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Gas Chromatograph Uses Varactor Bridge Flame Detector Amplifier for Enhanced Performance

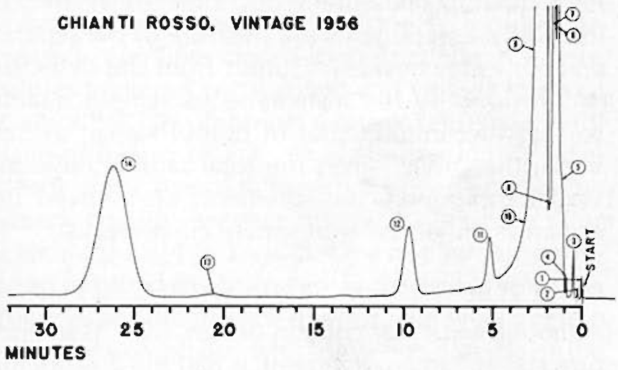
By **Harry Gill**, *Perkin-Elmer Corp.*

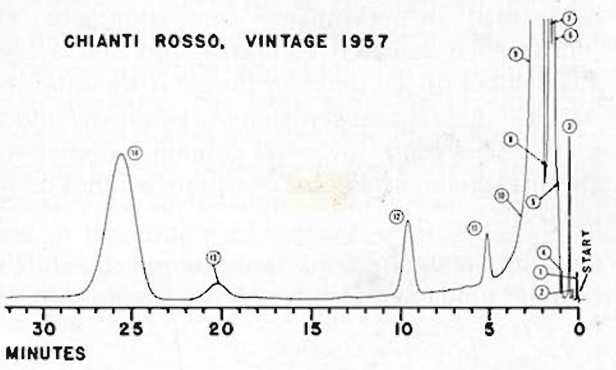
Current amplifier circuit based on parametric (varactor bridge) op amp increases gas chromatograph's useful sensitivity, stability, and dynamic range. New design furnishes 5 x 10^{-12} amp full scale output for recorder and integrator drive.

Electronic engineers have no monopoly on sophisticated instruments for measuring the different components of an unknown input. Gas chromatographs, which have evolved as rapidly since 1950 as microwave spectrum analyzers, enable research chemists to separate tenths of microlitres of any vaporizable sample into its individual constituents, and to measure the quantity of each sample-constituent with better than 2% accuracy.

Not all gas chromatographs find their way into research laboratories or advanced chemical plants, as motorists convicted of dangerous driving can frequently testify. Many police departments use these instruments for qualitative measurements of blood-borne alcohol. In other novel applications, the gas chromatograph can distinguish vintage wine from last years' crop; detect from a sample of "minced earthworms" that pesticides wash into the soil and stay there (vide, Rachel Carson's SILENT SPRING); sniff the noxious fumes in an automobile's exhaust; or bolster an Englishman's conviction that teabags prevent Americans from ever tasting a civilized cup o' tea.

Chianti, vintage 1957, is introduced into the gas chromatograph. Comparison of differences in trace components on the chromatograms may be related to differences in relative quality of individual vintages.





A gas chromatograph's resolution depends primarily upon the quality of the separator column used to segregate the sample into its individual components. Many different types of separator column can be "plugged in" to commercial chromatographs for a wide choice of analytical characteristics. Some of the more exotic separator columns are 300 foot coils, but more conventional types are wound into helixes that fit conveniently into the temperature controlled oven of most commercial instruments.

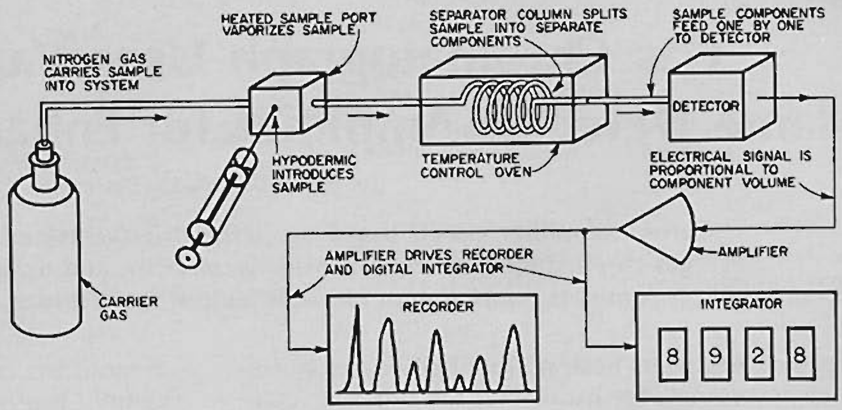


Figure 1

Samples introduced by hypodermic needle at heated sample port are vaporized then carried into the separator column by inert "carrier" gas. Adsorption or related process selectively delays different sample components, feeds them one-by-one to the detector. Gas Chromatograph technique can measure sample components down to 10^{-12} grams.

