

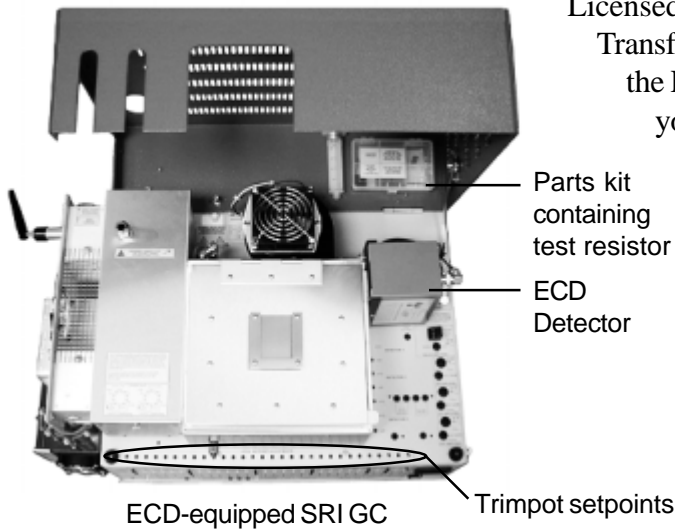
DETECTORS

Electron Capture Detector - ECD

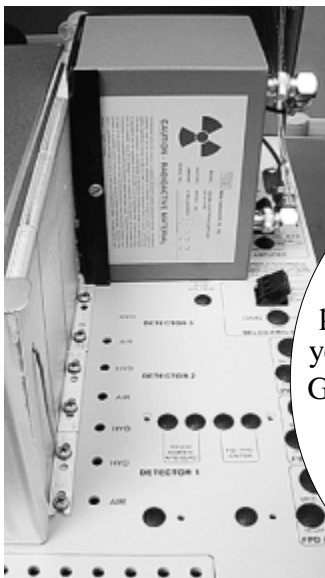
OVERVIEW

The Electron Capture Detector (ECD) is selective to electronegative compounds, especially chlorinated, fluorinated, or brominated molecules. It is sensitive to some of these compounds in the parts per trillion (ppt) range. The ECD detector requires nitrogen or argon / 5% methane (P5) to operate. The ECD detector is mounted immediately adjacent to the right rear column oven wall on your SRI GC chassis. Two BNC cables connect the anode and cathode, respectively, to the ECD amplifier. The ECD detector consists of a stainless steel cylinder containing 5 millicuries of radioactive Nickel 63 in an oven enclosure that is thermostatically controllable from ambient temperature to 375°C. Since the detector contains only 5 millicuries of Nickel-63, the ECD is covered by a "General License" requiring a periodic wipe test and the filing of a form with your state's Department of Health. The documentation necessary to authorize your possession of a radioactive source is included in the ECD manual from Valco, the manufacturers. This documentation transfers possession of the ECD directly to you from Valco; SRI provides the ECD installation service and the GC. There are four important documents to look for: 1) Certification of Sealed Source, 2) Conditions for Acceptance of a Generally Licensed Device, 3) Test Specifications, and 4) Record of Source Transfer.

