Agilent G1533-90790 Rebuilt Electron Capture Detector

User Information

Agilent Technologies

Notices

Manual Part Number

G1533-90790

Edition

Third edition, March 2002

Printed in USA

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Palo Alto, CA 94304 USA

Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

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1
About Your Refurbished Detector

The enclosed Electron Capture Detector (ECD) was refurbished and checked to meet a set of specifications that will guarantee that the detector will perform as well as a brand new ECD on a new instrument.

The enclosed chromatogram shows the typical response and noise expected from any of the refurbished ECDs that pass through our process and are run under the typical check-out conditions as specified in the Performance Verification Procedures for this model of **new** chromatographic instrument, or an instrument that is clean, leak free and supplied with clean gases.

Since the condition of the instrument to be used to mount this refurbished detector is unknown, we cannot predict its performance when installed. Several instrument conditions may prevent this detector from performing to the desired specifications – such as contamination entering the detector from any source, i.e., the column, the injection port, the support gases, etc.

If the enclosed detector does not meet your requirements when installed, please use proper troubleshooting procedures to try to determine and isolate the source of the problem. It is likely that some of the other hardware that plays a vital role in the proper operation of the analytical system may be the source of contamination to the ECD.

If the performance of this rebuilt ECD is questionable, please perform the performance verification (PV) according to the instructions for your instrument and compare that response to the expected response and noise levels for your configuration of the instrument.

If the performance does not meet the guidelines given in the PV procedures, please refer to the enclosed troubleshooting guidelines or the troubleshooting guidelines in the service section of your instrument manual as an aid in your troubleshooting procedures. You may also wish to refer extended troubleshooting of a difficult problem to your Agilent service representative.

If a detector is to be left unused for a period of time, purge it with a small flow of inlet or purge gas (helium or nitrogen, etc.). This will prevent other contaminants (such as laboratory air) from entering the detector and degrading the ultimate performance.

2
Typical Operation of an ECD Over Long Periods

Figure 1 illustrates how ECD performance can change with “typical” operation over extended periods of time.

The “Y” axis represents the frequency or response of the detector and the “X” axis is a pseudo time axis. Please note that neither axis is meant to represent a linear scale but only the general concept of the response or time.

Region “A” represents the operation of the detector when the instrument or detector is new and/or recently cleaned or refurbished. The “peak” would represent the chromatographic response to a standard sample under those conditions.

The successive “peaks” across the time direction (in the “B” region) represent a typical response to the same standard sample at various times over the lifetime of the ECD. Note that the “response” tends to increase as the baseline increases with time. The baseline increase may result from a contamination build-up inside the detector over time with either sample components or other contamination fed to the detector via the support gases or column bleed.

Note that the noise of the baseline increases over time as a direct result of the contamination build-up and baseline increase. However in some customers’ practice the increased noise may be unnoticeable because of the range and attenuation that the instrument is being operated at, as well as the fact that the noise increase is quite gradual and does not affect the quantitation from a calibrated detector response.

Region “C” represents the approach to the end of the useful lifetime of the ECD due to the excessive response as well as the excessive noise of the baseline. In these cases the baseline noise begins to interfere with the peak identification (especially at low levels of detection for the desired components). The response from higher levels of sample may even saturate the frequency output of the detector resulting in flat-topped peaks.

It is in the “C” region that the necessity of cleaning the detector becomes obvious and a user may remove the ECD from service and return it to the factory for refurbishment. When received, Agilent will “clean” the detector, replace any necessary components and test it for functionality to the same specifications as the original detector. We will return a detector to you in a similar condition as represented in the “A” region.

The relative difference between the “peak height” response of a detector operating in the “C” region and the “A” region can be as great as a factor of 8–10. The signal noise will also be increased by the same (or greater) amount, so the sensitivity (as defined by the signal to noise ratio) is not greater in the “C” region and the overall operation of the detector is improved with the cleaner detector.

