Innovations in Gas Chromatography

from Varian, Inc. -

a Historical Perspective

by:

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Oftentimes we take the present design of gas chromatographs for granted and presume that all manufacturers have developed the same hardware and have had similar features and performance. The evolution of the current GC instrumentation is based on a 40 year history, with many key developments created and marketed by Varian since the beginning. Fortunately this process has been cumulative; many of the innovations by Varian listed below are quite visible and often are featured prominently on current models by Varian, as well as instruments from other GC manufacturers.

Some GC companies have made claims that they are the "innovators" in this instrumentation genre. Although Varian has been relatively shy about their unique developments in GC, Varian always has been a major contributor to the current state-of-the-art technology and has made GCs easier to use, more powerful and significantly more automated.

General

Modular electronics – Moduline [1964]
- easier to service by simple exchange of electronic modules

Platinum resistance temperature probes (RTD) – Model 1700 [1968]
- self-calibrating
- physical break avoids thermal runaway
- accurate over wide temperature range

5 independent temperature zones – Model 3700 [1974]

Multi-line entry/status display – Vista 6000 [1980]

User-selectable temperature limits for all thermal zones – Model 3400 [1984]

Zero-resistance circuit board insertion – Model 3400 [1984]

6 independent temperature zones – Model 3600 [1984]

Dedicated capillary GC – Model 3500 [1985]

7 independent temperature zones – Model 3800 [1997]

Injectors

Four installed injectors in two temperature zones – Model 3700 [1974]

Spatially separated injectors – Model 3700 [1974]

Syringe guide in injector nut – Model 3700 [1974]
- fewer bent needles

Knife-edged injector nut – Model 3700 [1974]
- less tension on septa for longer life

Subambient on-column capillary injector – Model 3700/1095 [1979]

Subambient temperature programmable injector zone – Model 3700 [1979]

VTAT – Variable Temperature Absorption Trap – Model 3700 [1979]
- built-in air concentrator
- controlled through system operations

Injector switch – Vista 6000 [1980]
- automatic start of GC temperature programming and data system upon injection

Display of capillary linear velocity – Model 3500 [1985]

Display of capillary split ratio – Model 3500 [1985]

Three independent injectors – Model 3800 [1997]

SPT – Sample Preconcentration Trap – Model 3800 [1999]
- second generation air concentrator
- direct electrical heating of trap
- controlled through system operations

Automated Sampler

AutoSampler™ – Model 8000 [1972]
- Varian-registered trademark

Linear travel for syringe – Model 8000 [1972]
- fewer bent needles and easy alignment

«0.01% sample carry-over – Model 8000 [1972]
- side-loading syringe permits complete flushing of syringe with new sample prior to injection

Vial position documentation – Model 2700/8000/CDS101 [1974]

Dual automated sampler – Model 3700/8000 [1974]
- (120 vial capacity)

GC method change with A/S position – Model 3700/8000/CDS111 [1974]

LC/GC Interface – Vista 6000 [1982]
- automatically injected LC column effluent portion into GC for GC separation and detection

Automated sampler for on-column narrow bore capillary injector – Model 8035 [1985]

Automated sampler for SPME – Model 8100 [1992]

Pneumatics

Column headpressure gauge – Model 1700 [1968]

Heated pneumatics – Model 3700 [1974]
- maintains accurate flow independent of room temperature

Digital flow controller – Model 3700 [1974]

First generation automatic flow control – Model 3700 [1976]

Viton ferrules – Model 3400 [1984]
- easy adaptation of pneumatics without special fittings

Pneumatics (cont.)