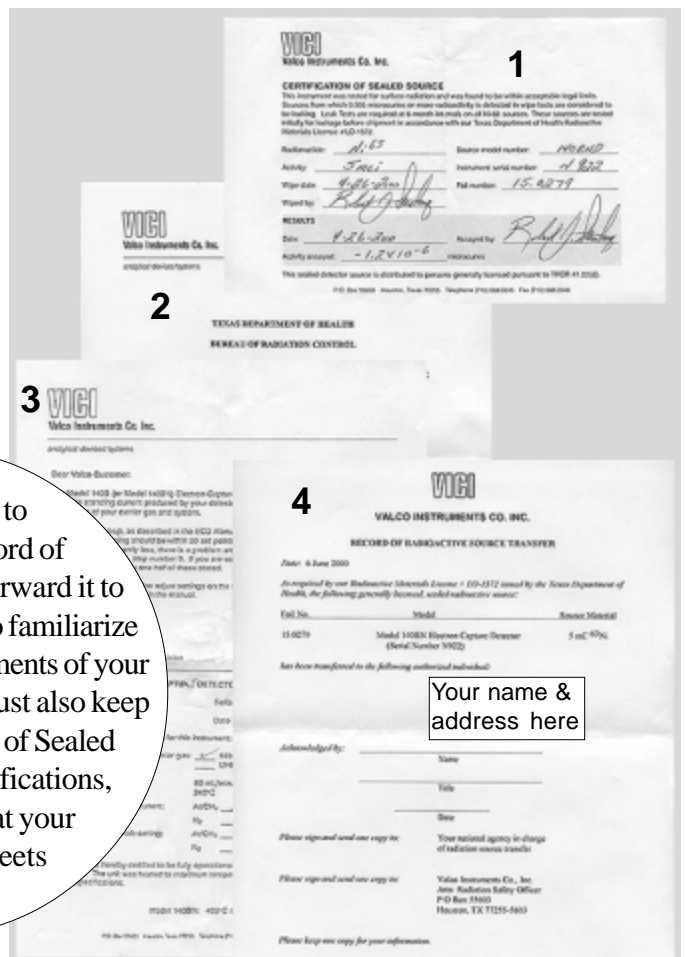
Valco may print your address on the multiple copies of the Record of Source Transfer, which are to be completed by you and filed with the appropriate state and local authorities. The other documents remain with the ECD detector, and are necessary to prove authorized possession of the ECD.



ECD-equipped SRI GC



Close-up view of the ECD detector on an SRI GC



It is imperative to complete the Record of Source Transfer and forward it to proper authorities, and to familiarize yourself with the requirements of your General License. You must also keep on file the Certification of Sealed Source and Test Specifications, which are proof that your ECD detector meets regulations.