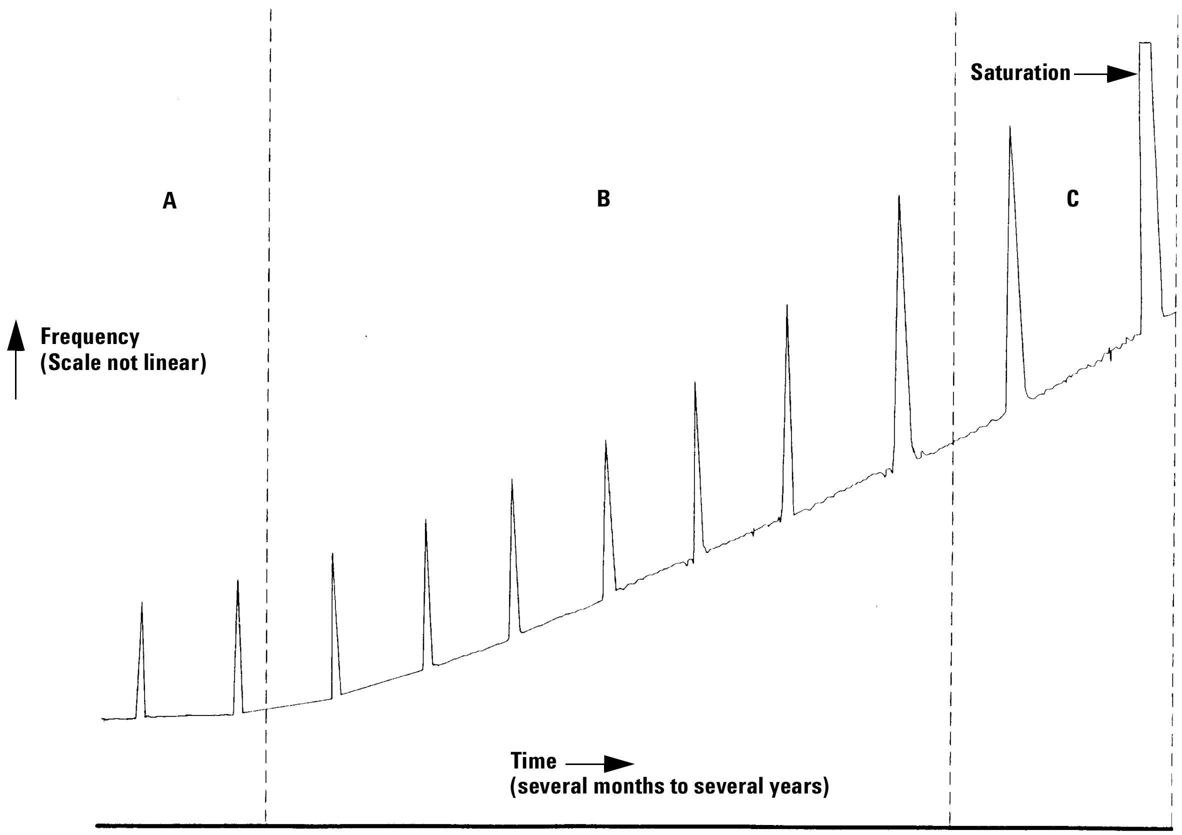

Figure 1 “Typical” operation of an ECD over extended periods of time

3
Checkout Conditions for Common Configurations

This section lists the checkout conditions and typical expected performance for 5890 and 6890 GCs with split/splitless inlets. For other configurations please see the latest revision of the Standard Operating Procedures (SOP) documents or Performance Verification Procedures (PV) documents. See Table 1 for the appropriate document.

Table 1 Checkout conditions for 5890 and 6890 instruments with S/S Inlets and ECDs

	SOP	PV
5890 instruments	05890-90390	5965-6628
6890 instruments	G1530-61210	5963-7184

6890 Noise Test

The tables below summarize the test conditions and typical expected results. For the detailed test procedure and more information, see the SOP or PV.