A simplified gas chromatograph block diagram is given in Fig. 1. Samples are introduced by hypodermic needle at the heated input port, where they are instantly vaporized, thence carried into the separator column by a stream of nitrogen, helium or other inert "carrier" gas. By selectively delaying the different sample components, the separator column feeds them one-by-one to the detector for component quantity measurements. Time delay between individual components is a measure of the separator column's effectiveness. Output from the detector is proportional to the instantaneous sample quantity, so that the time-integral of output signal, or "area under the curve," gives the total sample flow. Individual component measurements are derived from the areas under the appropriate curve-peaks.

DETECTOR

Although separator column design, with its temperature controlled environment, is half the development battle for high performance chromatographs (early fundamental research earned Martin and Synge a 1952 Nobel prize), detector design is the other half. An ideal detector must match the sensitivity and stability of the most advanced column, otherwise the chromatograph's inherent capability will not be fully realized.

Gas chromatographs may use thermal conductivity or other fundamental parameters for measuring sample concentration. However, the flame ionization detector generally gives highest sensitivity, detecting the presence of components weighing less than 10^{-12} gram.

Although gas chromatograph detector considerations might seem a somewhat specialized topic for this magazine's broad readership, in reality the problem of stable, noise-free, picoampere measurement recurs in many branches of industry and research.

DETECTOR PRINCIPLES

A hydrogen flame within the detector assembly, Fig. 2, ionizes sample components emerging from the separator column, and develops a proportional

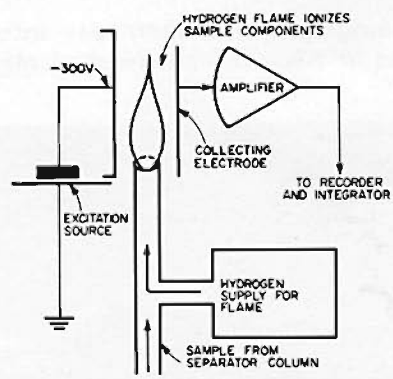


Figure 2

Flame ionization detector ionizes sample components with hydrogen flame, develops output current proportional to sample flow. Amplifier must resolve down to 2 x 10^{-14} ampere flame noise level, give accurate measurement for picoampere signals.

current flow between excitation electrode and collector plate. A high gain amplifier completes the external circuit and provides the drive signal necessary for recorder and integrator operation.

At highest sensitivity levels, detector resolution is limited by flame noise to about 5 x 10^{-14} amperes; consequently, there is little advantage in using amplifier systems with higher sensitivity or lower noise. On the other hand, amplifiers unable to resolve down to this level because of poor signal-to-noise ratio would be unable to respond to the full range of detector output, hence would cramp the chromatograph's wide output range.

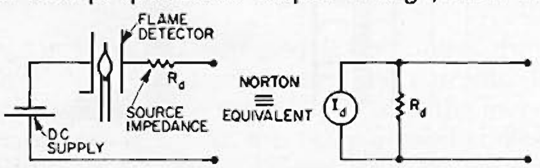
Past chromatography instruments have used electrometer tubes and post amplifiers to measure flame detector current. Warmup delays, aging, shift in operating point, noise, and humidity effects are some of the problems associated with such amplifiers. In particular, noise is the limiting factor with electrometer tubes, because it restricts dynamic range, and forms the weak link in the detector-amplifier-recorder-integrator chain. Consequently, we set out to design a new amplifier system that eliminated the electrometer tube, and which would raise overall chromatograph performance in the process.

A commercial varactor bridge operational amplifier solved the problem. This unit provided picoampere sensitivity, very low noise levels, and gave wider dynamic range than we could use. Additional virtues were its solid state 3 cu. inch P.C. mounting construction, high open-loop gain, high input impedance, modest warmup time, and ample output for direct recorder and integrator drive.

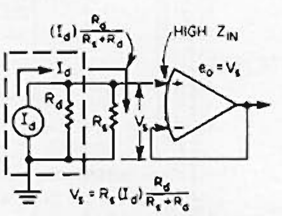
Key specifications for the selected varactor bridge amplifier are 2 pA max bias current and 0.1 pA/°C bias drift at 25°C; 0.01 pA noise from DC to 1 Hz; 10^{10} ohms and 10^{12} ohms differential and common mode input impedance; 10^6 gain; ±10 volt, 20mA output rating; and potential 10^9:1 dynamic range.

