Detector cleaning should only be performed after one is certain that gases, traps and plumbing connections have been verified to be adequate for operation.

WARNING

THE DETECTOR EXIT MUST ALWAYS BE CONNECTED TO A FUME HOOD (TO PREVENT POSSIBLE HAZARDOUS CONTAMINATION OF THE LAB WITH RADIOACTIVE MATERIAL) OR OTHERWISE VENTED IN COMPLIANCE WITH THE LATEST REVISION OF TITLE 10 CFR PART 20 OR WITH THE REGULATIONS OF A STATE WITH WHICH THE NRC HAS ENTERED INTO AN AGREEMENT. FOR COUNTRIES OUTSIDE THE U.S. CONSULT WITH THE APPROPRIATE AGENCY TO DETERMINE THE EQUIVALENT REGULATIONS.

Remove any existing column between the injection port and ECD. Install an empty glass column. Establish normal oven temperature (about 250 °C) and carrier flow rate (20-30 ml/min). Elevate the ECD zone to 350 °C to thermally clean for several (3-4) hours.

WARNING

DETECTOR DISASSEMBLY AND/OR CLEANING PROCEDURES OTHER THAN THERMAL CLEANING SHOULD BE ATTEMPTED ONLY BY APPROPRIATELY TRAINED PERSONNEL WHO HAVE BEEN DULY AUTHORIZED AND SPECIFICALLY LICENSED BY THE NRC, AGREEMENT STATES, OR OTHER APPROPRIATE REGULATORY AGENCIES TO HANDLE RADIOACTIVE MATERIALS. TRACE AMOUNTS OF Ni63 MAY BE REMOVED DURING OTHER CLEANING PROCEDURES, EXPOSURE TO BETA AND X-RADIATION (BREMSSTRAHLUNG) MAY BE HAZARDOUS UNLESS PROPER HANDLING PROCEDURES ARE OBSERVED.

NPD MAINTENANCE

In addition to the actual detector cleaning, some parts of other systems associated with the detector may require maintenance.

WARNING

NITROGEN-PHOSPHORUS DETECTORS UTILIZE HYDROGEN GAS. IF THE FLOW IS ON AND NO COLUMN IS CONNECTED TO THE DETECTOR INLET FITTING, HYDROGEN GAS CAN FLOW INTO THE OVEN AND CREATE AN EXPLOSION HAZARD. ALL INLET FITTINGS MUST HAVE EITHER A COLUMN OR A CAP CONNECTED TO THEM AT ALL TIMES.

Flow system leaks can dramatically affect overall NPD performance.

CAUTION

Leak detection solutions should be used cautiously. The contamination produced may reduce NPD sensitivity.

Another potential area of problems can be the electrical connection between the detector board and the detectors spring contact.

If the detector signal shows noise spikes this is usually the result of poor contact, or dirt, between the spring contact and the contact rod tip on the detector electronics board. Polish both parts with crocus cloth and wipe with a suitable degreasing solvent (i.e. hexane).

Detector Access

Remove the instruments top panel and the metal cover over the detector. Loosen the two holding screws of the air shield and lift the shield straight up and away from the instrument. Disconnect the two pin transformer connector (J2) from the detector board.

REPAIR

Operator repairs to Ni63 electron capture detectors will usually consist of replacing defective components or assemblies. By referring to the appropriate illustrations in the Parts Identification pages of this section the defective part(s) can be identified and replacements obtained. Replacing the part can be accomplished by using the illustration as a guide. If step-by-step instructions are required to remove an assembly, refer to RELOCATING DETECTORS in Section C of this manual.

For further assistance, contact the appropriate sales and service office of Hewlett-Packard. Electron capture detectors can be returned to specific Hewlett-Packard locations for repair provided that specified accompanying forms are completed in accordance with prevailing regulations.

RETURN OF DETECTOR TO HEWLETT-PACKARD

An electron capture detector (ECD) that is returned to Hewlett-Packard for any reason must include the entire fully assembled detector cell assembly. It must be accompanied by properly filled out radioactive license verification (part no. 5959-8209). The card must include all data and, when required, should be signed by your radiation safety officer.

NOTE

Electron capture detector assemblies are identified by a special number attached to the detector assembly. This number is required to facilitate their location in compliance with U.S. Nuclear Regulatory Commission regulations.

In the United States, return the completed card with the properly packaged detector to:

Hewlett-Packard Company
Avondale Division
Route 41
Avondale, Pennsylvania 19311
Attn. Electron Capture Detector Repair
Facility

Proper packaging, shipping, and labeling must comply with appropriate local, federal, and international laws and regulations. Consult your radiation safety officer or equivalent about local regulations.