Table 2 6890 parameters for detector noise test

	Detector	
Conditions	**ECD**	**µECD**
6890		
Detector offset	<100*	<400
6890 range	0	6
3396B/3396C/3397A		
ATTN^2	0	3
Zero	50	10
Chart speed	1	1

* 100 display units = 500 Hz at Ref. current of 1 nA

Figure 2 and Figure 3 show typical noise test results.

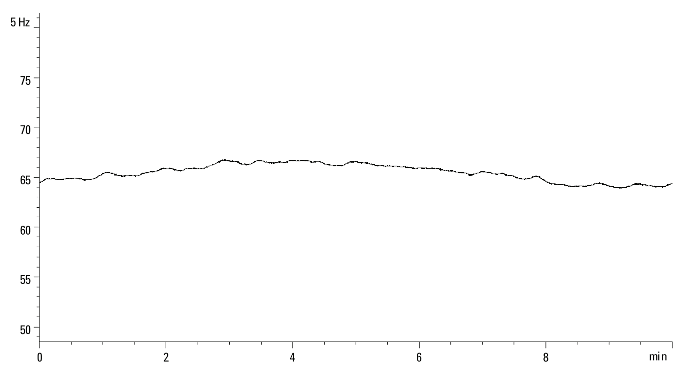

Figure 2 ECD with capillary split/splitless inlet noise test

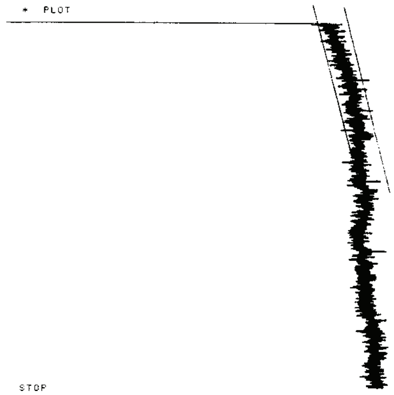

Figure 3 µECD with capillary split/splitless inlet noise test

Table 3 6890 detector noise, wander and drift test limits

	Detector	
Range of Operation	**ECD**	**µECD**
Eval time (min)	10	11
Noise (min)	<9	<37
Wander (mm)	<90	<90
Drift	<90	<90

6890 Performance

Table 4 summarizes the chemical test method parameters.

Table 4 6890 chemical test parameters

		Detector	ECD				µECD			
		Inlet	**Cap S/SL**	**PP**	**COC**	**PTV**	**Cap S/SL**	**PP**	**COC**	**PTV**
Temperatures, °C		Oven temp.	80	80	80	80	80	80	80	80
		Initial temp.	80	80	80	80	80	80	80	80
		Initial time, min	0	0	0	0	0	0	0	0
		Rate, °C/min	15	15	15	15	15	15	15	15
		Final temp.	180	180	180	180	180	180	180	180
		Final time, min	10	10	10	10	10	10	10	10
		Detect temp.	300	300	300	300	300	300	300	300
		Inlet temp.	200	200	OT	80[*]	200	200	OT	80[*]
Flow/Pressure Rates	Inlet	Pressure (EPC) (psi)	25[†]	25[†]	25[†]	25[†]	25[†]	25[†]	25[†]	25[†]
		Mode	SL/CP	CF	CP	SL/CP	SL/CP	CF	CP	SL/CP
		Purge time, min	0.75	N/A	N/A	0.75	0.75	N/A	N/A	0.75
		Purge flow, mL/min	60	N/A	N/A	60	60	N/A	N/A	60
	Detector	Ref gas	N_2[‡]	N_2[‡]	N_2[‡]	N_2[‡]	N_2[‡]	N_2[‡]	N_2[‡]	N_2[‡]
		Ref flow (mL/min)	6±1[‡]	6±1[‡]	6±1[‡]	6±1[‡]				
		Makeup gas	N_2	N_2	N_2	N_2	N_2	N_2	N_2	N_2
		C. mkup flow (mL/min)	60±3	60±3	60±3	60±3	30±3	30±3	30±3	30±3
		Offset[**] (display units)	<100	<100	<100	<100	<400	<400	<400	<400
Other		Range	0	0	0	0	6	6	6	6
		Column	19091J-413 HP-5 30 m x 0.32 mm x 0.25 mm							
		Sample	ECD	ECD	ECD	ECD	ECD	ECD	ECD	ECD