CHOICE OF CIRCUITS

Probably the most obvious method for measuring detector response is to convert the output current signal to a proportional output voltage, then use a

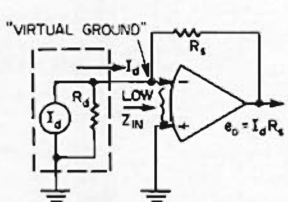


Detector (left) can be characterized by a Norton equivalent current source, I_d, in parallel with source impedance R_d (right).



Amplifier input impedance must be very high compared with sampling resistor to minimize loading effect, also to reduce errors caused by temperature-induced input impedance variations.

Fig. 3A



Source impedance, R_d, is paralleled by amplifier's very low closed-loop input impedance. Consequently, R_d does not enter into the output equation.

Fig. 3B

Figure 3

Noninverting voltage amplifier (left), uses sampling resistor R_s to develop signal voltages in response to detector output current. Alternative circuit (right), uses no sampling resistor, eliminates common mode and impedance errors, converts detector output directly to voltage with superior stability and noise performance.

high gain voltage amplifier to bring signals up to the right level. A circuit such as Fig. 3A uses a sampling resistor R_s to develop voltage V_s proportional to detector current; amplifier A then buffers this voltage to drive the recorder and integrator. A noninverting amplifier configuration has highest input impedance, hence places maximum input impedance in parallel with R_s and minimizes loading errors.

However, the most obvious amplification techniques are not necessarily the best! A current-to-voltage converter, or current amplifier, Fig3B, provides many advantages compared with the conventional voltage amplifier, even though both circuits can be based on the same operational amplifier. Instead of using sampling resistor R_s to convert detector current to equivalent voltage signals, the current amplifier can convert and amplify in one simple arrangement.

The primary advantage to the current amplifier is that feedback holds the amplifier input voltage at "virtual ground" so that input impedance is very low. Actually the circuits of Fig. 3A and 3B can be viewed as current meters. An ideal current meter would have zero impedance so that the measuring instrument would have no effect on the circuit being measured. While the circuit of Fig. 3B approaches this ideal, the circuit of Fig. 3A introduces a large impedance, R_s, into the circuit being measured, hence introduces measuring errors that depend upon the relative magnitudes of the sampling resistor, R_s, and source resistance R_d.

Any attempt to reduce measuring errors by reducing the magnitude of sampling resistor R_s requires an increase in amplifier closed-loop gain, which in turn amplifies the input voltage drift and voltage noise of the amplifier. By contrast, voltage-noise-gain and voltage-drift-gain of Fig. 3B is always unity*, independent of the size of R_s (which is now placed in the feedback circuit). Another disadvantage to the sampling technique of Fig. 3A is that the noninverting connection required to obtain very high input impedance introduces another source of error, namely, common mode rejection errors, which are not present in the single-ended circuit of Fig. 3B. Furthermore, the common mode impedance (which sets input impedance for the noninverting amplifier) changes with ambient temperature. For large sampling resistors this causes errors due to variable loading of the voltage amplifier. On the other hand, the current amplifier offers very low closed loop input impedance, as compared to the source impedance, so that the amplifier's impedance variation for this configuration has negligible effect on total accuracy.

$* \left[\frac{(R_s + R_d)}{R_d} \doteq 1\right]$

ACTUAL CIRCUIT

A simplified flame detector circuit, based on Analog Devices' Model 301 Varactor Bridge (parametric) amplifier* connected as a current-to-voltage converter, is shown in Fig. 4. The circuit's overall specifications include 5×10^{-12} amperes full-scale reading on the most sensitive range, 2.5×10^{-14} amperes maximum equivalent input noise, 5×10^{-14} amperes long-term drift, 7.5×10^{-15} amperes/°C drift over the range 15°C to 32°C, and 5×10^{-14} amperes offset change for line voltage variation from 105 to 130 volts.

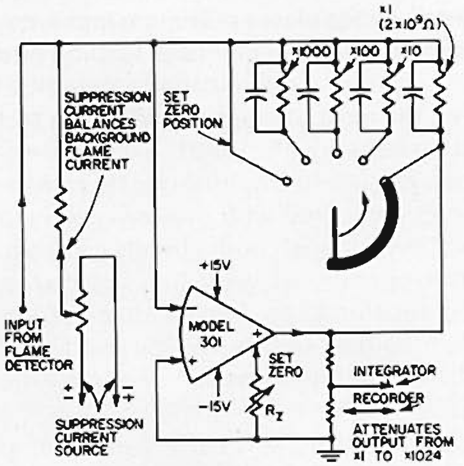


Figure 4

Current-to-voltage converter based on varactor bridge (parametric) op amp uses a 2×10^{9} ohm feedback resistor for 5×10^{-12} amperes full-scale reading. Dynamic range is 10^{5}:1 on most sensitive scale; 10^{6}:1 on all others.

