

# walter c. mocrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS \* MICROSCOPY \* SMALL PARTICLE PROBLEMS \* SOLID-STATE CHEMISTRY

5 June 1985

Mr. James W. Patterson U. S. N. R. C. Matrials License Section 799 Roosevelt Road Glen Ellyn, Illinois 60137

#### Ref: BML #12-10632-1

Dear Mr. Patterson:

We have returned the H3 foil from our Varian E.C. detector to Varian Associates for disposal. Currently we hold a Valco Instruments Company Model 140B S/N152 electron capture detector.

I have included pages from their manual and a picture of the notice on the surface of the chamber. These indicate that the source is normally not accessible. We will not attempt to repair or replace the foil should such service be necessary, but will return the detector to Valco for service.

I have included a copy of Record of Source Transfer. Upon receipt of the detector the blue copy was sent to the Region 1 USNRC. At that time I thought that that was all that was necess v. I am now requesting than an amendment be made to our lice v to delete the Varian source and include this source.

If you have any questions, please write and we will respond promptly.

Sincerely,

Robert Z. Murgh PAD

JUN

7 1985

Robert Z. Muggli, PhD Senior Research Chemist

RZM:cb Encls.

8601020439 851125 REG3 LIC30 12-10632-01 PDR

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#### APPENDIX A

#### RADIATION SAFETY

# Read before installation of Model 140 detector

#### 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 Valco 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, 3H, decays with a half life of 12.26 years by the emission of beta particles to a stable nuclide of helium, 3HE. These beta particles are emitted with a maximum energy of 0.018 MeV and are absorbed by less than 1 mg/cm<sup>2</sup> 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 rotation of 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 resconsible for radiation safety enforcement.

#### Venting

To avoid lab contamination with traces of tritium, it is recommended that the detector be vented via the outlet tubes connection to a fume hood or outside building air trough, a window or vent.

## 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 Valco Instruments Co., Inc. (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 Texas and the name, address, and telephone number of your local radiation control agency are appended.

YOUR LOCAL RADIATION CONTROL AGENCY IS:

## 4.2 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, <sup>3</sup>H (tritium) and <sup>63</sup>Ni. Almost all of the EC detectors available use one of these isotopes.

Tritiated-Titanium Source. Tritium holds several major advantages over Nickel-53 as an ECD source material. Its betas have much lower energies (18 ke' maximum compared with 63 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 larger 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 titaniumplated metal surface. This technique produces high-intensity (0.25 curies/ cm<sup>2</sup>) sources which will maintain the tritium surface bond at tempera irus 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.

4.2.2 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.

4.2.3 Tritiated-Scandium Sources. Tritiated scandium combines all of the best features of tritium with a maximum temperature limit of  $320^{\circ}$  C, which is quite adequate for essentially all ECD applications. Source activities are available that approach one curie/cm<sup>2</sup>, producing a higher specific activity and thus a higher baseline current than any other beta source available for ECD use. The Valco Model 140 Wide-Range Electron-Capture Detector employs a l-curie tritiated-scandium source with a surface area of 1.6 cm<sup>2</sup>, and achieves a baseline current of at least 2 x 10<sup>-8</sup> amperes in a detector volume of only 180 microliters.

Valco Model-140 Wide Range Electron-Capture Detector

RECORD OF SOURCE TRANSFER (License No. 8-2584G) (Generally Licensed)

Date: 6-24-83

(Qty) Model 1408	detector assemblies with the following serial numbers Detector S/N 152
16-11-11-11-11-11-11-11-11-11-11-11-11-1	Controller S/N 112
and concaining a	total of 1000 millicuries of H3 Tritium were
shipped on6-23	3-83 via UPS
to:	HNU Systems 160 Charlemont St. Newton, MA 02161
The cognizant Rac	fiation Control Agency for the recipient is: Region 1, USNRC Office of Inspection & Enforcement 631 Park Ave. King of Prussia, PA 19406