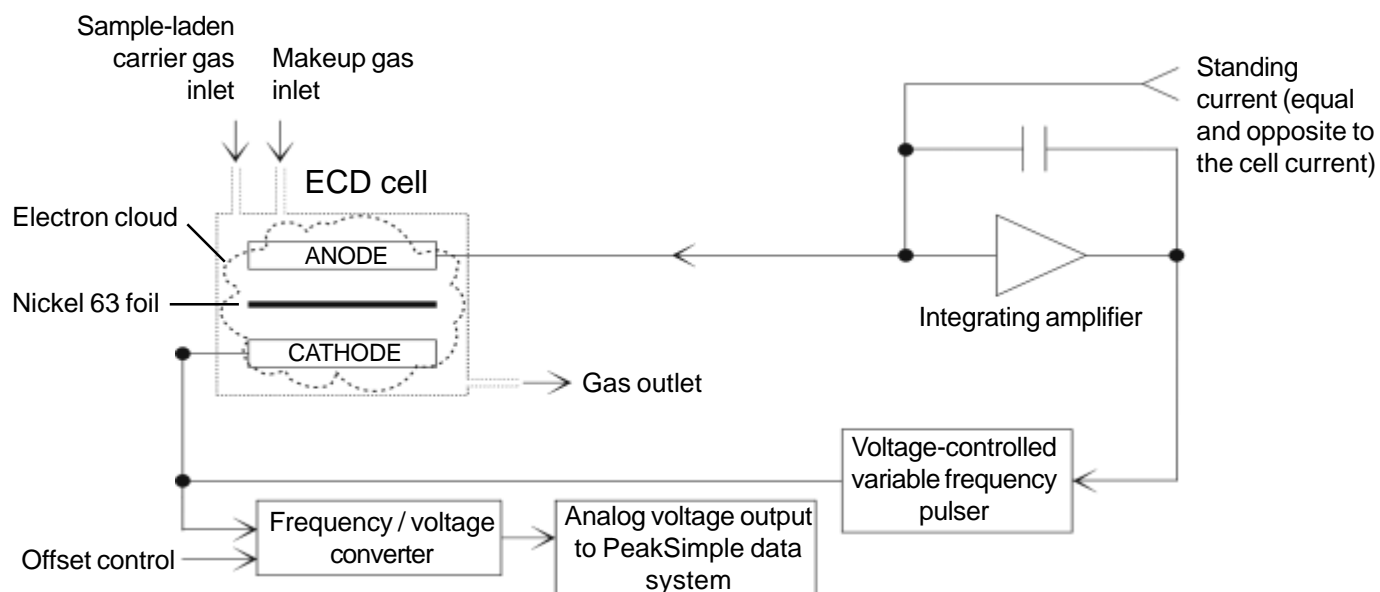
DETECTORS

Electron Capture Detector - ECD

Theory of Operation

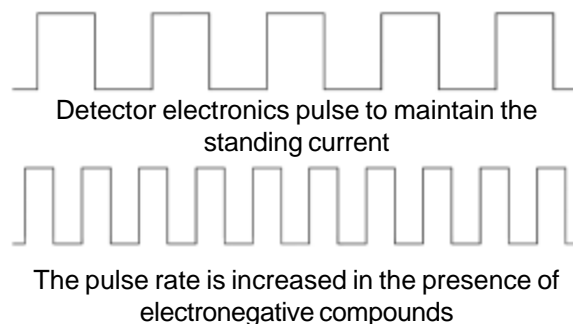
The radioactive Nickel 63 sealed inside the ECD detector emits electrons (beta particles) which collide with and ionize the make-up gas molecules (either nitrogen or P5). This reaction forms a stable cloud of free electrons in the ECD detector cell. The ECD electronics work to maintain a constant current equal to the standing current through the electron cloud by applying a periodic pulse to the anode and cathode. The standing current value is selected by the operator; the standing current value sets the pulse rate through the ECD cell. A standing current value of 300 means that the detector electronics will maintain a constant current of 0.3 nanoamperes through the ECD cell by periodically pulsing. If the current drops below the set standing current value, the number of pulses per second increases to maintain the standing current.

ECD Detector Operational Diagram



When electronegative compounds enter the ECD cell from the column, they immediately combine with some of the free electrons, temporarily reducing the number remaining in the electron cloud. When the electron population is decreased, the pulse rate is increased to maintain a constant current equal to the standing current. The pulse rate is converted to an analog output, which is acquired by the PeakSimple data system. Unlike other detectors which measure an increase in signal response, the ECD detector electronics measure the pulse rate needed to maintain the standing current.

Example Pulse Trains



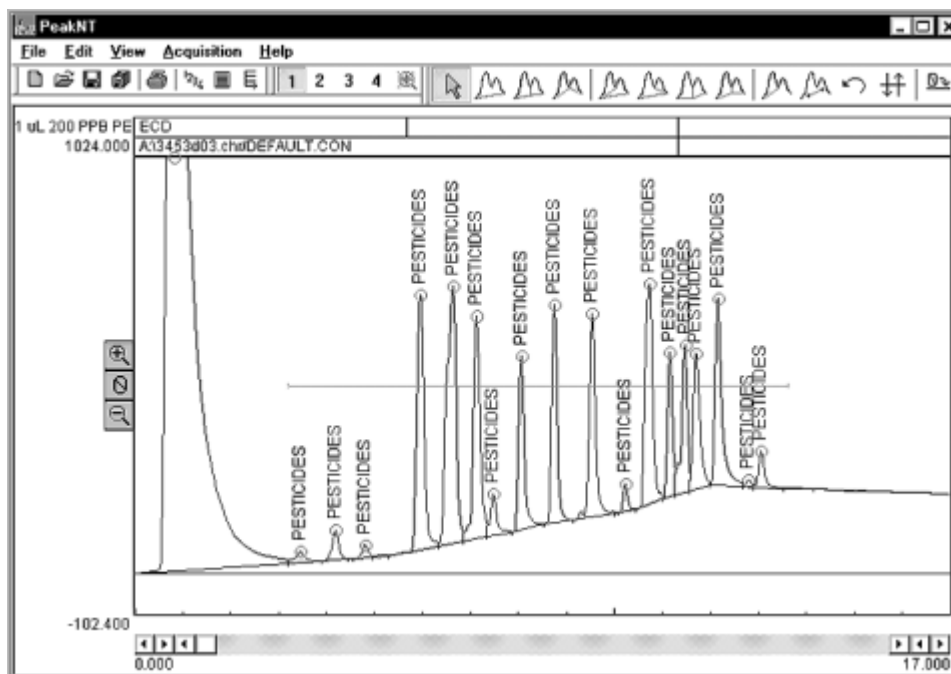
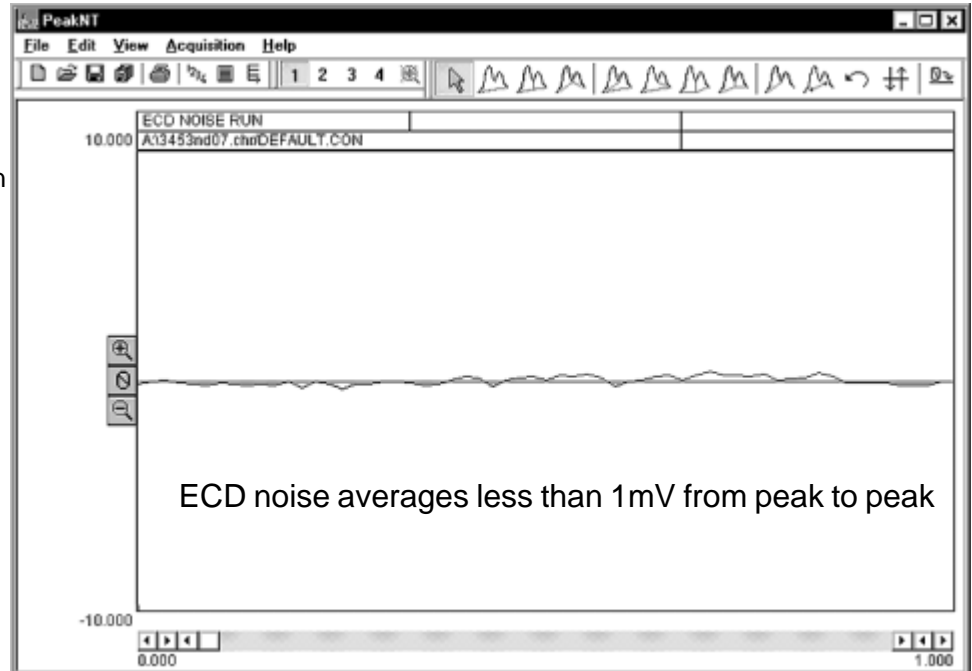
DETECTORS