Four levels of current sensitivity, all developing up to 10 volts at the amplifier output, are selected by range switch S_1. The switch connects additional resistors in parallel with the 2,000 megohm highest-sensitivity feedback resistor to reduce net feedback resistance, hence sensitivity, in steps of ten. Output voltage, V_o, for a given input current, I_{in}, is simply related to feedback resistance, R_f, by $V_o = I_{in} \times R_f$. This gives a 2 megohm total feedback resistance for the least sensitive range. Parallel capacitors used with each feedback resistor ensure amplifier closed loop stability and give correct operating bandwidth for each sensitivity range. Amplifier output to the recorder can be attenuated by x1 to x1024 in multiples of 2.

The most sensitive scale yields a potential 100,000:1 dynamic signal range through its ability to handle input signal swings from 5×10^{-14} amperes (twice the noise level) to 5×10^{-9} amperes at maximum output attenuation. The higher input level develops an output voltage $I_{in} \times R_f$ equal to $(5 \times 10^{-9}) \times (2 \times 10^{9}) = 10$ volts, which is the maximum Model 301 output before saturation.

See article, page 6, L. R. Smith, "A Parametric Operational Amplifier," NEREM, Boston, 1966.

Key to the detector amplifier's temperature stability of 7.5×10^{-15} amperes/°C drift for normal laboratory environments is the temperature-compensated amplifier enclosure. The heated enclosure reduces ambient temperature variations by a factor of approximately 60, prevents humidity from affecting high value resistors and associated insulation, and stabilizes the varactor bridge amplifier.

The varactor bridge Model 301 amplifier draws a maximum bias current of 2 pA at 25°C, which doubles for every 10°C temperature rise. Since the bias current is in effect drawn through the feedback resistor R_f, variations in bias current (i.e., bias current drift) develop spurious amplifier outputs. Although initial offsets can be externally zero'd by adjusting R_z, bias-current increase from 2 pA to 4 pA for a 25°C to 35°C rise creates an "unzero'd" offset error of $(2 \times 10^{-12}) \times (2 \times 10^{9}) = 4$ millivolts. This is equal to 40% of the sensitive range's 5×10^{-12}A full scale recorder output. However, the amplifier enclosure effectively insulates against ambient variations, so that drift is less than 1.5% of full scale for the same 10°C temperature rise.

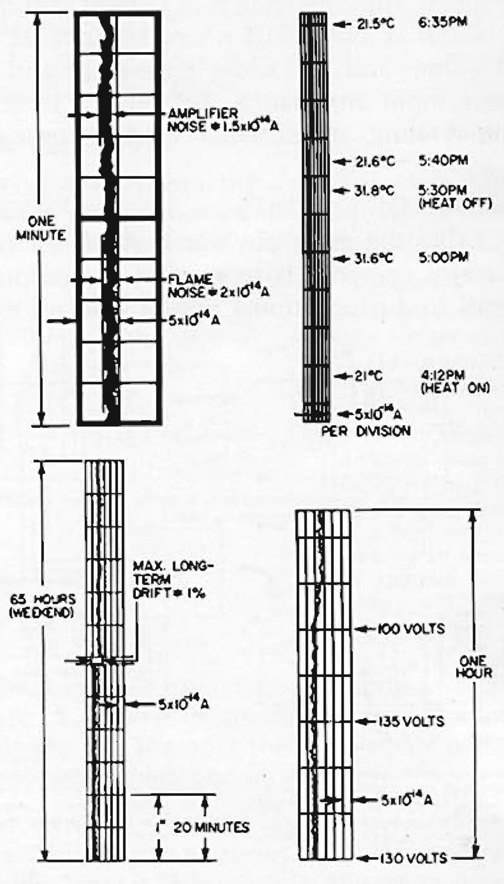


Figure 5

Actual measurements show how chromotograph amplifier easily meets specifications. Noise (top left) of 1.5×10^{-14} amps is well below flame noise level of roughly 2×10^{-14} amps; ambient temperature variation from 21°C to 31.8°C produces about 5×10^{-14} amp offset (top right). Long term stability test (bottom left), produces less than 1% drift during 65-hour weekend, while effect of supply voltage variation from 130 to 110 volts is approximately 5×10^{-14} amps offset (bottom right).