* See the 6890 PV for PTV temperature program

† 7.5 to 7.9 mL/min (manual) at oven temperature of 40 °C

‡ ECD anode purge flow

** ECD–0.2 x Hz

ECD checkout sample - part no. 18713-60040

SL/CP - Splitless Constant/Pressure for EPC inlets

CF - Constant Flow for EPC inlets

OT - Oven Track

ECD

Table 5 lists the checkout method and typical expected 6890 ECD performance.

Table 5 ECD with capillary split/splitless inlet

Oven	
Initial temp	80°C
Initial time	0 min
Rate 1	15°C/min
Final temp 1	180°C
Final time 1	10 min
Inlet	
Temperature	200°C
Inlet pressure	25 psi (cons. press. for EPC inlets), 7.5 — 7.9 mL/min (manual) at an oven temp of 40°C
Mode	Splitless
Purge time	0.75 min
Purge flow	60 mL/min
Detector	
Temperature	300°C
Anode purge flow (N_2)	6 ± 1 mL/min
Cons. mkup flow (N_2)	60 ± 3 mL/min
Ref. current	1 nA
Offset	Should be < 100 display counts (< 500 Hz)
Column and sample	
Type	HP-5 30 m x 0.32 mm x 0.25 mm -- part no. 19091J-413
Sample	ECD checkout sample -- part no. 18713-60040
Composition	33 pg/mL (0.33 ppm) of each lindane and aldrine in isooctane
Injection volume	1 µL
6890 signal parameters	
Range	0

Table 5 ECD with capillary split/splitless inlet (continued)

3396B or 3396C or 3397A integrator conditions with analog	
Set date and time	
ZERO	10
ATT 2^	7
CHT SP	1
PK WD	0.04
THRSH	7
STOP TIME	11
Chromatogram specifications	
Sensitivity	Lindane Area counts >757,000
Reproducibility	RSD of Lindane retention times, for inj. 1 to 6, <1%*
	RSD of Lindane Area, for injections 1 to 6, <3%*
MDL	MDL (Lindane pg/s) = (3366 x Noise/(Area Lindane) with the area measured at range 2^) and the noise in mm at attn 2^0 (noise measure peak to peak) <=0.4 pg/s

* 6890 Series Automatic Liquid Sampler only

Figure 4 and Table 6 show typical 6890 ECD checkout chromatogram and results.

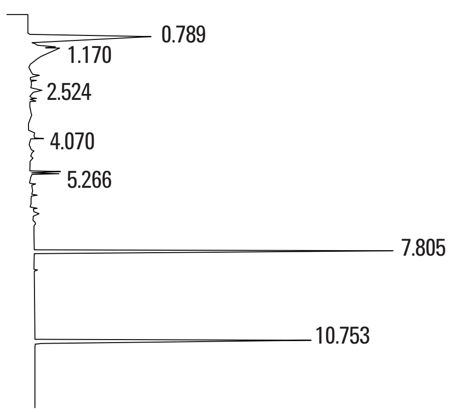

Figure 4 6890 ECD with capillary split/splitless inlet checkout chromatogram

Table 6 Typical expected checkout results

RT	Area	Type	Width	Area%
0.789	1843432	BV	0.132	27.22585
1.080	196465	VV	0.064	2.90161
1.170	903974	VP	0.256	13.35089
2.524	146218	BP	0.097	2.15951
4.070	86832	VB	0.055	1.28243
5.190	113038	PV	0.032	1.66947
5.266	106328	VV	0.032	1.57037
7.805	1361074	PB	0.034	20.10185
10.753	2013529	PB	0.064	29.73802

µECD

Table 7 lists the checkout method and typical expected 6890 µECD performance.