Electronic pressure readout of column headpressure – Model 3400 [1984]

Large pneumatic compartment – Model 3600 [1986]

True electronic flow control with EFC (Type 3) – Model 3800 [1997]

Super large pneumatic compartment – Model 3800 [1997]

Special EFC for valving - EFC Type 4 – Model 3800 [1999]

Special EFC for purge/trap – EFC Type 5 – Model 3800 [1999]

Valving

7 separate positions for valve mounting – Model 3700 [1974]

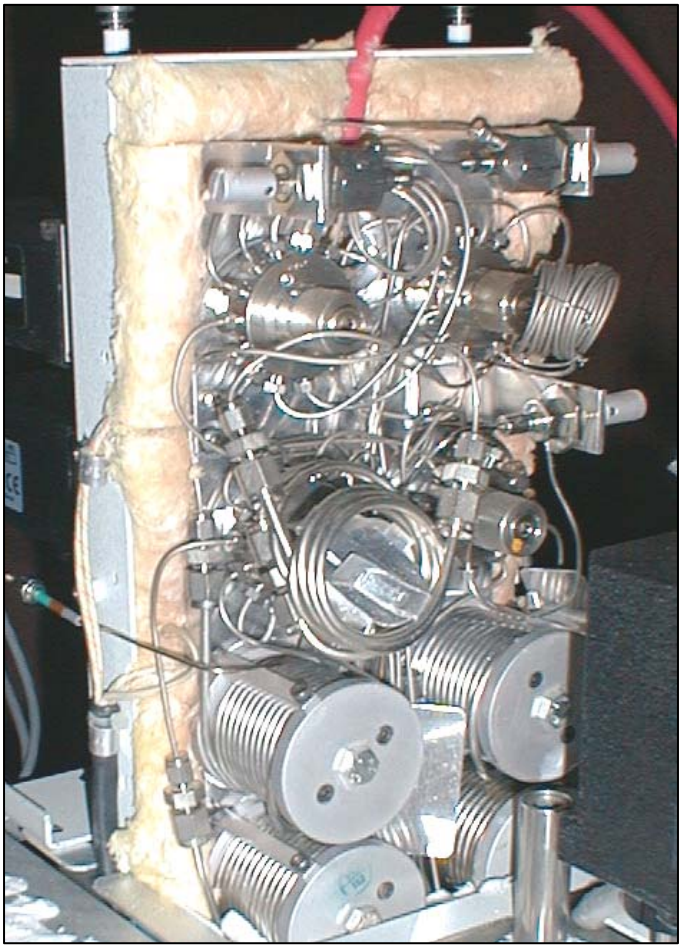
8 external events controlled by system – Vista 6000 [1980]

Pre-injection valve programming – Model 3400 [1986]

Full control of gas stream selector (SSV) through GC – Model 3400 [1986]

7-position valve oven
– Model 3800 [1997]

Full control over micro-electric multi-position valve (SSV)
– Model 3800 [1997]



Column Oven

Multi-level temperature programming
– Model 1520 [1965]

Space to mount columns side-by-side
– Model 1520 [1965]

Large Column Oven
– Model 1520 [1965]

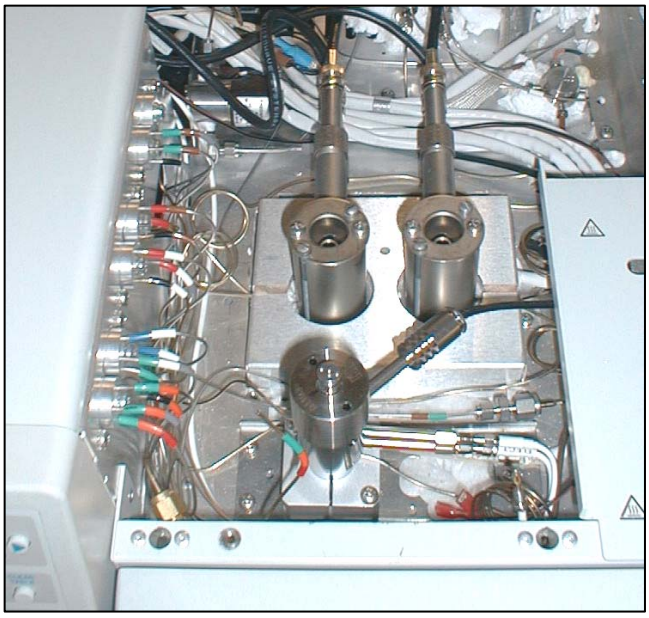
Huge column oven
– Model 3700 [1974]