Electron Capture Detector - ECD

Expected Performance

ECD Noise Run

Column: 15m MXT-5 capillary
Carrier: Helium @ 10mL/min
Makeup: Nitrogen @ 60mL/min
ECD Temp: 250°C
ECD standing current: 300
Offset before zeroing the data
system signal: 280mV



ECD Pesticide Analysis

Sample: 1 μ L 200ppb chlorinated pesticides
Column: 15m MXT-5 capillary
Carrier: Helium @ 10mL/min
Makeup: Nitrogen @ 60mL/min
ECD temp: 250°C
ECD standing current: 300

Results:
Component Retention Area
Pesticides 2.850 45792.4350

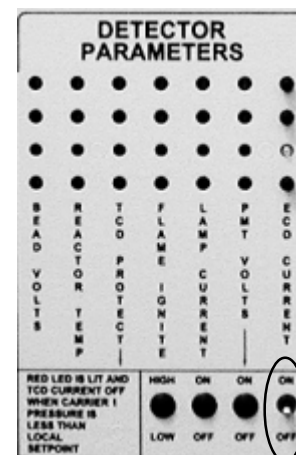
DETECTORS

Electron Capture Detector - ECD

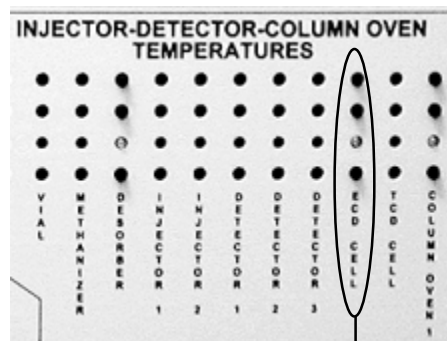
General Operating Procedure

The following suggestions are specific to your SRI ECD-equipped GC. Consult the Valco ECD detector manual for carrier gas purity requirements, carrier gas system configuration, and other general ECD detector information. Keep in mind that the electronics schematics in the Valco manual do not apply to your ECD-equipped SRI GC.