Table 7 µECD with capillary split/splitless inlet

Oven	
Initial temp	80°C
Initial time	0 min
Rate 1	15°C/min
Final temp 1	180°C
Final time 1	10 min
Inlet	
Temperature	200°C
Inlet pressure	25 psi (cons. press. for EPC inlets), 7.5 — 7.9 mL/min (manual) at an oven temp of 40°C
Mode	Splitless
Purge time	0.75 min
Purge flow	60 mL/min
Detector	
Temperature	300°C
Anode purge flow (N_2)	6 ± 1 mL/min
Cons. mkup flow (N_2)	30 ± 3 mL/min
Ref. current	1 nA (fixed)
Offset	Should be < 400 display counts
Column and sample	
Type	HP-5 30 m x 0.32 mm x 0.25 mm -- part no. 19091J-413
Sample	ECD checkout sample -- part no. 18713-60040
Composition	33 pg/mL (0.33 ppm) of each lindane and aldrine in isooctane
Injection volume	1 µL
6890 Signal parameters	
Range	6

Table 7 µECD with capillary split/splitless inlet (continued)

3396B or 3396C or 3397A integrator conditions with analog		
Set date and time		
ZERO	10	
ATT 2^	5	
CHT SP	1	
AR REJ	100000	
PK WD	0.04	
THRSH	5	
STOP TIME	11	
Chromatogram specifications		
Sensitivity	Lindane Area Counts	> 52,000 X noise
Reproducibility	RSD of Lindane Retention Times, for inj. 1 to 6*	< 1%*
	RSD of Lindane Area, for injections 1 to 6	< 3%*
MDL	MDL (Lindane fg/sec) = (47,950 x Noise/(Area Lindane) (with the area measured at range 2^6), the noise in mm at attn 2^3 (noise measure peak to peak), and Range^0 should be <37 mm (1 min).	< 8 fg/s

* 6890 Series Automatic Liquid Sampler only

Figure 5 and Table 8 show typical 6890 µECD checkout chromatogram and results.

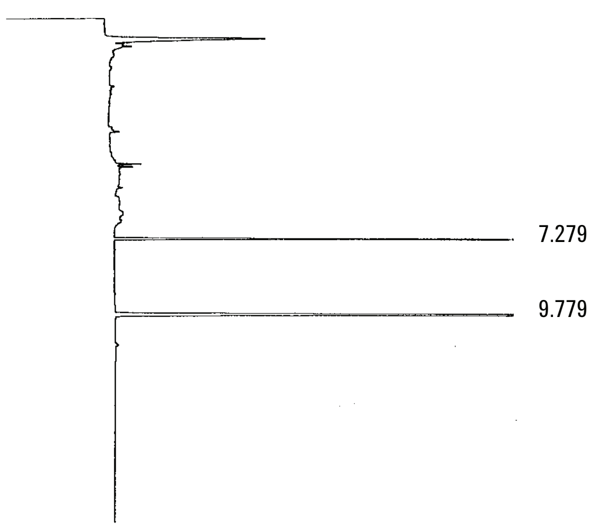

Figure 5 µECD with capillary split/splitless inlet checkout chromatogram

Table 8 Typical expected checkout results

RT	Area	Type	Width	Area%
0.653	556300	PV	0.103	16.93781
7.279	1272275	PB	0.028	38.73728
9.779	1455794	VB	0.055	44.32493

5890 Noise Test

The tables below summarize the test conditions and typical expected results. For the detailed test procedure and more information, see the SOP or PV.

Table 9 5890 parameters for detector noise test

	Setting
5890	
Range Sig 1 or Sig 2	0
3396	
ATT 2	-2 for 2 min 0 for 10 min
Zero	50
Chart speed	1

Table 10 ECD noise specifications

Noise	For any 2-minute interval over the entire plot, the noise in mm should be 0.415 x the observed signal offset
Wander	For any 10-minute interval over the entire plot, the wanders should remain within the confines of the page
Drift	For any 10-minute interval over the entire plot, the drift should remain within the confines of the page

Figure 6 shows a typical noise test result.