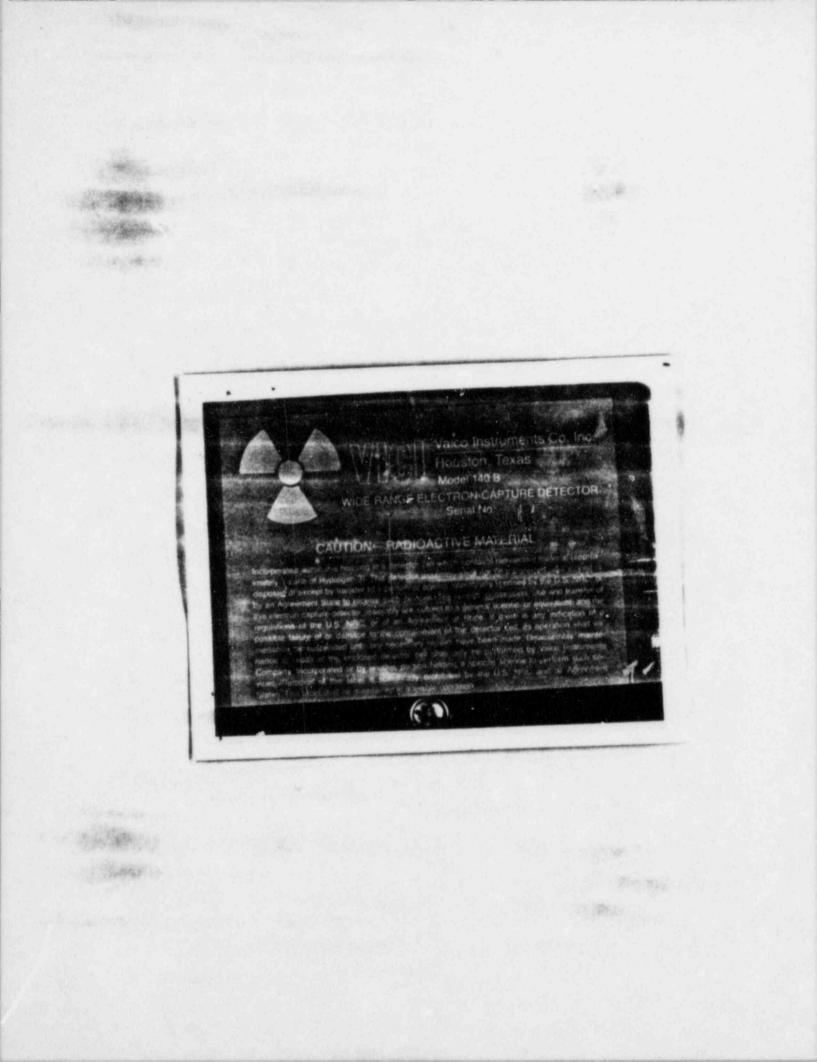
Receipt acknowledged by:

Cobert Z. Muggh: (Name) Job. Dieter (Title/Position) 4 Oct & 3

Date)

Sign and send BLUE COPY to Radiation Control Agency (address given above) Sign and return YELLOW COPY to: Valco Instruments Co., Inc. Attn: Radiation Safety Officer 7812 Bobbitt Lane Houston, Texas. 77055

Retain WHITE COPY for your information



TIME DATE CONVERSATION RECORD 5721/85 11. 30Am. TYPE ROUTING CONFERENCE TELEPHONE VISIT NAME/SYMBOL INT INCOMING OUTGOING Location of Visit/Conference: ORGANIZATION (Office, dept., bureau, NAME OF PERSON(8) CONTACTED OR IN CONTACT TELEPHONE NO. WITH YOU Walter C. MI Crone Asso, In Robert Z. Muggli, Phd SUBJECT Submitting the detector Cell information SUMMARY Mr. Muggle was told that he would have to submit detector cell cleaning information to the Commission. He agreed to forward such information as needed 1012 11/08/85 Talked to Dr Muggli - Se had no problem is authorized them for 2 - Deurie H3 foils. ACTION REQUIRED None NAME OF PERSON DOCUMENTING CONVERSATION SIGNATURE DATE Janes W. Gatters -5/21/85 JAMES W. PATTERSON ACTION TAKEN SIGNATURE TITLE DATE 50271-101 OUS. Gavernment Minting Office: 1980-341-526/6230 CONVERSATION RECORD OPTIONAL FORM 271 (12-76) DEPARTMENT OF DEFENSE