1. Cap off the carrier inlet to the ECD cell (in the column oven).
2. Connect the makeup gas and let it flow through and purge the ECD cell. Makeup flow is 40-100mL; typically 60mL.
3. Heat the ECD detector to 150°C to verify that the baseline noise and offset are normal. 150°C is hot enough to evaporate off water but low enough to avoid oxidation of the nickel foil which can occur at high temperatures in the presence of oxygen. Once you have verified the ECD's operation at this temperature, you may heat it to higher temperatures.
4. Turn on the ECD standing current (the ECD current ON / OFF switch is located on the front control panel of the GC, under "DETECTOR PARAMETERS"). As a rule of thumb, an ECD detector requires enough nitrogen makeup flow (40-100mL/min) to significantly dilute the carrier in order to help keep detector noise down; the ECD can tolerate a 6:1 ratio of nitrogen to helium.



ECD standing current ON/OFF switch



ECD cell temperature display

With the carrier and makeup gas connected and flowing, check the offset from zero. The millivolt reading should be between 100 and 500mV. If the signal offset is less than 100mV, the standing current needs to be increased. If the signal offset is higher than 500mV, the standing current needs to be decreased. Once the signal is relatively quiet and stable, set the temperature to whatever is appropriate for your analysis by adjusting the trimpot setpoint with the flat blade screwdriver provided.

5. When the ECD detector cell reaches temperature, let the system stand until you get a stable milliVolt reading. Once the system exhibits a stable baseline, reconnect the column. Observe the signal in the presence of the carrier flow. If it is significantly higher, it indicates contamination introduced on the carrier flow. If the milliVolt reading is still relatively stable in the presence of carrier flow, then sample may be injected. Avoid samples with high concentrations of electronegative compounds; they may effect ECD operation for some time thereafter, as they could take too long to dissipate.
6. You may need to adjust the ECD standing current using its trimpot setpoint. The trimpot setpoints are located on the top edge of the front control panel, directly above the display push-buttons for each controlled zone. Remember, increasing the standing current increases the ECD's sensitivity and raises the baseline offset.

ECD Troubleshooting

If you are experiencing baseline offset and noise problems with your ECD detector, try the following two diagnostic tests:

1. Verify that the ECD amplifier electronics are working properly by removing the detector from the circuit and inserting a 1000M Ω test resistor in its place. The parts kit in the tackle box included with your GC under the red lid contains a 1000M Ω resistor for this test. Turn the ECD current off. The anode and cathode connections are BNC connectors located on the GC chassis near the base of the ECD detector housing. Disconnect these two BNC connectors from the detector electronics, and install the 1000M Ω test resistor as a jumper between the center conductor in the anode BNC jack and the center conductor in the cathode BNC jack. Zero the data system signal. Turn the ECD current back on, and check the signal offset (observe the mV reading in the upper right area of the PeakSimple chromatogram window. With the test resistor in the detector's place, the signal offset should be 120-150mV with the standing current at 300. If the signal offset is pegged up or down (5000mV or 1500mV, respectively), there is a problem with your ECD detector electronics. Try turning off the GC power for at least 30 seconds, with the test resistor still in place, then turning it back on to see if the signal offset still indicates a problem. If the signal offset is at zero with the test resistor in place, check to make sure that you are looking at the correct detector channel. If you are observing a signal offset of zero in the ECD detector channel, call technical support.

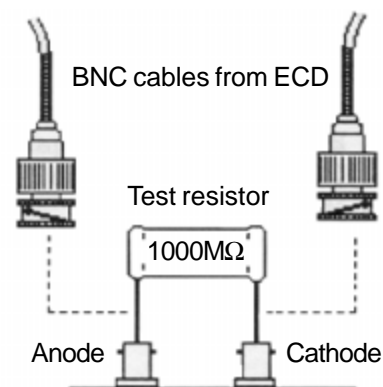
The ECD anode and cathode are connected to the ECD amplifier via BNC cables



Anode and cathode BNC connectors



ECD electronics test



2. Operate the ECD on make-up gas only by disconnecting the column from the ECD. With the standing current still set at 300, observe the signal offset and noise. If it drops, then the problem is being introduced into the GC and ECD by the carrier gas through the column.

Tip: In most situations, the ECD will be used to detect sample components that are reactive with metal. Use glass, fused silica, or fused silica lined metal capillary columns to help avoid reactive sites and ghost peaks.