Control to +12 °C above room temperature
– Model 3700 [1974]

Access light in column oven
– Varian 3400CX [1991]

Control to +8 °C above room temperature
– Model 3800 [1997]

Detectors



Universal ionization detector base
– Model 1700 [1968]
- permits easy interchange of detectors

Control of detector gases at instrument – Model 1700 [1968]

4-detector mounting
– Model 3700 [1978]

TCD and FID in series
– Model 3700 [1978]

3-detector simultaneous operation
– Model 3800 [1997]

Flame Ionization Detector (FID)

JFET electrometer – Model 1700 [1968]
- Johnson Field Effect Transistors
- simplified electrometer circuit
- improved signal conversion

10^{-12} electrometer range – Model 1700 [1968]
- gives us “Range 12”

$1x10^{-14}$ FID noise – Model 3700 [1974]

Auto flame-out /reignition – Model 3700 [1976]

Nickel reduction catalyst – Model 3700 [1978]
- improved detection and selectivity for CO and CO_2

Ceramic flame tip – Model 3400 [1992]
- patented

Thermal Conductivity Detector (TCD)

Amplified TCD signal - 20X – Model 3700 [1974]
- enhance signal without affecting filament lifetime

Constant mean temperature filament control – Model 3700 [1974]
- longer filament lifetime, as temperature is limited by setting

Filament protection circuit – Model 3700 [1974]
- shuts detector down with loss of carrier to protect filaments

Dual TCD on single instrument – Model 3700 [1976]
- permits simultaneous detection of H2 with argon carrier *and* other gases with helium as carrier

Electron Capture Detector (ECD)

300 μl cell volume – Model 3700 [1975]
- miniature cell maximizes sensitivity

Detection to 0.1 pg lindane – Model 3700 [1975]

Nitrogen carrier gas for ECD – Model 3700 [1975]
- not limited to Argon/Methane

Sealed ECD foil – Model 3700 [1975]
- no "site" license for radioactivity required by end user

Rapid interchange with FID – Model 3700 [1975]

Thermal-ionic Specific Detector (TSD or NPD[1])

Ceramic bead – Model 3700 [1976]
- major improvement in lifetime over coated bead
- previous versions were made from glass

Impregnated rubidium bead – Model 3700 [1976]

Self aligning bead – Model 3700 [1976]

Rapid interchange with FID – Model 3700 [1976]

Flame Photometric Detector (FPD)

Dual flame FPD – Model 3700 [1977]
- very selective detection of sulfur compounds
- minimal suppression by hydrocarbons

Pulsed FPD – Model 3400 [1994]
- sulfur/phosphorus emission separated from hydrocarbon signal by time
- applicable to many other chemical species

Computer display of emission profile – Model 3800 [1999]

[1] Varian has traditionally labeled detectors by their functionality and not by the species measured. Thus, the TSD is not called a "nitrogen-phosphorus" detector (NPD), just as the FID is not a "hydrocarbon" detector.

Data Collection

First commercial application of a microprocessor in a chromatography data system – CDS 101 [1974]

4 channel simultaneous/independent data collection and full instrument control – Vista 401 [1980]

Unique data files names automatically assigned, with overwriting prohibited – Vista 401 [1980]

Simultaneous GC/LC control from single data system – Vista 401 [1980]

Multi-tasking data system – Vista 401 [1980]
- even before PCs were multi-tasking

Multi-tasking floppy drive – Vista 401 [1980]
- permits storage of data while reading off data
- even PCs today cannot do this

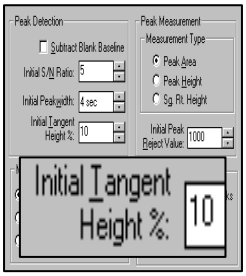
Multi-tasking hard drive – DS654 [1985]

In-board data handling (IBDH) – Model 3400 [1986]

"Wild card" files names – Star Ver 5 [1998]
- automatic file labeling by date/time/sample name