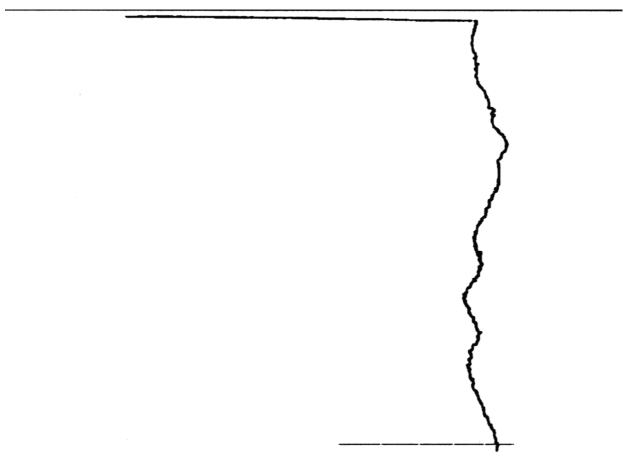

Figure 6 ECD noise test

5890 Performance

Table 11 ECD with split-only or split/splitless capillary inlet

Temperatures	
Oven	170 °C
Oven equilib. time	3 min
Initial temp	170 °C
Initial Time	8 min
Rate	0
Final temp	0
Final time	0
Detector	300 °C
Inlet	200 °C
5890 Signal Parameters	
Range	0
Flow Rates	
Carrier (Helium)	15 ± 1 mL/min
Makeup (N_2)	60 ± 2 mL/min
Split vent	60 ± 2 mL/min
Septum purge	5 ± mL/min
Sample	
Type	ECD Sample
Part number	18713-60040
Composition	33 pg/µL (0.33) ppm) of each of Lindane and Aldrine in Isooctane
Injection volume	1 µL
3396 Integrator Conditions	
ZERO	10
Attn	5
Chart speed	1
Peak width	0.04
Threshold	5
Area reject	0

Table 11 ECD with split-only or split/splitless capillary inlet (continued)

Stop time	8		
Chromatogram Specifications			
Sensitivity	Lindane Area Counts	>11,400 x Noise (mm)	
Reproducibility	RSD of Aldrin Retention Times, for inj. 2 to 6		< 1% (7673 only)
	RSD of Aldrin Area, for injections 2 to 6		< 3% (7673 only)

Figure 7 and Table 12 show typical 5890 ECD checkout chromatogram and results.

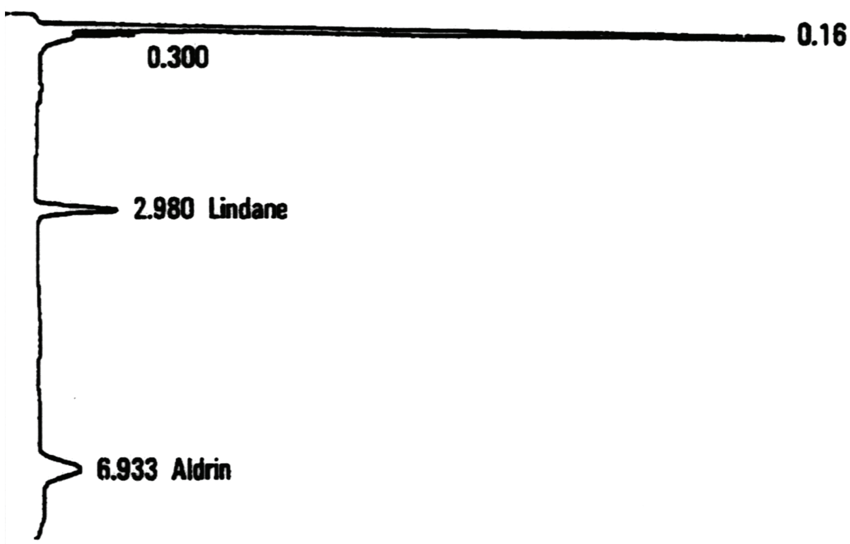

Figure 7 ECD with capillary inlet checkout chromatogram

Table 12 Typical expected checkout results

RT	Area	Type	Width	Area%
0.167	2195754	SBB	0.033	84.70592
0.300	32279	TBB	0.027	1.24523
2.980	172491	BB	0.116	6.65422
6.933	191684	BB	0.261	7.39463