PARTS IDENTIFICATION, ECD

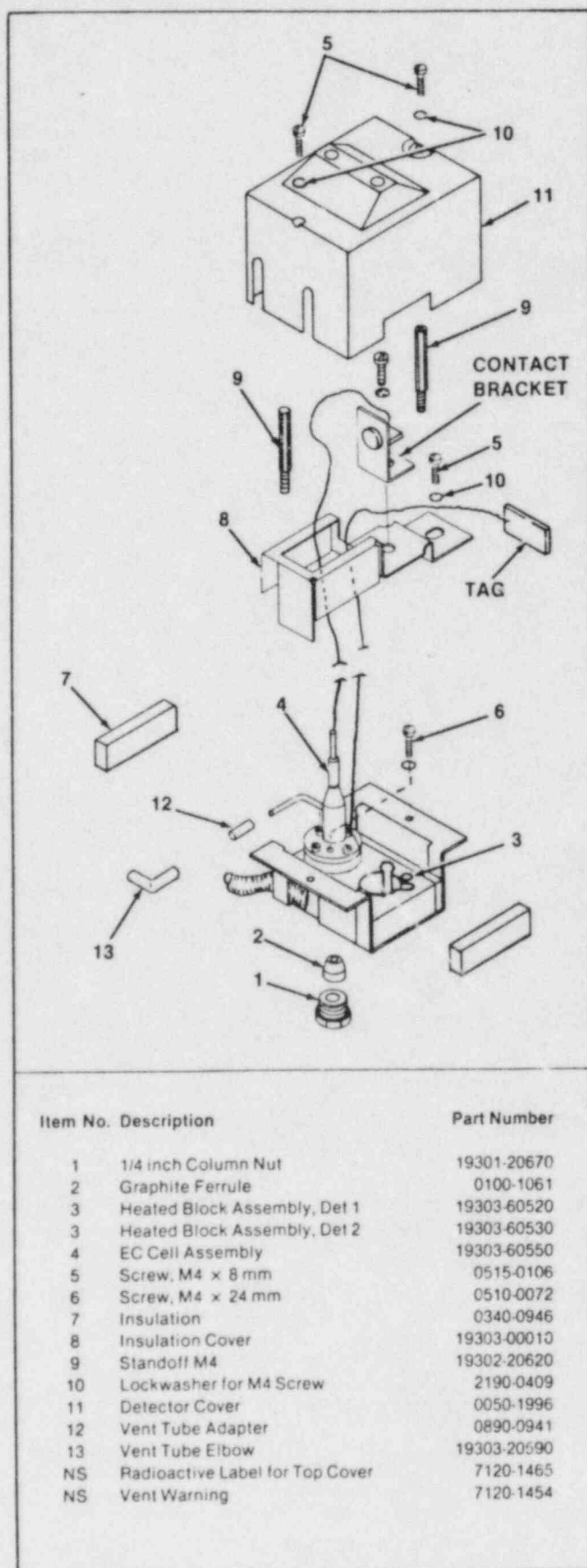


Figure 164. ECD Assembly

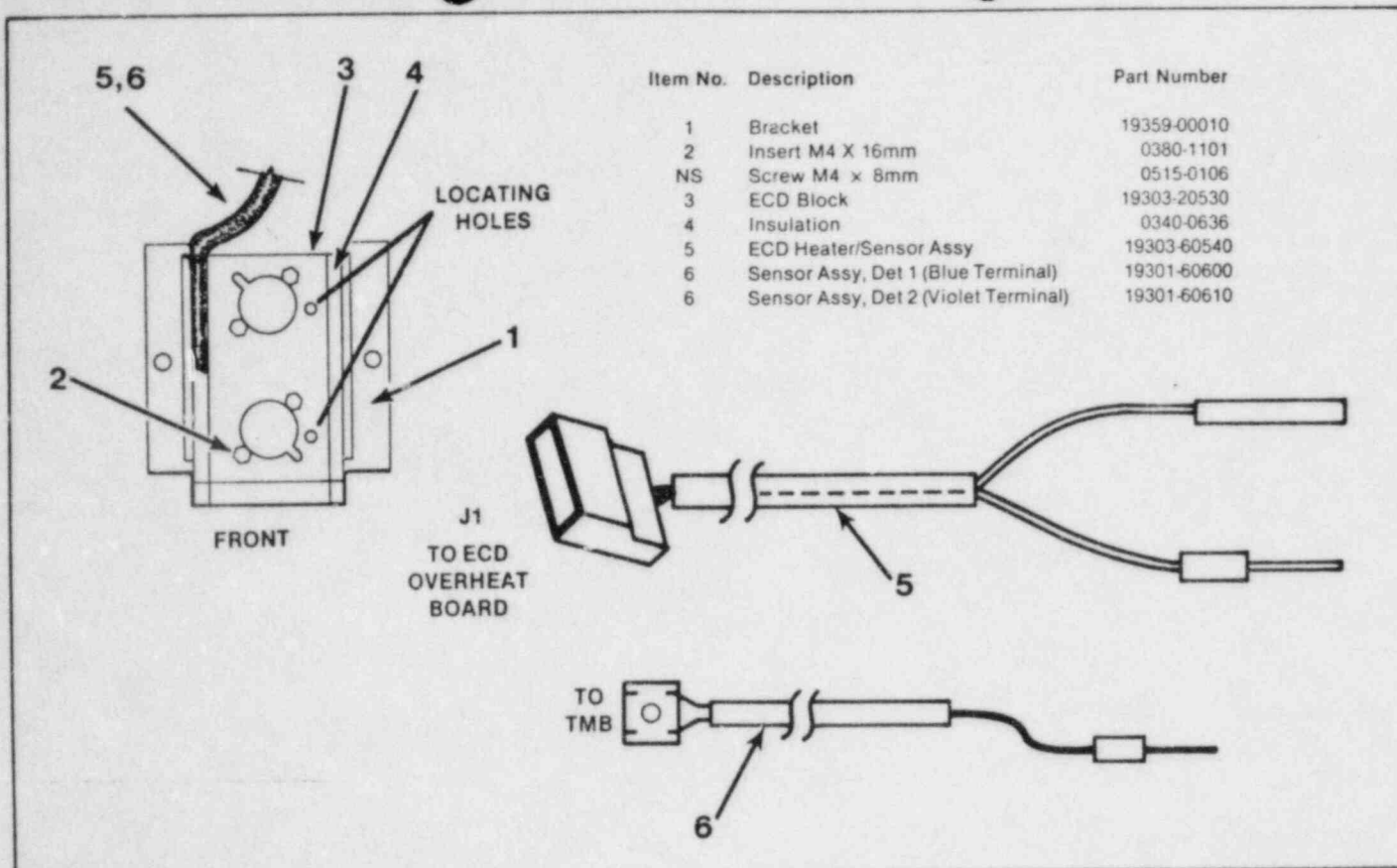


Figure 166. ECD Heated Block Assembly