Data Computations

Peak detection based on S/N and peak width, not slope sensitivity – CDS101 [1974]
- parameters meaningful to chromatographers
- easier to optimize peak detection



Multiple internal standards in one run – CDS101 [1974]

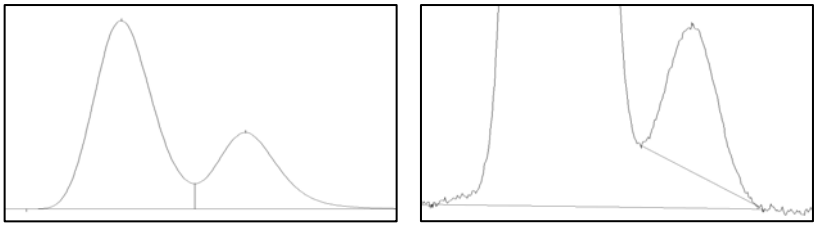
Multiple reference peaks – CDS101 [1974]

Automated PCB pattern matching – CDS101 [1974]

Tan% judgment of fused peaks – CDS101 [1974]

Point-to-point baseline subtraction – Vista 401 [1980]

User-modifiable baseline subtraction file – Vista 401 [1980]



Cannot alter sample names postrun – Vista 401 [1980]

Cannot alter time/date of injection postrun – Vista 401 [1980]

Software square-root computation for sulfur FPD – Vista 401 [1980]

"Verification" sample type – DS654 [1985]
- compares QC sample results to expected values, with error flags

Data Archiving and Display

First implementation of disk storage in a chromatography data system – Vista 401 [1980]

Raw data saved on disk for replot/recalculations – Vista 401 [1980]

Dual channel plotting – live vs live, live vs stored, stored vs stored – Vista 401 [1980]

High speed line printer (120 characters per second) – Vista 401 [1980]

Built-in printer/plotter in GC – Model 3400 [1984]

Automation

GC control from data system – CDS111 [1976]

Simultaneous GC and LC control from single data system – Vista 401 [1980]

Extensive error monitoring – Vista 401 [1980]
- automatic halt to sample sequence with "fatal" error detection

Autolink to external computer – Vista 401/Apple 2e [1980]
- permits automatic execution of special programs for data reduction and formatting

Single cable with multiple instrument channels (RS485) – DS654 [1985]
- early version of Ethernet concept

"Wait" function in sample list – Star WorkStation [1988]
- allows automatic execution of data collection at specific times

"Goto *sample line***" function in sample list** – Star WorkStation [1988]
- sets up repetitive cycling of sample list

Ethernet connection for full instrument control and data collection – Model 3800 [1997]

Documentation

Runlog GC/LC documentation – CDS111 [1976]

Custom report formatting – Vista 401/Apple2e [1980]

Single run file possesses raw data, results, reports, method employed, error log, run log, calibration data and curves – Star WorkStation [1988]

```
3400 GC Event Log:
       0.00  Change in Run Status : INJECT
       0.00  Initial Detector B Type : FID
       0.00  Initial Detector B Attenuation : 8
       0.00  Initial Detector B Range : 11
       0.00  Initial Detector B Autozero : YES
       0.00  Initial Column Temperature (degrees C) : 70
       0.00  Initial Injector Temperature (degrees C) : 250
       0.00  Initial Detector Temperature (degrees C) : 250
       0.00  Initial Auxiliary Temperature (degrees C) : 250
       0.01  Initial Pressure A (psi) : 15.5
       0.01  Initial Flow A (ml/minute) : 3.7
       0.04  AutoSampler Volume (ul) : 2.0
       0.04  Start Column Temperature Ramp (degrees C/minute) : 10.0
       0.13  Injection Residence Time (minutes) : 0.10
      13.01  End Column Temperature Ramp (degrees C) : 200
      15.00  Change in Run Status : END
```

The author offers acknowledgements to the many Varian "old-timers" who assisted with the compilation of this listing, with a special thanks to Herb Neumann, Norm Kirshen and Rich DeLew.

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