4
Troubleshooting Contamination Problems on ECD

Persistent problems with high ECD background noise or “Ghost Peaks” during temperature program are almost always due to contamination introduced to the GC system from outside the GC, either from dirty samples, consumables, or from the carrier/make-up gas supplies.

Insure clean gas supplies

Before beginning the troubleshooting process, make sure the GC system is installed with supply gases of sufficient purity.

- ✔ The carrier and makeup gas purity should be >99.999%.
  See the site prep section in the 6890 user documentation.
- ✔ After you are sure of the purity of the carrier gas, insure that the tank regulators have STAINLESS STEEL diaphragms, equivalent to Agilent p/n 8507-0407 (CGA 580 in USA).
- ✔ Install new 1/8-inch copper supply tubing – Agilent p/n 5180-4196. (Many times “clean” tubing from other vendors has caused high ECD background). At the same time install new traps in both the carrier and makeup supplies – moisture trap p/n 5060-9084 closest to the tank, and indicating O_2 trap p/n 3150-0528 closest to the GC. Be very careful to leak test the entire plumbing manifold.

Isolate problem to carrier or makeup gas supplies

Determine what components of the apparent ECD contamination are from the carrier vs. makeup systems in the GC. Typically, sharp, well-resolved peaks that elute during a temperature program with no injection are from the carrier/inlet system. Broader “humps” in the baseline are usually from the makeup system. Overall high background can be contaminated gas from either the carrier or makeup gas supply or a contaminated ECD cell. To determine the source of contamination, perform the following procedure:

1 Remove the column from the ECD, and inspect the installation of the makeup gas adapter in the ECD cell. It is very common to install the adapter too low in the detector. To determine proper placement of the adapter measure from the bottom of the 1/4-inch swagelok nut to the bottom of the hex of the makeup gas adapter. The measurement should be 19-20 mm. If it is >22 mm, then the adapter was installed incorrectly. A ridge that is machined into the entrance of the ECD cell prevents the

adapter from easily seating all the way. Wiggling the adapter while installing will allow it to go all the way in. If the adapter is not all the way in, it can leak during a temperature program and can adversely affect linearity for pesticide methods.