An auxiliary reference supply furnishes suppression currents needed to balance the flame detector's background current, and is adjustable from 1×10^{-14} to 1×10^{-8} amperes. Both suppression current supply and the amplifier's ±15 volt DC input are provided by regulated power supplies rather than batteries.

Actual amplifier performance falls well within the specifications, as the actual test charts, Fig. 5, show. In fact, measured amplifier noise was about 1.5×10^{-14} amperes, which is less than the 2.5×10^{-14} amperes usually cited for flame noise. Consequently, there is clearly little advantage in using elaborate and expensive low-noise techniques to reduce amplifier noise below its present level.

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NEW PRODUCTS

Model 147A/B/C FET Operational Amplifier excels on every major specification.

Most FET operational amplifiers offer advantages of high input impedance and low bias current but at a sacrifice in other specifications, usually voltage drift and common mode rejection. The Model 147A/B/C is the first general purpose FET op amp which excels in every performance category.

Model 147, with voltage drift of only 2μV/°C (max) and input bias current of 15pA (max), approaches the performance of chopper stabilized amplifiers and offers advantages of smaller size, lower price, lower noise and the versatility of differential inputs. The 147 boosts the inherently poor CMR of FET's to 300,000 — a 10 to 100 fold increase over most FET amplifiers.

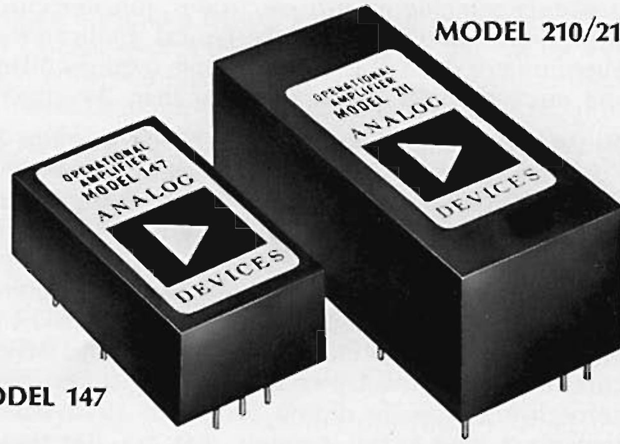
With 15pA bias current and 0.1pA noise, the 147 is excellent for measuring very low currents. With 10MHz and 10V/μsec response on both the inverting or noninverting inputs, the 147 is a good choice for sample/hold circuits, A-D and D-A convertors or wideband, high impedance ($10^{12}\Omega$, 3pF) noninverting amplifiers.

SPECIFICATIONS

Open Loop Gain, min.	10^6
Rated Output, min.	±10V @ 10mA
Unity Bandwidth	10MHz
Full Power Response, min.	150kHz
Slewing Rate, min.	10V/μsec
Common Mode Rejection	300,000
Input Impedance, C.M.	$10^{12}\Omega$, 3pF
Voltage Noise	3μV, p-p, DC to 1 Hz
Current Noise	0.1pA, p-p, DC to 1Hz
Size	2" x 1.2" x .62"

	Model A	Model B	Model C
Bias Current, max.*	30pA	15pA	15pA
Voltage Drift, max.			
(+10 to +60°C)	15μV/°C	5μV/°C	2μV/°C
(−25°C to +85°C)	15μV/°C	10μV/°C	5μV/°C
Price (1-9)	$110.	$120.	$135.

* At 25°C, doubles each 10°C



Model 210/211 Chopper Stabilized Op Amp's Drift is reduced with no increase in price.

Refinements in circuit design have netted two-fold reduction in voltage and current drift of now famous Model 210/211, making these units the industry's best buy in chopper stabilized operational amplifiers. For only $120, Model 211 offers maximum drift of 1μV/°C and 3pA/°C, while the Model 210 guarantees drift of 0.5μV/°C and 1pA/°C for $157. Other high performance specifications (see below) are retained as before.

SPECIFICATIONS

Open Loop Gain, min.	10^8
Rated Output, min.	±10V @ 20mA
Unity Bandwidth	20MHz
Slewing Rate, min.	100V/μsec
Overload Recovery	0.2μsec
Voltage Noise	5μV, p-p, DC to 1Hz
Size	2.8" x 1.3" x .95"

	Model 210	Model 211
Voltage Drift, max.	0.5μV/°C	1μV/°C
Current Drift, max.	1pA/°C	3pA/°C
Price (1-9)	$157.	$120.