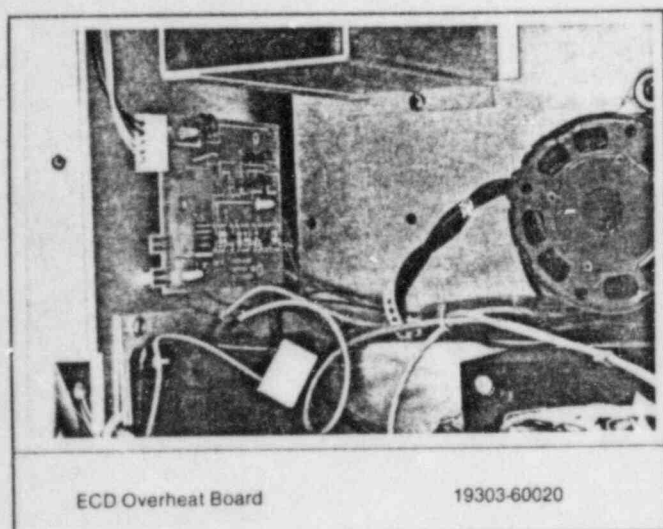


Figure 167. ECD Overheat Board

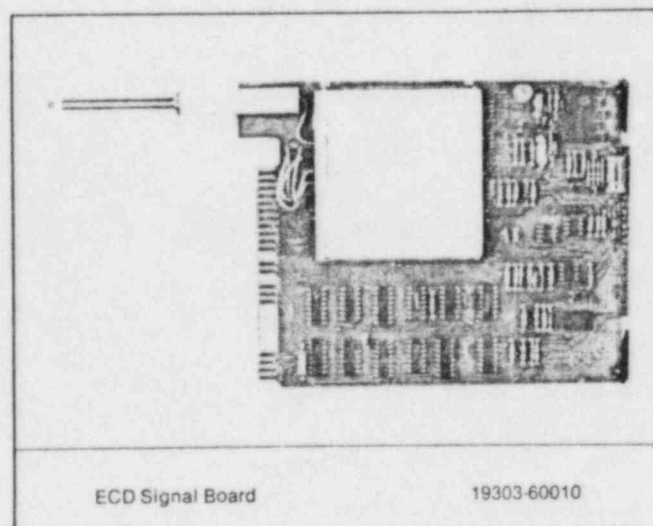


Figure 168. ECD Signal Board