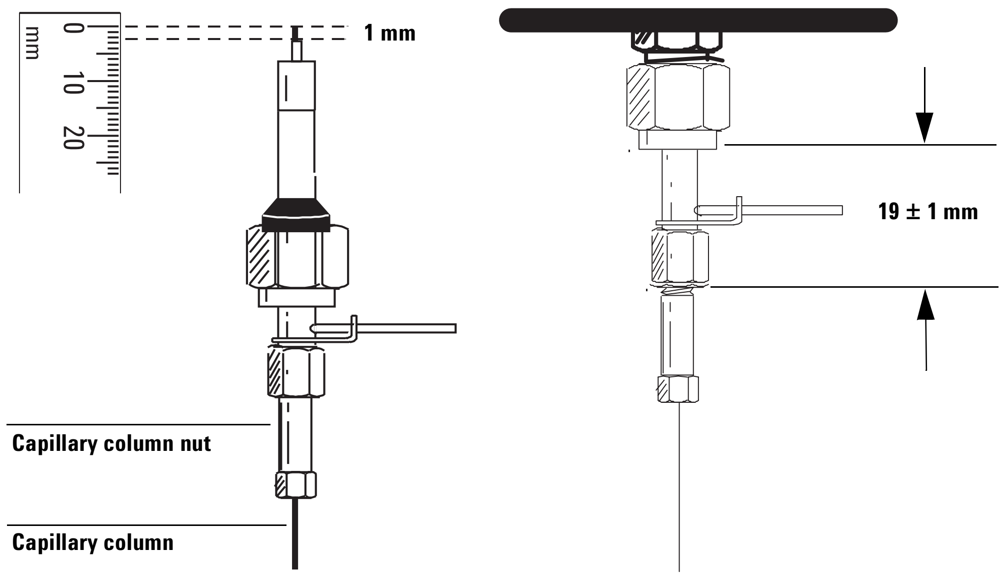

Figure 1 Correct installation of ECD makeup adapter

2 Remove the makeup adapter from the ECD and then unscrew the tip and take out the Gigabore liner. Inspect the makeup adapter body for graphite deposits. Remove all graphite deposits and clean it thoroughly with methanol. Soak the removable tip in methanol.

3 Reassemble the adapter WITHOUT the Gigabore liner. Cap off the adapter using a 5-inch piece of narrow bore column, capped at one end with a new septum. The other end of the column should extend one mm past the tip of the adapter. When troubleshooting contamination problems, it is wise to use column ferrules of Vespel instead of graphite. Tighten the tip just past finger tight with a clean pair of pliers and clean the whole assembly with methanol before installing.

4 Reinstall the capped off makeup adapter, making sure it is fully inserted into the ECD cell. Use a new ¼" Vespel ferrule to allow for easy installation. Retighten after heating the ECD.

5 Reset the makeup flow to the original setpoint and bakeout the detector at 350 degrees for 1 hour. During this detector bakeout, put the inlet into split mode with 200-300 mL/min of split vent flow (gas saver off) and bakeout the injection port at 275 degrees and the column at its appropriate bakeout temperature.

6 After the bakeout, run a series of blank runs using the customer method. If the baseline is acceptable – free of humps or peaks, and stays under 1000 hz during the temperature program, then the detector and makeup side of the system is clean.

7 Any unacceptable baseline problems could indicate contaminated makeup gas, EPC module, makeup adapter or ECD cell. These must be addressed before proceeding. If the 6890 was manufactured prior to 6/97, then the EPC modules are suspect to having O-ring contamination. See service note G1530-14 and follow the procedure outlined – the EPC module should also be replaced. The O-ring contamination problem was resolved after 7/97.

Evaluate the carrier side

Once the ECD and the makeup system are determined to be clean, then the carrier, inlet and column should be evaluated.

1 Reinstall the column in the makeup adapter by the same procedure used to install the piece of column used to cap the detector:

  a Remove the makeup adapter from the ECD and remove the short piece of column

  b Install new ferrule on the column and trim the column end

  c Install the column in the makeup adapter such that <1 mm of column extends past the tip of the adapter. It is best not to use the Gigabore liner. A better liner solution is the new mixing liner (p/n G2397-20540) from the µECD. It is backwards compatible to 5890 SER II and 6890 ECDs. Column installation is per 6890 µECD directions.

  d Wipe-off the entire makeup adapter with methanol and install the adapter fully inserted into the ECD.

2 Bakeout the entire system for another hour with the ECD at 350 degrees and the inlet in split mode at 275 degrees. The column should be baked at the manufacturer’s recommended bakeout temperature.

3 After bakeout, reload the customer’s method and make a series of blank runs (no injection) to determine if the contamination problem persists. Again a single, well resolved peak could be due to EPC module O-ring contamination mentioned previously. Address per service note.

4 If the contamination persists then perform a complete injection port maintenance, including a thorough cleaning of the shell weldment, replacing gold seal and liner. Also install a known good 30 meter/320 µm HP-5 checkout column to rule out column contamination.

5 Typically these peaks from the inlet side are due to contaminated carrier gas supply, EPC module, insert weldment, injection port/liner or column.

6 On the 6890 it is best to turn “Auto-Prep Run” to off so that the inlet is being purged when the GC is not in use.

Agilent Technologies, Inc.

Printed in USA, March 2002

G1533-90790